

APPENDIX B
UNIFORM FEDERAL POLICY -QUALITY ASSURANCE
PROJECT PLAN

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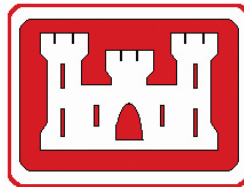
Final

**Quality Assurance Project Plan
Additional Sampling for
CC RVAAP-69 Building 1048 Fire Station,
CC RVAAP-70 East Classification Yard, and
CC RVAAP-74 Building 1034-Motor Pool Hydraulic Lift
Ravenna Army Ammunition Plant Restoration Program
Camp Ravenna, Portage and Trumbull Counties, Ohio**

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Prepared for:



**U.S. Army Corps of Engineers,
Louisville District
600 Dr. Martin Luther King Jr. Place
Louisville, Kentucky 40202-2267**

Prepared by:

PARSONS
401 Diamond Drive NW
Huntsville, AL 35806
256-837-5200

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ACRONYMS AND ABBREVIATIONS

%R	percent recovery
%D	percent dilution
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
ADR	Automated Data Review
AES	Atomic Emission Spectroscopy
amsl	above mean sea level
amu	atomic mass unit
AOC	Area of Concern
ARNG	Army National Guard
bgs	below ground surface
BUSTR	Bureau of Underground Storage Tanks
°C	Celsius
CAS	Chemical Abstracts Services
CC	Army Environmental Compliance-Related Cleanup Program
CCB	continuing calibration blanks
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CoC	Chain of Custody
COR	Contracting Officer's Representative
CSM	Conceptual Site Model
DNAPL	dense non-aqueous phase liquid
DoD	United States Department of Defense
DL	detection limits
DQO	Data Quality Objective
DRO	Diesel Range Organic
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
FCR	Field Change Request
FSP	Field Sampling Plan
FWCUG	Facility-Wide Human Health Cleanup Goals
FWSAP	Facility-Wide Sampling and Analysis Plan
GC	Gas Chromatograph
GC/MS	Gas Chromatograph/Mass Spectrometer
GC/FID	Gas Chromatograph/Flame Ionizing Detector
HDPE	high-density polyethylene
HHR	Historical Records Review
HPLC	High-performance liquid chromatography
ICAL	initial calibration
ICB	initial calibration blanks
ICP	inductively coupled plasma
ICP/MS	inductively coupled plasma mass spectrometry

ACRONYMS AND ABBREVIATIONS (CONTINUED)

ICV	initial calibration verification
IDW	investigation derived waste
LC/MS/MS	Liquid chromatography -tandem mass spectrometry
LCS	Laboratory Control Sample
LDR	Linear Dynamic Range
LNAPL	light non-aqueous phase liquid
LOD	limit of detection
LOQ	Limit of Quantitation
MB	Method Blank
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
MPC	Measurement Performance Criteria
mph	miles per hour
MRO	Motor Oil Range Organics
MS	Matrix Spike
MSL	Munitions Response Site
N/A	Not applicable
ng/kg	nanograms per kilogram
ng/L	nanograms per liter
OHARNG	Ohio Army National Guard
Ohio EPA	Ohio Environmental Protection Agency
PAH	polycyclic aromatic hydrocarbon
PAL	Project Action Limits
PCBs	polychlorinated biphenyls
PDT	Project Delivery Team
PID	photoionizing detector
PM	Project Manager
PMP	Project Management Plan
PWS	Performance Work Statement
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality System Manual
RCRA	Resource Conservation and Recovery Act
REIMS	Ravenna Environmental Information Management System
RI	Remedial Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RRT	Relative Response Factor
RSD	relative standard deviation
RSL	Regional Screening Level
RT	Retention Time

ACRONYMS AND ABBREVIATIONS (CONTINUED)

RVAAP	Ravenna Army Ammunition Plant
SAIC	Science Applications International Corporation
SEDD	Superfund Electronic Data Deliverable
SI	Site Investigation
SOP	Standard Operating Procedure
SSCV	Second Source Calibration Verification
SVOC	Semi-volatile Organic Compound
TA	Test America
TAL	target analyte list
TAT	turnaround time
TBD	To Be Determined
TCLP	Toxicity Characteristic Leaching Procedure
TPH	Total Petroleum Hydrocarbon
UFP-QAPP	Uniform Federal Policy-Quality Assurance Project Plan
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
USP&FO	United States Property and Fiscal Officer
VOC	Volatile Organic Compound
WP	Work Plan

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EXECUTIVE SUMMARY

ES.1 Introduction

This Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP) is the primary operations document for implementation of Performance-Based Remediation for the Ravenna Army Ammunition Plant (RVAAP) Restoration Program. The Work Plan associated with this UFP-QAPP describes data gap investigations to complete the Site Inspection (SI) at CC RVAAP-70 East Classification Yard and Remedial Investigations (RI) at CC RVAAP-69 Building 1048 Fire Station and CC RVAAP-74 Building 1034-Motor Pool Hydraulic Lift at Camp Ravenna under Delivery Order 0003 of Contract No. W912QR-12-D-0002.

The former RVAAP, now known as the Camp Ravenna Joint Military Training Center (Camp Ravenna), located in northeastern Ohio within Portage and Trumbull counties, is approximately three (3) miles east/northeast of the City of Ravenna and one (1) mile north/northwest of the City of Newton Falls. Camp Ravenna is federally owned and is approximately 11 miles long and 3.5 miles wide. Camp Ravenna is bounded by State Route 5, the Michael J. Kirwan Reservoir, and the CSX System Railroad to the south; Garret, McCormick, and Berry Roads to the west; the Norfolk Southern Railroad to the north; and State Route 534 to the east. In addition, Camp Ravenna is surrounded by the communities of Windham, Garrettsville, Charlestown, and Wayland.

ES.2 Work Plan Objectives

The Work Plan for this project (see main text of report) describes data gap investigations to complete the SI at CC RVAAP-70 East Classification Yard and RI at CC RVAAP-69 Building 1048 Fire Station and CC RVAAP-74 Building 1034-Motor Pool Hydraulic Lift at Camp Ravenna. The Work plan provides a detailed summary of previous investigations at the three Areas of Concern (AOCs) (Section 2.0 of the Work Plan), evaluates the existing data to identify data gaps (Section 3.0 of the Work Plan), and proposes a sampling approach to address the data gaps (Section 4.0 of the Work Plan). The data gaps and approach to address the data gaps are summarized for each AOC below.

CC RVAAP-69 Building 1048 Fire Station

The fire station was previously located in the former RVAAP Administration Area in the northwest quadrant of the intersection of George Road and South Service Road. No documented evidence was found regarding specific years of service for the Building 1048 Fire Station. A site schematic dated 1941 was found as part of the historical records review, so it is assumed services commenced shortly after the 1941 building construction. The fire station building was demolished in late 2008, and the site currently remains undeveloped. Possible sources of chemicals of potential concern (COPCs) are unreported spills or leaks from firefighting equipment. Carbon tetrachloride was commonly used through the 1950s to extinguish fires as the chemical was readily available, nonflammable, easily volatilized, and inhibited the combustion process. Former employees reported that it was common practice for the fire department to clean out fire extinguishers behind (i.e., the west side) the fire building, and to allow the contents of the fire extinguishers (carbon tetrachloride) to spill onto the ground surface (SAIC, 2011d).

RI sampling was conducted in 2012 and 2015. Sampling included 4 surface and 16 subsurface incremental sampling methodology (ISM) samples, which were analyzed for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), and target analyte list (TAL) metals. Sampling also included 57 discrete subsurface soil samples from 9 borings which were

analyzed for VOCs. Full suite analyses were also performed on one surface sample and four subsurface samples. Data were screened by comparing concentrations to the most stringent Resident Receptor Facility Wide Cleanup Goals (FWCUGs). June 2017 United States Environmental Protection Agency (USEPA) Regional Screening Levels (RSLs) for Residential Soil were used for those analytes without established Resident Receptor FWCUGs at cancer risk of 1×10^{-6} or a hazard quotient (HQ) of 0.1. The following COPCs were identified for surface soil: two inorganics (chromium and cobalt) and five SVOCs (benzo[a]anthracene; benzo[a]pyrene; benzo[b]fluoranthene; dibenz[a,h]anthracene; and indeno[1,2,3-c,d]pyrene). One inorganic (arsenic), one SVOC (benzo[a]pyrene), and two VOCs (carbon tetrachloride and chloroform) were identified as COPCs for subsurface soil. However, only the VOCs have not been delineated.

The existing data was evaluated to identify the following data gaps and propose actions to address those data gaps:

- o The vertical extent of carbon tetrachloride in soil at SB101 is not defined. The deepest sample collected from boring 69-1048-SB101 (SB101) at 15-16 feet below ground surface (bgs) exceeded the June 2017 USEPA RSL of 0.65 milligram per kilogram (mg/kg) for carbon tetrachloride. Carbon tetrachloride detected in samples from 5-16 feet bgs in this boring consistently exceeded the June 2017 USEPA Residential RSL. Concentrations in this boring appear to decrease below 10 feet bgs. Soil concentrations in soil borings 72-1048-RV5-SB2, 69-1048-SB101, 69-1048-SB104, and 69-1048-SB105 range from 0.7 mg/kg to 14 J mg/kg, which exceed the June 2017 USEPA Residential RSL. The boring log shows that SB101 extended to 20 feet bgs; however, no samples were collected for laboratory analysis below 16 feet bgs. A new boring will be installed in the immediate vicinity of the earlier borings SB101 and RV5-SB2. The boring will extend from the ground surface to bedrock, which is estimated to be 45 feet bgs. The length of the core will be screened with a PID monitor. If no evidence of a chemical release is noted (no odors, staining, or elevated PID readings), then discrete soil samples will be collected every 5 feet from the length of the core in each boring beginning at 10 feet bgs. However, if evidence of potential chemical release is observed, then discrete soil samples will be collected from the interval exhibiting the highest odors, staining, or elevated PID readings. Soil samples will be analyzed for carbon tetrachloride and its decay products (i.e., chlorinated methanes). All new borings will be continuously logged.
- o The lateral extent of carbon tetrachloride contamination in soil is not well defined below a depth of 1018 feet above meal sea level (amsl). Carbon tetrachloride concentrations from borings SB101 and SB104 were highest in the vertical interval of 5 and 10 feet bgs (1015 and 1020 feet amsl). The soil borings immediately to the north (SB105), south (SB103) and east (SB102) of SB101 were only sampled to a depth of 10 feet bgs (1018 or 1019 feet amsl). Additional soil borings will be installed north (near SB105), east (near SB102), and south (near SB103) of the maximum concentrations. These soil borings will extend to the water table, which is approximately 10 to 20 feet bgs at CC RVAAP-69. All new borings will be continuously logged. The length of the core will be screened with a PID monitor. If no evidence of a chemical release is noted (no odors, staining, or elevated PID readings), then discrete soil samples will be collected every 5 feet from the length of the core in each boring beginning at 10 feet bgs (i.e., samples at 10, 15 and 20 feet bgs). However, if evidence of potential chemical release is observed, then discrete soil samples will be collected from the interval exhibiting the highest odors, staining, or elevated PID readings.

Soil samples will be analyzed for carbon tetrachloride and its decay products. The ground surface elevation at CC RVAAP-69 varies between 1025 and 1029 feet amsl. These proposed samples will define the extent of carbon tetrachloride and its decay products in the vadose zone and upper portions of the saturated zone, which may extend as deep as 1005 feet amsl. Contamination is defined to a depth of about 13 feet bgs (1012 feet amsl) in distal sample locations (SB106, SB109, and SB107), so additional step out samples will not be necessary regardless of whether carbon tetrachloride is detected in any of the proposed soil samples.

- o Groundwater was not evaluated as part of the previous investigations. The potentiometric surface for Camp Ravenna aquifers is mapped annually from groundwater elevation measurements in monitoring wells. The groundwater flow direction in the unconsolidated aquifer beneath AOC CC RVAAP-69 is generally to the east at an elevation between 1010 and 1020 feet amsl (10 to 20 feet bgs) (TEC-WESTON Joint Venture, 2016). Groundwater grab samples will be collected, and then groundwater monitoring wells will be installed and monitored to establish the local groundwater flow direction and delineate carbon tetrachloride in groundwater, if any. In order to efficiently optimize the number of monitoring wells needed to define the direction of groundwater flow and monitor a plume, first grab samples will be collected using a Screenpoint 16 sampler. If groundwater grab samples cannot be collected from well points, then temporary wells will be installed, developed, and sampled. Groundwater grab samples will be collected from the upper portions of the saturated zone within the unconsolidated aquifer. These screening samples will be used to select monitoring well locations. Initial groundwater grab samples will be collected from near the maximum concentrations detected in soil (near RV5-SB2) and in locations approximately 50 to 70 feet downgradient (east to southeast) of that location (i.e., between the former Building 1048 Fire Station and George Road/South Service Road). Groundwater grab samples will be analyzed for chlorinated methanes on a quick turn (24 hour) basis, and additional step-in or step-out samples will be collected until the plume is delineated or until sampling establishes that carbon tetrachloride or its decay products are not present in groundwater at concentrations above Residential Receptor FWCUGs in groundwater using the carcinogenic risk level of 10^{-6} or the hazard quotient of 0.1. If a carbon tetrachloride groundwater plume is detected and delineated with grab samples, a minimum of three monitoring wells will be installed; one near the maximum concentrations detected (near RV5-SB2), one upgradient of the source area, and one at the downgradient extent of the plume. More than the minimum of three monitoring wells may be needed to confirm the extent of potential carbon tetrachloride contamination beneath CC RVAAP-69. Borings from the new monitoring wells will be continuously logged. If a carbon tetrachloride groundwater plume is not indicated by the grab samples, at minimum one monitoring well will be installed near RV5-SB2 (where maximum concentrations have been detected in soil). At least two additional monitoring wells will be installed to ensure that the local potentiometric surface can be mapped. Monitoring wells will be installed with screens set across the water table (estimated at 10 to 20 feet bgs) to maximize the likelihood of intercepting any COPCs in groundwater because the maximum concentrations in soil to date are in the interval of 5 to 10 feet bgs (1015 to 1020 feet amsl). Monitoring wells will be developed and sampled quarterly during the first year for carbon tetrachloride and its decay products (chlorinated methanes). The RI report will evaluate and recommend any post-RI monitoring requirements. Post-RI monitoring will be addressed as part of the

facility-wide groundwater monitoring program. If dense non-aqueous phase liquid (DNAPL) is present in the bottom of the CC RVAAP-69 monitoring wells, its thickness will be measured with an oil/water interface probe capable of detecting the presence of non-aqueous phase liquid within the well casing.

CC RVAAP-70 East Classification Yard

The former RVAAP was originally equipped with east and west classification yards during the facility's early operational years. The East Classification Yard is located east of Load Line 1 and the Main Defense Logistics Agency Ore Storage Area in close proximity to the intersection of Ramsdell Road and Irons Road. No documentation was found during the historical records review to define the specific years of operation of the AOC. The East Classification Yard AOC consists of Building 47-40 (Round House, still exists but is not actively used), the former herbicide storage shed (former Building 47-60), the containment area for a former aboveground storage tank (AST) (documented spill of No. 5 fuel oil occurred within the containment area in 1986), and an outdoor open wash rack south of the East Classification Yard (north of Butts-Kistler Road). A railroad track complex is located east of the site and is currently used by the OHARNG. Two former 15,000-gallon diesel fuel USTs, RV-11 and RV-22, were located west of the wash rack, but were removed in February 1990 and received No Further Action in April 1992 (SAIC, 2011d).

SI sampling was accomplished in 2012. The AOC was divided into seven decision units (DU) to identify potential chemical releases associated with historical practices conducted at the East Classification Yard. The 2012 SI sampling included surface soil, subsurface soil, and dry sediment samples:

- o Former Fuel Oil Spill Area (DU01) was established as the area within the containment berm that surrounded the former AST where No. 5 fuel oil was released. One surface soil ISM sample, and 9 subsurface soil ISM samples were collected and analyzed for VOCs, methyl tertiary-butyl ether (MTBE), SVOCs, 2 TPH diesel range organics (DRO) carbon chain compounds (C10-C20, C20-C34), and 1 TPH-gasoline range organics (GRO) carbon chain. Additionally, one subsurface sample was analyzed the full analytical suite.
- o Drainage Ditch West of Building 47-40 (DU02) was established to evaluate the dry sediment in the AOC drainage ditches. One dry sediment sample was collected from the drainage ditch to the west of Building 47-40 and analyzed for VOCs, MTBE, SVOCs, TPH-GRO, and TPH-DRO.
- o Building 47-40 Round House (DU03) was established as a 15-foot zone surrounding the exterior perimeter of Building 47-40 to sample for potential contamination resulting from locomotive maintenance activities conducted at Building 47-40. One surface soil ISM sample and 7 subsurface soil ISM samples were collected and analyzed for SVOCs, TAL metals and PCBs.
- o Building 47-40 Interior Repair Pit (DU04) was established to sample the soil beneath the concrete floor of the Interior Repair Pit. The Interior Repair Pit lies underneath the area where locomotives were maintained and is a low point for any releases, which increases the likelihood of finding potential contamination from activities conducted within Building 47-40. The soil samples collected from the bottom of the Interior Repair Pit (beneath the concrete floor) were considered to be subsurface samples because the concrete floor prevents contact directly with the underlying soil. The SI photo log indicates black grime (called sludge in this Work Plan[WP]) in the bottom of the pit. Five soil borings were

advanced through the concrete floor of the pit. Auger refusal (due to encountering sandstone bedrock) was encountered at less than 4 feet depth in all five borings, therefore subsurface soil ISM samples were collected at vertical intervals ranging from 0-1 feet to 0-3.83 feet (below the repair pit bottom). All samples were analyzed for SVOCs, TAL metals and PCBs.

- o Former Herbicide Storage Shed (DU05) was established to sample soil around the former herbicide storage shed. One surface soil ISM sample and 9 subsurface soil ISM samples were collected and samples were analyzed for SVOCs and herbicides. One sample was also analyzed for full suite analyses. Additional analyses were performed for VOCs and TPH-GRO on vertical ISM soils collected from DU05 SB02 due to field observations and photoionization detector field screening vapor headspace results of 75 parts per million indicating the likelihood of potential contamination in soil at this boring.
- o Outdoor Wash Rack Area (DU06) was established to investigate the potential release of chemicals from site operations to surface soil and by infiltration to subsurface soil, and by overland conveyance to nearby drainage ditches. One surface soil ISM sample and 7 subsurface soil ISM samples were collected and analyzed for SVOCs, PCBs, and explosives. Additional analyses were performed for VOCs and TPH-GRO on a vertical ISM soil sample collected from DU06 SB05 due to field observations and photoionization detector field screening vapor headspace results of 40 parts per million indicating the likelihood of potential contamination in soil at this boring.
- o Drainage Ditch East of Building 47-40 (DU07) was established to evaluate the dry sediment in the AOC drainage ditches. Two dry sediment ISM samples (including a field duplicate) were collected from the drainage ditch to the east of Building 47-40 and analyzed for the full analytical suite including VOCs, MTBE, SVOCs, metals, TPH-GRO and TPH-DRO, herbicides, explosives, PCBs, and propellants.

Data were screened by comparing concentrations to the most stringent Resident Receptor FWCUGs or June 2017 USEPA Residential Receptor RSLs for those analytes without established FWCUGs at cancer risk of 1×10^{-6} or a HQ of 0.1 (Chapter 3 Tables). Petroleum hydrocarbons were screened using Bureau of Underground Storage Tank Regulation (BUSTR) Soil Class 2 Action Levels (Ohio Department of Commerce, 2017). Data were screened as part of this WP using FWCUGs (SAIC, 2010) and updated (June 2017) Residential RSLs. Concentrations of petroleum constituent VOCs and SVOCs at Former Fuel Oil Spill Area DU01 were also compared to appropriate FWCUGs or RSL. Chemicals exceeding these screening levels in surface soil and/or dry sediment are benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, indeno(1,2,3-cd)pyrene, and Aroclor 1242. These results are consistent with the Draft SI Report (ECC, 2015a), except that benzo(a)pyrene exceeded its FWCUG at the Former Fuel Oil Spill Area DU01. These chemicals do not have Camp Ravenna background values. Inorganics were below Camp Ravenna background levels, except arsenic in the dry sediment samples from the ditches. In the Draft SI Report (ECC, 2015a), arsenic was eliminated because concentrations were below the wet sediment background value. Chemicals exceeding these screening levels in subsurface soil are benzo(a)anthracene, benzo(a)pyrene, and benzo(b)fluoranthene. Inorganics in subsurface soil were below Camp Ravenna background levels. These results are consistent with the Draft SI Report (ECC, 2015a). Petroleum constituent VOCs and SVOCs at the Former Fuel Oil Spill Area (DU01) had formerly been compared only to BUSTR Soil Class 2 Action Levels. VOC and SVOC data at the Former Fuel Oil Spill Area (DU01) were re-evaluated and compared to FWCUGs

(SAIC, 2010) or to June 2017 Residential RSLs if FWCUGs were not available. Benzo(a)anthracene, benzo(a)pyrene, and benzo(b)fluoranthene in subsurface soil at the Former Fuel Oil Spill Area (DU01) exceeded the screening criteria.

Existing data was evaluated as part of this WP to identify the following data gaps and propose actions to address those data gaps:

- o Soil underneath the floor inside Building 47-40 has not been sampled at locations that are stained and cracked. Prior to sampling, oily sludge will be removed from the floor of Building 47-40 and Interior Repair Pit. The sludge will be removed to reveal areas of the floor that are stained and cracked and therefore the most likely routes that chemicals could enter the subsurface, and to prevent cross contamination when borings are advanced through the floor. After removing the sludge from the floor of Building 47-40, the concrete floor will be inspected for cracks and stains. Additional soil sampling within this building is proposed to target soil underneath visible cracks and stains on the concrete floor of Building 47-40 in order to confirm/deny evidence of impact below the concrete floor. Two soil borings will be installed near cracks and biased towards locations which exhibit staining or had sludge on the floor. One of these borings will be near the former oil storage rack on the East wall of Building 47-40 through the wooden floor in that area. Discrete soil samples will be collected from each boring at 2 foot intervals down to 7 feet bgs or until sandstone is reached (from 0-2, 2-4, 4-6, and 6-7 feet bgs), and samples will be analyzed for VOCs, SVOCs, PCBs, and TAL metals. All new borings will be continuously logged.
- o Soil underneath the concrete floor of the Interior Repair Pit (DU04) has not been sampled for VOCs. After the sludge is removed as described above, the concrete floor of the Interior Repair Pit will be inspected for any visible cracks and staining after the sludge is removed. Two soil borings will be drilled inside the Interior Repair Pit with preference given to any locations with cracks, staining, or sludge noted on the concrete floor. The length of the cores will be screened with a photoionization detector (PID) monitor. If no evidence of a chemical release is noted (no odors, staining, or elevated PID readings), then discrete soil samples will be collected every 2 feet from the length of the core in each boring. However, if evidence of contamination is observed, then discrete soil samples will be collected from any intervals with odors, staining, or elevated PID readings. Samples from each boring will be analyzed to confirm/deny the presence of VOCs. Previous boring logs indicate that sandstone was reached from approximately 1-4 feet bgs in the pit; therefore, the proposed soil borings will be drilled in 2 foot intervals until sandstone is reached. All new borings will be continuously logged.
- o Discrete soil samples have not been sampled and analyzed for VOCs from locations at the Outdoor Wash Rack Area at the vertical interval where odor and elevated PID readings were noted in boring logs. Boring logs for 70-4759-DU6-SB1 and 70-4759-DU6-SB5 at the wash rack area indicated elevated PID readings in 2012. Proposed additional soil sampling will target the soil near these previous borings with elevated PID readings at the wash rack to confirm/deny the presence of VOCs. Borings will be drilled near 70-4759-DU6-SB1 and 70-4759-DU6-SB5, and soil cores will be collected from 1 to 7 feet bgs. The entire soil core will be screened with a PID monitor and discrete samples will be collected from the depth intervals with field indications of potential contamination (staining, odors, or elevated PID readings). However, if no field evidence of contamination is noted, then samples will be collected from the 6 to 7 feet bgs interval which indicated

elevated PID readings and odor during the 2012 field investigation. The soil samples will be analyzed for VOCs. All new borings will be continuously logged.

- o Sediment from the storm drain at the southwest corner of Building 47-40 has not been sampled for potential contamination that may have resulted from the Outdoor Wash Rack. The storm drain at the southwest corner of Building 47-40 will be inspected where wash water may have drained from the wash rack for staining, odors, or elevated PID readings. A sediment sample will be collected from the storm drain and analyzed for PCBs, SVOCs, and explosives. One boring will be drilled as close to the drain as possible and soil samples will be collected to determine if subsurface soil near this storm drain was impacted by historical activities at the wash rack. Subsurface soil samples will be collected from 1-3, 3-5, and 5-7 feet bgs from the boring and analyzed for VOCs, PCBs, SVOCs, and explosives. All new borings will be continuously logged.

CC RVAAP-74 Building 1034 Motor Pool Hydraulic Lift

Building 1034 Motor Pool Hydraulic Lift is located south of the intersection of George Road and South Service Road just south of Building 1037 in the Administration Area in the south-central portion of Camp Ravenna. The Building 1034 Motor Pool Hydraulic Lift consists of the former underground hydraulic lift at Building 1034. Design drawings for the building and lift are dated 1941. There is one in-ground hydraulic lift that is composed of two cylinders; each in its own underground vault. The smaller (eastern) vault contains a hydraulic cylinder the portion of the lift that fitted under the rear axle of a vehicle. The larger, “L” shaped (western) vault contained a separate hydraulic cylinder for the front axle and a storage tank that stored the hydraulic fluid. The position of the front axle lift could be adjusted to accommodate different sized vehicles. When it was realized that the hydraulic system was losing fluid, an attempt was made to find the leak and the lift was taken out of service. The date that the lift was taken out of service was not recorded. The underground hydraulic lift is in place but no longer active. Building 1034 is currently unoccupied and used for storage, however, the Army plans to remodel the building for use as a workspace.

RI sampling was conducted in 2013. Only subsurface soil was sampled because the AOC is inside an existing building and is covered by the concrete floor. A total of 21 soil borings were advanced through the floor to depths ranging from 6 to 15 feet bgs, and soil samples were collected from intervals where hydrocarbon odor or staining was noted. If there was no indication of hydrocarbons, soil samples were collected from the bottom of the boring. A total of 33 discrete samples and one composite sample were collected. All soil samples were analyzed for polycyclic aromatic hydrocarbons (PAHs), DRO, and motor oil range organics (MRO) because the hydraulic fluid used in the lift contained mineral oil (a petroleum by-product). One sample was analyzed for the full suite of analytes. All 33 soil samples collected in 2013 were analyzed for PAHs. A total of 18 PAHs were detected and identified as site-related chemicals (including all 7 PAHs regulated under BUSTR). However, none of the PAH site-related chemicals exceeded their BUSTR Soil Class 1 Action Levels FWCUGs. Therefore, the horizontal and vertical extent of SVOCs/PAHs has been delineated at this AOC, and delineation of PAHs in soil is not a data gap. No further sampling or analysis for PAHs in soil is proposed. DRO and MRO petroleum hydrocarbons (which together encompass the range of mineral oil-based hydraulic fluid) were detected in 10 of 21 borings sampled at depths up to 14 feet bgs. MRO detections in two soil borings 5,900 mg/kg at 6-6.5 feet bgs in 74-1034-HL-SB17 and 26,000 mg/kg at 8-8.5 feet bgs in 74-1034-HL-SB18, located on opposite sides of the rear axle portion of the hydraulic lift exceeded the BUSTR Soil

Class 1 Action Level of 5,000 mg/kg. The boring log for 74-1034-HL-SB18 indicated “free product” at 7.5-8.0 feet bgs. MRO was the only COPC identified.

To avoid confusion, the TPH-C₁₀ to C₂₀ distillate range previously described in the Draft RI as "diesel range organics" or "DRO" will be more accurately described in this WP as "middle distillates range." Also, the TPH-C₂₀ to C₃₄ distillate range described in the Draft RI is "motor oil range" or "MRO" (not to be confused with middle range organics or distillates) will be more accurately described in this WP as "heavy distillates range." Existing data were evaluated to identify the following data gaps and propose actions to address those data gaps:

- o It is unclear if the “free product” identified in 74-1034-HL-SB18 at 7.5 to 8.5 feet bgs is mobile light non-aqueous phase liquid (LNAPL) or residual LNAPL. The historic boring log for SB18 noted “free product,” and the concentration of TPH-C₂₀ to C₃₄ heavy distillates range in soil exceeds the saturation level for Class 1 soil. These results could indicate the presence of mobile LNAPL. However, the results could also represent residual LNAPL only. A new boring will be installed in the immediate vicinity of the earlier boring 74-1034-HL-SB18; the soil will be photographed, visually inspected, and scanned with an ultraviolet light (UV) light and PID to evaluate if mobile LNAPL flows out of the formation versus residual LNAPL or oil that is essentially immobile and held in capillary spaces. An UV light will be used to help screen for the presence of hydraulic oil. All new borings will be continuously logged. A soil sample will be collected from 7.5 to 8.5 feet bgs to confirm what was found by evaluating the chromatograph to a known sample of hydraulic fluid (i.e., mineral oil). Soil samples will be analyzed for VOCs, TPH-C₁₀ to C₂₀ and C₂₀ to C₃₄ distillate ranges, and PCBs. A temporary well will be installed in the borehole to a depth of approximately 10 feet bgs and screened across the vertical interval where “free product” was historically noted in the gravel layer on the SB18 boring log. The temporary well will be developed. Measurements (depth to LNAPL and/or water) will be made to determine if mobile LNAPL flows into the well, indicating the presence or absence of mobile LNAPL.
- o The horizontal extent of COPCs in soil is not defined east and south of 74-1034-HL-SB18. The elevated TPH-C₂₀ to C₃₄ heavy distillates range detections at SB18 and SB17 were detected at approximately 6 to 8.5 feet bgs. However, to the east and south of SB18, soil in borings 1034-HL-SB-14 and 74-1034-HL-SB-19 did not indicate elevated PID readings, odor or staining at this interval. Soil was only sampled at the bottom of these at boreholes about 14 feet bgs, and therefore, the extent of COPCs in soil was not bounded east and south of SB18. Soil borings will be advanced in the immediate vicinity of the earlier borings 1034-HL-SB-14 and 74-1034-HL-SB-19, and collect soil samples from 7.5 to 8.5 feet to evaluate the horizontal extent in east and south of 74-1034-HL-SB18. Soil samples will be analyzed for VOCs, TPH-C₁₀ to C₂₀ and C₂₀ to C₃₄ distillate ranges. All new soil boings will be continuously logged.
- o Groundwater was not evaluated as part of the previous investigations, and it is unknown if groundwater is impacted or if enough hydraulic fluid was released to accumulate as LNAPL at the water table. The potentiometric surface for Camp Ravenna aquifers is mapped annually from groundwater elevation measurements in monitoring wells. The groundwater flow direction in the unconsolidated aquifer beneath AOC CC RVAAP-74 is generally to the east at an elevation between 1010 and 1020 feet amsl (TEC-WESTON Joint Venture, 2016). Depth to groundwater is expected to be between 10 to 20 feet bgs. Groundwater grab samples will be collected and analyzed, and then groundwater

monitoring wells will be installed and sampled to establish the local groundwater flow direction and delineate potential hydraulic fluid (LNAPL and/or dissolved phase) in groundwater, if any. In order to efficiently optimize the number of monitoring wells needed to define the direction of groundwater flow and monitor a potential plume, first collect grab samples will be collected using a Screenpoint 16 sampler. If groundwater grab samples cannot be collected from well points, then temporary wells will be installed, developed, and sampled. Groundwater grab samples will be collected from the upper portions of the saturated zone within the unconsolidated aquifer. The results from these screening samples will be used to select monitoring well locations. Initial groundwater grab samples will be collected approximately 3 feet to the southeast and 20 feet downgradient (east and southeast) of soil boring 74-1034-HL-SB18 where the maximum TPH-C₂₀ to C₃₄ heavy distillates range concentrations were detected in 2013 (i.e., outside the southeast corner of Building 1034). Groundwater grab samples will be analyzed for VOCs and PAHs on a quick turn (24 hour) basis. Additional step-out samples will be collected until the plume is delineated or until groundwater grab samples establish that VOCs and PAHs are not present in groundwater above BUSTR Action Levels. Constituents that do not have BUSTR Action Levels will be compared to Resident Receptor FWCUGs using the carcinogenic risk level of 10⁻⁶ or the HQ of 0.1 or current USEPA Residential RSLs (June 2017). A minimum of three monitoring wells will be installed and developed; one near the maximum detection of TPH-C₂₀ to C₃₄ heavy distillates range in soil (near SB18), one upgradient well near the cylinder that held the hydraulic fluid (near SB02), and one at the downgradient extent of the plume (if a plume exists). More than the minimum of three monitoring wells may be needed to confirm the extent of potential contamination beneath CC RVAAP-74. Borings from new monitoring wells will be continuously logged. Monitoring wells will be installed with screens set across the water table (estimated at 10 to 20 feet bgs) to maximize the likelihood of intercepting LNAPL or a hydrocarbon plume. The fluid levels in the wells will be measured to detect LNAPL (if any), and groundwater samples will be analyzed for VOCs, PAHs, and PCBs. The local potentiometric surface will be mapped using groundwater elevations in wells installed at CC RVAAP-74 and adjacent AOC CC RVAAP-69. Monitoring wells will be developed and sampled quarterly during the first year. The RI report will evaluate and recommend any post-RI monitoring requirements. Post-RI monitoring will be addressed as part of the facility-wide groundwater monitoring program.

- o Only one soil sample was analyzed for VOCs. Although hydraulic fluid is not expected to contain volatile components, it is possible that VOCs were used and released as part of automobile maintenance activities in Building 1034. All soil samples from this AOC will be analyzed for VOCs in addition to TPH-C₁₀ to C₂₀ and C₂₀ to C₃₄ distillate ranges. One soil sample will be analyzed for PCBs. Groundwater samples from this AOC will be analyzed for VOCs and PAHs. Groundwater samples from groundwater monitoring wells will also be analyzed for PCBs.

ES.3 Document Content

This UFP-QAPP was prepared in accordance with UFP-QAPP guidance to ensure environmental data collected are scientifically sound, of known and documented quality, and suitable for their intended purposes. This UFP-QAPP focuses on the site-specific details for the site to include investigation methods, general analytical services, data management and validation procedures,

and field SOPs. Supporting plans and other information are included in the appendices to the Work Plan and this UFP-QAPP.

REFERENCES

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- Ohio Environmental Protection Agency, 2015. *Conceptual Site Models*, Guidance Document, Division of Environmental Response and Revitalization. April.
- ECC, October 2012. *Final Site Inspection and Remedial Investigation Work Plan at Compliance Restoration Sites, Former Ravenna Army Ammunition Plant, Portage and Trumbull Counties, Ohio*. October 3.
- ECC and AMEC Environment and Infrastructure, Inc. (AMEC), May 2014. *Preliminary Draft Remedial Investigation Report CC RVAAP-69 Building 1048 Fire Station, Former Ravenna Army Ammunition Plant, Portage and Trumbull Counties, Ohio*. May.
- ECC and AMEC, March 2015. *Draft Remedial Investigation Report CC RVAAP-74 Building 1034 Motor Pool Hydraulic Lift, Former Ravenna Army Ammunition Plant, Portage and Trumbull Counties, Ohio*. March 6.
- Science Applications International Corporation (SAIC), 2011a. *Facility-Wide Sample and Analysis Plan for Environmental Investigations at the Ravenna Army Ammunition Plant, Ravenna, Ohio*, February 24.
- SAIC, 2011b. *Historical Records Review for the 2010 Phase I Remedial Investigation Services at Compliance Restoration Sites (9 Areas of Concern)*, December 22.
- United States Army Corps of Engineers (USACE), Louisville District, 2012. *United States Army Corps of Engineers Ravenna Army Ammunition Plant (RVAAP) Position Paper for the Application and Use of Facility-Wide Human Health Cleanup Goals, Ravenna Army Ammunition Plant, Ravenna, Ohio*. Revised February 2012.
- Code of Federal Regulations*, 40 CFR 261, Appendix II, 1993 ed., as amended by 58 FR 46040, August 31, 1993.
- United States Environmental Protection Agency (USEPA), 1988, *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA (Interim Final)*, EPA/540/G-89/004. October.
- USEPA, 2005. *Uniform Federal Policy for Quality Assurance Project Plans: Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs, Part 1: UFP-QAPP Manual*, Intergovernmental Data Quality Task Force, March.
- USEPA, 2017. *Regional Screening Level (RSL) Summary Table (TR=1E-06, HQ=0.1)*, June.

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WORKSHEET #1, 2: TITLE AND APPROVAL PAGE

[Uniform Federal Policy – Quality Assurance Project Plan (UFP-QAPP) Manual, Section 2.1]

1.1 Project Identifying Information

Site Name/Project Name: Ravenna Army Ammunition Plant (RVAAP) Restoration Program,
Former Camp Ravenna, Portage and Trumbull Counties, Ohio

Site Location/No: CC RVAAP-69 Building 1048 Fire Station
CC RVAAP-70 East Classification Yard
CC RVAAP-74 Building 1034-Motor Pool Hydraulic Lift

Contract No/ W912QR-12-D-0002
Task Order No.: Delivery Order: 0003
Preparation Date: November 2017

1.2 Concurrence Signatures

The below signatures indicate the representatives of the subject organizations have reviewed this Uniform Federal Policy – Quality Assurance Project Plan (UFP-QAPP) and concur with its implementation as written.



Dan Griffiths, PG, CPG, Parsons
Parsons Technical Manager

27 November 2017

Date



Ed Heyse, PhD, PE, Parsons
Parsons Project Manager (PM)

27 November 2017

Date

Relevant plans and reports from previous investigations are included in the Reference list of this UFP-QAPP.

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WORKSHEET #3, 5: PROJECT ORGANIZATION AND UFP-QAPP DISTRIBUTION

(UFP-QAPP Guidance Manual, Section 2.4.1)

This worksheet identifies key project personnel, as well as lines of authority and lines of communication among the lead agency, prime contractor, and subcontractors, regulatory agencies, and stakeholders. Project Organization is presented in **Figure B-3-1** and contact information for the purposes of UFP-QAPP distribution is listed in **Table B-3-1**.

Figure B-3-1: Project Organizational Chart

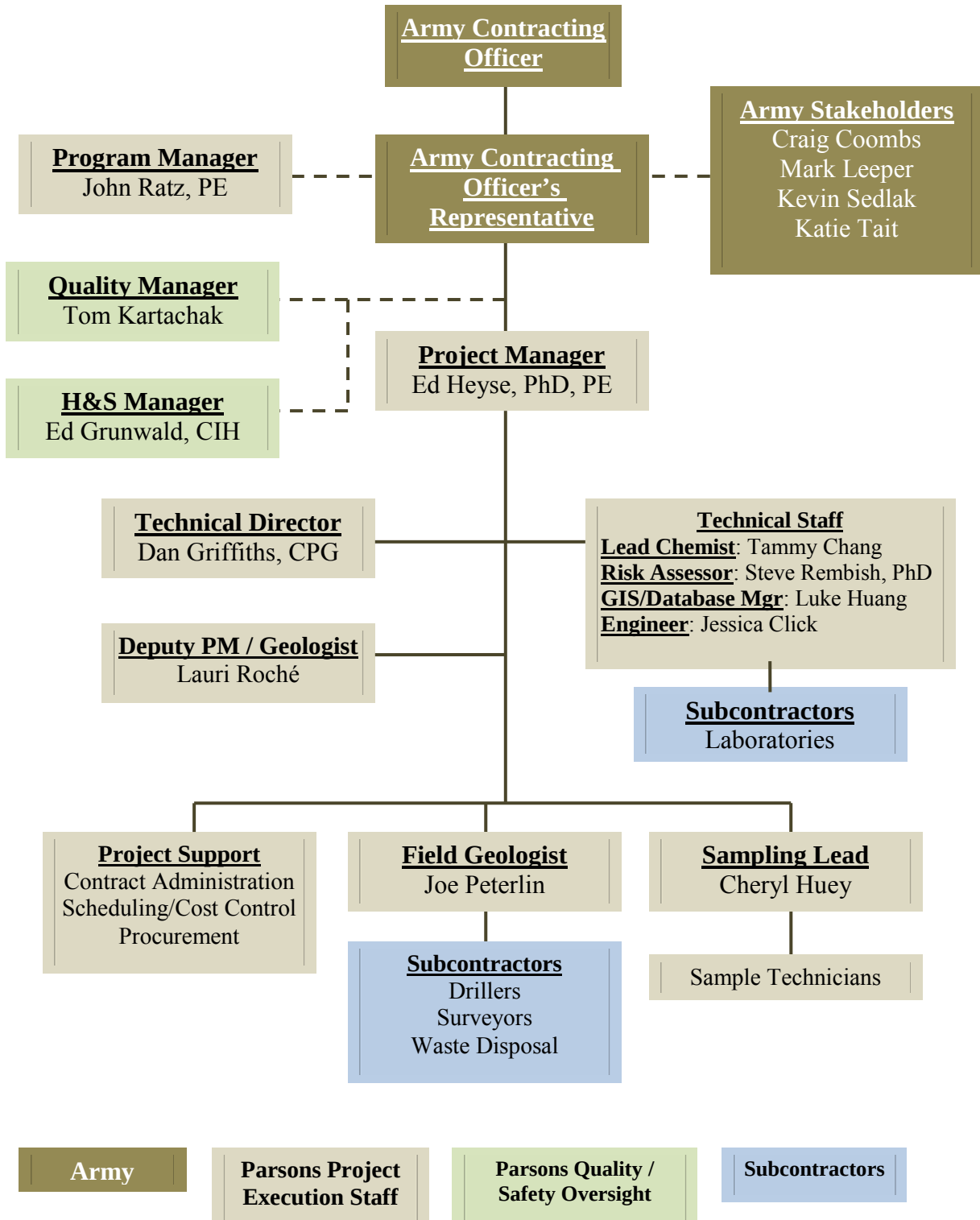


Table B-3-1: UFP-QAPP Distribution List

Recipient	Title	Organization	Telephone Number	E-mail Address
Mark Leeper	RVAAP Restoration Program Manager	ARNG	703-607-7899	Mark.S.Leeper.gov@mail.mil
Kevin Sedlak	RPM	Camp Ravenna	614-336-6000 X2053	Kevin.M.Sedlak.ctr@mail.mil
Katie Tait	Environmental Specialist	OHARNG	614-336-6136	Kathryn.s.tait.nfg@ mail.mil
Gail Harris	RVAAP Administrative Records	Vista Sciences Corporation	330-872-8003	Gail.harris@vistasciences.com
Pat Ryan	Senior Environmental Scientist/Ravenna Environmental Information Management System (REIMS) Support	Leidos-REIMS	865-481-4664	Patrick.F.Ryan@leidos.com
Kevin Mieczkowski	COR	USACE – Louisville District	502-381-6061	Kevin.M.Mieczkowski@usace.army.mil
Craig Coombs	USACE PM	USACE – Louisville District	502-315-6324	Craig.a.coombs@usace.army.mil

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WORKSHEET #4, 7, & 8: PERSONNEL QUALIFICATIONS AND SIGN-OFF SHEET

(UFP-QAPP Manual Sections 2.3.2 – 2.3.4)

This worksheet identifies key project personnel for each organization performing tasks defined in this QAPP. These organizations include the prime contractor, and primary laboratory.

Signatures indicate personnel have read and agree to implement this QAPP as written.

KEY PROJECT PERSONNEL

Title/Role	Name/ Organization	Contact Information Telephone/Email	Experience (1)	Specialized Training/ Certifications (2)	Signature/Date (3)
USACE PM	Craig Coombs	(502) 315-6324 Craig.a.coombs@usace.army.mil	n/a	n/a	Signature on Worksheets #1 & 2
USACE COR	Kevin Mieczkowski	(502) 381-6061 Kevin.m.mieczkowski@usace.army.mil	n/a	n/a	Signature on Worksheets #1 & 2
RVAAP Restoration Program Manager	Mark Leeper ARNG	(703) 607-7899 Mark.S.Leeper.gov@mail.mil	n/a	n/a	Signature on Worksheets #1 & 2
Contractor PM	Ed Heyse, PhD, PE Parsons	(256) 217-2573 Ed.Heyse@Parsons.com	Thirty-five years of experience in environmental research, teaching, project and program management, consulting, and technical direction.	B.S. Civil Engineering, 1980 M.S. Civil/Environmental Engineering, 1982 Ph.D. Environmental Engineering Sciences, 1995	Signature on Worksheets #1 & 2

KEY PROJECT PERSONNEL (continued)

Title/Role	Name/ Organization	Contact Information Telephone/Email	Experience (1)	Specialized Training/ Certifications (2)	Signature/Date (3)
Contractor Technical Manager	Daniel Griffiths Parsons	(303) 764-1940 (303) 710-9419 Daniel.R.Griffiths@parsons.com	Over 20 years' experience in managing environmental projects with a focus on streamlining site delineation and innovative remedy design and implementation.	B.S. Geological Sciences, 1996 M.S. Unstable Isotope Mass Spectrometry, 1996.	Signature on Worksheets #1 & 2
Contractor Project Chemist	Tammy Chang Parsons	(512) 719-6092 (512) 590-1126 (cell) Tammy.Chang@parsons.com	Over 25 yrs of analytical laboratory and chemistry-related experience	M.S., Chemistry, 1977	
Quality Assurance (QA) Laboratory PM	Erika Gish/TestAmerica Laboratories, Inc. (TA)-St Louis	(314) 787-8276 erika.gish@testamericainc.com	10+ years of environmental laboratory experience.	B.A. Biology, 2002	
QA Laboratory QA/Quality Control (QC) Manager	Kristen Ely/ TA-St. Louis	(314) 298-8566 Kristin.ely@testamericainc.com	10+ years of environmental laboratory experience	B.S. Biology, 2005	

- (1) Experience shown is limited to that relating to Performance Work Statement (PWS) requirements.
(2) Specialized training and certifications shown are those required by the PWS, relevant guidance, or other parts of this UFP-QAPP.
(3) Signatures indicate personnel have read this UFP-QAPP and agree to implement the procedures as written.

WORKSHEET #6: COMMUNICATION PATHWAYS

(UFP-QAPP Manual Section 2.4.2)

This worksheet documents specific issues (i.e., communication drivers) that will trigger the need to communicate with project personnel or stakeholders. It ensures procedures are in place for providing the appropriate notifications and generating the appropriate documentation when handling important communications.

Communication Driver	Initiator (role)	Recipient(s) (role)	Procedure
General communication between Lead Agency and other Project Delivery Team (PDT) members	USACE District PM or designee	Appropriate PDT member(s)	Communicates directly as needed (verbally and/or in writing).
General communication between USACE District and other PDT members	USACE District PM	Appropriate PDT member(s)	Communicates directly as needed (verbally and/or in writing).
Communication regarding Project Work Statement (PWS) or contractual Matters	USACE COR	Contractor PM	Communicates directly as needed (verbally and/or in writing).
Regulatory interface	USACE District PM	Regulator	Provides project update via e-mail at least every other week during field activities.
Regulatory oversight	Regulator	USACE District PM	Communicates directly as needed (verbally and/or in writing).
Project management, Task Order administration and logistics, UFP-QAPP changes prior to field/laboratory work	Contractor PM	USACE District PM, and/or USA Team lead technical and site management personnel	Communicates directly as needed (verbally and/or in writing).
Field progress reports	Contractor Site Manager	Contractor PM and site personnel	Documents progress in daily report and submits to Contractor PM for onward distribution to PDT. Daily reports will be submitted to Contractor PM within 24 hours of work completion that day.
Stop work due to safety issues	All site personnel	USACE PM	Follow-up notification to USACE PM as soon as possible either verbally or by email after work stoppage.

Communication Driver	Initiator (role)	Recipient(s) (role)	Procedure
UFP-QAPP changes in the field	Contractor Site Manager	Contractor PM	Communicates directly as needed (verbally and/or in writing) and submits draft Field Change Request (FCR) form for discussion; does not implement change until approval is granted; consults with other personnel as needed.
	Contractor PM	USACE Lead Agency and District PMs	Submits Field Change Request (FCR) form to USACE for approval; does not implement change until approval is granted.
	USACE Lead Agency and District PM	Regulatory PM and Stakeholder	Communicates directly with regulatory agency and stakeholders and notifies them of the changes as necessary.
Field corrective actions	Contractor Site Manager	Contractor PM	The need for field corrective actions will be determined by the Site Manager in consultation with the Contractor PM, and/or contractor technical personnel. The Site Manager will notify the Contractor PM of any needed field corrective actions and the Contractor PM will respond within 24 hrs.
Reporting laboratory quality variances or sample receipt variances	Laboratory Project Manager	Contractor Project Chemist	Applicable Laboratory PM will notify Contractor Project Chemist verbally and in writing. Contractor Project Chemist will notify Contractor PM. All sample receipt variances will be communicated within 24 hrs. of sample receipt.
Analytical corrective actions and data validation corrective actions	Contractor Project Chemist	Contractor PM	The need for Corrective Actions for analytical and data validation issues will be determined by the Contractor Project Chemist, who will notify the PM verbally or in writing of any laboratory issues that may cause Data Quality Objectives (DQOs) to be unattainable or cause delivery issues such that the project schedule cannot be met.
	Contractor PM	USACE Lead Agency and District PMs	The Contractor PM will notify USACE of analytical and data validation issues if they cause DQOs to be unattainable or cause delivery issues such that the project schedule cannot be met.
Reporting Data Validation Issues	Contractor Project Chemist	Laboratory Project Manager	All completeness and data issues will be addressed with the laboratory directly verbally and in writing. The validated data package will be due within approximately 14 calendar days of receipt.

WORKSHEET #9: PROJECT PLANNING SESSION SUMMARY

(UFP-QAPP Manual Section 2.5.1 and Figures 9 through 12)

This worksheet provides a concise record of participants, key decisions or agreements reached, and action items.

PROJECT MEETINGS

Client meetings and significant decisions and action items are documented in the Monthly Progress Report. Following is a summary of project teleconferences:

September 26, 2016: Parsons held a conference call with USACE to discuss the data gap sampling strategy for the Work Plan. USACE provided feedback to Parsons regarding our proposed approach and Ohio Environmental Protection Agency (Ohio EPA) comments on CC-RVAAP-73 Draft RI and their request for additional sampling. Based on the large amount of coal on the surface at the North Line Road Coal Tipple, USACE decided to ask for an extension on responding to comments while they consider options.

List of Attendees

Organization	Name / Role	Telephone / Email
Parsons	Ed Heyse, PhD, PE / PM	303-563-9452 Ed.heyse@parsons.com
Parsons	Lauri Roché / Deputy Project Manager	818-281-6630 Lauri.roche@parsons.com
Parsons	Jessica Click / Associate Engineer	256-295-4814 Jessica.click@parsons.com
Parsons	Janelle Bartscherer / Senior Engineer	303-386-2414 Janelle.bartscherer@parsons.com
USACE	Craig Coombs / USACE PM	502-315-6061 craig.a.coombs@usace.army.mil
USACE	Nathaniel Peters / Senior Environmental Engineer	502-315-2624 nathaniel.peters.II@usace.army.mil
USACE	Angela Schmidt / Risk Assessor	502-315-6313 angela.l.schmidt@usace.army.mil

January 18, 2017: USACE held a conference call to discuss comments on the Work Plan. Parsons was asked to provide recommendations on editing the Work Plan text to Ohio EPA's comments, which Ed Heyse provided on the same day. Craig Coombs said he would contact the Ohio EPA reviewer for clarification on how to proceed before finalizing the document.

List of Attendees

Organization	Name / Role	Telephone / Email
Parsons	Ed Heyse, PhD, PE / PM	303-563-9452 Ed.heyse@parsons.com
USACE	Craig Coombs / USACE PM	502-315-6324 Craig.a.coombs@usace.army.mil
USACE	Kevin Mieczkowski / USACE COR	502-381-6061 Kevin.M.Mieczkowski@usace.army.mil
ARNG	Kevin Sedlak / RPM	614-336-6136 Kevin.M.Sedlak.ctr@mail.mil
Camp Ravenna	Kathryn Tait / Environmental Specialist	614-336-6136 Kathryn.s.tait@us.army.mil

January 21, 2017: Parsons discussed quality issues with the CC RVAAP-79 database and advised the COR that these issues represent a change in conditions from the information available at the time of the proposal. In addition to data quality issues, Parsons also noted that there is considerably more data to be incorporated into the draft Remedial Investigation (RI) report than was apparent at the time of the proposal. The PWS identified a preliminary draft RI report for CC RVAAP-79, however, after contract award, Parsons learned that there was also a draft RI report with additional sampling data that was not mentioned in the PWS or provided during the proposal phase. Incorporating additional data into the CC RVAAP-79 RI Report represents a change in conditions.

List of Attendees

Organization	Name / Role	Telephone / Email
Parsons	Ed Heyse, PhD, PE / PM	303-563-9452 Ed.heyse@parsons.com
USACE	Kevin Mieczkowski / USACE COR	502-381-6060 Kevin.M.Mieczkowski@usace.army.mil

February 21, 2017: Ed Heyse spoke with Kevin Mieczkowski regarding the Ravenna database issues (laboratory and survey data). Mr. Mieczkowski requested that Parsons provide details as to why Parsons would have assumed that the data would be provided in a complete and error-free database based on the information provided with the Request for Proposal. Parsons provided this information in emails to Mr. Mieczkowski in emails dated 21 and 23 February 2017. Issues include (1) there was additional data for Sites CC RVAAP-79 and CC RVAAP-69 in draft reports that were not provided to Parsons at the time of the proposal, and (2) the previous contractors work plan outlined how data would be provided as a complete, error-free electronic format suitable for incorporation into the Army and Camp Ravenna databases.

List of Attendees

Organization	Name / Role	Telephone / Email
Parsons	Ed Heyse, PhD, PE / Project Manager	303-563-9452 Ed.heyse@parsons.com
USACE	Kevin Mieczkowski / USACE COR	502-381-6060 Kevin.M.Mieczkowski@usace.army.mil

Variable Dates: The Parsons Camp Ravenna project team holds weekly internal project meetings to discuss project approach, field and lab methodologies, and proposed sampling and analysis to be conducted at each AOC.

List of Attendees

Organization	Name / Role	Telephone / Email
Parsons	Ed Heyse, PhD, PE / PM	303-563-9452 Ed.heyse@parsons.com
Parsons	Lauri Roché / Deputy PM	818-281-6630 Lauri.roche@parsons.com
Parsons	Jessica Click / Associate Engineer	256-295-4814 Jessica.Click@parsons.com
Parsons	Janelle Bartscherer / Senior Engineer	303-386-2414 Janelle.bartscherer@parsons.com
Parsons	Cindy Conway / Project Geologist	720-519-5019 Cynthia.conway@parsons.com

March 28, 2017: Parsons Project Chemist and lab key staff discussed the project needs regarding the total petroleum hydrocarbon (TPH) analysis. Lab staff can recognize the pattern of mineral oil and/or hydraulic fluid in the soil samples via TPH-diesel range organics (DRO) + TPH-motor oil range organics (MRO) analysis of SW8015M method. However, without knowing the exact type of mineral oil and hydraulic fluid historically used on site, the lab cannot quantitatively determine the concentration of these parameters. The lab agreed to state the type of heavy TPH on the result page of soil samples, upon request.

September 6, 2017: Update on TPH analyses. A project chemist contacted Test America by email to request the TPH results be reported as “C10-C20” and “C20-C34”, instead of referring to these hydrocarbon ranges as “DRO” and “MRO”. Test America project manager, Erika Gish, agreed to our request and updated the project files accordingly.

List of Attendees

Organization	Name / Role	Telephone / Email
Parsons	Tammy Chang / Project Chemist	512-719-6092 Tammy.chang@parsons.com
TestAmerica Laboratories Inc.	Elaine Wild / Laboratory Director	314-298-8566 elaine.wild@testamericainc.com
TestAmerica Laboratories Inc.	Terry Romanko / Technical Director	314-298-8566 terry.romanko@testamericainc.com
TestAmerica Laboratories Inc.	Dennis Konopka / Gas chromatography/GC–mass spectrometry (GC/GCMS) Extractable Organics Manager	314-298-8566 dennis.konopka@testamericainc.com

April 10, 2017: Parsons Project Manager met with the RPM for a site walk. A summary of issues discussed include the following:

- o Parsons to provide Contractor Access Request Form to Becky Shreffler at Rebecca.shreffler@vistasciences.com at least 48 hours in advance. Parsons may submit a list of all personnel who are expected to access the facility for a 6-month time period.
- o Kevin Sedlak's staff will provide a safety orientation.
- o Parsons to observe base speed limit of 20 miles per hour (mph) and use caution around people and heavy vehicles.
- o The Post closes at 4:30 pm, and work shall not be conducted after this time.
- o Building 1036 may be used to process samples.
- o No toilet facilities are available but hydrant water is available.
- o If Parsons needs on-site support, Parsons should contact Vista for a bid. The Vista PM is Allan Brillinger who can be reached at 330-872-8009 or Allan.brillinger@vistasciences.com.
- o Parsons needs to add volatile organic compounds (VOCs) to the analytical list for CC-74.
- o At CC RVAAP-70 there is a storm water drain located at the southwest corner of the building adjacent to the road. It is clear that wash water from the Outdoor Wash Rack Area could have drained to this location. It may not be possible to remove the grate but Parsons may be able to take a hand auger or grab sample at this location.
- o Parsons needs to look for VOCs in the building.
- o Boring logs should state the cause of refusal at any location (e.g. bedrock, concrete).

List of Attendees

Organization	Name / Role	Telephone / Email
Parsons	Ed Heyse, PhD, PE / PM	303-563-9452 Ed.heyse@parsons.com
USACE	Kevin Sedlak, RPM	614-336-6136 Kevin.M.Sedlak.ctr@mail.mil

WORKSHEET #10: CONCEPTUAL SITE MODEL

(UFP-QAPP Manual Section 2.5.2)

CONCEPTUAL SITE MODEL (CSM)

The CSM is a description of a site and its environment that can be used to depict the nature of potential contamination, its location, and the possible interactions of human and environmental receptors with that contamination. The CSM summarizes which potential receptor exposure pathways for contaminated media are (or may be) complete and which are (and are likely to remain) incomplete. An exposure pathway is considered incomplete unless *all four* of the following elements are present (United States Environmental Protection Agency [USEPA], 1988):

- o A source of contamination
- o An environmental transport and/or exposure medium
- o A point of exposure at which the contaminant can interact with a receptor
- o A receptor and a likely route of exposure at the exposure point.

If any single factor was not present, the pathway would be incomplete. An incomplete exposure pathway indicates there are no current means by which a receptor (human or ecological) can be exposed to either contamination. In this case, no hazards or risks from exposure to contamination would be expected. This information can be used to focus the investigation of the site by suggesting which complete or potentially complete exposure pathways need to be evaluated. The CSM is a “living document” that is based on existing knowledge and, therefore, can and should be updated throughout the course of the project as more data become available. For the purposes of this Work Plan, a preliminary CSM was developed for the three AOCs at Camp Ravenna.

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Table B-10-1: Conceptual Site Models

Known/Suspected Contamination Source(s)	Potential/Suspected Location and Distribution	Source or Exposure Media	Current and Future Receptors	Potentially Complete Exposure Pathways
CC RVAAP-69 Building 1048 Fire Station				
Carbon tetrachloride, used in fire extinguishers, was the only contaminant for this AOC identified in the Historical Records Review (HRR) (SAIC, 2011b). Primary (continuing) contaminant sources (discharge of fire extinguishers and stored chemicals) were eliminated and the site is no longer used as a fire station. Secondary sources (contaminated soil) were identified during the previous remedial investigations. The most notable secondary sources were carbon tetrachloride and chloroform in subsurface soil. Groundwater has not yet been sampled at this AOC.	Soil and groundwater behind and within the footprint of the former fire station building, and downgradient groundwater.	Soil and groundwater	Hypothetical residents, industrial workers, and construction workers.	Exposure to contaminated soil and groundwater.
CC RVAAP-70 East Classification Yard, Building 47-40 (Round House) below the concrete floor and below the floor of the Interior Repair Pit				
Past operations at the AOC may have resulted in a release of chemicals from site operations. No primary (continuing) sources of contamination exist as operations have been discontinued and most of the infrastructure (except for the Round House Building 47-40 and rails) has been demolished. Secondary sources such as chemicals in soil and dry sediment may exist from releases related to maintenance operations, petroleum storage, or herbicide storage and mixing operations. To date, the only COPCs identified at this decision unit are polycyclic aromatic hydrocarbons in surface and subsurface soil samples around the perimeter of the building.	Soil below the floor, especially in areas with oily sludge, staining, and/or cracks in the concrete floor. The interior repair pit and oil storage area are areas with staining or accumulated oily sludge.	Soil	Hypothetical residents, industrial workers, construction workers	Exposure to contaminated soil

Table B-10-1: Conceptual Site Models

CC RVAAP-70 East Classification Yard, Outdoor Wash Rack Area				
Historical activities at the Outdoor Wash Rack Area used to wash box cars which carried explosives. The wash rack was also reportedly used to wash engines. To date, the only COPCs identified at this decision unit are polycyclic aromatic hydrocarbons in surface and subsurface soil samples. However, odor and elevated PID measurements were noted on boring logs at approximately 6 to 7 feet bgs.	Soil and sediment at the Outdoor Wash Rack Area and associated storm drain.	Soil and sediment	Hypothetical residents, industrial workers, construction workers	Exposure to contaminated soil and sediment
CC RVAAP-74 Building 1034 Motor Pool Hydraulic Lift				
Historical leaks of hydraulic fluid from the former lift system. Potential for release of VOCs.	Soil and possibly groundwater below the concrete floor of Building 1034 near the hydraulic lift.	Soil and groundwater	Hypothetical residents, industrial workers, construction workers, future workers in Building 1034.	Exposure to contaminated soil and groundwater. Potential soil vapor intrusion if VOCs are present.

WORKSHEET #11: PROJECT QUALITY OBJECTIVES

(UFP-QAPP Manual Section 2.6.1)

The DQOs are presented in **Tables B-11-1** through **B-11-3**.

Table B-11-1: Data Quality Objectives and Summary of Technical Approach
CC RVAAP-69 Building 1048 Fire Station
Camp Ravenna

State the Problem	Identify the Decision to be Made	Identify Inputs to the Decision	Define the Study Boundaries	Develop Decision Rules	Specify Limits On Decision Errors	Optimize the Design for Obtaining Data (Technical Approach Summary)
Data gaps have been identified that need to be addressed to complete the RI for the site. The following data gaps have been identified for AOC CC RVAAP-69:						
The vertical extent of carbon tetrachloride in soil at SB101 is not defined.	Define the vertical extent of carbon tetrachloride and its decay products (chlorinated methanes) in soil.	Existing data and new analytical results from proposed discrete soil samples collected at depth near where the maximum carbon tetrachloride concentrations in soil have been detected historically.	All proposed soil borings and in the unconsolidated aquifer.	- If carbon tetrachloride and its decay products (chlorinated methanes) are detected in a sample above PALs (Worksheet #15), then that sample will be considered within the area of contamination for defining the vertical extent. - If carbon tetrachloride and its decay products (chlorinated methanes) are not detected in a sample above PALs (Worksheet #15), then that sample will be considered outside the area of contamination for defining the vertical extent.	All sampling and analysis will be performed in accordance with the procedures outlined in the Appendix A, the Field Sampling Plan (FSP)< and this UFP-QAPP.	Install a new boring in the immediate vicinity of the earlier borings SB101 and RV5-SB2. The boring will extend from the ground surface to bedrock (estimated at 45 feet below ground surface [bgs]). The length of the core will be screened with a PID monitor. If no evidence of a chemical release is noted (no odors, staining, or elevated PID readings), then discrete soil samples will be collected at 5-foot intervals beginning at 10 feet bgs (i.e., samples at 10, 15, 20, 25, 30, 35, 40 and 45 feet bgs). However, if evidence of potential chemical release is observed, then discrete soil samples will be collected from the interval exhibiting the highest odors, staining, or elevated PID readings. Soil samples will be analyzed for carbon tetrachloride and its decay products (chlorinated methanes). The new boring will be continuously logged.

Table B-11–1: Data Quality Objectives and Summary of Technical Approach
CC RVAAP-69 Building 1048 Fire Station
Camp Ravenna

State the Problem	Identify the Decision to be Made	Identify Inputs to the Decision	Define the Study Boundaries	Develop Decision Rules	Specify Limits On Decision Errors	Optimize the Design for Obtaining Data (Technical Approach Summary)
o The lateral extent of carbon tetrachloride contamination in soil is not well defined below a depth of 1018 feet above mean sea level (amsl).	o Define the horizontal extent of carbon tetrachloride and its decay products (chlorinated methanes) in soil.	Existing data and new analytical results from proposed discrete soil samples collected at depth near where the maximum carbon tetrachloride concentrations in soil have been detected historically.	All proposed soil borings and in the unconsolidated aquifer.	o If carbon tetrachloride and its decay products (chlorinated methanes) are detected in a sample above PALs (Worksheet #15), then that sample will be considered within the area of contamination for defining the horizontal extent. o If carbon tetrachloride and its decay products (chlorinated methanes) are not detected in a sample above PALs (Worksheet #15), then that sample will be considered outside the area of contamination for defining the horizontal extent.	All sampling and analysis will be performed in accordance with the procedures outlined in the FSP and this UFP-QAPP.	Install additional soil boring locations north (near SB105), east (near SB102) and south (near SB103) of the presumed release area (RV5-SB2). All new borings will be continuously logged. These soil borings will extend to the water table (approximately 10 to 20 feet bgs at CC RVAAP-69). The length of the cores will be screened with a PID monitor. If no evidence of a chemical release is noted (no odors, staining, or elevated PID readings), then discrete soil samples will be collected at 5-foot intervals beginning at 10 feet bgs (i.e., samples at 10, 15 and 20 feet bgs). However, if evidence of potential chemical release is observed, then discrete soil samples will be collected from the interval exhibiting the highest odors, staining, or elevated PID readings. Soil samples will be analyzed for carbon tetrachloride and its decay products (chlorinated methanes). The ground surface at CC RVAAP-69 varies between 1025 feet and 1029 feet amsl. These proposed samples will define the extent of carbon tetrachloride (and decay product) contamination in the vadose zone and upper portions of the saturated zone to an elevation as deep as 1005 feet amsl. Contamination is defined to an elevation of about 1012 feet amsl in distal sample locations (SB106, SB109, and SB104), so additional step out samples will not be necessary regardless of whether carbon tetrachloride is detected in any of the proposed soil samples. If carbon tetrachloride extends deeper into the subsurface, delineation will focus on delineating any resulting groundwater plume.

Table B-11–1: Data Quality Objectives and Summary of Technical Approach
CC RVAAP-69 Building 1048 Fire Station
Camp Ravenna

State the Problem	Identify the Decision to be Made	Identify Inputs to the Decision	Define the Study Boundaries	Develop Decision Rules	Specify Limits On Decision Errors	Optimize the Design for Obtaining Data (Technical Approach Summary)
Groundwater was not evaluated as part of the previous investigations.	- What is the direction of groundwater flow? - What is the extent of carbon tetrachloride, if present, at concentrations above the Adult Resident Facility-Wide Human Health Cleanup Goals (FWCUG) (0.204 micrograms per liter [µg/L] in groundwater?	Groundwater elevations from wells in at CC RVAAP-69 and adjacent AOC CC RVAAP-74. Screening level analytical results from groundwater grab samples and analytical results from monitoring wells to delineate carbon tetrachloride contamination in groundwater (if any).	All proposed groundwater grab samples and monitoring wells.	- If carbon tetrachloride concentrations are detected in groundwater grab samples in groundwater above the Adult Resident FWCUG (0.204 µg/L), then the sampling location will be considered within the area of contamination and step-out grab samples will be collected. - If carbon tetrachloride concentrations are not detected in groundwater grab samples in groundwater above the Adult Resident FWCUG (0.204 µg/L), then the sampling location will be considered outside of the area of contamination no further grab samples will be collected in that direction. - If a carbon tetrachloride groundwater plume is detected and delineated with grab samples, a minimum of three monitoring wells will be installed; one in the near the maximum carbon tetrachloride concentration in soil (near RV5-SB2), one upgradient of the former fire station, and one at the downgradient extent of the plume. More than the minimum of three monitoring wells may be needed to confirm the extent of potential carbon tetrachloride contamination beneath CC RVAAP-69. - If a carbon tetrachloride groundwater plume is not indicated in the grab samples, at a minimum one monitoring well will be installed at the near the maximum carbon tetrachloride concentration in soil (near RV5-SB2). At least two additional monitoring wells will be installed to ensure that the local potentiometric surface can be mapped. - If dense non-aqueous phase liquid (DNAPL) is present in the bottom of the CC RVAAP-69 monitoring wells, its thickness will be measured with an oil/water interface probe capable of detecting the presence of non-aqueous phase liquid within the well casing.	All sampling and analysis will be performed in accordance with the procedures outlined in the FSP and this UFP-QAPP.	In order to efficiently optimize the number of monitoring wells needed to define the direction of groundwater flow and monitor a plume, Parsons will first collect grab samples using a Screenpoint 16 sampler (or install temporary wells if groundwater grab samples cannot be collected from well points, and all temporary wells will be developed prior to sampling). Groundwater grab samples will be collected from the upper portions of the saturated zone within the unconsolidated aquifer. These screening samples will be used to select monitoring well locations. Initial groundwater grab samples will be collected from near the presumed release point (near RV5-SB2) and in locations approximately 50 to 70 feet downgradient (east to southeast) of the presumed release point (i.e., between the former Building 1048 Fire Station and George Road/South Service Road). Groundwater grab samples will be analyzed for chlorinated methanes on a quick turn (24 hour) basis, and additional step-in or step-out samples will be collected until the plume is delineated or until groundwater grab samples establish that groundwater is not contaminated with carbon tetrachloride at concentrations above the Resident Receptor FWCUG (0.204 µg/L). If a carbon tetrachloride groundwater plume is detected and delineated, the orientation of the plume will provide an indication of the local groundwater flow direction. Monitoring wells will be installed with screens set across the water table to maximize the likelihood of intercepting the contaminant plume because the maximum concentrations in soil to date are in the interval of 1015 to 1020 feet amsl. However, if it is determined that carbon tetrachloride extends deeper in the unconsolidated soil, it may be necessary to install additional deeper wells. The local potentiometric surface will be mapped using groundwater elevations in wells installed at CC RVAAP-69 and adjacent AOC CC RVAAP-74. Monitoring wells will be developed and sampled quarterly for the first year.

Table B-11-2: Data Quality Objectives and Summary of Technical Approach
CC RVAAP-70 East Classification Yard
Camp Ravenna

State the Problem	Identify the Decision to be Made	Identify Inputs to the Decision	Define the Study Boundaries	Develop Decision Rules	Specify Limits On Decision Errors	Optimize the Design for Obtaining Data (Technical Approach Summary)
Data gaps have been identified that need to be addressed to complete the SI for the site. The following data gaps have been identified for CC RVAAP-70:						
o Soil underneath the floor inside Building 47-40 has not been sampled at locations that are stained and cracked.	o Do contaminant concentrations in soil below the concrete floor exceed PALs (Worksheet #15)	o Analytical results from both historical data and biased sampling below the floor.	Two biased soil borings installed below the floor.	- o If chemicals are detected above PALs (Worksheet #15), then a remedial investigation may be warranted. - o If chemicals are not detected at concentrations above PALs (Worksheet #15), then no additional investigation will be warranted.	All sampling and analysis will be performed in accordance with the procedures outlined in the FSP and this UFP-QAPP.	After sludge removal, install two soil borings below the floor at locations biased towards areas with sludge, staining, and/or cracks in the concrete. Four discrete soil samples (0-2', 2-4', 4-6', and 6-7' or until sandstone is reached) will be collected at each location and analyzed for VOCs, semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), and TAL metals. All new borings will be continuously logged.
o Soil underneath the floor of the Interior Repair Pit (DU04) has not been sampled for VOCs.	o Do contaminant concentrations in soil within the pit exceed PALs (Worksheet #15)?	o Analytical results from proposed soil sampling.	Two biased soil borings installed within the interior pit.	- o If chemicals are detected above PALs (Worksheet #15), then a remedial investigation may be warranted. - o If chemicals are not detected at concentrations above PALs (Worksheet #15), then no additional investigation will be warranted.	All sampling and analysis will be performed in accordance with the procedures outlined in the FSP and this UFP-QAPP.	After sludge removal, install two soil borings within the pit at locations biased towards areas with sludge, staining, and/or cracks in the concrete. The length of the core will be screened with a photoionizing detector (PID), and if no evidence of contamination is noted, then a discrete soil sample will be collected from 0-2 and 2-4 feet bgs or until sandstone is reached. If evidence of contamination is observed a discrete soil sample will be collected from that interval. The discrete soil samples will be analyzed for VOCs. All new borings will be continuously logged.
o Discrete soil samples have not been sampled and analyzed for VOCs from locations at the Outdoor Wash Rack Area at the vertical interval where odor and elevated PID readings were noted in boring logs.	o Do VOC concentrations in soil in the Outdoor Wash Rack Area exceed PALs (Worksheet #15)?	o Analytical results for VOC analysis.	Two biased soil borings to be installed adjacent to historical borings (SB01 and SB05) which had elevated PID readings in the 6-7' bgs depth interval.	- o If chemicals are detected above PALs (Worksheet #15), then a remedial investigation may be warranted. - o If chemicals are not detected at concentrations above PALs (Worksheet #15), then no additional investigation will be warranted.	All sampling and analysis will be performed in accordance with the procedures outlined in the FSP and this UFP-QAPP.	Install two soil borings adjacent to historical soil borings SB01 and SB05. The length of the core will be screened with a PID and if no evidence of contamination is noted a discrete soil sample will be collected from the 6-7' interval which corresponds to the historical elevated PID readings. If evidence of contamination is observed a discrete soil sample will be collected from that interval. The discrete soil samples will be analyzed for VOCs. All new borings will be continuously logged.
o Sediment from the storm drain at the southwest corner of Building 47-40 has not been sampled for contamination from the Outdoor Wash Rack.	o Do contaminant concentrations at the culvert exceed PALs (Worksheet #15)?	o Analytical results for surface sediment and subsurface soil.	Sediment sample from storm drain and one soil boring to be installed adjacent to the storm drain.	- o If chemicals are detected above PALs (Worksheet #15), then remedial investigation may be warranted. - o If chemicals are not detected at concentrations above PALs (Worksheet #15), then no additional investigation will be warranted.	All sampling and analysis will be performed in accordance with the procedures outlined in the FSP and this UFP-QAPP.	Collect sediment sample from within the storm drain. The surface sediment sample (0-1' bgs) will be analyzed for PCBs, SVOCs, and explosive; VOCs are not expected to be present at the ground surface because they are readily volatile. Install one boring adjacent to the storm drain. Subsurface soil (1-4' and 4-7' bgs) samples will be collected and analyzed for PCBs, VOCs, SVOCs, and explosives. All new borings will be continuously logged.

**Table B-11-3: Data Quality Objectives and Summary of Technical Approach
CC RVAAP-74 Building 1034 Motor Pool Hydraulic Lift
Camp Ravenna**

State the Problem	Identify the Decision to be Made	Identify Inputs to the Decision	Define the Study Boundaries	Develop Decision Rules	Specify Limits On Decision Errors	Optimize the Design for Obtaining Data (Technical Approach Summary)
Data gaps have been identified that need to be addressed to complete the SI for the site. The following data gaps have been identified for CC RVAAP-74:						
o It is unclear if the “free product” historically identified in 74-1034-HL-SB18 at 7.5 to 8.5 feet bgs is mobile LNAPL or residual LNAPL.	o What is the nature of the “free product” identified in 74-1034-HL-SB18 at 7.5 to 8.5 feet bgs? Nature includes (1) is the organic contaminant mobile or free flowing, and (2) is it hydraulic fluid or some other organic liquid?	Existing data and new analytical results from proposed discrete soil samples collected near where the “free product” identified in 74-1034-HL-SB18 at 7.5 to 8.5 feet bgs.	Soil boring to be completed adjacent to former boring SB18.	- o If the organic contaminant appears to flow freely from the soil core, it is “free product.” If the organic contaminant does not flow freely from the soil core, it is “residual saturation.” - o If the organic contaminant is identified as mineral oil based on the chromatograph, then the June 2017 USEPA RSL for mineral oils will be used to screen for COPC. - o If the “free product” is not identified as mineral oil based on the chromatograph, then the BUSTR Action Level will be used to screen for COPC.	All sampling and analysis will be performed in accordance with the procedures outlined in the FSP and this UFP-QAPP.	Install a new boring in the immediate vicinity of the earlier boring 74-1034-HL-SB18; the soil will be photographed, visually inspected, and scanned with a PID to evaluate if there is mobile LNAPL (i.e., oil that flows out of the formation versus residual saturation or oil that is essentially immobile and held in capillary spaces). An ultraviolet light (UV) light will also be used to help screen for the presence of hydraulic oil. A soil sample will be collected from 7.5 to 8.5 feet bgs to confirm what was found by evaluating the chromatograph to a known sample of hydraulic fluid (i.e., mineral oil). Soil samples will be analyzed for VOCs, TPH-C ₁₀ to C ₂₀ and C ₂₀ to C ₃₄ distillate ranges, and PCBs. The new boring will be continuously logged.
o The horizontal extent of soil contamination is not defined east and south of 74-1034-HL-SB18.	o Define the horizontal extent of contamination in soil.	Existing data and new analytical results from proposed discrete soil samples.	Proposed soil borings.	- o If chemicals are detected in a sample above PALs (Worksheet #15), then that sample will be considered within the area of contamination and this information will be used in defining the horizontal extent. - o If chemicals are not detected in a sample above PALs (Worksheet #15), then that sample will be considered outside the area of contamination and this information will be used in defining the horizontal extent.	All sampling and analysis will be performed in accordance with the procedures outlined in the FSP and this UFP-QAPP.	Parsons will advance soil borings in the immediate vicinity of the earlier borings 1034-HL-SB-14 and 74-1034-HL-SB-19, and collect soil samples from 7.5 to 8.5 feet to evaluate the horizontal extent in east and south of 74-1034-HL-SB18. Soil samples will be analyzed for VOCs, TPH-C ₁₀ to C ₂₀ and C ₂₀ to C ₃₄ distillate ranges. All new borings will be continuously logged.
o Only one soil sample was analyzed for VOCs.	o Are VOCs present in soil above PALs (Worksheet #15)	New analytical results from proposed discrete soil samples.	All proposed soil borings and groundwater monitoring wells.	- o If VOCs are detected above PALs (Worksheet #15), then VOCs will be considered a COPC at the AOC and included in future analysis and decision-making. - o If VOCs are not detected above PALs (Worksheet #15), then VOCs will not be considered a COPC at the AOC and not included in future analysis or decision-making.	All sampling and analysis will be performed in accordance with the procedures outlined in the FSP and this UFP-QAPP.	All new soil samples from this AOC will be analyzed for VOCs in addition to TPH-C ₁₀ to C ₂₀ and C ₂₀ to C ₃₄ distillate ranges. One soil sample will also be analyzed for PCBs. Groundwater samples analyzed as part of this data gap investigation at CC RVAAP-74 will be analyzed for VOCs and PAHs. Groundwater samples from groundwater monitoring wells will also be analyzed for PCBs.

**Table B-11–3: Data Quality Objectives and Summary of Technical Approach
CC RVAAP-74 Building 1034 Motor Pool Hydraulic Lift
Camp Ravenna**

State the Problem	Identify the Decision to be Made	Identify Inputs to the Decision	Define the Study Boundaries	Develop Decision Rules	Specify Limits On Decision Errors	Optimize the Design for Obtaining Data (Technical Approach Summary)
o Groundwater was not evaluated as part of the previous investigations, and it is unknown if groundwater is impacted or if enough hydraulic fluid was released to accumulate as LNAPL at the water table.	o What is the direction of groundwater flow? o What is the horizontal extent of VOCs PAHs, or PCBs, if present, at concentrations above PALs (Worksheet #15) in groundwater?	Groundwater elevations from wells in at CC RVAAP-74 and adjacent AOC CC RVAAP-69. Screening level analytical results from groundwater grab samples and analytical results from monitoring wells delineate hydraulic fluid contamination in groundwater (if any).	All proposed groundwater grab samples and monitoring wells.	o If contaminant concentrations are detected in groundwater grab samples in groundwater above PALs (Worksheet #15) for VOCs, PAHs or PCBs, then the sampling location will be considered within the area of contamination and step-out grab samples will be collected. o If contaminant concentrations are not detected in groundwater grab samples in groundwater above PALs (Worksheet #15) for VOCs PAHs, or PCBs, then the sampling location will be considered outside of the area of contamination no further grab samples will be collected in that direction. o If a groundwater plume is detected above PALs (Worksheet #15) and delineated with grab samples, a minimum of three monitoring wells will be installed; one in the greatest TPH-C₂₀ to C₃₄ detection in soil (near 74-1034-HL-SB18), one upgradient of the hydraulic lift, and one at the downgradient extent of the plume. o If a groundwater plume is detected above PALs (Worksheet #15), then at a minimum one monitoring well will be installed at the maximum TPH-C₂₀ to C₃₄ detection in soil (near 74-1034-HL-SB18). At least two additional monitoring wells will be installed to ensure that the local potentiometric surface can be mapped.	All sampling and analysis will be performed in accordance with the procedures outlined in the FSP and this UFP-QAPP.	In order to efficiently optimize the number of monitoring wells needed to define the direction of groundwater flow and monitor a plume (if any), Parsons will first collect grab samples using a Screenpoint 16 sampler (or install temporary wells if groundwater grab samples cannot be collected from well points, and all temporary wells will be developed prior to sampling). Groundwater grab samples will be collected from the upper portions of the saturated zone within the unconsolidated aquifer. These screening samples will be used to select monitoring well locations. Initial groundwater grab samples will be collected approximately 20 feet downgradient (east and southeast) of soil boring 74-1034-HL-SB18 where the maximum TPH-C₂₀ to C₃₄ concentrations were detected in 2013 (i.e., outside the southeast corner of Building 1034). Groundwater grab samples will be analyzed for VOCs and PAHs on a quick turn (24 hour) basis. Additional step-out samples will be collected until the plume is delineated or until groundwater grab samples establish that groundwater is not contaminated at concentrations above PALs (Worksheet #15) for VOCs or PAHs. If a plume is detected, a third well will be installed near the downgradient edge of the plume. Monitoring wells will be installed with screens set across the water table to maximize the likelihood of intercepting LNAPL or a contaminant plume. The fluid levels in the wells will be measured to detect LNAPL (if any), and groundwater samples will be analyzed for VOCs, PAHs, and PCBs. The local potentiometric surface will be mapped using groundwater elevations in wells installed at CC RVAAP-74 and adjacent AOC CC RVAAP-69. Monitoring wells will be developed and sampled quarterly for the first year. The RI report will evaluate and recommend any post-RI monitoring requirements. Post-RI monitoring will be addressed as part of the facility-wide groundwater monitoring program.

WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA (MPC)

(UFP-QAPP Guidance Manual, Section 5.2.1)

The below table summarizes the MPCs that have been established for laboratory data assessment. The quality of the sampling procedures and laboratory results will be evaluated for compliance with DQOs through a review in accordance with the procedures described in [Worksheet #37](#). Sample collection procedures and analytical methods/standard operating procedures (SOPs) are summarized on [Worksheet #21](#) and [Worksheet #23](#), respectively.

Table B-12-1: Measurement Performance Criteria (MPC) for Sample Collection

Data Quality Indicator (DQIs)	MPC	QC Sample and/or Activity Used to Assess Measurement Performance
Completeness	Planned no. of samples matches no. of samples collected.	Project Chemist reviews CoC forms vs. Work Plan
Completeness	Planned QC samples matches no. of QC samples collected	Project Chemist reviews CoC forms vs. Work Plan
Completeness	CoC form lists correct samples and analyses	Project Chemist reviews CoC forms vs. Work Plan
Accuracy	CoC form correctly filled out and signed	Project Chemist reviews CoC forms

Table B-12-2: MPC for VOCs, SVOCs, PAHs, PCBs, Explosives, TAL Metals, and TPH-C₁₀ to C₂₀ and C₂₀ to C₃₄ in Soil, Sediment, and Water

Laboratory: TA-St. Louis

Matrix: Soil, Sediment, and Water

Procedure: Grab/Discrete /sediment and groundwater

DQIs	QC Sample and/or Activity Used to Assess Measurement Performance	MPC
Accuracy	Method Blank (MB)	< ½ Limit of Quantitation (LOQ) or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.
Accuracy	Laboratory Control Sample (LCS)	%Recovery (%R), See Worksheet #28
Accuracy/Precision/Bias	Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	%R, See Worksheet #28
Precision/Bias	matrix spike/matrix spike duplicate (MS/MSD)	%RPD, see Worksheet #28
Accuracy/Bias/Precision	Field Duplicate (FD)	The relative percent difference (RPD) for results above the LOQ must not exceed 60% for soil/sediment and 40% for water
Accuracy/Bias Matrix effects	Serial Dilution (if MS/MSD fail) for metal analysis	±10% Difference (D)
Accuracy/Bias Matrix effects	Post-Digestion Spike Analysis (if MS/MSD fail)	80-120%R
Accuracy	Interference Check Standard	±20% of the true value
Sensitivity	Low Level Calibration Verification	±20% of the true value
Completeness	Data Completeness Check	>90% sample collection, >90% laboratory analysis

Table B-12-3: MPC for Waste Characterization Analyses for Investigative-Derived Waste (IDW) samples

Laboratory: TA-St. Louis

Matrix: Soil and Water

Procedure: Grab/discrete for IDW soil or IDW water

DQIs	QC Sample and/or Activity Used to Assess Measurement Performance	MPC
Accuracy/Bias	MB	$< \frac{1}{2}$ LOQ or $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit, whichever is greater.
Accuracy/Bias	LCS	%R, See Worksheet #28
Accuracy/Bias	Surrogate Spike	%R, See Worksheet #28
Completeness	Data Completeness Check	>90% sample collection, >90% laboratory analysis

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WORKSHEET #13: SECONDARY DATA USES AND LIMITATIONS

(UFP-QAPP Manual Section 2.7)

This work sheet identifies sources of secondary data (i.e., data generated for purposes other than this specific project or data pertinent to this project generated under a separate QAPP) and summarizes information relevant to their uses for the current project.

Data Type	Source	Data Uses Relative to SI	Factors Affecting the Reliability of Data and Limitations on Data Use
Past DoD military history at Camp Ravenna	Historical Records Review Report for the 2010 Phase I Remedial Investigation Services at Compliance Restoration Sites (9 Areas of Concern). SAIC, 2011b	Locations and historical use data on the three AOCs addressed in this Work Plan	Limited data available regarding potential releases
Results of previous investigations	Preliminary Draft Remedial Investigation Report CC RVAAP-69 Building 1048 Fire Station. ECC and AMEC, 2014 Draft Site Inspection Report CC RVAAP-70 East Classification Yard ECC, 2015 Draft Remedial Investigation Report CC RVAAP-74 Building 1034 Motor Pool Hydraulic Lift. ECC and AMEC, 2015.	AOC-specific data	Limited data collected.

WORKSHEET #14 AND 16: PROJECT TASKS & SCHEDULE

(UFP-QAPP Manual Section 2.8.2)

14.1 Project Tasks

The field operations involve multiple elements, or “definable features of work (DFWs),” that are required to achieve the DQOs/project quality objectives (PQOs). This worksheet summarizes these DFWs and the associated component tasks. A detailed discussion of each of the DFWs is included on Worksheet #17 and the specific field procedures are described in Section 4 of the FSP provided as Appendix A to the Work Plan.

14.2 Project Schedule

The initial project schedule is detailed in the Project Management Plan (PMP) and will be routinely updated and distributed as directed by the COR primarily as part of the monthly status reports.

Definable Features of Work and Associated Tasks

Definable Feature of Work	Associated Tasks
Mobilization	- o Preparation (review plans, make travel arrangements, etc.) - o Arrange for equipment and vehicles, and get them delivered to the site - o Set up site communications - o Conduct site-specific training and briefing for required field personnel - o Request all laboratory supplies to be delivered to the hotel near the site
Site Preparation	- o Conduct vegetation removal and remove oily sludge from the Round House at CC RVAAP-70 East Classification Yard - o Conduct underground utility locate
Sampling	- o Collect soil, groundwater, and sediment samples - o Label all sample containers and pack them properly in coolers - o Ship all samples to the laboratory with chain-of-custody
Demobilization	o Upon completion of field activities all personnel, equipment and materials will be removed from the sites

WORKSHEET #15: PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION / QUANTITATION LIMITS

(EPA UFP-QAPP Guidance Manual, Section 2.8.1)

This worksheet provides the parameters to be analyzed and their associated detection limits (DLs), limits of detection (LODs), and limits of quantitation (LOQs) in order to satisfy the overall DQOs. The Project Action Limits (PALs), as referenced in the DQOs, are also included and were determined as follows:

- o PALs for chemicals in groundwater were determined using the following hierarchy: 1. Facility-Wide Human Health Cleanup Goals (FWCUGs) (SAIC 2010), 2. USEPA Maximum Contaminant Level (MCL), if available, and 3. June 2017 USEPA Regional Screening Levels (RSLs) for chemicals at Superfund Sites Tapwater Screening Levels used with a target risk of 1E-6 and a target HQ of 1 (USEPA, 2016c) (available at: <https://www.epa.gov/risk/regional-screening-levels-rsls-generic-tables-june-2017>).
- o PALs for TPH in soil were determined using Bureau of Underground Storage Tanks (BUSTR) (Class 1 Soil Action Levels, Table 3-8, 2017). PALs for other chemicals in soil were determined using the following hierarchy: 1) FWCUGs (SAIC 2010) and 2) June 2017 USEPA RSLs for Resident Soil Screening Levels used with a target risk of 1E-6 and a target HQ of 1 (USEPA, 2016c) (available at: <https://www.epa.gov/risk/regional-screening-levels-rsls-generic-tables-june-2017>).
- o PALs for IDW Solid/Water Toxicity Characteristic Leaching Procedure (TCLP) came from 40 Code of Federal Regulations (CFR) §261.24 (available at: https://www.ecfr.gov/cgi-bin/text-idx?node=se40.26.261_124&rgn=div8).

If the sensitivity requirements are not met for a particular analyte, the Parsons Team will evaluate whether the data can still be used for project decisions. If non-detect values exceed the June 2017 Regional RSLs, data is considered usable if the analyte is not a site-related chemical. Analytes that are not site-related chemicals, and thus are not expected to be found, do not impact decision making. For analytes that are site-related, the Parsons team will use a “weight of evidence” approach to evaluate the likelihood of the chemical’s presence. This approach uses available data that does meet sensitivity requirements to evaluate the presence or absence of the compound in other samples or other similar compounds and/or degradation products for the analyte in question. If sufficient evidence exists to determine a compound was likely not present due to the absence of similar compounds and breakdown products, the Parson Team will conclude that the analyte was not present, and thus data are usable for decision making. If it cannot be determined that the compound is likely not present, then the risk will be calculated assuming that the compound is present at the DL. This calculated risk will be presented as an uncertainty in the risk assessment, and the risk will not be included in the estimation of cumulative risk.

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**Table B-15-1: Project Action Limits for TA-St. Louis
Matrix: Water**

Analytical Group: VOCs in Water using 8260C							
Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
1,1,1-Trichloroethane	71-55-6	200	MCL	2.0	1.0	0.25	0.17
1,1,2,2-Tetrachloroethane ²	79-34-5	0.069	FWCUG	0.069	1.0	0.25	0.10
1,1,2-Trichloroethane	79-00-5	5	MCL	2.0	1.0	0.25	0.13
1,1-Dichloroethane	75-34-3	2.8	RSL	2.0	1.0	0.25	0.07
1,1-Dichloroethene	75-35-4	7	MCL	2.0	1.0	0.25	0.10
1,2-Dibromoethane ²	106-93-4	0.05	MCL	0.05	1.0	0.50	0.13
1,2-Dichloroethane ²	107-06-2	0.155	FWCUG	0.155	1.0	0.25	0.21
1,2-Dichloropropane	78-87-5	5	MCL	2.0	1.0	0.25	0.10
1,2,4 - Trimethylbenzene	95-63-6	5.6	RSL	2.0	1.0	0.25	0.17
2-Butanone (MEK)	78-93-3	560	RSL	10.0	5.0	1.0	0.47
2-Hexanone ²	591-78-6	3.8	RSL	10.0	5.0	0.50	0.25
4-Methyl-2-pentanone (MIBK)	108-10-1	630	RSL	10.0	5.0	0.50	0.22
Acetone	67-64-1	1400	RSL	4.0	2.0	1.0	0.55
Benzene ¹	71-43-2	0.431	FWCUG	0.431	1.0	0.25	0.10
Bromochloromethane	74-97-5	8.3	RSL	2.0	1.0	0.50	0.14
Bromodichloromethane ²	75-27-4	0.13	RSL	0.13	1.0	0.25	0.14
Bromoform	75-25-2	3.3	RSL	2.0	1.0	0.25	0.17
Bromomethane ¹	74-83-9	0.75	RSL	0.75	2.0	0.50	0.25
Carbon disulfide	75-15-0	81	RSL	4.0	2.0	0.25	0.10
Carbon tetrachloride ¹	56-23-5	0.204	FWCUG	0.204	1.0	0.25	0.18
Chlorobenzene	108-90-7	100	MCL	4.0	2.0	0.25	0.11
Chloroethane	75-00-3	2100	RSL	4.0	2.0	0.25	0.16
Chloroform ¹	67-66-3	0.207	FWCUG	0.207	1.0	0.25	0.10

**Table B-15-1: Project Action Limits for TA-St. Louis
Matrix: Water (continued)**

Analytical Group: VOCs in Water using 8260C							
Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Chloromethane	74-87-3	19	RSL	4.0	2.0	0.25	0.10
<i>cis</i> -1,2-Dichloroethene	156-59-2	70	MCL	2.0	1.0	0.25	0.10
<i>cis</i> -1,3-Dichloropropene	10061-01-5	NE	NE	2.0	1.0	0.25	0.16
Dibromochloromethane ¹	124-48-1	0.87	RSL	0.87	1.0	0.25	0.14
Ethylbenzene	100-41-4	700	MCL	2.0	1.0	0.25	0.12
Methyl tert-Butyl Ether (MTBE)	1364-04-4	14	RSL	4.0	2.0	0.25	0.15
Methylene Chloride	75-09-2	5.34	FWCUG	2.0	1.0	0.50	0.27
Styrene	100-42-5	100	MCL	2.0	1.0	0.25	0.13
Tetrachloroethene ²	127-18-4	0.098	FWCUG	0.098	1.0	0.50	0.18
Toluene	108-88-3	1000	MCL	2.0	1.0	0.25	0.14
<i>trans</i> -1,2-Dichloroethene	156-60-5	100	MCL	2.0	1.0	0.25	0.10
<i>trans</i> -1,3-Dichloropropene	10061-02-6	NE	NE	2.0	1.0	0.25	0.10
Trichloroethene ²	79-01-6	0.031	FWCUG	0.031	1.0	0.50	0.25
Vinyl chloride	75-01-4	2	MCL	2.0	2.0	0.25	0.19
Xylenes, Total	1330-20-7	10000	MCL	2.0	3.0	0.50	0.26

1 = The LOQ is greater than the project action level, but the DL is lower than the project action level. Project criteria are met since data will be flagged and reported down to the DL.

2 = Sensitivity requirements are not met for this analyte so data will be evaluated as described at the beginning of Worksheet #15.

**Table B-15-1: Project Action Limits for TA-St. Louis
Matrix: Water (continued)**

Analytical Group: VOCs Chlorinated Methanes Only in Water using 8260C							
Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Carbon tetrachloride ¹	56-23-5	0.204	FWCUG	0.204	1.0	0.25	0.18
Chloroform ¹	67-66-3	0.207	FWCUG	0.207	1.0	0.25	0.10
Chloromethane	74-87-3	19	RSL	4.0	2.0	0.25	0.10
Methylene Chloride	75-09-2	5.34	FWCUG	2.0	1.0	0.50	0.27

¹ = The LOQ is greater than the project action level, but the DL is lower than the project action level. Project criteria are met since data will be flagged and reported down to the DL.

Analytical Group: PAHs in Water using 8270D-SIM							
Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Acenaphthene	83-32-9	53	RSL	0.4	0.2	0.1	0.035
Acenaphthylene	208-96-8	NE	NE	0.4	0.2	0.1	0.038
Anthracene	120-12-7	180	RSL	0.4	0.2	0.1	0.039
Benzo[a]anthracene ²	56-55-3	0.004	FWCUG	0.004	0.2	0.1	0.031
Benzo[a]pyrene ²	50-32-8	0.00023	FWCUG	0.00023	0.2	0.1	0.053
Benzo[b]fluoranthene ²	205-99-2	0.002	FWCUG	0.002	0.2	0.1	0.055
Benzo[g,h,i]perylene	191-24-2	NE	NE	0.4	0.2	0.1	0.043
Benzo[k]fluoranthene	207-08-9	2.5	RSL	0.4	0.2	0.1	0.073
Chrysene	218-01-9	25	RSL	0.4	0.2	0.1	0.039
Dibenz(a,h)anthracene ²	53-70-3	0.00015	FWCUG	0.00015	0.2	0.1	0.046
Fluoranthene	206-44-0	80	RSL	0.4	0.2	0.1	0.035
Fluorene	86-73-7	29	RSL	0.4	0.2	0.1	0.032
Indeno[1,2,3-cd]pyrene ²	193-39-5	0.002	FWCUG	0.002	0.2	0.1	0.04

**Table B-15-1: Project Action Limits for TA-St. Louis
Matrix: Water (continued)**

Analytical Group: PAHs in Water using 8270D-SIM							
Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Naphthalene ¹	91-20-3	0.17	RSL	0.17	0.2	0.1	0.068
Phenanthrene	85-01-8	NE	NE	0.4	0.2	0.1	0.065
Pyrene	129-00-0	12	RSL	0.4	0.2	0.1	0.037

1 = The LOQ is greater than the project action level, but the DL is lower than the project action level. Project criteria are met since data will be flagged and reported down to the DL.

2 = Sensitivity requirements are not met for this analyte so data will be evaluated as described at the beginning of Worksheet #15.

Analytical Group: Explosives in Water using 8330B							
Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
1,3,5-Trinitrobenzene	99-35-4	59	RSL	0.40	0.20	0.20	0.057
1,3-Dinitrobenzene ¹	99-65-0	0.104	FWCUG	0.104	0.20	0.20	0.10
2,4,6-Trinitrotoluene	118-96-7	0.521	FWCUG	0.50	0.25	0.20	0.080
2,4-Dinitrotoluene ¹	121-14-2	0.12	FWCUG	0.12	0.25	0.20	0.081
2,6-Dinitrotoluene ²	606-20-2	0.122	FWCUG	0.122	0.40	0.20	0.13
2-Amino-4,6-dinitrotoluene ¹	35572-78-2	0.209	FWCUG	0.209	0.30	0.20	0.12
2-Nitrotoluene ¹	88-72-2	0.37	FWCUG	0.37	0.50	0.50	0.095
3-Nitrotoluene ¹	99-08-1	0.17	RSL	0.17	0.50	0.20	0.12
4-Amino-2,6-dinitrotoluene ¹	19406-51-0	0.209	FWCUG	0.209	0.40	0.20	0.12
4-Nitrotoluene	99-99-0	5.01	FWCUG	1.0	0.50	0.20	0.14
HMX	2691-41-0	100	RSL	0.70	0.35	0.20	0.11
Nitrobenzene	98-95-3	0.521	FWCUG	0.50	0.25	0.20	0.082
Nitroglycerin	55-63-0	5.01	FWCUG	3.0	1.5	1.0	0.54
PETN	78-11-5	3.9	RSL	3.0	2.0	1.0	0.61

**Table B-15-1: Project Action Limits for TA-St. Louis
Matrix: Water (continued)**

Analytical Group: Explosives in Water using 8330B							
Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific		
RDX	121-82-4	0.774	FWCUG	0.60	0.30	0.20	0.094
Tetryl	479-45-8	3.9	RSL	0.50	0.25	0.20	0.059

1 = The LOQ is greater than the project action level, but the DL is lower than the project action level. Project criteria are met since data will be flagged and reported down to the DL.
2 = Sensitivity requirements are not met for this analyte so data will be evaluated as described at the beginning of Worksheet #15.

Analytical Group: PCBs in Water using 8082A							
Analyte¹	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
PCB-1016	12674-11-2	0.14	RSL	0.14	1.0	0.80	0.35
PCB-1221	11104-28-2	0.0047	RSL	0.0047	1.0	0.80	0.35
PCB-1232	11141-16-5	0.0047	RSL	0.0047	1.0	0.80	0.35
PCB-1242	53469-21-9	0.213	FWCUG	0.213	1.0	0.80	0.35
PCB-1248	12672-29-6	0.0078	RSL	0.0078	1.0	0.80	0.35
PCB-1254	11097-69-1	0.021	FWCUG	0.021	1.0	0.30	0.22
PCB-1260	11096-82-5	0.213	FWCUG	0.213	1.0	0.30	0.22

1 = Sensitivity requirements are not met for this analyte so data will be evaluated as described at the beginning of Worksheet #15.

**Table B-15-1: Project Action Limits for TA-St. Louis
Matrix: Water (continued)**

Analytical Group: Target Analyte List (TAL) Metals in Water using 6020A/7470A							
Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Aluminum	7429-90-5	1028	FWCUG	100	50	40	20
Antimony ²	7440-36-0	0.389	FWCUG	0.389	5.0	4.0	2.0
Arsenic ²	7440-38-2	0.056	FWCUG	0.056	10	8.0	4.0
Barium	7440-39-3	204	FWCUG	4.0	2.0	1.8	0.90
Beryllium	7440-41-7	2.5	MCL	1.0	0.50	0.40	0.20
Cadmium ¹	7440-43-9	0.456	FWCUG	0.456	0.50	0.40	0.20
Calcium	7440-70-2	NE	NE	200	100	90	45
Chromium	7440-47-3	1214	FWCUG	20	10	8.0	4.0
Cobalt	7440-48-4	20.8	FWCUG	4.0	2.0	1.8	0.90
Copper	7440-50-8	80	RSL	6.0	3.0	0.80	0.40
Iron	7439-89-6	310	FWCUG	100	50	40	20
Lead	7439-92-1	15	RSL	6.0	3.0	2.0	1.0
Magnesium	7439-95-4	NE	NE	100	50	40	20
Manganese	7439-96-5	46.3	FWCUG	4.0	2.0	1.8	0.90
Nickel	7440-02-0	20.8	FWCUG	10.0	5.0	4.0	2.0
Potassium	7440-09-7	NE	NE	200	100	90	45
Selenium	7782-49-2	10	RSL	5.0	5.0	4.0	2.0
Silver	7440-22-4	9.4	MCL	4.0	2.0	1.8	0.90
Sodium	7440-23-5	NE	NE	100	50	40	20
Thallium ²	7440-28-0	0.083	FWCUG	0.083	2.0	1.8	0.90
Vanadium ¹	7440-62-2	6.38	FWCUG	6.38	10	8.0	4.0
Zinc	7440-66-6	312	FWCUG	40	20	15	7.5

**Table B-15-1: Project Action Limits for TA-St. Louis
Matrix: Water (continued)**

Analytical Group: Target Analyte List (TAL) Metals in Water using 6020A/7470A							
Analyte¹	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific		
Mercury ²	7439-97-6	0.063	RSL	0.063	0.20	0.20	0.10

1 = The LOQ is greater than the project action level, but the DL is lower than the project action level. Project criteria are met since data will be flagged and reported down to the DL.

2 = Sensitivity requirements are not met for this analyte so data will be evaluated as described at the beginning of Worksheet #15.

**Table B-15-2: Project Action Limits (PAL) for TA–St. Louis
Matrix: Soil**

Analytical Group: VOCs in Solids using 8260C							
Analyte	CAS Number	PAL (µg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/kg)	Laboratory-specific		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
1,1,1-Trichloroethane	71-55-6	810,000	RSL	10	5.0	1.0	0.43
1,1,2,2-Tetrachloroethane	79-34-5	600	RSL	10	5.0	1.0	0.41
1,1,2-Trichloroethane	79-00-5	150	RSL	10	5.0	1.0	0.57
1,1-Dichloroethane	75-34-3	3,600	RSL	10	5.0	1.0	0.39
1,1-Dichloroethene	75-35-4	23,000	RSL	10	5.0	5.0	1.6
1,2-Dibromoethane	106-93-4	36	RSL	10	5.0	1.0	0.70
1,2-Dichloroethane	107-06-2	460	RSL	10	5.0	1.0	0.87
1,2-Dichloropropane	78-87-5	1,000	RSL	10	5.0	1.0	0.38
1,2,4-Trimethylbenzene	95-63-6	280	RSL	10	5.0	1.0	0.60
2-Butanone (MEK)	78-93-3	2,700,000	RSL	40	20	5.0	1.9
2-Hexanone	591-78-6	20,000	RSL	40	20	5.0	1.8
4-Methyl-2-pentanone (MIBK)	108-10-1	3,300,000	RSL	40	20	5.0	0.73
Acetone	67-64-1	6,100,000	RSL	40	20	10	6.5
Benzene	71-43-2	1,200	RSL	10	5.0	1.0	0.25
Bromochloromethane	74-97-5	15,000	RSL	10	5.0	1.0	0.82
Bromodichloromethane	75-27-4	290	RSL	10	5.0	1.0	0.25
Bromoform	75-25-2	19,000	RSL	10	5.0	1.0	0.37
Bromomethane	74-83-9	680	RSL	20	10	5.0	1.1
Carbon disulfide	75-15-0	77,000	RSL	10	5.0	1.0	0.69
Carbon tetrachloride	56-23-5	650	RSL	10	5.0	1.0	0.52
Chlorobenzene	108-90-7	28,000	RSL	10	5.0	1.0	0.38
Chloroethane	75-00-3	1,400,000	RSL	20	10	1.0	0.52
Chloroform	67-66-3	320	RSL	10	5.0	1.0	0.38

**Table B-15-2: Project Action Limits (PAL) for TA–St. Louis
Matrix: Soil (continued)**

Analytical Group: VOCs in Solids using 8260C							
Analyte	CAS Number	PAL (µg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/kg)	Laboratory-specific		
Chloromethane	74-87-3	11,000	RSL	20	10	5.0	0.65
cis-1,2-Dichloroethene	156-59-2	16,000	RSL	10	5.0	1.0	0.60
cis-1,3-Dichloropropene	10061-01-5	NE	NE	10	5.0	1.0	0.59
Dibromochloromethane	124-48-1	8,300	RSL	10	5.0	1.0	0.41
Ethylbenzene	100-41-4	5,800	RSL	10	5.0	1.0	0.30
MTBE	1634-04-4	47,000	RSL	10	5.0	1.0	0.48
Methylene Chloride	75-09-2	35,000	RSL	20	10	5.0	1.6
Styrene	100-42-5	600,000	RSL	10	5.0	1.0	0.35
Tetrachloroethene	127-18-4	8,100	RSL	10	5.0	1.0	0.32
Toluene	108-88-3	490,000	RSL	10	5.0	1.0	0.70
trans-1,2-Dichloroethene	156-60-5	160,000	RSL	10	5.0	1.0	0.94
trans-1,3-Dichloropropene	10061-02-6	NE	NE	10	5.0	1.0	0.35
Trichloroethene	79-01-6	410	RSL	10	5.0	1.0	0.39
Vinyl chloride	75-01-4	59	RSL	20	10	1.0	0.43
Xylenes, Total	1330-20-7	58,000	RSL	20	10	5.0	0.85

Analytical Group: VOCs Chlorinated Methanes Only in Solids using 8260C							
Analyte	CAS Number	PAL (µg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/kg)	Laboratory-specific		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Carbon tetrachloride	56-23-5	650	RSL	10	5.0	1.0	0.52
Chloroform	67-66-3	320	RSL	10	5.0	1.0	0.38
Chloromethane	74-87-3	11,000	RSL	20	10	5.0	0.65
Methylene Chloride	75-09-2	35,000	RSL	20	10	5.0	1.6

**Table B-15-2: Project Action Limits (PAL) for TA–St. Louis
Matrix: Soil (continued)**

Analytical Group: SVOCs in Solids using 8270D							
Analyte	CAS Number	PAL (µg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/kg)	Laboratory-specific		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
1,2,4-Trichlorobenzene	120-82-1	5,800	RSL	660	330	99	33.3
1,2-Dichlorobenzene	95-50-1	180,000	RSL	660	330	99	33.3
1,3-Dichlorobenzene	541-73-1	NE	NE	660	330	99	33.3
1,4-Dichlorobenzene	106-46-7	2,600	RSL	660	330	99	33.3
2,4,5-Trichlorophenol	95-95-4	630,000	RSL	660	330	99	33.3
2,4,6-Trichlorophenol	88-06-2	6,300	RSL	660	330	99	33.3
2,4-Dichlorophenol	120-83-2	19,000	RSL	660	330	99	33.3
2,4-Dimethylphenol	105-67-9	130,000	RSL	660	330	99	33.3
2,4-Dinitrophenol	51-28-5	13,000	RSL	660	1600	660	330
2,4-Dinitrotoluene	121-14-2	753	FWCUG	660	330	99	33.3
2,6-Dinitrotoluene	606-20-2	769	FWCUG	660	330	99	33.3
2-Chloronaphthalene	91-58-7	480,000	RSL	660	330	99	33.3
2-Chlorophenol	95-57-8	39,000	RSL	660	330	99	33.3
2-Methylnaphthalene	91-57-6	30,600	FWCUG	660	330	99	33.3
2-Methylphenol	95-48-7	320,000	RSL	660	330	99	33.3
2-Nitroaniline	88-74-4	63,000	RSL	3,200	1600	99	33.3
2-Nitrophenol	88-75-5	NE	NE	660	330	99	33.3
3 & 4 Methylphenol	15831-10-4	NE	NE	1,320	660	99	66.6
3,3'-Dichlorobenzidine ¹	91-94-1	1,200	RSL	1,200	1600	660	330
3-Nitroaniline	99-09-2	NE	NE	3,200	1600	99	33.3
4,6-Dinitro-2-methylphenol ¹	534-52-1	510	RSL	510	1600	660	330
4-Bromophenyl phenyl ether	101-55-3	NE	NE	660	330	99	33.3

**Table B-15-2: Project Action Limits (PAL) for TA–St. Louis
Matrix: Soil (continued)**

Analytical Group: SVOCs in Solids using 8270D							
Analyte	CAS Number	PAL (µg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/kg)	Laboratory-specific		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
4-Chloro-3-methylphenol	59-50-7	630,000	RSL	660	330	99	33.3
4-Chloroaniline	106-47-8	2,700	RSL	660	330	99	33.3
4-Chlorophenyl phenyl ether	7005-72-3	NE	NE	660	330	99	33.3
4-Nitroaniline	100-01-6	25,000	RSL	3,200	1600	660	330
4-Nitrophenol	100-02-7	61,200	FWCUG	3,200	1600	660	330
Acenaphthene	83-32-9	360,000	RSL	660	330	99	33.3
Acenaphthylene	208-96-8	NE	NE	660	330	99	33.3
Anthracene	120-12-7	1,800,000	RSL	660	330	99	33.3
Benzo[a]anthracene ¹	56-55-3	221	FWCUG	221	330	99	33.3
Benzo[a]pyrene ²	50-32-8	22	FWCUG	22	330	99	33.3
Benzo[b]fluoranthene ¹	205-99-2	221	FWCUG	221	330	99	33.3
Benzo[g,h,i]perylene	191-24-2	NE	NE	660	330	99	33.3
Benzo[k]fluoranthene	207-08-9	2,210	FWCUG	660	330	99	33.3
Benzoic acid	65-85-0	25,000,000	RSL	3,200	1600	660	330
Benzyl alcohol	100-51-6	630,000	RSL	660	330	99	51.8
bis (2-chloroisopropyl) ether	108-60-1	310,000	RSL	660	330	99	33.3
Bis(2-chloroethoxy)methane	111-91-1	23,000	FWCUG	660	330	99	33.3
Bis(2-chloroethyl)ether ¹	111-44-4	230	RSL	230	330	99	33.4
Butyl benzyl phthalate	85-68-7	290,000	RSL	660	330	99	33.3
Carbazole	86-74-8	44,600	FWCUG	660	330	99	33.3
Chrysene	218-01-9	22,100	FWCUG	660	330	99	33.3
Dibenz(a,h)anthracene ²	53-70-3	22	FWCUG	22	330	99	33.3
Dibenzofuran	132-64-9	15,300	FWCUG	660	330	99	33.3

**Table B-15-2: Project Action Limits (PAL) for TA–St. Louis
Matrix: Soil (continued)**

Analytical Group: SVOCs in Solids using 8270D							
Analyte	CAS Number	PAL (µg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/kg)	Laboratory-specific		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Diethyl phthalate	84-66-2	5,100,000	RSL	660	330	99	33.3
Dimethyl phthalate	131-11-3	NE	NE	660	330	99	33.3
Di-n-butyl phthalate	84-74-2	630,000	RSL	660	330	99	33.3
Di-n-octyl phthalate	117-84-0	63,000	RSL	660	330	99	33.3
Fluoranthene	206-44-0	163,000	FWCUG	660	330	99	33.3
Fluorene	86-73-7	243,000	FWCUG	660	330	99	33.3
Hexachlorobenzene ¹	118-74-1	210	RSL	210	330	99	33.3
Hexachlorobutadiene	87-68-3	1,200	RSL	660	330	99	33.3
Hexachlorocyclopentadiene ²	77-47-4	180	RSL	180	1600	660	330
Hexachloroethane	67-72-1	1,800	RSL	660	330	99	33.3
Indeno[1,2,3-cd]pyrene ¹	193-39-5	221	FWCUG	221	330	99	33.3
Isophorone	78-59-1	570,000	RSL	660	330	99	33.3
N,N-Diphenylamine	122-39-4	630,000	RSL	660	330	99	33
Naphthalene	91-20-3	122,000	FWCUG	660	330	99	33.3
Nitrobenzene	98-95-3	5,100	RSL	660	330	99	33.3
N-Nitrosodi-n-propylamine ¹	621-64-7	120	FWCUG	120	330	99	33.3
Pentachlorophenol	87-86-5	2,120	FWCUG	2,000	1600	330	330
Phenanthrene	85-01-8	NE	NE	660	330	99	33.3
Phenol	108-95-2	1,900,000	RSL	660	330	99	33.3
Pyrene	129-00-0	122,000	FWCUG	660	330	99	33.3

1 = The LOQ is greater than the project action level, but the DL is lower than the project action level. Project criteria are met since data will be flagged and reported down to the DL.

2 = Sensitivity requirements are not met for this analyte so data will be evaluated as described at the beginning of Worksheet #15.

**Table B-15-2: Project Action Limits (PAL) for TA–St. Louis
Matrix: Soil (continued)**

Analytical Group: TPH in Solids using 8015B							
Analyte	CAS Number	PAL (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
TPH-C10-C20	NA	2,000	BUSTR	50	25	1.0	0.34
TPH-C20-C34	NA	5,000	BUSTR	50	25	1.0	0.64

BUSTR –Bureau of Underground Storage Tanks (BUSTR), 2017. Class 1 Soil Action Levels, Table 3-8; mg/kg = milligram per kilogram.

Analytical Group: Explosives in Solids using 8330B							
Analyte	CAS Number	PAL (µg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/kg)	Laboratory-specific		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
1,3,5-Trinitrobenzene	99-35-4	225,000	FWCUG	500	250	80	28
1,3-Dinitrobenzene	99-65-0	765	FWCUG	500	250	80	44
2,4,6-Trinitrotoluene	118-96-7	3,650	FWCUG	500	250	80	36
2,4-Dinitrotoluene	121-14-2	753	FWCUG	500	250	80	38
2,6-Dinitrotoluene	606-20-2	769	FWCUG	500	250	80	64
2-Amino-4,6-dinitrotoluene	35572-78-2	1,540	FWCUG	500	250	80	43
2-Nitrotoluene	88-72-2	3,880	FWCUG	500	250	80	65
3-Nitrotoluene	99-08-1	630	RSL	500	250	80	56
4-Amino-2,6-dinitrotoluene	19406-51-0	1,540	FWCUG	600	300	120	94
4-Nitrotoluene	99-99-0	52,500	FWCUG	500	250	120	81
HMX	2691-41-0	359,000	FWCUG	500	250	80	39
Nitrobenzene	98-95-3	5,100	RSL	500	250	80	43
Nitroglycerin	55-63-0	52,500	FWCUG	2600	1300	380	270
PETN	78-11-5	13,000	RSL	5000	2500	380	350
RDX	121-82-4	8,030	FWCUG	500	250	80	62
Tetryl	479-45-8	16,000	RSL	500	250	80	46

**Table B-15-2: Project Action Limits (PAL) for TA–St. Louis
Matrix: Soil (continued)**

Analytical Group: PCBs in Solids using 8082A							
Analyte	CAS Number	PAL (µg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/kg)	Laboratory-specific		
					LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
PCB-1016	12674-11-2	203	FWCUG	66	33	16	9.6
PCB-1221	11104-28-2	200	RSL	66	33	16	9.6
PCB-1232	11141-16-5	170	RSL	66	33	16	9.6
PCB-1242	53469-21-9	230	RSL	66	33	16	9.6
PCB-1248	12672-29-6	203	FWCUG	66	33	16	9.6
PCB-1254	11097-69-1	120	FWCUG	66	33	10	8.0
PCB-1260	11096-82-5	203	FWCUG	66	33	10	8.0

Analytical Group: TAL Metals in Solids using 6020A/7471B							
Analyte	CAS Number	PAL (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aluminum	7429-90-5	7,380	FWCUG	10.0	5.0	4.0	2.0
Antimony	7440-36-0	2.82	FWCUG	1.0	0.50	0.40	0.20
Arsenic ¹	7440-38-2	0.425	FWCUG	0.425	1.0	0.80	0.40
Barium	7440-39-3	1,413	FWCUG	4.0	2.0	1.0	0.50
Beryllium	7440-41-7	16	RSL	0.20	0.10	0.080	0.040
Cadmium	7440-43-9	6.41	FWCUG	0.10	0.050	0.048	0.024
Calcium	7440-70-2	NE	NE	100	50	20	10
Chromium	7440-47-3	8,147	FWCUG	2.0	1.0	0.90	0.45
Cobalt	7440-48-4	131	FWCUG	0.40	0.20	0.15	0.075
Copper	7440-50-8	311	FWCUG	2.0	1.0	0.80	0.40
Iron	7439-89-6	2,313	FWCUG	10.0	5.0	4.0	2.0

**Table B-15-2: Project Action Limits (PAL) for TA–St. Louis
Matrix: Soil (continued)**

Analytical Group: TAL Metals in Solids using 6020A/7471B							
Analyte	CAS Number	PAL (µg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/kg)	Laboratory-specific		
Lead	7439-92-1	400	RSL	0.60	0.30	0.25	0.13
Magnesium	7439-95-4	NE	NE	100.0	50.0	5.0	2.5
Manganese	7439-96-5	293	FWCUG	1.0	0.50	0.40	0.20
Nickel	7440-02-0	155	FWCUG	1.0	0.50	0.40	0.20
Potassium	7440-09-7	NE	NE	20.0	10	8.0	4.0
Selenium	7782-49-2	39	RSL	1.0	0.50	0.40	0.32
Silver	7440-22-4	38.6	FWCUG	0.40	0.20	0.15	0.075
Sodium	7440-23-5	NE	NE	40	20	15	7.5
Thallium	7440-28-0	0.612	FWCUG	0.50	0.50	0.40	0.20
Vanadium	7440-62-2	44.9	FWCUG	2.0	1.0	0.80	0.40
Zinc	7440-66-6	2,321	FWCUG	10.0	5.0	4.00	2.0
Mercury	7439-97-6	2.27	FWCUG	0.066	0.033	0.030	0.011

1 = The LOQ is greater than the project action level, but the DL is lower than the project action level. Project criteria are met since data will be flagged and reported down to the DL.

**Table B-15-3: Project Action Limits and TA–St Louis
Matrix: IDW Solid/Water Toxicity Characteristic Leaching Procedure (TCLP)**

Analytical Group: TCLP VOCs using 1311/8260C							
Analyte	CAS Number	PAL (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Laboratory-specific		
					LOQs (mg/L)	LOD (mg/L)	DLs (mg/L)
Benzene	71-43-2	0.5	TCLP	0.10	0.050	0.010	0.0025
2-Butanone (MEK)	78-93-3	200	TCLP	0.10	0.050	0.050	0.0038
Carbon tetrachloride	56-23-5	0.5	TCLP	0.10	0.050	0.010	0.0036
Chlorobenzene	108-90-7	100	TCLP	0.10	0.050	0.010	0.0038
Chloroform	67-66-3	6	TCLP	0.10	0.050	0.010	0.00092
1,2-Dichloroethane	107-06-2	0.5	TCLP	0.10	0.050	0.010	0.0037
1,1-Dichloroethene	75-35-4	0.7	TCLP	0.10	0.050	0.010	0.0036
Trichloroethene	79-01-6	0.5	TCLP	0.10	0.050	0.010	0.0029
Vinyl chloride	75-01-4	0.2	TCLP	0.10	0.10	0.010	0.0043
Tetrachloroethene	127-18-4	0.7	TCLP	0.10	0.050	0.010	0.0028

Analytical Group: TCLP SVOC using 1311/8270D							
Analyte	CAS Number	PAL (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Laboratory-specific		
					LOQs (mg/L)	LOD (mg/L)	DLs (mg/L)
1,4-Dichlorobenzene	106-46-7	7.5	TCLP	0.10	0.050	0.015	0.0050
2,4,5-Trichlorophenol	95-95-4	400	TCLP	0.10	0.050	0.015	0.010
2,4,6-Trichlorophenol	88-06-2	2	TCLP	0.10	0.050	0.015	0.010
2,4-Dinitrotoluene	121-14-2	0.13	TCLP	0.10	0.050	0.015	0.0050
2-Methylphenol	95-48-7	200	TCLP	0.10	0.050	0.015	0.010
3 & 4 Methylphenol	15831-10-4	200	TCLP	0.10	0.10	0.015	0.0050
Hexachlorobutadiene	87-68-3	0.13	TCLP	0.10	0.050	0.015	0.0050

Table B-15-3: Project Action Limits and TA–St Louis
Matrix: IDW Solid/Water Toxicity Characteristic Leaching Procedure (TCLP) (continued)

Analytical Group: TCLP SVOC using 1311/8270D							
Analyte	CAS Number	PAL (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Laboratory-specific		
Hexachlorobenzene	118-74-1	0.5	TCLP	0.10	0.050	0.015	0.0050
Hexachloroethane	67-72-1	3	TCLP	0.10	0.050	0.015	0.0050
Nitrobenzene	98-95-3	2	TCLP	0.10	0.050	0.015	0.0050
Pentachlorophenol	87-86-5	100	TCLP	0.50	0.250	0.050	0.010
Pyridine	110-86-1	5	TCLP	0.10	0.10	0.050	0.025

Analytical Group: TCLP Pesticides using 1311/8081B and TCLP Herbicides using 1311/8151A							
Analyte	CAS Number	PAL (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Laboratory-specific		
					LOQs (mg/L)	LOD (mg/L)	DLs (mg/L)
gamma-BHC (Lindane)	58-89-9	0.4	TCLP	0.001	0.00050	0.000020	0.000015
Endrin	72-20-8	0.02	TCLP	0.001	0.00050	0.000050	0.000050
Heptachlor	76-44-8	0.008	TCLP	0.001	0.00050	0.000050	0.000015
Heptachlor epoxide	1024-57-3	0.008	TCLP	0.001	0.00050	0.000050	0.000050
Methoxychlor	72-43-5	10	TCLP	0.002	0.0010	0.000050	0.000050
Toxaphene	8001-35-2	0.5	TCLP	0.040	0.020	0.020	0.000050
Chlordane	12789-03-6	0.03	TCLP	0.01	0.0050	0.0050	0.00020
2,4-D	94-75-7	10	TCLP	0.080	0.040	0.025	0.020
Silvex (2,4,5-TP)	93-72-1	1	TCLP	0.020	0.010	0.0050	0.0030

Table B-15-3: Project Action Limits and TA–St Louis
Matrix: IDW Solid/Water Toxicity Characteristic Leaching Procedure (TCLP) (continued)

Analytical Group: TCLP Metals using 1311/6010C/7470A							
Analyte	CAS Number	PAL (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Laboratory-specific		
					LOQs (mg/L)	LOD (mg/L)	DLs (mg/L)
Arsenic	7440-38-2	5	TCLP	0.05	0.025	0.022	0.010
Barium	7440-39-3	100	TCLP	0.26	0.13	0.12	0.0375
Cadmium	7440-43-9	1	TCLP	0.026	0.013	0.012	0.0038
Chromium	7440-47-3	5	TCLP	0.05	0.025	0.022	0.0075
Lead	7439-92-1	5	TCLP	0.05	0.025	0.022	0.0075
Selenium	7782-49-2	1	TCLP	0.074	0.038	0.035	0.020
Silver	7440-22-4	5	TCLP	0.05	0.025	0.022	0.0075
Mercury	7439-97-6	0.2	TCLP	0.003	0.0015	0.00065	0.000079

Analytical Group: Reactivity, Corrosivity, and Ignitibility (RCI)							
Analyte¹	CAS Number	PAL (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific		
					LOQs (mg/kg)	LOD (mg/kg)	DLs (mg/kg)
Cyanide, Total using 9012B	57-12-5	NE	NE	1.0	0.50	0.11	0.11
Sulfide, Total using 9034	18496-25-8	NE	NE	120	60	48	24
pH using 9045D	NA	NA	NA	<2 or >12	NA	NA	NA
Flashpoint using 1010A	NA	NA	NA	<60° C	NA	NA	NA

TCLP = Code of Federal Regulations, 40 CFR §261.24.

1 = A solid waste exhibits the characteristic of reactivity if it is a cyanide or sulfide bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment

WORKSHEET #17: SAMPLING DESIGN AND RATIONALE

(UFP-QAPP Manual Section 3.1.1)

This worksheet describes the sampling design and rationale for proposed sampling at CC RVAAP-69, CC RVAAP-70, and CC RVAAP-74. This includes the tasks that will be required to successfully complete field operations and achieve the DQOs described on Worksheet #11. A summary of the samples to be collected, including Sample IDs, is provided in Worksheet #18.

17.1 CC RVAAP-69 Building 1048 Fire Station

The primary objective of this work plan is to address data gaps to complete the RI Report for this site. As discussed in Sections 3.1.5 and 4.1 of the Work Plan, the following data gaps have been identified for AOC RVAAP-69 Building 1048 Fire Station:

- o The vertical extent of carbon tetrachloride in soil at SB101 is not defined.
- o The lateral extent of carbon tetrachloride contamination in soil is not well defined below a depth of 1018 feet amsl.
- o Groundwater was not evaluated as part of the previous investigations.

To fill these data gaps, the following activities will be carried out in the sequence described below:

- 1) Mobilization, including all necessary notifications and utility clearances.
- 2) Collect groundwater grab samples.
- 3) Advance and collect soil samples from a deep boring to bedrock (estimated at 45 feet bgs) near previous borings SB101 and RV5-SB2 (the maximum carbon tetrachloride concentration in soil was detected in RV5-SB2).
- 4) Advance and collect soil samples from additional soil borings located north (near SB105), east (near SB102) and south (near SB103) of the maximum carbon tetrachloride detections (i.e., near RV5-SB2) and determine the lateral extent of carbon tetrachloride contamination deeper than 1018 amsl in soil.
- 5) Install a minimum of three groundwater monitoring wells based on the results of the groundwater grab samples.
- 6) Measure groundwater elevation and collect first quarter groundwater monitoring well samples.
- 7) After initial fieldwork, survey all sample locations and demobilize.
- 8) Return to measure groundwater elevation and sample wells quarterly for three more quarters.

Field procedures are provided in Appendix A. Groundwater grab samples, soil sampling, and groundwater monitoring well installation and sampling activities are discussed in more detail below.

17.1.1 Groundwater Grab Samples

In order to efficiently optimize the number of monitoring wells needed to define the direction of groundwater flow and monitor a plume, Parsons will first collect grab samples using a Screenpoint 16 sampler (or install temporary wells if groundwater grab samples cannot be collected from well

points, and all temporary wells will be developed prior to sampling). Groundwater grab samples will be collected from the upper portions of the saturated zone within the unconsolidated aquifer. These screening samples will be used to select monitoring well locations. Initial groundwater grab samples will be collected from near the maximum carbon tetrachloride concentrations in soil (near RV5-SB2) and in locations approximately 50 to 70 feet downgradient (east to southeast) of that location (i.e., between the former Building 1048 Fire Station and George Road/South Service Road), as shown on Work Plan, Figure 4-1.

Groundwater grab samples will be analyzed for VOCs Chlorinated Methanes, as detailed in Worksheet 15 **Table B-15-1**, on a quick turn (24 hour) basis (**Table B-18-1**), and additional step-in or step-out samples will be collected until the plume is delineated or until groundwater grab samples establish that groundwater is not contaminated with carbon tetrachloride at concentrations above the Adult Resident FWCUG (0.204 µg/L) (see Worksheet 15, **Table B-15-1**).

17.1.2 Soil Sampling

To evaluate the vertical extent of carbon tetrachloride in soil, Parsons will advance a new boring (69-1048-SB110) in the immediate vicinity of the earlier borings SB101 and RV5-SB2 (the maximum carbon tetrachloride concentration in soil was detected at RV5-SB2), as shown on Work Plan, Figure 4-1. The boring will extend from the ground surface to bedrock (estimated at 45 feet bgs). The length of the core will be screened with a PID monitor. If no evidence of a chemical release is noted (no odors, staining, or elevated PID readings), then discrete soil samples will be collected at 5 foot intervals beginning at 10 feet bgs (i.e., samples at 10, 15, 20, 25, 30, 35, 40 and 45 feet bgs). However, if evidence of potential chemical release is observed, then discrete soil samples will be collected from the interval exhibiting the highest odors, staining, or elevated PID readings. Sample IDs are provided in Worksheet 18, **Table B-18-1**.

Earlier investigations analyzed all soil samples for VOCs, SVOCs and metals, as well as 10 percent of soil samples for organochlorine pesticides, PCBs, and explosives/propellants. In subsurface soil, only carbon tetrachloride and chloroform were detected above June 2017 USEPA Residential RSLs in more than one sample. Further analysis for compounds other than carbon tetrachloride and its decay products is not necessary to address data gaps. Therefore, soil samples will be analyzed for VOCs Chlorinated Methanes, as detailed in Worksheet 15 **Table B-15-2**.

To evaluate the lateral extent of carbon tetrachloride contamination deeper than 1018 amsl in soil, Parsons will advance additional soil borings (69-1048-SB111, 69-1048-SB112, and 69-1048-SB113) located north (near SB105), east (near SB102) and south (near SB103) of the maximum detections of carbon tetrachloride in soil (near RV5-SB2), as shown on Work Plan Figure 4-1. These soil borings will extend to the water table (approximately 10 to 20 feet bgs at CC RVAAP-69). The length of the core will be screened with a PID monitor. If no evidence of a chemical release is noted (no odors, staining, or elevated PID readings), then discrete soil samples will be collected at 5-foot intervals beginning at 10 feet bgs (i.e., samples at 10, 15 and 20 feet bgs). However, if evidence of potential chemical release is observed, then discrete soil samples will be collected from the interval exhibiting the highest odors, staining, or elevated PID readings. Contamination is defined to an elevation of about 1012 feet amsl in distal sample locations (SB106, SB109, and SB104), so additional step out samples will not be necessary regardless of whether carbon tetrachloride is detected in any of the proposed soil samples. If carbon tetrachloride extends deeper into the subsurface, then delineation will focus on delineating any resulting groundwater plume.

Soil samples will be analyzed for carbon tetrachloride and its decay products (VOCs Chlorinated Methanes), as detailed on **Table B-18-1**. The ground surface at CC RVAAP-69 varies between 1030 feet and 1020 feet amsl (Figure 1-2). These proposed samples will define the extent of carbon tetrachloride (and decay product) contamination in the vadose zone and upper portions of the saturated zone to an elevation as deep as 1005 feet amsl. All new borings will be continuously logged.

17.1.3 Groundwater Monitoring Well Installation and Sampling

The groundwater grab sample results will be evaluated to determine optimum well placement. If a carbon tetrachloride groundwater plume is detected, then the orientation of the plume will provide an indication of the local groundwater flow direction. A minimum of three monitoring wells will be installed; one near the maximum concentrations of carbon tetrachloride in soil (i.e., possible source area near RV5-SB2), one upgradient of that location, and one at the downgradient extent of the plume. If a carbon tetrachloride groundwater plume is not indicated by the grab samples, then at minimum one monitoring well will be installed near RV5-SB2. At least two additional monitoring wells will be installed to ensure that the local potentiometric surface can be mapped. More than the minimum of three monitoring wells may be needed to confirm the extent of potential carbon tetrachloride contamination beneath CC RVAAP-69. Monitoring wells will be installed with screens set across the water table to maximize the likelihood of intercepting the contaminant plume because the maximum concentrations in soil to date are in the interval of 1015 to 1020 feet amsl. However, if it is determined that carbon tetrachloride extends deeper in the unconsolidated soil, it may be necessary to install additional deeper wells. The local potentiometric surface will be mapped using groundwater elevations in wells installed at CC RVAAP-69 and adjacent AOC CC RVAAP-74.

Monitoring wells will be developed, and then the groundwater elevations measured and the wells sampled quarterly for the first year. Initial groundwater samples will be analyzed for VOCs Chlorinated Methanes, as detailed on **Table B-18-1**.

17.2 CC RVAAP-70 East Classification Yard

The primary objective of this work plan is to address data gaps to complete the SI Report for this site. As discussed in Sections 3.2.5 and 4.2 of the Work Plan, the following data gaps have been identified for AOC CC RVAAP-70 East Classification Yard:

- o Soil underneath the floor inside Building 47-40 has not been sampled at locations where the floor is stained and cracked.
- o Soil underneath the floor of the Interior Repair Pit (DU04) has not been sampled for VOCs.
- o Discrete soil samples have not been sampled and analyzed for VOCs from locations at the Outdoor Wash Rack Area at the vertical interval where odor and elevated PID readings were noted in boring logs.
- o Sediment from the storm drain at the southwest corner of Building 47-40 has not been sampled for contamination from the Outdoor Wash Rack.

To fill these data gaps, the following activities will be carried out in the sequence described below:

- 1) Mobilization, including all necessary notifications and utility clearances.
- 2) Clear brush, as necessary to allow access to the building and sample locations.

- 3) Remove oily sludge from the floor and pit inside Building 47-40.
- 4) Inspect the concrete floor of the Interior Repair Pit for any visible cracks and staining after the sludge is removed
- 5) Advance and collect soil samples from two soil borings near cracks and biased towards locations which exhibit staining or had sludge on the floor.
- 6) Advance and collect soil samples from two soil borings near cracks and biased towards locations which exhibit staining or had sludge in the pit.
- 7) Advance and collect soil samples from two soil borings near former borings that had elevated PID readings near the former wash rack (70-4759-DU6-SB1 and 70-4759-DU6-SB5).
- 8) Collect a sediment sample from the storm drain and drill one boring near the storm drain for subsurface soil sampling.
- 9) After fieldwork, survey all sample locations and demobilize.

Field procedures are provided in Appendix A. Brush clearing, oily sludge removal, soil sampling inside the building and soil sampling near the former wash rack are discussed in more detail below.

17.2.1 Clear Brush and Remove Oily Sludge

Brush will be cleared, as necessary, to access Building 47-40 and the outside sample locations. Only brush required to allow access for the investigation will be cleared.

Prior to soil sampling, oily sludge (estimated at approximately 10 cubic yards) will be removed from the floor of Building 47-40 and Interior Repair Pit to prevent cross contamination when borings are advanced through the floor and to reveal areas of the floor that may be stained and/or cracked, which would be the most likely routes that chemicals could enter the subsurface. The sludge will be removed using a mini excavator, vacuum truck, or other method by the Waste Handling Subcontractor. As it is removed, the sludge will be containerized in a roll-off bin and sampled for waste characterization and disposal purposes. Sludge samples will be analyzed for Toxicity Characteristic Leaching Procedure (TCLP) (Method 1311) metals, TCLP VOCs, TCLP SVOCs, TCLP herbicides, TCLP pesticides, total sulfide, total cyanide, corrosivity (pH), and flashpoint (**Table B-18-2**).

17.2.2 Soil Sampling Inside Building 47-40

After removing the sludge from the floor of Building 47-40, Parsons will inspect the concrete floor for cracks and stains. Two soil borings (70-4740-SB101 and 70-4740-SB102) will be installed near cracks and biased towards locations which exhibit staining or had sludge on the floor to confirm/deny whether soil beneath the concrete floor has been contaminated above FWCUGs (Worksheet 15.2). Approximate locations are shown on Work Plan Figure 4-2, but actual locations will be determined after visually inspecting the floor. One of these borings is proposed near the former oil storage rack on the East wall of Building 47-40 through the wooden floor in that area. Discrete soil samples will be collected from each boring at 2-foot intervals down to 7 feet bgs or until sandstone is reached (from 0-2, 2-4, 4-6, and 6-7 feet bgs), and samples will be analyzed for VOCs, SVOCs, PCBs, and TAL metals (**Table B-18-2**).

After the sludge is removed as described above, Parsons will inspect the concrete floor of the Interior Repair Pit for any visible cracks and staining. Two soil borings (70-4740-SB103 and 70-

4740-SB104) will be drilled inside the Interior Repair Pit to confirm/deny whether soil beneath the concrete floor has been contaminated above FWCUGs (Worksheet 15.2), with preference given to any locations with cracks, staining, or sludge noted on the concrete floor. Approximate locations are shown on Work Plan Figure 4-2, but actual locations will be determined after visually inspecting the floor. Continuous cores will be collected and visually logged, and the length of the cores will be screened with a PID monitor. If no evidence of contamination is noted (no odors, staining, or elevated PID readings), then discrete soil samples will be collected from 0-2 and 2-4 feet bgs and analyzed for VOCs (**Table B-18-2**). However, if evidence of soil contamination is observed, then discrete soil samples will be collected from any intervals with odors, staining, or elevated PID readings. Previous boring logs indicate that sandstone was reached from approximately 1 to 4 feet bgs in the pit; therefore, the proposed soil borings will be drilled until sandstone is reached. All new borings will be continuously logged.

17.2.3 Soil Sampling at Outdoor Wash Rack and Storm Drain

Two soil borings (70-4740-SB105 and 70-4740-SB106) will be drilled near former locations 70-4759-DU6-SB1 and 70-4759-DU6-SB5 where elevated PID readings were noted during the 2012 field investigation, as shown on Work Plan Figure 4-2. Continuous soil cores will be collected from 1 to 7 feet bgs, and the entire soil core will be logged and screened with a PID monitor. Discrete soil samples will be collected for laboratory analysis from the depth intervals with field indications of contamination (staining, odors, or elevated PID readings). However, if no evidence of contamination is noted, then samples will be collected from the 6 to 7 feet bgs interval for laboratory analysis to coincide with the elevated PID readings described previously. The soil samples will be analyzed for VOCs (**Table B-18-2**).

Parsons will inspect the storm drain at the southwest corner of Building 47-40 where wash water may have drained from the wash rack. A sediment sample (70-4770-SS107) will be collected from the storm drain and analyzed for PCBs, SVOCs, and explosives. One soil boring (70-4770-SB107) will be drilled as close to the drain as possible and soil samples will be collected to determine if subsurface soil near this storm drain was impacted by historical activities at the wash rack. Discrete subsurface soil samples will be collected from each boring at 2-foot intervals down to 7 feet bgs (from 1-3, 3-5, and 5-7 feet bgs) and analyzed for VOCs, PCBs, SVOCs, and explosives. Proposed sample locations are shown on Work Plan Figure 4-2 and proposed analyses are shown on **Table B-18-2**. All new borings will be continuously logged.

17.3 CC RVAAP-74 Building 1034-Motor Pool Hydraulic Lift

The primary objective of this work plan is to address data gaps to complete the RI Report for this site. As discussed in Sections 3.3.5 and 4.3 of the Work Plan, the following data gaps have been identified for AOC CC RVAAP-74 Building 1034-Motor Pool Hydraulic Lift:

- o It is unclear if the “free product” historically noted on the 74-1034-HL-SB18 boring log at 7.5 to 8.5 feet bgs is mobile LNAPL or residual LNAPL.
- o The horizontal extent of soil contamination is not defined east and south of 74-1034-HL-SB18.
- o Groundwater was not evaluated as part of the previous investigations and it is unknown if groundwater is impacted or if enough hydraulic fluid was released to accumulate as LNAPL at the water table.
- o Only one soil sample was analyzed for VOCs.

To fill these data gaps, the following activities will be carried out in the sequence described below:

- 1) Mobilization, including all necessary notifications and utility clearances.
- 2) Collect groundwater grab samples downgradient of the site.
- 3) Advance a new boring in the immediate vicinity of the earlier boring 74-1034-HL-SB18; the soil will be photographed, visually inspected, and scanned with a PID to evaluate if there is LNAPL. A soil sample will be collected from the interval where “free product” was noted (7.5 to 8.5 feet bgs). A temporary well will be installed in the borehole to a depth of approximately 10 feet bgs and screened across the vertical interval where “free product” was noted in the gravel layer on the SB18 boring log. The temporary well will be developed. Measurements (depth to organic liquid and/or water) will be made to determine if organic liquid flows into the well, indicating “free product.”
- 4) Advance soil borings in the immediate vicinity of the earlier borings 1034-HL-SB-14 and 74-1034-HL-SB-19, and collect soil samples from 7.5 to 8.5 feet to evaluate the horizontal extent in east and south of 74-1034-HL-SB18.
- 5) Install a minimum of three groundwater monitoring wells based on the results of the groundwater grab samples.
- 6) Measure groundwater elevation and collect first quarter groundwater monitoring well samples.
- 7) After initial fieldwork, survey all sample locations and demobilize.
- 8) Return to measure groundwater elevation and sample wells quarterly for three more quarters.

Field procedures are provided in Appendix A. Groundwater grab samples, soil sampling, and groundwater monitoring well installation and sampling activities are discussed in more detail below.

17.3.1 Groundwater Grab Samples

In order to efficiently optimize the number of monitoring wells needed to define the direction of groundwater flow and monitor a plume (if any), Parsons will first collect grab samples using a Screenpoint 16 sampler (or install temporary wells if groundwater grab samples cannot be collected from well points, and all temporary wells will be developed prior to sampling). Groundwater grab samples will be collected from the upper portions of the saturated zone within the unconsolidated aquifer. These screening samples will be used to select monitoring well locations. Initial groundwater grab samples will be collected approximately 3 feet southeast and 20 feet downgradient (east and southeast) of soil boring 74-1034-HL-SB18 where the maximum TPH-C₂₀ to C₃₄ heavy distillates range concentrations were detected in 2013 (i.e., outside the southeast corner of Building 1034), as shown on Work Plan Figure 4-4.

Groundwater grab samples will be analyzed for VOCs and PAHs on a quick turn (24 hour) basis (**Table B-18-3**). Additional step-out samples will be collected until the plume is delineated or until groundwater grab samples establish that VOCs and SVOCs are not present in groundwater above BUSTR Action Levels. Constituents that do not have BUSTR Action Levels will be compared to Resident Receptor FWCUGs using the carcinogenic risk level of 10⁻⁶ or the HQ of 0.1 or current USEPA Residential RSLs (June 2017). (Worksheet 15.1).

17.3.2 Soil Sampling

To evaluate whether the “free product” identified in 74-1034-HL-SB18 at 7.5 to 8.5 feet bgs is mobile organic liquid or immobile residual saturation, Parsons will install a new boring (74-1034-SB23) in the immediate vicinity of the earlier boring 74-1034-HL-SB18, as shown on Work Plan Figure 4-3. The soil will be photographed, visually inspected, and scanned with a PID to evaluate if there is LNAPL (i.e., oil that flows out of the formation versus residual saturation or oil that is essentially immobile and held in capillary spaces). A soil sample will be collected from 7.5 to 8.5 feet bgs to confirm what was found by evaluating the chromatograph to a known sample of hydraulic fluid (i.e., mineral oil). The soil sample will be analyzed for VOCs, TPH-C₁₀ to C₂₀ and C₂₀ to C₃₄ distillate ranges, and PCBs (**Table B-18-3**). A temporary well will be installed in the borehole to a depth of approximately 10 feet bgs and screened across the vertical interval where “free product” was noted in the gravel layer on the SB18 boring log. The temporary well will be developed. Measurements (depth to organic liquid and/or water) will be made to determine if organic liquid flows into the well, indicating “free product.”

To evaluate the horizontal extent of soil contamination east and south of 74-1034-HL-SB18, Parsons will advance soil borings in the immediate vicinity of the earlier borings 1034-HL-SB-14 and 74-1034-HL-SB-19 (Work Plan Figure 4-3), and collect soil samples from 7.5 to 8.5 feet to evaluate the horizontal extent in east and south of 74-1034-HL-SB18. Soil samples will be analyzed for VOCs, TPH-C₁₀ to C₂₀ and C₂₀ to C₃₄ distillate ranges (**Table B-18-3**). All new borings will be continuously logged.

17.3.3 Groundwater Monitoring Well Installation and Sampling

A minimum of three monitoring wells will be installed; one near the maximum detection of TPH-C₂₀ to C₃₄ heavy distillates range in soil (near SB18), one upgradient well near the cylinder that held the hydraulic fluid (near SB02), and one at the downgradient extent of the plume (if a plume exists). Monitoring wells will be installed with screens set across the water table to maximize the likelihood of intercepting LNAPL or a contaminant plume.

Parsons will install and develop groundwater monitoring well 74MW-001 approximately three feet southeast of the soil sampling location exhibiting the maximum amount of contamination (74-1034-HL-SB18) to determine if LNAPL exists at the water table and establish depth to groundwater. The monitoring well will be located a few feet from 74-1034-HL-SB18 to avoid pushing contamination through a potential confining layer into groundwater.

Parsons will install and develop groundwater monitoring well 74MW-002 located near soil boring 74-1034-HL-SB02. This location is in the presumed upgradient direction and only very low TPH-C₁₀ to C₂₀ and C₂₀ to C₃₄ distillate range concentrations were detected in soil in 2013. This location is close to the cylinder that contained hydraulic fluid, and is likely to be the most upgradient location where hydraulic fluid could be present.

The groundwater grab sample results will be evaluated to determine optimum well placement for the third location. If a groundwater plume is detected, then the orientation of the plume will provide an indication of the local groundwater flow direction and groundwater monitoring well 74MW-003 will be installed near the downgradient edge of the plume. The local potentiometric surface will be mapped using groundwater elevations in wells installed at CC RVAAP-69 and adjacent AOC CC RVAAP-74.

Monitoring wells will be developed, and then the groundwater elevations measured and sampled quarterly for the first year. During each event, the fluid levels in the wells will be measured to detect LNAPL (if any), and groundwater samples will be analyzed for VOCs, PAHs, and PCBs (**Table B-18-1**). The local potentiometric surface will be mapped using groundwater elevations in wells installed at CC RVAAP-74 and adjacent AOC CC RVAAP-69.

WORKSHEET # 18: SAMPLING LOCATIONS AND METHODS/SOP REQUIREMENTS

(UFP-QAPP Manual Section 3.1.1)

**Table B-18-1: Sampling Locations and Methods
at CC RVAAP-69 Building 1048 Fire Station**

Location ID	Sample ID	Depth ¹ (feet bgs)	Matrix	Sample Type		Analysis ²	Sampling SOP Reference
69-1048-SB110	069SB-110-0088-SO	10-15	Soil	Discrete	N	VOCs Chlorinated Methanes Only (See Worksheet 15)	Appendix A, Section 4.4
	069SB-110-9088-SO	10-15			FD		
	069SB-110-0089-SO	15-20			N	VOCs Chlorinated Methanes Only (See Worksheet 15)	
	069SB-110-0090-SO	20-25					
	069SB-110-0091-SO	25-30					
	069SB-110-0092-SO	30-35					
	069SB-110-0093-SO	35-40					
	069SB-110-0094-SO	40-45					
	069SB-110-0095-SO	45-50					
69-1048-SB111	069SB-111-0096-SO	10-15	Soil	Discrete	N	VOCs Chlorinated Methanes Only (See Worksheet 15)	Appendix A, Section 4.4
	069SB-111-0097-SO	15-20					
	069SB-111-0098-SO	20-25					
69-1048-SB112	069SB-112-0099-SO	10-15	Soil	Discrete	N	VOCs Chlorinated Methanes Only (See Worksheet 15)	Appendix A, Section 4.4
	069SB-112-9099-SO	10-15			FD		
	069SB-112-0099-SO-MS	10-15			MS		
	069SB-112-0099-SO-MSD	10-15			MSD		
	069SB-112-0100-SO	15-20			N		
	069SB-112-0101-SO	20-25			N		

Location ID	Sample ID	Depth ¹ (feet bgs)	Matrix	Sample Type		Analysis ²	Sampling SOP Reference
69-1048-SB113	069SB-113-0102-SO	10-15	Soil	Discrete	N	VOCs Chlorinated Methanes Only (See Worksheet 15)	Appendix A, Section 4.4
	069SB-113-0103-SO	15-20					
	069SB-113-0104-SO	20-25					
069WP-001	069WP-001-0001-GW	20-25	Groundwater	Discrete Grab (Screening Samples) ³	N	VOCs Chlorinated Methanes Only (See Worksheet 15)	Appendix A, Section 4.3.6
069WP-002	069WP-002-0001-GW	20-25					
069WP-003	069WP-003-0001-GW	20-25					
069WP-004	069WP-004-0001-GW	20-25					
069MW-001	069MW-001-0001-GW	20-30	Groundwater	Discrete	N	VOCs Chlorinated Methanes Only (See Worksheet 15)	Appendix A, Section 4.3.6
	069MW-001-9001-GW	20-30			FD		
	069MW-001-0002-GW	20-30			N	VOCs Chlorinated Methanes Only (See Worksheet 15)	
	069MW-001-0003-GW	20-30			N		
	069MW-001-0004-GW	20-30			N		
069MW-002	069MW-002-0001-GW	20-30	Groundwater	Discrete	N	VOCs Chlorinated Methanes Only (See Worksheet 15)	Appendix A, Section 4.3.6
	069MW-002-0002-GW	20-30					
	069MW-002-0003-GW	20-30					
	069MW-002-0004-GW	20-30					
069MW-003	069MW-003-0001-GW	20-30	Groundwater	Discrete	N	VOCs Chlorinated Methanes Only (See Worksheet 15)	Appendix A, Section 4.3.6
	069MW-003-0002-GW	20-30			N		
	069MW-003-0003-GW	20-30			N		
	069MW-003-0004-GW	20-30			N		
	069MW-003-9004-GW	20-30			FD		
	069MW-003-0004-GW-MS	20-30			MS		

Location ID	Sample ID	Depth ¹ (feet bgs)	Matrix	Sample Type		Analysis ²	Sampling SOP Reference
	069MW-003-0004-GW-MSD	20-30			MSD		

Notes:

Examples of Sample IDs to be used for blanks are as follows: Field Blanks (069FB-DDMMYY-01), Equipment Blanks (069EB-DDMMYY-01), and Trip Blanks (069TB-DDMMYY-01).

1 = Groundwater grab samples will be collected near the water table (anticipated to be 20-25 feet bgs) and groundwater monitoring wells samples will be collected from the within the screened interval (anticipated to be 20-30 feet bgs); however, actual depth will be determined in the field.

2 = Because this sampling effort is designed to address specific data gaps, no samples will be analyzed for the Ravenna full-suite of analysis.

3 = Four groundwater grab samples (screening samples) will be initially collected. Based on screening results, additional groundwater grab samples may be collected.

bgs = below ground surface; FD = field duplicate; N = normal; MS = matrix spike; MSD = matrix spike duplicate; SOP = standard operating procedure; VOCs = volatile organic compounds.

**Table B-18-2: Sampling Locations and Methods
at CC RVAAP-70 East Classification Yard Interior Repair Pit**

Location ID	Sample ID	Depth (feet bgs)	Matrix	Sample Type		Analysis ¹	Sampling SOP Reference
70-4740-SB101	070SB-101-0061-SO	0-2	Soil	Discrete ²	N	VOCs, SVOCs, PCBs, TAL metals	Appendix A, Section 4.4
	070SB-101-0062-SO	2-4					
	070SB-102-0063-SO	4-6					
	070SB-102-0064-SO	6-7					
70-4740-SB102	070SB-102-0065-SO	0-2	Soil	Discrete ²	N	VOCs, SVOCs, PCBs, TAL metals	Appendix A, Section 4.4
	070SB-102-9065-SO	0-2			FD		
	070SB-102-0065-SO-MS	0-2			MS		
	070SB-102-0065-SO-MSD	0-2			MSD		
	070SB-102-0066-SO	2-4			N		
	070SB-104-0067-SO	4-6			N		
	070SB-104-0068-SO	6-7			N		
70-4740-SB103	070SB-103-0069-SO	0-2	Soil	Discrete ²	N	VOCs	Appendix A, Section 4.4
	070SB-103-0070-SO	2-4					
70-4740-SB104	070SB-104-0071-SO	0-2	Soil	Discrete ²	N	VOCs	Appendix A, Section 4.4
	070SB-104-0072-SO	2-4					
70-4740-SB105	070SB-105-0073-SO	6-7 unless field data indicate a different depth interval is impacted	Soil	Discrete	N	VOCs	Appendix A, Section 4.4

Location ID	Sample ID	Depth (feet bgs)	Matrix	Sample Type		Analysis ¹	Sampling SOP Reference
70-4740-SB106	070SB-106-0074-SO	6-7 unless field data indicate a different depth interval is impacted	Soil	Discrete	N	VOCs	Appendix A, Section 4.4
70-4770-SS107	070SS-107-0075-SO	0-1	Sediment	Discrete	N	PCBs, SVOCs, Explosives	Appendix A, Section 4.4
	070SS-107-9075-SO	0-1			FD		
70-4770-SB107	070SB-107-0076-SO	1-3	Soil	Discrete	N	VOCs, PCBs, SVOCs, Explosives	Appendix A, Section 4.4
	070SB-107-0077-SO	3-5					
	070SB-107-0078-SO	5-7					

Notes:

Examples of Sample IDs to be used for blanks are as follows: Equipment Blanks (070EB-DDMMYY-01) and Trip Blanks (070TB-DDMMYY-01).

1 = Because this sampling effort is designed to address specific data gaps, no samples will be analyzed for the Ravenna full-suite of analysis.

2 = Discrete soil samples will be collected in 2-foot intervals or until sandstone is reached.

bgs = below ground surface; FD = Field Duplicate; MS = Matrix Spike; MSD = Matrix Spike Duplicate; PCBs = polychlorinated biphenyls; SOP = standard operating procedure; SVOCs = semi-volatile organic compounds; TAL = target analyte list; VOCs = volatile organic compounds.

**Table B-18-3: Sampling Locations and Method
at CC RVAAP-74 Building 1034-Motor Pool Hydraulic Lift**

Location ID	Sample ID	Depth ¹ (feet bgs)	Matrix	Sample Type		Analysis ²	Sampling SOP Reference
74-1034-SB23	074SB-023-0039-SO	7.5-8.5	Soil	Discrete	N	TPH-C ₁₀ to C ₂₀ , TPH-C ₂₀ to C ₃₄ , VOCs, and PCBs	Appendix A, Section 4.4
74-1034-SB24	074SB-024-0040-SO	7.5-8.5			N	TPH-C ₁₀ to C ₂₀ , TPH-C ₂₀ to C ₃₄ , and VOCs	
	074SB-024-9040-SO	7.5-8.5			FD		
	074SB-024-0040-SO-MS	7.5-8.5			MS		
	074SB-024-0040-SO-MSD	7.5-8.5			MSD		
74-1034-SB25	074SB-025-0041-SO	7.5-8.5			N		
074-1034-WP1	074WP-001-0001-GW	20-25	Groundwater	Discrete Grab (Screening Samples) ³	N	VOCs and PAHs	Appendix A, Section 4.3.6
074-1034-WP2	074WP-002-0001-GW	20-25			N		
074-1034-WP3	074WP-003-0001-GW	20-25			N		
074-1034-WP4	074WP-004-0001-GW	20-25			N		
074MW-001	074MW-001-0001-GW	20-30	Groundwater	Discrete	N	VOCs, PAHs, and PCBs	Appendix A, Section 4.3.6
	074MW-001-0002-GW	20-30			N		
	074MW-001-0003-GW	20-30			N		
	074MW-001-0004-GW	20-30			N		
074MW-002	074MW-002-0001-GW	20-30	Groundwater	Discrete	N	VOCs, PAHs, and PCBs	Appendix A, Section 4.3.6
	074MW-002-0002-GW	20-30			N		
	074MW-002-9002-GW	20-30			FD		
	074MW-002-0002-GW-MS	20-30			MS		
	074MW-002-0002-GW-MSD	20-30			MSD		
	074MW-002-0003-GW	20-30	Groundwater	Discrete	N	VOCs, PAHs, and PCBs	

Location ID	Sample ID	Depth ¹ (feet bgs)	Matrix	Sample Type		Analysis ²	Sampling SOP Reference
	074MW-002-0004-GW	20-30			N		Appendix A, Section 4.3.6
074MW-003	074MW-003-0001-GW	20-30	Groundwater	Discrete	N	VOCs, PAHs, and PCBs	Appendix A, Section 4.3.6
	074MW-003-0002-GW	20-30			N		
	074MW-003-0003-GW	20-30			N		
	074MW-003-9003-GW	20-30			FD		
	074MW-003-0003-GW-MS	20-30			MS		
	074MW-003-0003-GW-MSD	20-30			MSD		
	074MW-003-0004-GW	20-30			N		

Notes:

Examples of Sample IDs to be used for blanks are as follows: Field Blanks (074FB-DDMMYY-01), Equipment Blanks (074EB-DDMMYY-01), and Trip Blanks (074TB-DDMMYY-01).

1 = Groundwater grab samples will be collected near the water table (anticipated to be 20-25 feet bgs) and groundwater monitoring wells samples will be collected from the within the screened interval (anticipated to be 20-30 feet bgs); however, actual depth will be determined in the field.

2 = Because this sampling effort is designed to address specific data gaps, no samples will be analyzed for the Ravenna full-suite of analysis.

3 = Four groundwater grab samples (screening samples) will be initially collected. Based on screening results, additional groundwater grab samples may be collected.

bgs = below ground surface; FD = Field Duplicate; MS = Matrix Spike; MSD = Matrix Spike Duplicate; PAH = polycyclic aromatic hydrocarbons; PCBs = polychlorinated biphenyls; SOP = standard operating procedure; TPH = Total Petroleum Hydrocarbon; VOCs = volatile organic compounds.

WORKSHEETS #19 & 30: SAMPLE CONTAINERS, PRESERVATIONS, AND HOLD TIMES

(EPA UFP-QAPP Guidance Manual, Section 3.1.1)

This worksheet summarizes the analytical methods for each sampling matrix, including the required sample volume, containers, preservation, and holding time requirements. Details concerning sampling handling are included on [Worksheets #26 & 27](#). Data package turnaround time (TAT) for all analyses and laboratories is 21 calendar days from receipt of the samples.

**Table B-19-1: Analytical Requirements, Sample Containers, Preservation, and Hold Times
(TA-St. Louis)**

Analyte/Analyte Group	Matrix	Analytical and Preparation Method/ SOP Reference	Containers (Number, Size, and Type per Sample)	Preservation Requirements (Chemical, Temperature, Light Protected)	Preparation Holding Time	Analytical Holding Time
VOCs	Soil	8260C/5035A/ST-MS-0002	Terracore Kit	Cool to 4 ± 2 C	14 days	14 days
VOCs	Water	8260C/5030B/ST-MS-0002	3 x 40-mL glass vials	HCL, Cool to 4 ± 2 C	14 days	14 days
TPH-C ₁₀ to C ₂₀ and TPH-C ₂₀ to C ₃₄	Soil	8015B/3550C/ST-GC-0005	1 x 4-ounce glass jar	Cool to 4 ± 2 C	14 days	40 days
Explosives	Soil	8330B/ST-LC-0002	1 x 4-ounce glass jar	Cool to 4 ± 2 C	14 days	40 days
Explosives	Water	8330B/ST-LC-0002	2 x 1-L amber glass bottle	Cool to 4 ± 2 C	7 days	40 days
SVOCs	Soil	8270D/3550C/ST-MS-0001	1 x 4-ounce glass jar	Cool to 4 ± 2 C	14 days	40 days
PAHs	Water	8270D SIM/3550C/ST-MS-0001	2 x 1L Amber glass bottle	Cool to 4 ± 2 C	7 days	40 days
PCBs	Soil	8082A/3550C/ST-GC-0015	1 x 4-ounce glass jar	Cool to 4 ± 2 C	None	None
PCBs	Water	8082A/3510C/ST-GC-0015	2 x 1-L amber glass bottle	Cool to 4 ± 2 C	None	None
TAL Metals	Soil	6020A/3050B/ST-MS-0001	1 x 2-ounce glass jar	Cool to 4 ± 2°C	180 Days	180 Days

Analyte/Analyte Group	Matrix	Analytical and Preparation Method/ SOP Reference	Containers (Number, Size, and Type per Sample)	Preservation Requirements (Chemical, Temperature, Light Protected)	Preparation Holding Time	Analytical Holding Time
TAL Metals	Water	6020A/3010AB/ST-MS-0001	1 x 500-mL HDPE bottle	Cool to $4 \pm 2^{\circ}\text{C}$, preserved with HNO_3	180 Days	180 Days
Mercury	Soil	7471B/ST-MS-0007	1 x 2-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	28 Days	28 Days
Mercury	Water	7470A/ST-MS-0005	1 x 500-mL HDPE bottle	Cool to $4 \pm 2^{\circ}\text{C}$, preserved with HNO_3	28 Days	28 Days
TCLP VOCs	Soil	8260C/1311/ST-MS-0002	2 x 2-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	14 days	14 days
TCLP SVOCs	Soil	8270D/1311/ST-MS-0001	1 x 4-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	14 days	40 days
TCLP Pesticides	Soil	8081B/1311/ST-GC-0016	1 x 4-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	14 days	40 days
TCLP Herbicides	Soil	8151A/1311/ST-GC-0017	1 x 4-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	14 days	40 days
TCLP Resource Conservation and Recovery Act (RCRA) 7 Metals	Soil	6010C/1311/ST-MT-0003	1 x 4-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	180 days	180 days
TCLP Mercury	Soil	7470A/1311/ST-MT-005	1 x 4-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	28 days	28 days
pH	Soil	9045D/ST-WC-0011	1 x 4-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	N/A	ASAP
Total Cyanide	Soil	9012B/ST-WC-0002	1 x 4-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	N/A	14 days
Total Sulfide	Soil	9034/ST-WC-0012	1 x 4-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	N/A	7 days
Ignitability	Soil	1010A/ST-WC-0026	1 x 4-ounce glass jar	Cool to $4 \pm 2^{\circ}\text{C}$	N/A	180 days
TCLP VOCs	Water	8260C/1311/ST-MS-0002	3 x 40-mL glass vials	Cool to $4 \pm 2^{\circ}\text{C}$	14 days	14 days

Analyte/Analyte Group	Matrix	Analytical and Preparation Method/ SOP Reference	Containers (Number, Size, and Type per Sample)	Preservation Requirements (Chemical, Temperature, Light Protected)	Preparation Holding Time	Analytical Holding Time
TCLP SVOCs	Water	8270D/1311/ST-MS-0001	1 x 1-L amber glass bottle	Cool to 4 ± 2°C	7 days	40 days
TCLP Pesticides	Water	8081B/1311/ST-GC-0016	1 x 1-L amber glass bottle	Cool to 4 ± 2°C	7 days	40 days
TCLP Herbicides	Water	8151A/1311/ST-GC-0017	1 x 1-L amber glass bottle	Cool to 4 ± 2°C	7 days	40 days
TCLP RCRA 7 Metals	Water	6010C/1311/ST-MT-0003	1 x 500-mL HPDE bottle	Cool to 4 ± 2°C	180 days	180 days
TCLP Mercury	Water	7470A/1311/ST-MT-005	1 x 500-mL HPDE bottle	Cool to 4 ± 2°C	28 days	28 days
pH	Water	9040C/ST-WC-0011	1 x 250mL HDPE bottle	Cool to 4 ± 2°C	N/A	ASAP
Total Cyanide	Water	9012B/ST-WC-0002	1 x 250mL HDPE bottle	NaOH, Cool to 4 ± 2°C	N/A	14 days
Total Sulfide	Water	9034/ST-WC-0012	1 x 250mL HDPE bottle	NaOH & Zn Acetate, Cool to 4 ± 2°C	N/A	7 days
Ignitability	Water	1010A/ST-WC-0026	1 x 250mL HDPE bottle	Cool to 4 ± 2°C	N/A	180 days

Notes:

See Worksheet 23 for SOP reference details.

Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

Some analyses can be performed from the same bottle. The lab will minimize the exact number of bottles per sample.

Laboratory Standard Operating Procedures are subject to revision and updates during duration of the project, lab will use the most current revision of the SOP at the time of analysis.

Sample size is a minimum; the containers listed will be filled to compensate for any required re-analysis or re-extractions. For samples requiring MS/MSD containers listed should be tripled or at least enough volume or weight for lab to run three analyses.

WORKSHEET #20: FIELD QUALITY CONTROL

(UFP-QAPP Guidance Manual, Section 3.1.1)

This worksheet summarizes the field QC samples to be collected and analyzed for the project. It shows the relationship between the number of field samples and associated QC samples for each combination of analyte/analytical group and matrix. Note that if samples are collected over the estimated number shown, additional QC samples will be collected at the rate shown. Field QC samples are not collected for waste characterization samples. Due to the small number of samples proposed for collection, 10% of the total number of soil samples will be collected for field duplicates, and 5% of the total number of soil samples will be collected for matrix spike/matrix spike duplicates. Likewise, 10% of the total number of groundwater samples will be collected for field duplicates, and 5% of the total number of groundwater samples will be collected for matrix spike/matrix spike duplicates. At least 1 field duplicate sample will be collected per groundwater monitoring event from the monitoring wells. However, field duplicates and matrix spike/matrix spike duplicates will not be collected from screening groundwater grab samples.

Field QC measures are described on [Worksheet #12](#) and [Worksheet #17](#).

Matrix	Analytical Group	Estimated No. of Field Samples ⁽¹⁾	Field Duplicates	Matrix Spike / Matrix Spike Duplicates	Field Blanks ⁽²⁾	Equipment Blanks ⁽³⁾	Trip Blanks	Estimated Number of Total Analyses
Soil	VOCs	38	4 - Estimated	3 - Estimated	0	3	3	51 - Estimated
Soil / Sediment	SVOCs, PCBs	12	2 - Estimated	1 - Estimated	0	1	0	16 - Estimated
Soil / Sediment	Explosives	4	1 - Estimated	0 - Estimated	0	1	0	6 - Estimated
Soil	TPH-C ₁₀ to C ₂₀ and C ₂₀ to C ₃₄	3	1 - Estimated	1 - Estimated	0	1	0	6 - Estimated
Soil	TAL metals	8	1 - Estimated	1 - Estimated	0	1	0	11 - Estimated
Groundwater	PAHs, PCBs	12	2 - Estimated	2 - Estimated	0	4	0	20 - Estimated
Groundwater	VOCs	24	4 - Estimated	3 - Estimated	0	4	6	41 - Estimated

Notes:

- (1) Number of samples will be based on conditions encountered in the field. Number of samples does not include screening groundwater grab samples.
- (2) Field blanks will be collected only if contaminant sources (e.g. airborne dust, exhaust fumes) are expected to be present in the field.
- (3) Assuming 1 equipment blank per event, per matrix.

WORKSHEET #21: STANDARD OPERATING PROCEDURES (SOPS)

(UFP-QAPP Guidance Manual, Section 3.1.2)

In lieu of stand along SOPs, the standard operating procedures for this project are included in the Field Sampling Plan provided as Appendix A. Key procedures are listed in the table below.

FSP Section	Standard Operating Procedures	Originating Organization
Section 4	Geophysics	Parsons
Section 4	Drilling	Parsons
Section 4	Soil Sampling	Parsons
Section 4	Well Construction and Development	Parsons
Section 4	Surveying	Parsons
Section 4	Groundwater Sampling	Parsons
Section 4	Surface soil and sediment sampling	Parsons

All field activities and sampling procedures uses on this project will be conducted in accordance with the Facility-Wide Sampling and Analysis Plan (FWSAP) (SAIC 2011) or as described in Section 4 of the FSP provided as Appendix A to the Work Plan.

WORKSHEET #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION

(UFP-QAPP Manual Section 3.1.2.4)

This worksheet identifies all field equipment and instruments (other than analytical instrumentation) that require calibration, maintenance, testing, or inspection. In addition, it documents the frequency of activity, acceptance criteria, and corrective action requirements on the work sheet. In lieu of stand-alone SOPs, the standard operating procedures for this project are included as Section 4 of the project Field Sampling Plan included as Appendix A.

Field Equipment	Calibration, Verification, Testing, or Maintenance Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Water Quality Meter	Calibrate with pH, oxidation reduction potential (ORP), and conductivity solutions	Daily	Per manufacturer's manual	Recalibrate or replace defective meter	Operator	NA
PID	Calibrate with isobutylene	Daily	Per manufacturer's manual	Recalibrate or replace defective meter	Operator	NA
Soil and Groundwater Sampling Equipment	visual inspection	Between sampling locations	Equipment decontaminated prior to each use	Decontaminate item	Operator	NA

WORKSHEET #23: ANALYTICAL STANDARD OPERATING PROCEDURES

(UFP-QAPP Guidance Manual, Section 3.2.1)

The applicable SOPs to be used for analysis of environmental samples collected during the RI at Camp Ravenna are listed in the below tables. The laboratory SOP references were provided by TA-St. Louis and are presented in Attachment B-1.

Table B-23-1: TESTAMERICA Analytical Standard Operating Procedures

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
ST-IP-0002	Acid Digestion of Soils for inductively coupled plasma mass spectrometry (ICP) or ICP/MS, Rev.19,07/28/16	Definitive	Soil, Metals	ICP, ICP/MS	TestAmerica St. Louis	N
ST-IP-0013	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP or ICP/MS, Rev. 22 08/12/16	Definitive	Water, Metals	ICP, ICP/MS	TestAmerica St. Louis	N
ST-MS-0002	Determination of Volatile Organics by GC/MS, Rev. 26, 03/06/17	Definitive	Water and Soil, and IDW VOCs	GC/MS	TestAmerica St. Louis	N
ST-GC-0005	Extractable Total Petroleum Hydrocarbons by GC-FID, Rev. 20, 06/22/15	Definitive	Soil TPH-C ₁₀ to C ₂₀ and C ₂₀ to C ₃₄	GC/FID	TestAmerica St. Louis	N
ST-GC-0015	PCB GC Analysis, Rev. 20, 10/28/16	Definitive	Water and Soil PCBs	GC/FID	TestAmerica St. Louis	N
ST-GC-0016	Pesticide GC Analysis, Rev. 20, 10/28/16	Definitive	Soil and Water TCLP Pesticides	GC/FID	TestAmerica St. Louis	N
ST-GC-0017	Herbicide GC Analysis, Rev. 17, 10/31/16	Definitive	Soil and Water TCLP Herbicides	GC/FID	TestAmerica St. Louis	N
ST-LC-0002	Analysis of Nitroaromatic and Nitroamine Explosives by HPLC, Rev. 21, 08/03/16	Definitive	Water and Soil Explosives	GC/FID	TestAmerica St. Louis	N
ST-MS-0001	GC/MS Semi-Volatile Analysis, Rev.22, 10/28/16	Definitive	Soil and Water SVOCs, PAHs, and TCLP SVOCs	GC/MS	TestAmerica St. Louis	N

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work (Y/N)
ST-MT-0001	Analysis of Metals by Inductively Coupled Plasma / Mass Spectrometry, Rev. 25, 08/11/16	Definitive	Water and Soil TAL Metals	ICP/MS	TestAmerica St. Louis	N
ST-MT-0003	Inductively Coupled Plasma-Atomic Emission Spectroscopy (AES), Spectrometric Method for Trace Element Analysis, Rev. 18, 08/12/16	Definitive	Soil and Water TCLP Metals	ICP/AES	TestAmerica St. Louis	N
ST-MT-0005	Preparation and Analysis of Mercury in Aqueous Samples by CVAA, Rev. 17, 08/31/16	Definitive	Soil and Water TCLP Mercury	Cold Vapor AA	TestAmerica St. Louis	N
ST-MT-0007	Preparation and Analysis of Mercury in Solid Samples by CVAA, Rev. 16, 11/21/16	Definitive	Soil Mercury	Cold Vapor AA	TestAmerica St. Louis	N
ST-OP-0002	Extraction and Cleanup of Organic Compounds from Waters and Soils, Rev.27, 09/20/16	Definitive	Water and Soil Organics	GC, GC/MS	TestAmerica St. Louis	N
ST-OP-0007	Extraction of Herbicides from Waters and Soils 8151A, Rev. 17, 09/19/16	Definitive	Water and Soil Herbicides	GC	TestAmerica St. Louis	N
ST-OP-0008	Extraction of Nitroaromatic and Nitroamine Explosives [3535A] for Waters and Soils [8330B], Rec. 12, 05/23/16	Definitive	Water and Soil Explosives	HPLC	TestAmerica St. Louis	N
ST-OP-0009	Toxicity Characteristic Leaching Procedure (TCLP), Rev. 7, 10/28/16	Definitive	Water and Soil	CG, GC/MS, ICP, ICP/MS, CVAA	TestAmerica St. Louis	N
ST-WC-0011	pH Analysis in Water and Soil, Rev. 17, 07/29/16	Definitive	Soil and Water pH	Electrobe	TestAmerica St. Louis	N
ST-WC-0002	Analysis of Cyanide in Water and Soil, Rev. 18, 05/23/16	Definitive	Soil and Water Cyanide	Discrete Analyzer	TestAmerica St. Louis	N
ST-WC-0012	Analysis of Sulfide, Rev. 14, 03/31/16	Definitive	Soil and Water Sulfide	Titration	TestAmerica St. Louis	N
ST-WC-0026	Flash Point by Pensky-Martens Closed Cup Tester, Rev. 18, 11/21/16	Definitive	Soil and Water Ignitability	Pensky-Martens Closed Cup Tester	TestAmerica St. Louis	N

WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION

(UFP-QAPP Guidance Manual, Section 3.2.2)

The calibration procedures for the instruments to be used for analysis of environmental samples collected are presented in the below tables. The specific analytical SOPs referenced in these tables are provided in Attachment B-1.

Table B-24-1: Analytical Instrument Calibration, TA–St. Louis

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
Liquid chromatography–tandem mass spectrometry (LC/MS/MS)	Tune Check	Prior to initial calibration (ICAL) and after any mass calibration or maintenance is performed.	Tuning standard must contain analytes of interest or appropriate substitute. Mass assignments of tuning standard within 0.5 atomic mass unit (amu) of true value.	Retune instrument. If the tuning will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Lab Manager / Analyst ^a	WS-LC-0025
LC/MS/MS	Minimum five-point initial calibration for target analytes, lowest concentration standard at or below the reporting limit	Initial calibration prior to sample analysis	Each calibration point for each analyte must calculate to be within 75-125%, except the lowest cal point which must calculate to within 70-130%.	Evaluate standards, chromatography, and mass spectrometer response. If problem found with above, correct as appropriate, then repeat initial calibration.	Lab Manager / Analyst ^a	WS-LC-0025
LC/MS/MS	Second-source calibration verification	Once per initial calibration, following initial calibration.	All reported analytes and labeled compounds within $\pm 25\%$ of true value.	Evaluate data. If problem (e.g., concentrated standard, plugged transfer line) found, correct, then repeat second source verification. If it still fails, then repeat initial calibration.	Lab Manager / Analyst ^a	WS-LC-0025

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
LC/MS/MS	Daily calibration verification	Before sample analysis, after every 10 samples, and at the end of the sequence.	All reported analytes and labeled compounds within $\pm 25\%$ of true value.	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment. For closing continuing calibration verification (CCV), if compounds are not identified as critical compounds of concern report results with qualifiers. For closing CCVs, if the compound is identified as a critical compound of concern, then recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Lab Manager / Analyst ^a	WS-LC-0025
GC/MS	ICAL – five-point ICAL	Initial calibration prior to sample analysis	Percent relative standard deviation (%RSD) <20% all compounds, Relative Response Factor (RRT) meet method criteria	Repeat calibration	Lab Manager / Analyst ^a	ST-MS-0002

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
GC/MS	Second Source Calibration Verification (SSCV)	Once after each initial calibration	Value of second source for all analytes within $\pm 30\%$ of expected	Rerun initial calibration verification (ICV) one time, second failure requires recalibration	Lab Manager / Analyst ^a	ST-MS-0002
GC/MS	CCV	Daily, before sample analysis, and every 12 hours of analysis time	+/- 20% D criteria for all analytes	Re-inject CCV; if passes rerun previous ten samples and continue run; if 2nd CCV fails, recalibrate	Lab Manager / Analyst ^a	ST-MS-0002
GC/MS	Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis	Specific ion abundance criteria of 4-bromofluorobenzene (BFB) from method	Retune instrument and verify	Lab Manager / Analyst ^a	ST-MS-0002
GC/MS	Retention Time (RT) window position establishment	Once per ICAL and at the beginning of the analytical sequence	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed.	N/A	Lab Manager / Analyst ^a	ST-MS-0002
GC/MS	Evaluation of Relative Retention Times (RRT)	With each sample	RRT of each reported analyte within +/- 0.06 RRT units	Correct problem, then rerun ICAL	Lab Manager / Analyst ^a	ST-MS-0002
GC/MS	ICAL – five-point ICAL	Initial calibration prior to sample analysis	%RSD<20% all compounds, Relative Response Factor meet method criteria	Repeat calibration	Lab Manager / Analyst ^a	ST-MS-0001
GC/MS	SSCV	Once after each initial calibration	Value of second source for all analytes within $\pm 30\%$ of expected	Rerun ICV one time, second failure requires recalibration	Lab Manager / Analyst ^a	ST-MS-0001

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
GC/MS	CV	Daily, before sample analysis, and every 12 hours of analysis time	+/- 20%Dilution (%D) criteria for all analytes	Re-inject CCV; if passes rerun previous ten samples and continue run; if 2nd CCV fails, recalibrate	Lab Manager / Analyst ^a	ST-MS-0001
GC/MS	Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis	Specific ion abundance criteria of Decafluorotri-phenylphosphine (DFTPP) from method	Retune instrument and verify	Lab Manager / Analyst ^a	ST-MS-0001
GC/MS	Performance Check	At the beginning of each 12-hour period, prior to analysis of samples	Degradation $\leq$ 20% for Dichlorodiphenyltri chloroethane (DDT). Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks	Lab Manager / Analyst ^a	ST-MS-0001
GC/MS	Retention Time (RT) window position establishment	Once per ICAL and at the beginning of the analytical sequence	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	N/A	Lab Manager / Analyst ^a	ST-MS-0001
GC/FID	ICAL – five-point ICAL	Initial calibration prior to sample analysis	Linear mean RSD $\leq$ 20%	Recalibrate	Lab Manager / Analyst ^a	ST-GC-0005

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
GC/FID	SSCV	Once after each initial calibration	Value of second source for all analytes within $\pm 25\%$ of expected value (initial source)	Rerun ICV one time, second failure requires recalibration	Lab Manager / Analyst ^a	ST-GC-0005
GC/Flame ionizing detector (FID)	Initial Calibration Verification (ICV and CCV)	ICV: Daily, before sample analysis. CCV: Every 12 hours of analysis time and at the end of the analysis sequence	All analytes within $\pm 15\%$ of expected value from the ICAL	Re-inject CCV; if passes rerun previous 10 samples and continue run; if 2nd CCV fails, recalibrate	Lab Manager / Analyst ^a	ST-GC-0005
GC/FID	RT window position establishment	Once per ICAL and at the beginning of the analytical sequence	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used	N/A	Lab Manager / Analyst ^a	ST-GC-0005
GC/FID	RT window width	At method set-up and after major maintenance (e.g. column change)	RT width is ± 0.3 times standard deviation for each analyte RT from the 72-hour study	N/A	Lab Manager / Analyst ^a	ST-GC-0005
GC	RT window width	At method set-up and after major maintenance (e.g. column change)	RT width is ± 0.3 times standard deviation for each analyte RT from the 72-hour study	N/A	Lab Manager / Analyst ^a	ST-GC-0015, ST-GC-0016, ST-GC-0017
GC	ICAL – five-point ICAL	Initial calibration prior to sample analysis	Mean RSD for each compound $\leq 20\%$	Recalibrate	Lab Manager / Analyst ^a	ST-GC-0015, ST-GC-0016, ST-GC-0017

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
GC	SSCV	Once after each initial calibration	Value of second source for all analytes within $\pm$ 30% of expected value (initial source)	Rerun ICV one time, second failure requires re-calibration	Lab Manager / Analyst ^a	ST-GC-0015, ST-GC-0016, ST-GC-0017
GC	ICV and CCV	ICV: Daily, before sample analysis CCV: After every 12 hours of analysis time and at the end of the analysis sequence	All analytes within $\pm$ 20% of expected value from the ICAL	Re-inject CCV; if passes rerun previous 10 samples and continue run; if 2nd CCV fails, recalibrate	Lab Manager / Analyst ^a	ST-GC-0015, ST-GC-0016, ST-GC-0017
GC	Confirmation of positive results (second column)	All positive results must be confirmed	Calibration and QC criteria are the same for the second column. RPD<40% between primary and second column results	Report from both columns.	Lab Manager / Analyst ^a	ST-GC-0015, ST-GC-0016, ST-GC-0017
High-performance liquid chromatography (HPLC)	ICAL – five-point ICAL	Initial calibration prior to sample analysis	%RSD<20% all compounds, Relative Response Factor meet method criteria	Repeat calibration	Lab Manager / Analyst ^a	ST-LC-0002
HPLC	SSCV	Once after each initial calibration	Value of second source for all analytes within $\pm$ 20% of expected	Rerun ICV one time, second failure requires recalibration	Lab Manager / Analyst ^a	ST-LC-0002
HPLC	CCV	Daily, before sample analysis, and every 12 hours of analysis time	+/- 20%D criteria for all analytes	Re-inject CCV; if passes rerun previous 10 samples and continue run; if 2nd CCV fails, recalibrate	Lab Manager / Analyst ^a	ST-LC-0002

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
HPLC	Confirmation of positive results (second column)	All positive results must be confirmed	Calibration and QC criteria are the same for the second column. RPD<40% between primary and second column results	Report from both columns.	Lab Manager / Analyst ^a	ST-LC-0002
ICP/AES	Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months high a high standard at the upper limit of the range	Within + 10% of true value	Dilute samples within the calibration range, or re-establish/verify the LDR	Lab Manager / Analyst ^a	ST-MT-0003
ICP/AES	ICAL – minimum one high standard and a calibration blank	Daily initial calibration prior to sample analysis	3 standards and a blank. Correlation Coefficient of ≥ 0.998	Recalibrate	Lab Manager / Analyst ^a	ST-MT-0003
ICP/AES	SSCV	Once after each initial calibration, prior to sample analysis	Value of second source for all analyte(s) within $\pm 10\%$ of expected	Recalibrate	Lab Manager / Analyst ^a	ST-MT-0003
ICP/AES	CCV	After every 10 samples and at the end of the analysis sequence	All analytes within + 10% of expected value	Recalibrate – rerun 10 samples previous to failed CCV.	Lab Manager / Analyst ^a	ST-MT-0003
ICP/AES	Calibration Blanks (ICB/ Continuing Calibration Blanks [CCB])	After ICV (ICB) and after every CCV (CCB)	All analytes less than LOQ	Correct problem and rerun ICB or CCB and all bracketing samples	Lab Manager / Analyst ^a	ST-MT-0003

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
ICP/AES	Low-level Calibration Check Standard (Low-level ICV)	Daily	All analytes within + 30% of expected value	Correct problem and repeat ICAL	Lab Manager / Analyst ^a	ST-MT-0003
ICP/AES	Interference Check Solutions (ICS)	After ICAL and prior to sample analysis	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spike analytes). ICS-AB: within + 20% of true value	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples	Lab Manager / Analyst ^a	ST-MT-0003
ICP-MS	LDR or high-level check standard	At initial set up and checked every 6 months high a high standard at the upper limit of the range	Within + 10% of true value	Dilute samples within the calibration range, or re-establish/verify the LDR	Lab Manager / Analyst ^a	ST-MT-0001
ICP-MS	Tuning	Prior to ICAL	Mass calibration < 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height	Retune instrument and verify	Lab Manager / Analyst ^a	ST-MT-0001
ICP-MS	ICAL – minimum one high standard and a calibration blank	Daily initial calibration prior to sample analysis	3 standards and a blank. Correlation Coefficient of ≥ 0.998	Recalibrate	Lab Manager / Analyst ^a	ST-MT-0001

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
ICP-MS	SSCV	Once after each initial calibration, prior to sample analysis	Value of second source for all analyte(s) within $\pm 10\%$ of expected	Recalibrate	Lab Manager / Analyst ^a	ST-MT-0001
ICP-MS	CCV	After every 10 samples and at the end of the analysis sequence	All analytes within + 10% of expected value	Recalibrate – rerun 10 samples previous to failed CCV.	Lab Manager / Analyst ^a	ST-MT-0001
ICP-MS	ICB and CCB	After ICV (ICB) and after every CCV (CCB)	All analytes less than LOQ	Correct problem and rerun ICB or CCB and all bracketing samples	Lab Manager / Analyst ^a	ST-MT-0001
ICP-MS	Low-level Calibration Check Standard (Low-level ICV)	Daily	All analytes within + 20% of expected value	Correct problem and repeat ICAL	Lab Manager / Analyst ^a	ST-MT-0001
ICP-MS	ICS	After ICAL and prior to sample analysis	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spike analytes). ICS-AB: within + 20% of true value	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples	Lab Manager / Analyst ^a	ST-MT-0001
Cold Vapor AA	ICAL	Daily initial calibration prior to sample analysis	Correlation coefficient $R \geq 0.995$ for linear regression	Recalibrate	Lab Manager / Analyst ^a	ST-MT-0007 and ST-MT-0005

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
Cold Vapor AA	SSCV	Once after each initial calibration, prior to sample analysis	Value of second source for all analyte(s) within $\pm 10\%$ of expected value (second source)	Recalibrate	Lab Manager / Analyst ^a	ST-MT-0007 and ST-MT-0005
Cold Vapor AA	CCV	After every 10 samples and at the end of the analysis sequence.	All analytes within + 20% of expected value	Recalibrate – rerun 10 samples prior to the failed CCV.	Lab Manager / Analyst ^a	ST-MT-0007 and ST-MT-0005
Discrete Analyzer	ICAL	Daily – minimum of 5 points	Correlation coefficient of 0.995 or greater	Recalibrate	Lab Manager / Analyst ^a	ST-WC-0002
Discrete Analyzer	ICV	Immediately after the last calibration standard	Within +/- 15% of true value	Recalibrate	Lab Manager / Analyst ^a	ST-WC-0002
Discrete Analyzer	CCV	Every 10 samples during the sequence and after the last analytical sample.	$\pm 15\%$ of true value. The high and low distilled check standard must be $\pm 10\%$.	Recalibrate and reanalyze all analytical samples analyzed since the last compliant CCV	Lab Manager / Analyst ^a	ST-WC-0002
Pensky-Marten Closed Cup	Initial Calibration	Annual	Thermometers are verified annually. Rotation apparatus verified annually.	Maintenance required	Lab Manager / Analyst ^a	ST-WC-0026
Pensky-Marten Closed Cup	Calibration Check	Prior to running samples	The flash point of a check standard (p-xylene) must be obtained within a value of $27.2 \pm 1.1^{\circ}\text{C}$	Rerun check	Lab Manager / Analyst ^a	ST-WC-0026
Pensky-Marten Closed Cup	Continuing Calibration Check (second source)	Every 10 samples	Control limit is $27.2 \pm 1.1^{\circ}\text{C}$.	Recalibrate – rerun samples bracketed by failing CCV	Lab Manager / Analyst ^a	ST-WC-0026

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action (CA)	SOP Reference
Electronic Probe	ICAL	Prior to sample analysis	A three-point calibration is performed. Buffer solutions of 4, 7, and 10 are used. Readings must be within ± 0.05 unit for each buffer solution.	Recalibrate	Lab Manager / Analyst ^a	ST-WC-0011
Electronic Probe	CCV	Check the calibration by analyzing the calibration check standard (pH 7 buffer) after the initial calibration, every ten samples, and at the end of the analytical run	pH reading must be within ± 0.05 unit of the pH 7 buffer.	Recalibrate – rerun samples bracketed by failing CCV	Lab Manager / Analyst ^a	ST-WC-0011
Titration	Calibration Check	Prior to sample analysis	Recovery Standard Deviation of 20%	Recalibrate – rerun samples	Lab Manager / Analyst ^a	ST-WC-0012

^a The analyst initiates the corrective action (CA) and the lab manager and analysts are responsible for the CA.

WORKSHEET #25: ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION

(UFP-QAPP Guidance Manual, Section 3.2.3)

This worksheet provides information on analytical instruments and equipment, maintenance, testing, and inspection. To ensure that the analytical instruments and equipment are available and in working order when needed, all laboratory analytical equipment will undergo maintenance and testing procedure in accordance with the laboratory SOPs (provided in Attachment B-1).

Table B-25-1: Analytical Instrument and Equipment Maintenance, Testing, and Inspection for TA-St. Louis

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference ⁽¹⁾
LC/MS/MS	Replace columns as needed, check eluent reservoirs	Sensitivity check	Instrument performance and sensitivity	Daily or as needed	CCV pass criteria	Recalibrate	TestAmerica Chemist	WS-LC-0025
GC-MS	Clean sources, maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintenance as needed	Tune and CCV pass criteria	Recalibrate instrument	TestAmerica Chemist	ST-MS-0001 and ST-MS-0002
GC-MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Response factors and chromatogram review	Instrument performance and sensitivity	As needed	Tune and CCV pass criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	TestAmerica Chemist	ST-MS-0001 and ST-MS-0002
GC	Change septum, clean injection port, change or clip column, install new liner, replace column, filters and seals	Detector signals and chromatogram review	Instrument performance and sensitivity	As needed	CCV passes criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	TestAmerica Chemist	ST-GC-0005 ST-GC-0015 ST-GC-0016 ST-GC-0017

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference ⁽¹⁾
HPLC	Check column flow; check for leaks; Change purge valve frit; change guard cartridge and frit cap; replace column; replace UV source; replace pump seals.	Performance	QC check	As needed and day of use	Per instrument manufacturer	Replace; reanalyze any affected samples	TestAmerica Chemist	LC-0002 (HPLC)
ICP/MS	Replace disposables, clean/change nebulizer, torch, and cones	Tuning	Instrument performance and sensitivity	As needed	Tune and CCV pass criteria	Recalibrate	TestAmerica Chemist	ST-MT-0001
ICP/AES	Clean/Replace sample lines/probe, FAST valve, torch, nebulizer, spray chamber	Performance	Instrument performance and sensitivity	As needed	ICV and CCV pass criteria	Recalibrate	TestAmerica Chemist	ST-MT-0003
CVAA	Clean windows, optics, lamp. Inspect tubing and glassware	Performance	Instrument performance and sensitivity	As needed	ICV and CCV pass criteria	Recalibrate	TestAmerica Chemist	ST-MT-0005
pH probe	Clean and inspect	Performance	QC check	Day of use	Meet method criteria	Clean probe; reanalyze any affected samples	TestAmerica Chemist	ST-WC-0011
Flashpoint apparatus	Clean and inspect – annual check of rotator	Performance	QC check	Day of use	Meet method criteria	Call for repair; reanalyze any affected samples	TestAmerica Chemist	ST-WC-0026

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference ⁽¹⁾
Titration	Visual inspection of glassware used; pipette calibration	Performance	QC check	Day of use	Meet method criteria	Replace; reanalyze any affected samples	TestAmerica Chemist	ST-WC-0012
Discrete analyzer	Check air pressure, change tubing as needed, finish run with wash out	Performance	QC check	Day of use	Meet method criteria	Contact repair service – reanalyze any affected samples.	TestAmerica Chemist	ST-WC-0002

(1) Laboratory Standard Operating Procedures are subject to revision and updates during duration of the project, lab will use the most current revision of the SOP at the time of analysis.

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WORKSHEETS #26 & 27: SAMPLE HANDLING, CUSTODY, AND DISPOSAL

(UFP-QAPP Guidance Manual, Section 3.3)

26.1 Sample Numbering

A sample numbering system will be implemented to identify each sample collected during the investigation using a unique Sample ID. The numbering system will ensure each sample is uniquely labeled and will provide a tracking procedure to allow retrieval of information about each sample collected. QC samples will be numbered using the same system and notes will be made in the field notebook to record which samples are QC samples; however, duplicates will not be identified to the laboratory. The sample numbering system will follow the guidelines presented in the FWSAP (SAIC, February 2011).

Every Sample ID will be preceded by the AOC (i.e. CC RVAAP-69, CC RVAAP-70, and CC RVAAP-74 denoted by 069, 070, and 074, respectfully) where the sample was collected. The type of the sample location will be reflected in the sample number using “SS” for surface soil, “SB” for soil boring, “SD” for sediment, “MW” for monitoring well, “WP” for groundwater well point and “IDW” for IDW samples. Unique, sequential station numbers for each sample location beginning with the following number from the last number used from previous investigation stations and extending into any subsequent investigative phases. Sequential sample numbers will indicate the number assigned to the sample collected from the sampling station. The environmental medium collected will also be reflected in the sample number using “SO” for soil and “GW” for groundwater. Therefore, the sample number will be as follows:

AOC# Type – Sequential Station # – Sequential Sample # - Medium

For example:

The sample number “074SB-023-0001-SO” would indicate the first soil sample collected from AOC CC RVAAP-74 from soil boring at station 23.

The sample number “074MW-001-0002-GW” would indicate the second groundwater sample collected from AOC CC RVAAP-74 at monitoring well 1.

All new wells will be identified per the requirements of the FWSAP. There are no existing monitoring wells at CC RVAAP-69 or CC RVAAP-74; therefore, wells installed during the investigations will be numbered consecutively beginning with the designation 69MW-001 for wells installed at CC RVAAP-69, and 74MW-001 for wells installed at CC RVAAP-74. Boreholes drilled for monitor well installation, but subsequently abandoned, also will be numbered consecutively beginning with the designation XXXSB-001. If boreholes previously have been drilled at the AOC, then numbering will begin with the next highest unused number. The well identification system will be consistent with the location/sample identification naming convention specified in Section 6.3 of the FWSAP.

26.2 Sample Handling

To ensure sample authenticity and data defensibility, proper sample handing system procedures will be followed from the time of sample collection to final sample disposal. The Contractor Sampling Lead or designee is responsible for completing the sample bottle label and Chain-of-

Custody (CoC) form, sample collection, storage of samples in the field pending shipment to the laboratory, sample packing, and coordination of sample shipment via overnight courier.

The laboratory receiving staff and/or custodian will acknowledge the receipt of shipped samples upon their arrival at the laboratory. The laboratory analytical technicians will prepare and analyze the field samples in accordance with the analytical SOPs. The field samples and all extracts will be stored in the refrigerators or walk-in coolers at the laboratory for 30 days after a final report has been submitted to Contractor. The laboratory hazardous waste manager will be responsible for the final sample disposal upon notice from the Contractor Project Chemist or designee.

Table B-26-1: Responsibilities for Sample Handling, Custody, and Disposal

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT	
Sample Collection (Personnel/Organization)	Contractor Sampling Lead or designee
Sample Packaging (Personnel/Organization)	Contractor Sampling Lead or designee
Coordination of Shipment (Personnel/Organization)	Contractor Project Chemist
Type of Shipment/Carrier	Overnight (FedEx anticipated, but other carriers may be used as/if necessary)
SAMPLE RECEIPT AND ANALYSIS	
Sample Receipt (Personnel/Organization)	Sample receiving staff: TA-St. Louis
Sample Custody and Storage (Personnel/Organization)	Laboratory staff: TA-St. Louis
Sample Preparation (Personnel/Organization)	Laboratory Analyst: TA-St. Louis
Sample Determinative Analysis (Personnel/Organization)	Laboratory Analyst: TA-St. Louis
SAMPLE ARCHIVING	
Field Sample Storage (No. of days from final lab report submittal)	30 days
Sample Extract/Digestate Storage (No. of days from extraction/digestion)	30 days
SAMPLE DISPOSAL	
Personnel/Organization	Laboratory staff: TA-St. Louis
Number of Days from final lab report	30 days

26.3 Sample Labeling

Sample labels will include, at a minimum, project name, project number, sample ID, date/time collected, analysis group or method, preservative, and sampler's name. Labels will be taped to the sample container prior to sample collection to ensure that they do not separate.

26.4 Field Sample Custody Procedures (Sample Collection, Packaging, Shipment, and Delivery to Laboratory)

Samples will be collected by field team members under the supervision of Contractor Sample Team Lead. Sample will be preserved as described on Worksheets #19 & 30. Samples will be cushioned if necessary with packaging material and placed into coolers and the CoC form will be

placed in a plastic bag and taped to the inside of the cooler lid. Custody seals will be placed on each cooler in a manner that the cooler cannot be opened without breaking the seal. Each custody seal shall be signed and dated by the person packaging the cooler and the seals shall be covered by clear packing tape to prevent accidental loss or damage during shipping. Coolers will be shipped to the laboratory via overnight shipping, with the air bill number indicated on the CoC form (to relinquish custody). Upon delivery, the laboratory will log in each cooler and report the status of the samples.

The following addresses will be used for sample shipment:

Laboratory: TestAmerica Laboratories, Inc. – St. Louis
Attn.: Erika Gish
13715 Rider Trail North
St. Louis, MO 63045
Tel: 314-298-8566

26.5 Laboratory Sample Custody Procedures (Receipt of Samples, Archiving, Disposal)

All laboratory sample receipt, internal custody, sample archiving, and disposal procedures shall be completed in accordance with the laboratory specific SOPs. All documentation shall be available upon request or during a review of the laboratory for inspection.

26.6 Sample Identification Procedures

Upon removing the custody seals/tape and opening the cooler at the analytical laboratory, the receiving clerk will sign the CoC form and take the temperature using the temperature blank (if absent, a representative sample container shall be used). Then the sample containers in the cooler will be unpacked and checked against the client's CoC form. Any discrepancies noted with the samples will be noted on the sample receipt form. All cooler temperatures and the status of any broken or damaged sample containers shall be noted on the sample receipt form. The laboratory shall contact the Contractor Project Chemist or designee immediately upon discovery of non-compliant temperatures, broken or damaged sample containers, or any other issues that may possibly impact sample analysis and/or data quality. The clerk will deliver the CoC form (and any other paperwork) to the Laboratory PM for entry into the Laboratory Information Management System (LIMS) and for client notification.

The laboratory will assign each sample shipment to a unique SDG number. The laboratory shall log in the samples received and assign each field sample a laboratory sample ID based on the designated SDG and the information on the Chain-of-Custody Form. The laboratory shall send (via email) the sample login forms generated by the Laboratory Information Management System (LIMS) along with the signed Chain-of-Custody Forms and sample receipt forms/checklist to the Contractor Project Chemist or designee as soon as possible after sample receipt.

26.7 Chain-of-Custody Procedures

Chain-of-Custody Forms will include, at a minimum, laboratory contact information, client contact information, sample information, and relinquished by/received by information. Sample information will include sample ID, matrix, date/time collected, number and type of containers, preservative information, analysis method, and comments. The Chain-of-Custody Form will also have the sampler's name and signature. The Chain-of-Custody Form will link the location of the sample from the field logbook to the laboratory receipt of the sample. The laboratory will use the sample information to populate the LIMS database for each sample.

26.8 Non-Conformance

The Laboratory PM will contact Contractor Project Chemist to resolve issues encountered during sample receipt and login. Contractor Project Chemist will coordinate with Contractor Sampling Lead and other personnel as necessary to resolve the issues.

WORKSHEET #28: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION

(UFP-QAPP Guidance Manual, Section 3.4 and Tables 4, 5, and 6)

The tables in this worksheet provide information on QC samples, frequency, method or SOP QC acceptance limits, corrective actions, person responsible for the corrective actions, and MPCs for laboratory analyses.

Table B-28-1: TestAmerica – TA-St. Louis

Matrix: Aqueous and Solid						
Analytical Group: VOCs						
Analytical Method/ Method 8260C / ST-MS-0002						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
IS	Every field sample and QC samples	RT within ± 30 seconds from RT of initial calibration midpoint standard; area counts within - 50% to +100% of initial calibration midpoint standard	Correct problem, then re-analyze affected samples	Lab Manager/ Analyst	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than LOQ.	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits

Matrix: Aqueous and Solid						
Analytical Group: VOCs						
Analytical Method/ Method 8260C / ST-MS-0002						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD	One MS/MSD per preparation batch per matrix	Percent Recoveries and RPD per Department of Defense (DoD) QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4	Identify problem; if not related to matrix interference, re-analyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4
LCS/LCS duplicate (LCSD)	One LCS or LCS/LCSD pair per preparation batch per matrix	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4	Correct problem, then re-prepare and re-analyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4
Surrogate spiking	Every field sample and QC sample	%R per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4	Correct problem, then re-analyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4

Matrix: Solid						
Analytical Group: TPH-C₁₀ to C₂₀ and C₂₀ to C₃₄						
Analytical Method/ SOP Reference: Method 8015B / ST-GC-0005						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than LOQ.	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 13 & 14 and Appendix B Table 1	Identify problem; if not related to matrix interference, re-analyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 13 & 14 and Appendix B Table 1
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 13 & 14 and Appendix B Table 1	Correct problem, then re-prepare and re-analyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 13 & 14 and Appendix B Table 1

Matrix: Solid						
Analytical Group: TPH-C₁₀ to C₂₀ and C₂₀ to C₃₄						
Analytical Method/ SOP Reference: Method 8015B / ST-GC-0005						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate spiking	Every field sample and QC sample	%Rs per DoD QSM 5.0 Appendix C Tables 13 & 14 and Appendix B Table 1	Correct problem, then re-analyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Tables 13 & 14 and Appendix B Table 1

Matrix: Aqueous and Solid						
Analytical Group: Explosives						
Analytical Method/ SOP Reference: Method 8330B / ST-LC-0002						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 36 & 37 and Appendix B Table 3	Identify problem; if not related to matrix interference, re-analyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 36 & 37 and Appendix B Table 3
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 36 & 37 and Appendix B Table 3	Correct problem, then re-prepare and re-analyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 36 & 37 and Appendix B Table 3

Matrix: Aqueous and Solid						
Analytical Group: Explosives						
Analytical Method/ SOP Reference: Method 8330B / ST-LC-0002						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate standards	Every field sample and QC sample	%Rs per DoD QSM 5.0 Appendix C Tables 36 & 37 and Appendix B Table 3	Correct problem, then re-analyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Tables 36 & 37 and Appendix B Table 3

Matrix: Aqueous and Solid						
Analytical Group: PCBs						
Analytical Method/ SOP Reference: Method 8082A / ST-GC-0015						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 17 & 18 and Appendix B Table 1	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 17 & 18 and Appendix B Table 1
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 17 & 18 and Appendix B Table 1	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 17 & 18 and Appendix B Table 1

Matrix: Aqueous and Solid						
Analytical Group: PCBs						
Analytical Method/ SOP Reference: Method 8082A / ST-GC-0015						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate spiking	Every field sample and QC sample	%Rs per DoD QSM 5.0 Appendix C Tables 17 & 18 and Appendix B Table 1	Correct problem, then re-analyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Tables 17 & 18 and Appendix B Table 1

Matrix: Soil						
Analytical Group: Metals						
Analytical Method/ SOP Reference: Method 6020A / ST-MT-0001						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than LOQ.	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 4 & 5 and Appendix B Table 9	Identify problem; if not related to matrix interference, re-analyze MS/MSD and all associated batch samples	Lab Manager/Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 4 & 5 and Appendix B Table 9
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 4 & 5 and Appendix B Table 9	Correct problem, then re-prep and re-analyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/Analyst	Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 4 & 5 and Appendix B Table 9

Matrix: Soil						
Analytical Group: Metals						
Analytical Method/ SOP Reference: Method 6020A / ST-MT-0001						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial dilution	Each new sample matrix	1:5 dilution must agree within $\pm 10\%$ of original determination.	Perform post-digestion spike if serial dilution does not meet criteria	TestAmerica - St. Louis Analyst	Accuracy	1:5 dilution must agree within $\pm 10\%$ of original determination.
Post-digestion spike	When serial dilution or matrix spike fails	%Rs within 80-120%	Re-analyze post-digestion spike.	TestAmerica - St. Louis Analyst	Accuracy	Recovery within 80-120%

Matrix: Aqueous and Solid						
Analytical Group: Mercury						
Analytical Method/ SOP Reference: Method 7470A & 7471B / ST-MT-0005 & ST-MT-0007						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than LOQ.	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 11 & 12 and Appendix B Table 7	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 11 & 12 and Appendix B Table 7
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 11 & 12 and Appendix B Table 7	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 11 & 12 and Appendix B Table 7

Matrix: Aqueous and Solid						
Analytical Group: Mercury						
Analytical Method/ SOP Reference: Method 7470A & 7471B / ST-MT-0005 & ST-MT-0007						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial dilution	Each new sample matrix	1:5 dilution must agree within $\pm 10\%$ of original determination.	Perform post-digestion spike if serial dilution does not meet criteria	TestAmerica - St. Louis Analyst	Accuracy	1:5 dilution must agree within $\pm 10\%$ of original determination.
Post-digestion spike	When serial dilution or matrix spike fails	%Rs within 80-120%	Re-analyze post-digestion spike.	TestAmerica - St. Louis Analyst	Accuracy	Recovery within 80-120%

Table 28-2: TestAmerica – TA-St. Louis (Water Characterization Methods)

Matrix: Aqueous and Solid						
Analytical Group: TCLP VOCs						
Analytical Method/ SOP Reference: Method 8260C / ST-MS-0002						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
IS	Every field sample and QC samples	RT within ± 30 seconds from RT of initial calibration midpoint standard; area counts within - 50% to +100% of initial calibration midpoint standard	Correct problem, then re-analyze affected samples	Lab Manager/ Analyst	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than LOQ.	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4	Identify problem; if not related to matrix interference, re-analyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4

Matrix: Aqueous and Solid						
Analytical Group: TCLP VOCs						
Analytical Method/ SOP Reference: Method 8260C / ST-MS-0002						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4
Surrogate standards	Every field sample and QC sample	Percent Recoveries per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4	Correct problem, then re-reanalyze all affected samples	Lab Manager/Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Tables 23 & 24 and Appendix B Table 4

Matrix: Aqueous and Solid						
Analytical Group: SVOCs, PAHs, and TCLP SVOCs						
Analytical Method/ SOP Reference: Method 8270D / ST-MS-0001						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
IS	Every field sample and QC samples	RT within ± 30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard.	Correct problem, then re-analyze affected samples	Lab Manager/ Analyst	Accuracy	Acceptable recoveries per stated QC Acceptance Limits
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than LOQ.	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Table 26 and Appendix B Table 4	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 26 and Appendix B Table 4

Matrix: Aqueous and Solid						
Analytical Group: SVOCs, PAHs, and TCLP SVOCs						
Analytical Method/ SOP Reference: Method 8270D / ST-MS-0001						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Table 26 and Appendix B Table 4	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 26 and Appendix B Table 4
Surrogate standards	Every field sample and QC sample	%Rs per DoD QSM 5.0 Appendix C Table 26 and Appendix B Table 4	Correct problem, then re-reanalyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Table 26 and Appendix B Table 4

Matrix: Aqueous and Solid						
Analytical Group: TCLP Pesticides						
Analytical Method/ SOP Reference: Method 8081B / ST-GC-0016						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1

Matrix: Aqueous and Solid						
Analytical Group: TCLP Pesticides						
Analytical Method/ SOP Reference: Method 8081B / ST-GC-0016						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate standards	Every field sample and QC sample	%Rs per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1	Correct problem, then re-analyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Table 16 and Appendix B Table 1
Second column confirmation of positive results	All positive results must be confirmed	Calibration and QC criteria for second column are the same as for initial and primary column analysis. Results between primary and secondary column RPD $\leq$ 40%.	N/A	Lab Manager/ Analyst	Precision	RPD of results between two columns $\leq$ 40%.

Matrix: Aqueous and Solid						
Analytical Group: TCLP Herbicides						
Analytical Method/ SOP Reference: Method 8151A / ST-GC-0017						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Table 22 and Appendix B Table 1	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 22 and Appendix B Table 1
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Table 22 and Appendix B Table 1	Correct problem, then reprep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 22 and Appendix B Table 1

Matrix: Aqueous and Solid						
Analytical Group: TCLP Herbicides						
Analytical Method/ SOP Reference: Method 8151A / ST-GC-0017						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate standards	Every field sample and QC sample	%Rs per DoD QSM 5.0 Appendix C Table 22 and Appendix B Table 1	Correct problem, then re-analyze all affected samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries per DoD QSM 5.0 Appendix C Table 22 and Appendix B Table 1
Second column confirmation of positive results	All positive results must be confirmed	Calibration and QC criteria for second column are the same as for initial and primary column analysis. Results between primary and secondary column RPD $\leq$ 40%.	N/A	Lab Manager/ Analyst	Precision	RPD of results between two columns $\leq$ 40%.

Matrix: Aqueous and Solid						
Analytical Group: TCLP Metals						
Analytical Method/ SOP Reference: Method 6010C / ST-MT-0003						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than LOQ.	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Table 4 and Appendix B Table 8	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/Analyst	Precision/Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 4 and Appendix B Table 8
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Table 4 and Appendix B Table 8	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/Analyst	Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 4 and Appendix B Table 8

Matrix: Aqueous and Solid						
Analytical Group: TCLP Metals						
Analytical Method/ SOP Reference: Method 6010C / ST-MT-0003						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial dilution	Each new sample matrix	1:5 dilution must agree within $\pm 10\%$ of original determination.	Perform post-digestion spike if serial dilution does not meet criteria	TestAmerica - St. Louis Analyst	Accuracy	1:5 dilution must agree within $\pm 10\%$ of original determination.
Post-digestion spike	When serial dilution or matrix spike fails	%Rs within 80-120%	Re-analyze post-digestion spike.	TestAmerica - St. Louis Analyst	Accuracy	Recovery within 80-120%

Matrix: Aqueous and Solid						
Analytical Group: TCLP Mercury						
Analytical Method/ SOP Reference: Method 7470A / ST-MT-0005						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
MS/MSD	One MS/MSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Table 12 and Appendix B Table 7	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 12 and Appendix B Table 7
LCS/LCSD	One LCS or LCS/LCSD pair per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Table 12 and Appendix B Table 7	Correct problem, then re-prep and re-reanalyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Accuracy	Acceptable Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Table 12 and Appendix B Table 7

Matrix: Aqueous and Solid						
Analytical Group: TCLP Mercury						
Analytical Method/ SOP Reference: Method 7470A / ST-MT-0005						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial dilution	Each new sample matrix	1:5 dilution must agree within $\pm 10\%$ of original determination.	Perform post-digestion spike if serial dilution does not meet criteria	TestAmerica - St. Louis Analyst	Accuracy	1:5 dilution must agree within $\pm 10\%$ of original determination.

Matrix: Aqueous and Solid						
Analytical Group: TCLP Cyanide						
Analytical Method/ SOP Reference: Method 9012B / ST-WC-0002						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results per stated QC Acceptance Limits
LCS	One LCS per preparation batch per matrix	%Rs and RPD per DoD QSM 5.0 Appendix C Tables 38 & 39 and Appendix B Table 11	Correct problem, then re-prep and re-analyze the LCS and all associated batch samples for failed analytes, if sufficient sample volume is available and samples are within 2x the hold time. Qualify data accordingly if reprep & re-analysis cannot be performed or if reprep & reanalysis also has failed analytes	Lab Manager/ Analyst	Accuracy	Percent Recoveries and RPD per DoD QSM 5.0 Appendix C Tables 38 & 39 and Appendix B Table 11
MS	One MS per preparation batch per matrix	%Rs per DoD QSM 5.0 Appendix C Tables 38& 39 and Appendix B Table 11	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recovery per DoD QSM 5.0 Appendix C Tables 38& 39 and Appendix B Table 11

Matrix: Aqueous and Solid						
Analytical Group: TCLP Cyanide						
Analytical Method/ SOP Reference: Method 9012B / ST-WC-0002						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Duplicate	One DUP per preparation batch per matrix	RPD per DoD QSM 5.0 Appendix B Table 11	Correct problem, then re-analyze the DUP and all associated batch samples	TestAmerica - St. Louis Analyst	Precision	RPD per DoD QSM 5.0 Appendix B Table 11

Matrix: Aqueous and Solid						
Analytical Group: Total Sulfide						
Analytical Method/ SOP Reference: Method 9034 / ST-WC-0012						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch	No target analytes detected greater than one-half LOQ or 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re-analyze method blank and all samples processed with the contaminated blank	Lab Manager/ Analyst	Representativeness	Acceptable results
LCS	One LCS per preparation batch per matrix	%Rs: 70-130% (lab-derived statistical limits)	Correct problem, then re-analyze the LCS and all associated batch samples	Lab Manager/ Analyst	Accuracy	Percent Recoveries: 70-130% (lab-derived statistical limits)
MS	One MS per preparation batch per matrix	%Rs: 70-130% (lab-derived statistical limits)	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab Manager/ Analyst	Precision/Accuracy	Percent Recoveries: 70-130% (lab-derived statistical limits)
Duplicate	One DUP per preparation batch per matrix	RPD Limit: 20% (lab-derived statistical limits)	Correct problem, then re-analyze the DUP and all associated batch samples	TestAmerica - St. Louis Analyst	Precision	RPD Limit: 20% (lab-derived statistical limits)

Matrix: Aqueous and Solid						
Analytical Group: pH						
Analytical Method/ SOP Reference: Method 9040C & 9045D / ST-WC-0011						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Duplicate	One per prep batch	+/- 0.1 unit between the parent sample and lab duplicate	Correct problem, then re-analyze the DUP and all associated batch samples	TestAmerica - St. Louis Analyst	Precision	+/- 0.1 unit between the parent sample and lab duplicate
pH Buffer Ranges	Before beginning a sample run, at 4.0, 7.0, and 10.0 standard unit	Within +/- 0.05 unit of the standard	Correct problem, then recalibrate with the 3 different pH buffer standard solution and all samples	TestAmerica - St. Louis Analyst	Accuracy	Within +/- 0.05 unit of the standard

Matrix: Aqueous and Solid						
Analytical Group: Ignitability						
Analytical Method/ SOP Reference: Method 1010A / ST-WC-0026						
QC Sample	Frequency / Number	QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One LCS per preparation batch per matrix	99% Xylene is used as the LCS which has a known flash point of 27°C. Control limits are 24.3°C – 29.7°C.	Correct problem, then re-analyze the LCS and all associated batch samples. If still fails, order another batch of Xylene from vendor	Lab Manager/ Analyst	Accuracy	24.3°C – 29.7°C.
Duplicate	One per prep batch	RPD Limit: 20% (lab-derived limits)	Correct problem, then re-analyze the DUP and all associated batch samples	TestAmerica - St. Louis Analyst	Precision	RPD Limit: 20% (lab-derived limits)

WORKSHEET #29: PROJECT DOCUMENTS AND RECORDS

(UFP-QAPP Guidance Manual, Section 3.5.1)

This work sheet details information for all documents and records that will be generated for the project. It describes how information will be collected, verified, and stored. It supports data completeness, data integrity, and ease of retrieval.

Table B-29-1: Project Documents and Records

Document/Record	Purpose	Related Definable Feature of Work	Primary Generator (1)	Completion/ Update Frequency
Field Manager's Logbook	Record all important events	All	Site Manager	Daily
Team Leader Logbook(s)	Record important team-specific events	All	Team Leader(s)	Daily
Root Cause Analysis	Document equipment failures and causes, as well as corrective actions taken, actions taken to prevent recurrence, and actions taken to monitor effectiveness of corrective action	All	Site Manager	If equipment failures are noted
Photograph Log	Documents all photographs taken to document work and/or site conditions, and to record items of interest observed	All	Field Team	As needed

(1) The primary generator may designate another qualified individual to prepare the document or record; however, the primary generator is responsible for ensuring the quality and accuracy of that document/record and for providing the preparer's signature, when appropriate.

Table 29-2 and Table 29-3 list the data quality-related documentation prepared to support this project.

Table B-29-2: Project Sample Collection and Field Records

Record	Generation	Verification	Storage Location/Archival
Field logbook or data collection sheets	Parsons Sampling Lead	Parsons Sample Team Member	Project File/ Contractor Office
CoC Forms	Parsons Sample Team Member	Parsons Sampling Lead	Project File/ Contractor Office
Air Bills	Parsons Sample Team Member	Parsons Sampling Lead	Project File/ Contractor Office
Contractor Daily QC Reports	Parsons Sampling Lead	Parsons Sample Team Member	Project File/ Contractor Office
Deviations	Parsons Sampling Lead	Parsons Sample Team Member	Project File/ Contractor Office
Corrective Action Reports	Parsons Sampling Lead	Parsons Sample Team Member	Project File/ Contractor Office

Table B-29-3: Laboratory Records

Record	Generation	Verification	Storage Location/Archival
Sample Log-in	Sample Receiving	Laboratory PM	LIMS
Instrument Print-Out and Raw Data	Analyst	Analyst and peer reviewer	LIMS
Review Checklists (Analyst)	Analyst	Analyst and peer reviewer	LIMS
Laboratory Report/ Data Package	Laboratory PM	Peer reviewer/QA Manager	LIMS
PM Review Checklists	Laboratory PM	Peer reviewer/QA Manager	LIMS
Data Validation Report	Data Validator or Project Chemist	Project Chemist or PM	Project File/ Contractor Office

WORKSHEET #31, 32, & 33: ASSESSMENTS AND CORRECTIVE ACTION

(UFP-QAPP Guidance Manual, Section 4.1.1)

This worksheet provides information on the required periodic assessments that will be performed during the course of the project to ensure that the planned project activities are implemented in accordance with this UFP-QAPP.

The table below summarizes the type, frequency, and responsible parties of planned assessment activities to be performed for the project.

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing Corrective Actions	Person(s) Responsible for Monitoring Effectiveness of Corrective Actions
Quality Assessment Audit	Once during field event	Internal	Parsons	PM or designee	PM	PM	Field Team Leader

Based on the findings of the project assessments, corrective action may be required. For assessment findings that require corrective action, deficiencies will be documented and communicated to the appropriate project personnel. Corrective action will then be implemented and a follow- up assessment will be performed to verify the results of the corrective action. Procedures for handling UFP-QAPP deviations during each type of assessment are summarized in the table below.

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings	Time Frame of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Time Frame for Response
Quality Assessment Audit	Internal memo	PM	3-5 business days	Letter or memo	PM	3 to 5 business days

In order to demonstrate that PQOs (Worksheet #11) have been achieved, periodic QA management reports will be prepared to ensure that the project stakeholders are updated on project status and the results of QA assessments. The frequency and type of planned QA management reports, the delivery date, the personnel responsible for report preparation, and the report recipients are identified below.

Type of Report	Frequency	Projected Delivery Date(s)	Primary Person(s) Responsible for Report Preparation	Report Recipient(s)
Final Report	Once after data are generated and validated	TBD	Parsons	Recipients listed in the Distribution Memo (see Worksheet #3)

WORKSHEET #34: DATA VERIFICATION & VALIDATION INPUTS

(UFP-QAPP Guidance Manual, Section 5.2.1)

This worksheet lists the inputs that will be used during data verification and validation. Inputs include the planning documents, field records, and laboratory records that are listed on [Worksheet #29](#). Data verification is a check that all specified activities involved in collecting and analyzing samples have been completed and documented and that the necessary records (objective evidence) are available to proceed to data validation. Data validation is the evaluation of conformance to stated requirements, including those in the contract, methods, SOPs and the QAPP. Data validation includes evaluation of the data against the project-specific MPCs ([Worksheet #12](#)). Data verification and validation procedures and responsibilities are described on Worksheet #35 and Worksheet #36, respectively. Once verification and validation have been completed, a usability assessment is conducted to evaluate whether process execution and resulting data meet DQOs. Usability assessment procedures are described on Worksheet #37. The data verification, validation, and usability assessment process is summarized in **Figure 34.1**.

Figure B-34-1: Data Verification, Validation, and Usability Assessment Process

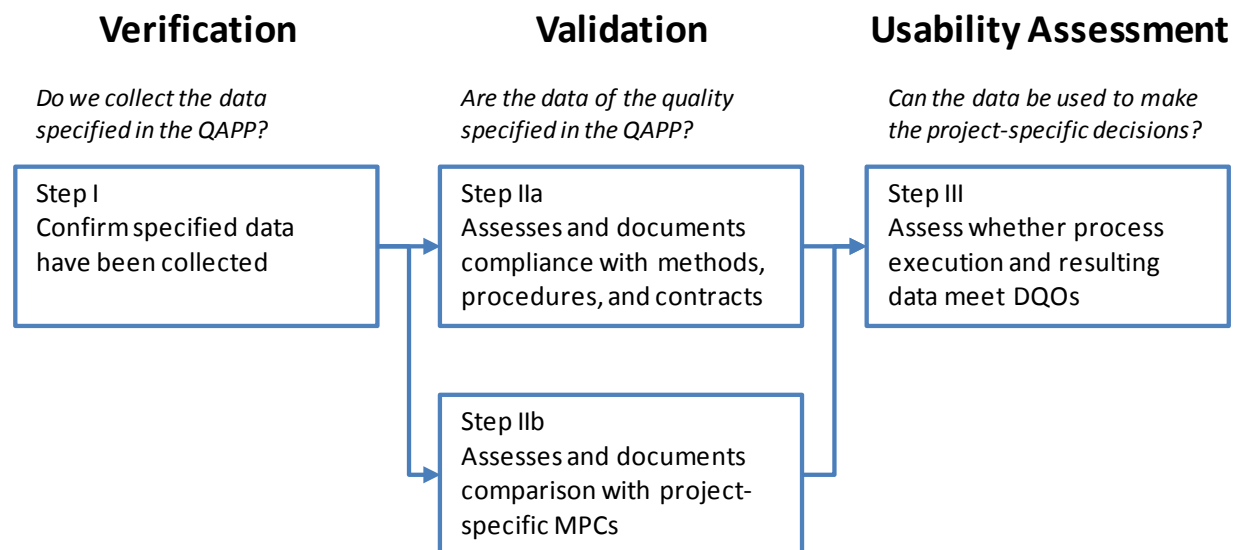


**Table B-34-1:
Verification and Validation Inputs**

Description	Verification (completeness)	Validation (conformance to specifications)
Field Records		
Field logbooks	X	
Chain-of-Custody (CoC) Forms	X	
Change orders/deviations, when applicable	X	
Field corrective action reports, when applicable	X	
Analytical Data Package		
Cover sheet (laboratory identifying information)	X	X
Case narrative	X	X
CoC and shipping forms	X	X
Sample receipt records	X	X
Sample chronology (i.e. dates/times of receipt, preparation, & analysis)		X
detection limit/limit of detection/limit of quantitation (DL/LOD/LOQ) establishment and verification	X	X
Detection and Quantitation Limits		X
Standards Traceability		X
Instrument calibration records		X
Definition of laboratory qualifiers	X	X
Analytical sample results	X	X
QC sample results	X	X
Raw data including manual integrations when applicable (1)		X
Electronic data deliverable		X
Corrective action reports, when applicable	X	X

(1) When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., the chromatograms obtained before and after the manual integration must be retained). This requirement applies to all analytical runs including calibration standards and QC samples. The person performing the manual integration must sign and date each manually integrated chromatogram and record the rationale for performing the manual integration.

WORKSHEET #35: DATA VERIFICATION PROCEDURES

(UFP-QAPP Guidance Manual, Section 5.2.2)

“Verification” is a completeness check that is performed before the data review process is conducted to determine whether the required information is available for validation. It involves a review of all data inputs to ensure that they are present. This step of the data review process answers whether or not the required data inputs are present.

The verification process for analytical related tasks is discussed in SOP# CHEM-01. The SOP lists and describes the analytical data-related verification procedures. These verification procedures are documented and included as part of the validation process.

The following section summarizes the verification procedures for field related tasks.

Table B-35-1: Verification Procedures for Field-Related Tasks

Verification Input	Description of Verification Process	Internal/ External	Responsible for Verification
Site Manager Logbook	Confirm logbooks on file cover entire duration of field effort	Internal	Site Manager
Daily Status Reports	Confirm Daily Status Reports on file cover entire duration of field effort	Internal	Site Manager
Daily QC Report	Verify Daily QC Reports on file cover entire duration of field effort	Internal	Site Manager
Team Leader Logbook(s)	Confirm logbooks on file cover entire duration of field effort	Internal	Site Manager
Field Change Request Form	Confirm that a Field Change Request on file for each QAPP change due to minor field-based QAPP revisions	Internal	Site Manager
Photograph Log	Verify photographs are listed on log and that it spans the duration of the project; verify that all photographs are on file	Internal	Site Manager
Daily Instrument Test Report	Verify that all magnetometer/metal detector test data are on file spanning the duration of the project	Internal	Site Manager
SOP Checklists	Confirm all SOP checklists are on file and cover entire duration of field work	Internal	Site Manager

Table B-35-2: Verification Inputs for Analytical Data-Related Tasks

Verification Input	Description of Verification Process	Internal/ External	Primary Person(s) Responsible
Cover Sheet	Confirm the cover sheet includes laboratory identifying information, contact information, assigned sample delivery group number, date issued, project name, and laboratory approval signature.	External and Internal	Laboratory PM or QA Manager Parsons Project Chemist
Case Narrative	Confirm the case narrative documents all non-conformance issues related to the samples and analytical data contained in the report.	External and Internal	Laboratory PM Parsons Project Chemist
CoC and Shipping Forms	CoC forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. A copy of the CoC form will be retained in the site file, and the original and remaining copies taped inside the cooler for shipment.	Internal	Site Manager Parsons Project Chemist
Sample Condition upon Receipt	Any discrepancies, missing, or broken containers will be communicated to the project chemist or designee along with the laboratory login information.	External	Laboratory Sample Receipt Staff
LOD/LOQ Verification	LOD and/or LOQ quarterly verifications must be provided for all required methods, matrices, and analytes. This information may be provided in the laboratory data report or as a separate deliverable.	External and Internal	Laboratory PM, Project Chemist or designee
Definition of Laboratory Qualifiers	Definitions of all laboratory applied data qualifiers must be included in the data package.	External and Internal	Laboratory PM, Project Chemist or designee
Analytical Sample Results	Review analytical sample results for completeness against the requested parameters listed on the CoC form.	External and Internal	Laboratory PM, Project Chemist or designee
QC Sample Results	Ensure all QC associated with sample analysis is included. (Laboratory should NOT include MS/MSD data associated with non-project samples.)	External and Internal	Laboratory PM, Project Chemist or designee
Corrective Action Reports	Corrective Action reports will be reviewed by the project chemist or PM and placed into the project file for archival at project closeout.	Internal	Parsons PM, Project Chemist

WORKSHEET #36: DATA VALIDATION PROCEDURES

(UFP-QAPP Guidance Manual, Section 5.2.2)

“Validation” is performed to identify and qualify data that do not meet the MPCs specified on [Worksheet #12](#). Data requiring validation are summarized on [Worksheet #34](#). The information in these tables shows what data inputs are required for data validation as well as the processes used to conduct the validation.

General procedures for chemistry data review and management are described in **SOP: CHEM-01, Chemistry Data Management**. Project specific data validation elements for this project are summarized in **Tables B-36-1 and B-36-2** below.

Table B-36-1: Overview of Analytical Data Validation for All Analytes

Data Validators:	Contractor Project Chemist and designee(s)
Analytical Group/Method:	All Chemical Analyses
Data deliverable requirements:	Level IV data packages and Electronic Data Deliverables (EDDs) (Superfund Electronic Data Deliverable Automated Data Review [SEDD ADR.net] format)
Analytical specifications:	Per UFP-QAPP and DoD QSM version 5.0
Measurement performance criteria:	Per UFP-QAPP (Worksheet #12)
Percent of data packages to be validated:	100%
Percent of raw data reviewed:	100%
Percent of results to be recalculated:	10%
Validation procedure:	Level 4 Validation (as described in SOP CHEM-01)
Data validation codes:	See Table B-36-2 below
Electronic validation program/version:	SEDD 2a/ADR.net

Table B-36-2: Data Validation Qualifiers and Definitions

Data Validation Codes	Definitions
U	Analyte was not detected and is reported as less than the LOD. The LOD has been adjusted for any dilution or concentration of the sample.
B	Blank contamination. The recorded result (<5x lab non-common contaminants or <10x lab common contaminants) is associated with a contaminated blank.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	The analyte was not detected above the reported sample LOQ. However, the reported LOQ is approximate and may or may not represent the actual LOQ necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Note: Analytical laboratories will report all concentrations down to the DL and flag any results between the DL and LOQ with “J”. All non-detected results will be reported as <LOD, per DoD QSM version 5.0.

WORKSHEET #37: USABILITY ASSESSMENT

(UFP-QAPP Guidance Manual, Section 5.2.3)

The data usability assessment is an evaluation based on the results of data verification and validation in the context of the overall project decisions or objectives. The assessment determines whether the project execution and resulting data meet the project DQOs (see [Worksheet #11](#)). All types of data (e.g., DGM, intrusive, sampling, analytical, etc.) will be considered with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data. The following sections summarize the processes to determine whether the collected data are of the right type, quality, and quantity to support the environmental decision-making for the project, and describes how data quality issues will be addressed and how limitations of the use of the data will be handled.

37.1 Usability Assessment for Analytical Data

37.1.1 Summary of Usability Assessment Processes

The data usability assessment for analytical data is performed at the conclusion of data collection activities, using the outputs from data verification and data validation. This data interpretation phase involves a qualitative and quantitative evaluation of environmental data to determine if the data are of the right type, quality, and quantity to support the decisions that need to be made. It involves a retrospective evaluation of the systematic planning process, and, like the systematic planning process, involves participation by key members of the project team. The data usability assessment evaluates whether underlying assumptions used during systematic planning are supported, sources of uncertainty have been accounted for and are acceptable, data are representative of the population of interest, and the results can be used as intended, with the acceptable level of confidence.

The Contractor's Project Chemist or designee will evaluate the sensitivity of the data collected to determine whether the required DLs, LODs, and LOQs were met. Data will be further evaluated to determine whether the reported DL, LOD, and LOQ are sufficient to meet the associated project action limits. Sensitivity of target compounds may be affected by matrix interference, high levels of target or non-target analytes, sample volume available for collection, etc. If the sensitivity requirements are not met for a particular analyte, the Contractor Team will evaluate whether the data can still be used for project decisions. If non-detect values exceed the PAL, data is considered usable if the analyte is a site-related chemical. Analytes that are not site-related chemicals are not expected to be found, and thus do not impact decision making. For analytes that are site-related, the Contractor team will use a "weight of evidence" approach to evaluate whether the chemical could have been present. This approach uses available data that does meet sensitivity requirements to evaluate the presence or absence of other similar compounds and/or degradation products for the analyte in question. If sufficient evidence exists to determine a compound was not present due to the absence of similar compounds and breakdown products, the Contractor Team can deduce that the analyte was not present, and thus data is usable for decision making.

Data validation will be the first step of the usability assessment. See Worksheet #28 for data quality indicators associated with the analytical measurements to be used on the project. All data qualifiers will be evaluated and any possible impact to the overall data quality will be discussed in the Data Usability Assessment Report. Any data gap due to the field and/or lab error will be pointed out in the report.

37.1.2 Documentation Generated

A data validation report will be created for each SDG including a summary of all QC results associated with the SDG to provide documentation whether data generated were in control throughout sample analysis. Topics of discussion include Accuracy/Biased Contamination, Precision of field duplicate and/or lab duplicate, Sensitivity, Representativeness, Completeness and Comparability of the data. Any deviations from planned procedures and analysis and the impact on the project objectives will be discussed, along with data usability and any data limitations. Criteria listed in the [Worksheet #12](#) will be examined to determine if the Measurement Performance Criteria were met. Any lab trending in the QC samples, such as high biased lab control sample for a particular compound or metal will be discussed. Data summary tables will be generated in order for the data reviewer to review the results in an organized manner.

37.1.3 Procedures to Assess Project-Specific Overall Measurement Error

Parsons will determine if quality control data is within specifications through the data assessment and data validation process ([Worksheet #36](#)).

37.1.4 Personnel Responsible for Performing Usability Assessment

The following personnel are responsible for performing usability assessments:

- o Contractor Project Manager
- o Contractor Project Chemist
- o Contractor Sampling Lead

37.1.5 Impacts of Qualified Data and Plan Deviations

Parsons will use all data not rejected during validation to determine the nature and extent of contamination, and to support the risk assessment. Parsons will work with USACE and project regulators if there is a concern about the statistical validity of the sample results or to determine if sample locations with rejected data need to be re-sampled.

37.1.6 Usability Assessment Documentation

All analytical results will be reported for an overall quality assessment in the Data Validation Report. The Data Validation Report will identify precision and accuracy exceedances with respect to the laboratory performance for each batch of samples, as well as comparability of field and laboratory duplicates. Data tables will be produced to reflect detected and non-detected analytes for each test. Data qualifiers will be provided in the data tables and discussed in the Data Validation Report.

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**ATTACHMENT B-1 LAB SOPS AND CERTIFICATIONS
(ELECTRONIC ONLY)**

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**LABORATORY
ACCREDITATION
BUREAU** a division of A-S-B



Certificate of Accreditation

ISO/IEC 17025:2005

Certificate Number L2468

TestAmerica Sacramento

880 Riverside Parkway
West Sacramento CA 95605

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation valid through: January 20, 2018

R. Douglas Leonard, Jr., President, COO
Laboratory Accreditation Bureau
Presented the 20th of January 2017

*See the laboratory's Scope of Accreditation for details of accredited parameters

**Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).
Form 403.14 - Rev 1 7/3/13

Scope of Accreditation For TestAmerica Sacramento

880 Riverside Parkway
West Sacramento, CA 95605
Ms. Lisa Stafford
916-373-5600

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (LABPR 403 DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM V5) based on the TNI Standard - Environmental Laboratory Sector, Volume 1 – Management and Technical Requirements for Laboratories Performing Environmental Analysis, Sept 2009 (EL-V1-2009); accreditation is granted to **TestAmerica Sacramento** to perform the following tests:

Accreditation granted through: **January 20, 2018**

Testing - Environmental

Non-Potable Water		
Technology	Method	Analyte
ICP-AES	EPA 6010B/6010C	Aluminum
ICP-AES	EPA 6010B/6010C	Antimony
ICP-AES	EPA 6010B/6010C	Arsenic
ICP-AES	EPA 6010B/6010C	Barium
ICP-AES	EPA 6010B/6010C	Beryllium
ICP-AES	EPA 6010B/6010C	Boron
ICP-AES	EPA 6010B/6010C	Cadmium
ICP-AES	EPA 6010B/6010C	Calcium
ICP-AES	EPA 6010B/6010C	Chromium (Total)
ICP-AES	EPA 6010B/6010C	Cobalt
ICP-AES	EPA 6010B/6010C	Copper
ICP-AES	EPA 6010B/6010C	Iron
ICP-AES	EPA 6010B/6010C	Lead
ICP-AES	EPA 6010B/6010C	Magnesium
ICP-AES	EPA 6010B/6010C	Manganese
ICP-AES	EPA 6010B/6010C	Molybdenum
ICP-AES	EPA 6010B/6010C	Nickel
ICP-AES	EPA 6010B/6010C	Potassium



Non-Potable Water		
Technology	Method	Analyte
ICP-AES	EPA 6010B/6010C	Selenium
ICP-AES	EPA 6010B/6010C	Silica
ICP-AES	EPA 6010B/6010C	Silicon
ICP-AES	EPA 6010B/6010C	Silver
ICP-AES	EPA 6010B/6010C	Sodium
ICP-AES	EPA 6010B/6010C	Thallium
ICP-AES	EPA 6010B/6010C	Tin
ICP-AES	EPA 6010B/6010C	Titanium
ICP-AES	EPA 6010B/6010C	Vanadium
ICP-AES	EPA 6010B/6010C	Zinc
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel
ICP-MS	EPA 6020/6020A	Phosphorus
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium
ICP-MS	EPA 6020/6020A	Strontium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Tin
ICP-MS	EPA 6020/6020A	Titanium
ICP-MS	EPA 6020/6020A	Uranium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
CVAAS	EPA 7470A	Mercury

Non-Potable Water		
Technology	Method	Analyte
Colorimetric	EPA 353.2	Nitrate
Colorimetric	EPA 353.2	Nitrate-nitrite
Colorimetric	EPA 353.2	Nitrite
Colorimetric	EPA 410.4	Chemical Oxygen Demand (COD)
LC/MS/MS	EPA 6850	Perchlorate
Colorimetric	EPA 7196A	Chromium (Hexavalent)
Probe	EPA 9040B/9040C	pH
Ion Chromatography	EPA 9056A/300.0	Bromide
Ion Chromatography	EPA 9056A/300.0	Chloride
Ion Chromatography	EPA 9056A/300.0	Fluoride
Ion Chromatography	EPA 9056A/300.0	Nitrate
Ion Chromatography	EPA 9056A/300.0	Nitrite
Ion Chromatography	EPA 9056A/300.0	Orthophosphate
Ion Chromatography	EPA 9056A/300.0	Sulfate
Titration	SM 2320B	Alkalinity
Gravimetric	SM 2540B	Solids, Total
Gravimetric	SM 2540C	Solids, Total Dissolved
Gravimetric	SM 2540D	Solids, Total Suspended
Colorimetric/Hydrolysis	EPA 353.2 Modified / WS-WC-0050	Nitrocellulose
GC/MS	EPA 8260B/8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/8260C	1,2-Dibromoethane
GC/MS	EPA 8260B/8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,3-Dichloropropane

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B/8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1-Chlorohexane
GC/MS	EPA 8260B/8260C	2,2-Dichloropropane
GC/MS	EPA 8260B/8260C	2-Butanone (MEK)
GC/MS	EPA 8260B/8260C	2-Chlorotoluene
GC/MS	EPA 8260B/8260C	2-Hexanone (MBK)
GC/MS	EPA 8260B/8260C	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA 8260B/8260C	4-Chlorotoluene
GC/MS	EPA 8260B/8260C	4-Isopropyltoluene
GC/MS	EPA 8260B/8260C	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 8260B/8260C	Acetone
GC/MS	EPA 8260B/8260C	Allyl Chloride
GC/MS	EPA 8260B/8260C	Benzene
GC/MS	EPA 8260B/8260C	Bromobenzene
GC/MS	EPA 8260B/8260C	Bromochloromethane
GC/MS	EPA 8260B/8260C	Bromodichloromethane
GC/MS	EPA 8260B/8260C	Bromoform
GC/MS	EPA 8260B/8260C	Bromomethane
GC/MS	EPA 8260B/8260C	Carbon Disulfide
GC/MS	EPA 8260B/8260C	Carbon Tetrachloride
GC/MS	EPA 8260B/8260C	Chlorobenzene
GC/MS	EPA 8260B/8260C	Chloroethane
GC/MS	EPA 8260B/8260C	Chloroform
GC/MS	EPA 8260B/8260C	Chloromethane
GC/MS	EPA 8260B/8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Cyclohexane
GC/MS	EPA 8260B/8260C	Dibromochloromethane
GC/MS	EPA 8260B/8260C	Dibromomethane
GC/MS	EPA 8260B/8260C	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C	Diisopropyl Ether (DIPE)
GC/MS	EPA 8260B/8260C	Ethylbenzene
GC/MS	EPA 8260B/8260C	Ethylmethacrylate
GC/MS	EPA 8260B/8260C	Ethyl tert-butyl Ether (ETBE)
GC/MS	EPA 8260B/8260C	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C	Hexane
GC/MS	EPA 8260B/8260C	Iodomethane
GC/MS	EPA 8260B/8260C	Isobutanol (2-Methyl-1-propanol)
GC/MS	EPA 8260B/8260C	Isopropylbenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B/8260C	m & p Xylene
GC/MS	EPA 8260B/8260C	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA 8260B/8260C	Methylene Chloride
GC/MS	EPA 8260B/8260C	Naphthalene
GC/MS	EPA 8260B/8260C	n-Butylbenzene
GC/MS	EPA 8260B/8260C	n-Propylbenzene
GC/MS	EPA 8260B/8260C	o-Xylene
GC/MS	EPA 8260B/8260C	sec-Butylbenzene
GC/MS	EPA 8260B/8260C	Styrene
GC/MS	EPA 8260B/8260C	t-Amyl methyl Ether (TAME)
GC/MS	EPA 8260B/8260C	t-1,4-Dichloro-2-Butene
GC/MS	EPA 8260B/8260C	tert-Butylbenzene
GC/MS	EPA 8260B/8260C	Tetrachloroethene
GC/MS	EPA 8260B/8260C	Toluene
GC/MS	EPA 8260B/8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Trichloroethene
GC/MS	EPA 8260B/8260C	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C	Vinyl Acetate
GC/MS	EPA 8260B/8260C	Vinyl Chloride
GC/MS	EPA 8260B/8260C	Xylenes, Total
GC/MS	EPA 8260B/AK101MS	Gasoline (GRO)
GC/MS	EPA 8270C/8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270C/8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270C/8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dimethylphenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrophenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2,6-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,6-Dinitrotoluene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/8270D	2-Chloronaphthalene
GC/MS	EPA 8270C/8270D	2-Chlorophenol
GC/MS	EPA 8270C/8270D	2-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2-Methylphenol
GC/MS	EPA 8270C/8270D	2-Nitroaniline
GC/MS	EPA 8270C/8270D	2-Nitrophenol
GC/MS	EPA 8270C/8270D	3&4-Methylphenol
GC/MS	EPA 8270C/8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/8270D	3-Nitroaniline
GC/MS	EPA 8270C/8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/8270D	4-Chloroaniline
GC/MS	EPA 8270C/8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Nitroaniline
GC/MS	EPA 8270C/8270D	4-Nitrophenol
GC/MS	EPA 8270C/8270D	Acenaphthene
GC/MS	EPA 8270C/8270D	Acenaphthylene
GC/MS	EPA 8270C/8270D	Aniline
GC/MS	EPA 8270C/8270D	Anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)pyrene
GC/MS	EPA 8270C/8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/8270D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/8270D	Benzoic Acid
GC/MS	EPA 8270C/8270D	Benzyl Alcohol
GC/MS	EPA 8270C/8270D	Benzyl butyl Phthalate
GC/MS	EPA 8270C/8270D	Biphenyl
GC/MS	EPA 8270C/8270D	Bis(2-chloroethoxy) Methane
GC/MS	EPA 8270C/8270D	Bis(2-chloroethyl) Ether
GC/MS	EPA 8270C/8270D	Bis(2-chloroisopropyl) Ether
GC/MS	EPA 8270C/8270D	Carbazole
GC/MS	EPA 8270C/8270D	Chrysene
GC/MS	EPA 8270C/8270D	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA 8270C/8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/8270D	Dibenzofuran
GC/MS	EPA 8270C/8270D	Diethyl Phthalate
GC/MS	EPA 8270C/8270D	Dimethyl Phthalate

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/8270D	Di-n-butyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-octyl Phthalate
GC/MS	EPA 8270C/8270D	Fluoranthene
GC/MS	EPA 8270C/8270D	Fluorene
GC/MS	EPA 8270C/8270D	Hexachlorobenzene
GC/MS	EPA 8270C/8270D	Hexachlorobutadiene
GC/MS	EPA 8270C/8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/8270D	Hexachloroethane
GC/MS	EPA 8270C/8270D	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA 8270C/8270D	Isophorone
GC/MS	EPA 8270C/8270D	Naphthalene
GC/MS	EPA 8270C/8270D	Nitrobenzene
GC/MS	EPA 8270C/8270D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/8270D	Pentachlorophenol
GC/MS	EPA 8270C/8270D	Phenanthrene
GC/MS	EPA 8270C/8270D	Phenol
GC/MS	EPA 8270C/8270D	Pyrene
GC/MS	EPA 8270C/8270D	Pyridine
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	1-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(b)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(g,h,i)perylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(k)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Chrysene

Non-Potable Water		
Technology	Method	Analyte
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluorene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Naphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Phenanthrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Pyrene
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0011	1,4-Dioxane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Dithiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Benzothiazole
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfoxide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Chloropicrin
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Acetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	2-Chloroacetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Oxathiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Dimethyl Disulfide
GC-IT/MS	EPA 521 Modified / WS-MS-0012	N-Nitrosodimethyl amine (NDMA)
GC-FID	EPA 8015B/8015C/8015D AK102	Diesel Range Organics (DRO)
GC-FID	AK103	Residual Range Organics
GC-FID	EPA 8015B/8015C/8015D	Motor Oil Range Organics (MRO)
GC-ECD	EPA 8081A/8081B	Aldrin
GC-ECD	EPA 8081A/8081B	a-BHC
GC-ECD	EPA 8081A/8081B	b-BHC

Non-Potable Water		
Technology	Method	Analyte
GC-ECD	EPA 8081A/8081B	d-BHC
GC-ECD	EPA 8081A/8081B	g-BHC (Lindane)
GC-ECD	EPA 8081A/8081B	a-Chlordane
GC-ECD	EPA 8081A/8081B	g-Chlordane
GC-ECD	EPA 8081A/8081B	4,4'-DDD
GC-ECD	EPA 8081A/8081B	4,4'-DDE
GC-ECD	EPA 8081A/8081B	4,4'-DDT
GC-ECD	EPA 8081A/8081B	Dieldrin
GC-ECD	EPA 8081A/8081B	Endosulfan I
GC-ECD	EPA 8081A/8081B	Endosulfan II
GC-ECD	EPA 8081A/8081B	Endosulfan sulfate
GC-ECD	EPA 8081A/8081B	Endrin
GC-ECD	EPA 8081A/8081B	Endrin Aldehyde
GC-ECD	EPA 8081A/8081B	Endrin Ketone
GC-ECD	EPA 8081A/8081B	Heptachlor
GC-ECD	EPA 8081A/8081B	Heptachlor Epoxide
GC-ECD	EPA 8081A/8081B	Methoxychlor
GC-ECD	EPA 8081A/8081B	Toxaphene
GC-ECD	EPA 8081A/8081B	Chlordane (technical)
GC-ECD	EPA 8082/8082A	PCB-1016
GC-ECD	EPA 8082/8082A	PCB-1221
GC-ECD	EPA 8082/8082A	PCB-1232
GC-ECD	EPA 8082/8082A	PCB-1242
GC-ECD	EPA 8082/8082A	PCB-1248
GC-ECD	EPA 8082/8082A	PCB-1254
GC-ECD	EPA 8082/8082A	PCB-1260
GC-ECD	EPA 8082/8082A	PCB-1262
GC-ECD	EPA 8082/8082A	PCB-1268
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDD
GC/MS	EPA 8280A/8280B	OCDD
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDF
GC/MS	EPA 8280A/8280B	2,3,4,7,8-PeCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDF

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDF
GC/MS	EPA 8280A/8280B	2,3,4,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8,9-HpCDF
GC/MS	EPA 8280A/8280B	OCDF
GC/MS	EPA 8280A/8280B	Total TCDD
GC/MS	EPA 8280A/8280B	Total PeCDD
GC/MS	EPA 8280A/8280B	Total HxCDD
GC/MS	EPA 8280A/8280B	Total HeptaCDD
GC/MS	EPA 8280A/8280B	Total TCDF
GC/MS	EPA 8280A/8280B	Total PeCDF
GC/MS	EPA 8280A/8280B	Total HxCDF
GC/MS	EPA 8280A/8280B	Total HpCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,7,8-TeCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8-PeCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,6,7,8-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8,9-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,6,7,8-HpCDD
GC/HRMS	EPA 8290/8290A/1613B	OCDD
GC/HRMS	EPA 8290/8290A/1613B	2,3,7,8-TeCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8-PeCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,4,7,8-PeCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,6,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8,9-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,4,6,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,6,7,8-HpCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8,9-HpCDF
GC/HRMS	EPA 8290/8290A/1613B	OCDF
GC/HRMS	EPA 8290/8290A/1613B	Total TCDD
GC/HRMS	EPA 8290/8290A/1613B	Total PeCDD
GC/HRMS	EPA 8290/8290A/1613B	Total HxCDD
GC/HRMS	EPA 8290/8290A/1613B	Total HpCDD
GC/HRMS	EPA 8290/8290A/1613B	Total TCDF
GC/HRMS	EPA 8290/8290A/1613B	Total PeCDF
GC/HRMS	EPA 8290/8290A/1613B	Total HxCDF
GC/HRMS	EPA 8290/8290A/1613B	Total HpCDF

Non-Potable Water		
Technology	Method	Analyte
HPLC/UV	EPA 8330A/8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	Glycerol trinitrate (Nitroglycerin)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitro- 1,3,5-triazine (Hexogen)
HPLC/UV	EPA 8330A/8330B	Methyl-2,4,6- trinitrophenylnitramine
HPLC/UV	EPA 8330A/8330B	Nitrobenzene
HPLC/UV	EPA 8330A/8330B	2-Nitrotoluene (o-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	3-Nitrotoluene (m-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	4-Nitrotoluene (p-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	Octahydro-1,3,5,7- tetranitro 1,3,5,7-tetracine (Octogen)
HPLC/UV	EPA 8330A/8330B	Picric acid
HPLC/UV	EPA 8330A/8330B	Pentaerythritol Tetranitrate
HPLC/UV	EPA 8330A/8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4,6-Trinitrotoluene
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3-dinitroso-5- nitro-1,3,5, triazine (DNX)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitroso- 1,3,5-triazine (TNX)
HPLC/UV	EPA 8330A/8330B	1-Nitroso-3,5-dinitro-1,3,5- triazacyclohexane (MNX)
HPLC/UV	EPA 8330A Modified /WS-LC-0010	Nitroguanidine
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-ethylperfluoro-1- octanesulfonamido)-ethanol (N-Et-FOSE)
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-Methylperfluoro-1- octanesulfonamido)-ethanol (N-Me-FOSE)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	6:2 Fluorotelomer sulfonate (6:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	8:2 Fluorotelomer sulfonate (8:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctane sulfonamide (EtFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctanesulfon amidacetic acid (EtFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctane sulfonamide (MeFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctanesulfon amidoacetic acid (MeFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctanoic acid (PFOA)

Non-Potable Water		
Technology	Method	Analyte
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctane Sulfonic Acid (PFOS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutyric acid (PFBA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoropentanoic acid (PFPA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexanoic acid (PFHxA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorononanoic acid (PFNA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecanoic acid (PFDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroundecanoic acid (PFUDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorododecanoic acid (PFDoDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotridecanoic acid (PFTriA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotetradecanoic acid (PDTeA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptane Sulfonic Acid (PFHpS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecane Sulfonic Acid (PFDS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctane Sulfonamide (FOSA)
GC/HRMS	EPA 1668A/1668C	PCB 1
GC/HRMS	EPA 1668A/1668C	PCB 2
GC/HRMS	EPA 1668A/1668C	PCB 3
GC/HRMS	EPA 1668A/1668C	PCB 4
GC/HRMS	EPA 1668A/1668C	PCB 5
GC/HRMS	EPA 1668A/1668C	PCB 6
GC/HRMS	EPA 1668A/1668C	PCB 7
GC/HRMS	EPA 1668A/1668C	PCB 8
GC/HRMS	EPA 1668A/1668C	PCB 9
GC/HRMS	EPA 1668A/1668C	PCB 10
GC/HRMS	EPA 1668A/1668C	PCB 11
GC/HRMS	EPA 1668A/1668C	PCB 12

Non-Potable Water		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 13
GC/HRMS	EPA 1668A/1668C	PCB 14
GC/HRMS	EPA 1668A/1668C	PCB 15
GC/HRMS	EPA 1668A/1668C	PCB 16
GC/HRMS	EPA 1668A/1668C	PCB 17
GC/HRMS	EPA 1668A/1668C	PCB 18
GC/HRMS	EPA 1668A/1668C	PCB 19
GC/HRMS	EPA 1668A/1668C	PCB 20
GC/HRMS	EPA 1668A/1668C	PCB 21
GC/HRMS	EPA 1668A/1668C	PCB 22
GC/HRMS	EPA 1668A/1668C	PCB 23
GC/HRMS	EPA 1668A/1668C	PCB 24
GC/HRMS	EPA 1668A/1668C	PCB 25
GC/HRMS	EPA 1668A/1668C	PCB 26
GC/HRMS	EPA 1668A/1668C	PCB 27
GC/HRMS	EPA 1668A/1668C	PCB 28
GC/HRMS	EPA 1668A/1668C	PCB 29
GC/HRMS	EPA 1668A/1668C	PCB 30
GC/HRMS	EPA 1668A/1668C	PCB 32
GC/HRMS	EPA 1668A/1668C	PCB 31
GC/HRMS	EPA 1668A/1668C	PCB 33
GC/HRMS	EPA 1668A/1668C	PCB 34
GC/HRMS	EPA 1668A/1668C	PCB 35
GC/HRMS	EPA 1668A/1668C	PCB 36
GC/HRMS	EPA 1668A/1668C	PCB 37
GC/HRMS	EPA 1668A/1668C	PCB 38
GC/HRMS	EPA 1668A/1668C	PCB 39
GC/HRMS	EPA 1668A/1668C	PCB 40
GC/HRMS	EPA 1668A/1668C	PCB 41
GC/HRMS	EPA 1668A/1668C	PCB 42
GC/HRMS	EPA 1668A/1668C	PCB 43
GC/HRMS	EPA 1668A/1668C	PCB 44
GC/HRMS	EPA 1668A/1668C	PCB 45
GC/HRMS	EPA 1668A/1668C	PCB 46
GC/HRMS	EPA 1668A/1668C	PCB 47
GC/HRMS	EPA 1668A/1668C	PCB 48
GC/HRMS	EPA 1668A/1668C	PCB 49
GC/HRMS	EPA 1668A/1668C	PCB 50
GC/HRMS	EPA 1668A/1668C	PCB 51

Non-Potable Water		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 52
GC/HRMS	EPA 1668A/1668C	PCB 53
GC/HRMS	EPA 1668A/1668C	PCB 54
GC/HRMS	EPA 1668A/1668C	PCB 55
GC/HRMS	EPA 1668A/1668C	PCB 56
GC/HRMS	EPA 1668A/1668C	PCB 57
GC/HRMS	EPA 1668A/1668C	PCB 58
GC/HRMS	EPA 1668A/1668C	PCB 59
GC/HRMS	EPA 1668A/1668C	PCB 60
GC/HRMS	EPA 1668A/1668C	PCB 61
GC/HRMS	EPA 1668A/1668C	PCB 62
GC/HRMS	EPA 1668A/1668C	PCB 63
GC/HRMS	EPA 1668A/1668C	PCB 64
GC/HRMS	EPA 1668A/1668C	PCB 65
GC/HRMS	EPA 1668A/1668C	PCB 66
GC/HRMS	EPA 1668A/1668C	PCB 67
GC/HRMS	EPA 1668A/1668C	PCB 68
GC/HRMS	EPA 1668A/1668C	PCB 69
GC/HRMS	EPA 1668A/1668C	PCB 70
GC/HRMS	EPA 1668A/1668C	PCB 71
GC/HRMS	EPA 1668A/1668C	PCB 72
GC/HRMS	EPA 1668A/1668C	PCB 73
GC/HRMS	EPA 1668A/1668C	PCB 74
GC/HRMS	EPA 1668A/1668C	PCB 75
GC/HRMS	EPA 1668A/1668C	PCB 76
GC/HRMS	EPA 1668A/1668C	PCB 77
GC/HRMS	EPA 1668A/1668C	PCB 78
GC/HRMS	EPA 1668A/1668C	PCB 79
GC/HRMS	EPA 1668A/1668C	PCB 80
GC/HRMS	EPA 1668A/1668C	PCB 81
GC/HRMS	EPA 1668A/1668C	PCB 82
GC/HRMS	EPA 1668A/1668C	PCB 83
GC/HRMS	EPA 1668A/1668C	PCB 84
GC/HRMS	EPA 1668A/1668C	PCB 85
GC/HRMS	EPA 1668A/1668C	PCB 86
GC/HRMS	EPA 1668A/1668C	PCB 87
GC/HRMS	EPA 1668A/1668C	PCB 88
GC/HRMS	EPA 1668A/1668C	PCB 89
GC/HRMS	EPA 1668A/1668C	PCB 90



Non-Potable Water		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 91
GC/HRMS	EPA 1668A/1668C	PCB 92
GC/HRMS	EPA 1668A/1668C	PCB 93
GC/HRMS	EPA 1668A/1668C	PCB 94
GC/HRMS	EPA 1668A/1668C	PCB 95
GC/HRMS	EPA 1668A/1668C	PCB 96
GC/HRMS	EPA 1668A/1668C	PCB 97
GC/HRMS	EPA 1668A/1668C	PCB 98
GC/HRMS	EPA 1668A/1668C	PCB 99
GC/HRMS	EPA 1668A/1668C	PCB 100
GC/HRMS	EPA 1668A/1668C	PCB 101
GC/HRMS	EPA 1668A/1668C	PCB 102
GC/HRMS	EPA 1668A/1668C	PCB 103
GC/HRMS	EPA 1668A/1668C	PCB 104
GC/HRMS	EPA 1668A/1668C	PCB 105
GC/HRMS	EPA 1668A/1668C	PCB 106
GC/HRMS	EPA 1668A/1668C	PCB 107
GC/HRMS	EPA 1668A/1668C	PCB 108
GC/HRMS	EPA 1668A/1668C	PCB 109
GC/HRMS	EPA 1668A/1668C	PCB 110
GC/HRMS	EPA 1668A/1668C	PCB 111
GC/HRMS	EPA 1668A/1668C	PCB 112
GC/HRMS	EPA 1668A/1668C	PCB 113
GC/HRMS	EPA 1668A/1668C	PCB 114
GC/HRMS	EPA 1668A/1668C	PCB 115
GC/HRMS	EPA 1668A/1668C	PCB 116
GC/HRMS	EPA 1668A/1668C	PCB 117
GC/HRMS	EPA 1668A/1668C	PCB 118
GC/HRMS	EPA 1668A/1668C	PCB 119
GC/HRMS	EPA 1668A/1668C	PCB 120
GC/HRMS	EPA 1668A/1668C	PCB 121
GC/HRMS	EPA 1668A/1668C	PCB 122
GC/HRMS	EPA 1668A/1668C	PCB 123
GC/HRMS	EPA 1668A/1668C	PCB 124
GC/HRMS	EPA 1668A/1668C	PCB 125
GC/HRMS	EPA 1668A/1668C	PCB 126
GC/HRMS	EPA 1668A/1668C	PCB 127
GC/HRMS	EPA 1668A/1668C	PCB 128
GC/HRMS	EPA 1668A/1668C	PCB 129



Non-Potable Water		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 130
GC/HRMS	EPA 1668A/1668C	PCB 131
GC/HRMS	EPA 1668A/1668C	PCB 132
GC/HRMS	EPA 1668A/1668C	PCB 133
GC/HRMS	EPA 1668A/1668C	PCB 134
GC/HRMS	EPA 1668A/1668C	PCB 135
GC/HRMS	EPA 1668A/1668C	PCB 136
GC/HRMS	EPA 1668A/1668C	PCB 137
GC/HRMS	EPA 1668A/1668C	PCB 138
GC/HRMS	EPA 1668A/1668C	PCB 139
GC/HRMS	EPA 1668A/1668C	PCB 140
GC/HRMS	EPA 1668A/1668C	PCB 141
GC/HRMS	EPA 1668A/1668C	PCB 142
GC/HRMS	EPA 1668A/1668C	PCB 143
GC/HRMS	EPA 1668A/1668C	PCB 144
GC/HRMS	EPA 1668A/1668C	PCB 145
GC/HRMS	EPA 1668A/1668C	PCB 146
GC/HRMS	EPA 1668A/1668C	PCB 147
GC/HRMS	EPA 1668A/1668C	PCB 148
GC/HRMS	EPA 1668A/1668C	PCB 149
GC/HRMS	EPA 1668A/1668C	PCB 150
GC/HRMS	EPA 1668A/1668C	PCB 151
GC/HRMS	EPA 1668A/1668C	PCB 152
GC/HRMS	EPA 1668A/1668C	PCB 153
GC/HRMS	EPA 1668A/1668C	PCB 154
GC/HRMS	EPA 1668A/1668C	PCB 155
GC/HRMS	EPA 1668A/1668C	PCB 156
GC/HRMS	EPA 1668A/1668C	PCB 157
GC/HRMS	EPA 1668A/1668C	PCB 158
GC/HRMS	EPA 1668A/1668C	PCB 159
GC/HRMS	EPA 1668A/1668C	PCB 160
GC/HRMS	EPA 1668A/1668C	PCB 161
GC/HRMS	EPA 1668A/1668C	PCB 162
GC/HRMS	EPA 1668A/1668C	PCB 163
GC/HRMS	EPA 1668A/1668C	PCB 164
GC/HRMS	EPA 1668A/1668C	PCB 165
GC/HRMS	EPA 1668A/1668C	PCB 166
GC/HRMS	EPA 1668A/1668C	PCB 167
GC/HRMS	EPA 1668A/1668C	PCB 168



Non-Potable Water		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 169
GC/HRMS	EPA 1668A/1668C	PCB 170
GC/HRMS	EPA 1668A/1668C	PCB 171
GC/HRMS	EPA 1668A/1668C	PCB 172
GC/HRMS	EPA 1668A/1668C	PCB 173
GC/HRMS	EPA 1668A/1668C	PCB 174
GC/HRMS	EPA 1668A/1668C	PCB 175
GC/HRMS	EPA 1668A/1668C	PCB 176
GC/HRMS	EPA 1668A/1668C	PCB 177
GC/HRMS	EPA 1668A/1668C	PCB 178
GC/HRMS	EPA 1668A/1668C	PCB 179
GC/HRMS	EPA 1668A/1668C	PCB 180
GC/HRMS	EPA 1668A/1668C	PCB 181
GC/HRMS	EPA 1668A/1668C	PCB 182
GC/HRMS	EPA 1668A/1668C	PCB 183
GC/HRMS	EPA 1668A/1668C	PCB 184
GC/HRMS	EPA 1668A/1668C	PCB 185
GC/HRMS	EPA 1668A/1668C	PCB 186
GC/HRMS	EPA 1668A/1668C	PCB 187
GC/HRMS	EPA 1668A/1668C	PCB 188
GC/HRMS	EPA 1668A/1668C	PCB 189
GC/HRMS	EPA 1668A/1668C	PCB 190
GC/HRMS	EPA 1668A/1668C	PCB 191
GC/HRMS	EPA 1668A/1668C	PCB 192
GC/HRMS	EPA 1668A/1668C	PCB 193
GC/HRMS	EPA 1668A/1668C	PCB 194
GC/HRMS	EPA 1668A/1668C	PCB 195
GC/HRMS	EPA 1668A/1668C	PCB 196
GC/HRMS	EPA 1668A/1668C	PCB 197
GC/HRMS	EPA 1668A/1668C	PCB 198
GC/HRMS	EPA 1668A/1668C	PCB 199
GC/HRMS	EPA 1668A/1668C	PCB 200
GC/HRMS	EPA 1668A/1668C	PCB 201
GC/HRMS	EPA 1668A/1668C	PCB 202
GC/HRMS	EPA 1668A/1668C	PCB 203
GC/HRMS	EPA 1668A/1668C	PCB 204
GC/HRMS	EPA 1668A/1668C	PCB 205
GC/HRMS	EPA 1668A/1668C	PCB 206
GC/HRMS	EPA 1668A/1668C	PCB 207

Non-Potable Water		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 208
GC/HRMS	EPA 1668A/1668C	PCB 209
Preparation	Method	Type
Acid Digestion (Aqueous)	EPA 3005A/3010A	Inorganics
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Non-Volatile Organics
Solid Phase Extraction	EPA 3535A	Semivolatile and Non-Volatile Organics
Purge and Trap	EPA 5030B/5030C	Volatile Organic Compounds
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs
Silica Gel Cleanup	EPA 3630C	Column Cleanup

Drinking Water		
Technology	Method	Analyte
LC/MS/MS	EPA 537	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	EPA 537	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 537	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	EPA 537	Perfluorononanoic acid (PFNA)
LC/MS/MS	EPA 537	Perfluorooctanoic acid (PFOA)
LC/MS/MS	EPA 537	Perfluorooctane Sulfonic Acid (PFOS)
Preparation	Method	Type
Solid Phase Extraction	EPA 537	Perfluoro compounds in Drinking Water

Solid and Chemical Materials		
Technology	Method	Analyte
ICP-AES	EPA 6010B/6010C	Aluminum
ICP-AES	EPA 6010B/6010C	Antimony
ICP-AES	EPA 6010B/6010C	Arsenic
ICP-AES	EPA 6010B/6010C	Barium
ICP-AES	EPA 6010B/6010C	Beryllium
ICP-AES	EPA 6010B/6010C	Boron
ICP-AES	EPA 6010B/6010C	Cadmium
ICP-AES	EPA 6010B/6010C	Calcium

Solid and Chemical Materials		
Technology	Method	Analyte
ICP-AES	EPA 6010B/6010C	Chromium (Total)
ICP-AES	EPA 6010B/6010C	Cobalt
ICP-AES	EPA 6010B/6010C	Copper
ICP-AES	EPA 6010B/6010C	Iron
ICP-AES	EPA 6010B/6010C	Lead
ICP-AES	EPA 6010B/6010C	Magnesium
ICP-AES	EPA 6010B/6010C	Manganese
ICP-AES	EPA 6010B/6010C	Molybdenum
ICP-AES	EPA 6010B/6010C	Nickel
ICP-AES	EPA 6010B/6010C	Potassium
ICP-AES	EPA 6010B/6010C	Selenium
ICP-AES	EPA 6010B/6010C	Silver
ICP-AES	EPA 6010B/6010C	Sodium
ICP-AES	EPA 6010B/6010C	Thallium
ICP-AES	EPA 6010B/6010C	Tin
ICP-AES	EPA 6010B/6010C	Titanium
ICP-AES	EPA 6010B/6010C	Vanadium
ICP-AES	EPA 6010B/6010C	Zinc
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel
ICP-MS	EPA 6020/6020A	Phosphorus
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium

Solid and Chemical Materials		
Technology	Method	Analyte
ICP-MS	EPA 6020/6020A	Strontium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Tin
ICP-MS	EPA 6020/6020A	Titanium
ICP-MS	EPA 6020/6020A	Uranium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
CVAAS	EPA 7471A/7471B	Mercury
Colorimetric	EPA 353.2	Nitrate
Colorimetric	EPA 353.2	Nitrate-nitrite
Colorimetric	EPA 353.2	Nitrite
Colorimetric/Hydrolysis	EPA 353.2 Modified /WS-WC-0050	Nitrocellulose
LC/MS/MS	EPA 6850	Perchlorate
Probe	EPA 9045C/9045D	pH
Ion Chromatography	EPA 9056A/300.0	Bromide
Ion Chromatography	EPA 9056A/300.0	Chloride
Ion Chromatography	EPA 9056A/300.0	Fluoride
Ion Chromatography	EPA 9056A/300.0	Sulfate
Ion Chromatography	EPA 9056A/300.0	Nitrate
Ion Chromatography	EPA 9056A/300.0	Nitrite
Gravimetric	ASTM D2216	%Moisture
GC/MS	EPA 8260B/8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/8260C	1,2-Dibromoethane
GC/MS	EPA 8260B/8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C	1,3,5-Trimethylbenzene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B/8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,3-Dichloropropane
GC/MS	EPA 8260B/8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1-Chlorohexane
GC/MS	EPA 8260B/8260C	2,2-Dichloropropane
GC/MS	EPA 8260B/8260C	2-Butanone (MEK)
GC/MS	EPA 8260B/8260C	2-Chlorotoluene
GC/MS	EPA 8260B/8260C	2-Hexanone (MBK)
GC/MS	EPA 8260B/8260C	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA 8260B/8260C	4-Chlorotoluene
GC/MS	EPA 8260B/8260C	4-Isopropyltoluene
GC/MS	EPA 8260B/8260C	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 8260B/8260C	Acetone
GC/MS	EPA 8260B/8260C	Allyl Chloride
GC/MS	EPA 8260B/8260C	Benzene
GC/MS	EPA 8260B/8260C	Bromobenzene
GC/MS	EPA 8260B/8260C	Bromochloromethane
GC/MS	EPA 8260B/8260C	Bromodichloromethane
GC/MS	EPA 8260B/8260C	Bromoform
GC/MS	EPA 8260B/8260C	Bromomethane
GC/MS	EPA 8260B/8260C	Carbon Disulfide
GC/MS	EPA 8260B/8260C	Carbon Tetrachloride
GC/MS	EPA 8260B/8260C	Chlorobenzene
GC/MS	EPA 8260B/8260C	Chloroethane
GC/MS	EPA 8260B/8260C	Chloroform
GC/MS	EPA 8260B/8260C	Chloromethane
GC/MS	EPA 8260B/8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Cyclohexane
GC/MS	EPA 8260B/8260C	Dibromochloromethane
GC/MS	EPA 8260B/8260C	Dibromomethane
GC/MS	EPA 8260B/8260C	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C	Diisopropyl Ether (DIPE)
GC/MS	EPA 8260B/8260C	Ethylbenzene
GC/MS	EPA 8260B/8260C	Ethylmethacrylate
GC/MS	EPA 8260B/8260C	Ethyl tert-butyl Ether (ETBE)
GC/MS	EPA 8260B/8260C	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C	Hexane
GC/MS	EPA 8260B/8260C	Iodomethane

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B/8260C	Isobutanol (2-Methyl-1-propanol)
GC/MS	EPA 8260B/8260C	Isopropylbenzene
GC/MS	EPA 8260B/8260C	m & p Xylene
GC/MS	EPA 8260B/8260C	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA 8260B/8260C	Methylene Chloride
GC/MS	EPA 8260B/8260C	Naphthalene
GC/MS	EPA 8260B/8260C	n-Butylbenzene
GC/MS	EPA 8260B/8260C	n-Propylbenzene
GC/MS	EPA 8260B/8260C	o-Xylene
GC/MS	EPA 8260B/8260C	sec-Butylbenzene
GC/MS	EPA 8260B/8260C	Styrene
GC/MS	EPA 8260B/8260C	t-Amyl methyl Ether (TAME)
GC/MS	EPA 8260B/8260C	t-1,4-Dichloro-2-Butene
GC/MS	EPA 8260B/8260C	tert-Butylbenzene
GC/MS	EPA 8260B/8260C	Tetrachloroethene
GC/MS	EPA 8260B/8260C	Toluene
GC/MS	EPA 8260B/8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Trichloroethene
GC/MS	EPA 8260B/8260C	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C	Vinyl Acetate
GC/MS	EPA 8260B/8260C	Vinyl Chloride
GC/MS	EPA 8260B/8260C	Xylenes, Total
GC/MS	EPA 8260B/AK101MS	Gasoline Range Organics (GRO)
GC/MS	EPA 8270C/8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270C/8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270C/8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dimethylphenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrophenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrotoluene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/8270D	2,6-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2-Chloronaphthalene
GC/MS	EPA 8270C/8270D	2-Chlorophenol
GC/MS	EPA 8270C/8270D	2-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2-Methylphenol
GC/MS	EPA 8270C/8270D	2-Nitroaniline
GC/MS	EPA 8270C/8270D	2-Nitrophenol
GC/MS	EPA 8270C/8270D	3&4-Methylphenol
GC/MS	EPA 8270C/8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/8270D	3-Nitroaniline
GC/MS	EPA 8270C/8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/8270D	4-Chloroaniline
GC/MS	EPA 8270C/8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Nitroaniline
GC/MS	EPA 8270C/8270D	4-Nitrophenol
GC/MS	EPA 8270C/8270D	Acenaphthene
GC/MS	EPA 8270C/8270D	Acenaphthylene
GC/MS	EPA 8270C/8270D	Aniline
GC/MS	EPA 8270C/8270D	Anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)pyrene
GC/MS	EPA 8270C/8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/8270D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/8270D	Benzoic Acid
GC/MS	EPA 8270C/8270D	Benzyl Alcohol
GC/MS	EPA 8270C/8270D	Benzyl butyl Phthalate
GC/MS	EPA 8270C/8270D	Biphenyl
GC/MS	EPA 8270C/8270D	Bis(2-chloroethoxy) Methane
GC/MS	EPA 8270C/8270D	Bis(2-chloroethyl) Ether
GC/MS	EPA 8270C/8270D	Bis(2-chloroisopropyl) Ether
GC/MS	EPA 8270C/8270D	Carbazole
GC/MS	EPA 8270C/8270D	Chrysene
GC/MS	EPA 8270C/8270D	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA 8270C/8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/8270D	Dibenzofuran

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/8270D	Diethyl Phthalate
GC/MS	EPA 8270C/8270D	Dimethyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-butyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-octyl Phthalate
GC/MS	EPA 8270C/8270D	Fluoranthene
GC/MS	EPA 8270C/8270D	Fluorene
GC/MS	EPA 8270C/8270D	Hexachlorobenzene
GC/MS	EPA 8270C/8270D	Hexachlorobutadiene
GC/MS	EPA 8270C/8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/8270D	Hexachloroethane
GC/MS	EPA 8270C/8270D	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA 8270C/8270D	Isophorone
GC/MS	EPA 8270C/8270D	Naphthalene
GC/MS	EPA 8270C/8270D	Nitrobenzene
GC/MS	EPA 8270C/8270D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/8270D	Pentachlorophenol
GC/MS	EPA 8270C/8270D	Phenanthrene
GC/MS	EPA 8270C/8270D	Phenol
GC/MS	EPA 8270C/8270D	Pyrene
GC/MS	EPA 8270C/8270D	Pyridine
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	1-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(b)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(g,h,i)perylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(k)fluoranthene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Chrysene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluorene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Naphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Phenanthrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Pyrene
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Dithiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Benzothiazole
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfoxide
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	p-Chlorophenyl methylsulfone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Chloropicrin
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Acetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	2-Chloroacetophenone
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	1,4-Oxathiane
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0003	Dimethyl Disulfide
GC/MS SIM	EPA 521 Modified / WS-MS-0012	N-Nitrosodimethyl amine (NDMA)
GC-FID	EPA 8015B/8015C/8015D AK102	Diesel Range Organics (DRO)
GC-FID	AK103	Residual Range Organics
GC-FID	EPA 8015B/8015C/8015D	Motor Oil Range Organics (MRO)
GC-ECD	EPA 8081A/8081B	Aldrin
GC-ECD	EPA 8081A/8081B	a-BHC
GC-ECD	EPA 8081A/8081B	b-BHC

Solid and Chemical Materials		
Technology	Method	Analyte
GC-ECD	EPA 8081A/8081B	d-BHC
GC-ECD	EPA 8081A/8081B	g-BHC (Lindane)
GC-ECD	EPA 8081A/8081B	a-Chlordane
GC-ECD	EPA 8081A/8081B	g-Chlordane
GC-ECD	EPA 8081A/8081B	4,4'-DDD
GC-ECD	EPA 8081A/8081B	4,4'-DDE
GC-ECD	EPA 8081A/8081B	4,4'-DDT
GC-ECD	EPA 8081A/8081B	Dieldrin
GC-ECD	EPA 8081A/8081B	Endosulfan I
GC-ECD	EPA 8081A/8081B	Endosulfan II
GC-ECD	EPA 8081A/8081B	Endosulfan sulfate
GC-ECD	EPA 8081A/8081B	Endrin
GC-ECD	EPA 8081A/8081B	Endrin Aldehyde
GC-ECD	EPA 8081A/8081B	Endrin Ketone
GC-ECD	EPA 8081A/8081B	Heptachlor
GC-ECD	EPA 8081A/8081B	Heptachlor Epoxide
GC-ECD	EPA 8081A/8081B	Methoxychlor
GC-ECD	EPA 8081A/8081B	Toxaphene
GC-ECD	EPA 8081A/8081B	Chlordane (technical)
GC-ECD	EPA 8082/8082A	PCB-1016
GC-ECD	EPA 8082/8082A	PCB-1221
GC-ECD	EPA 8082/8082A	PCB-1232
GC-ECD	EPA 8082/8082A	PCB-1242
GC-ECD	EPA 8082/8082A	PCB-1248
GC-ECD	EPA 8082/8082A	PCB-1254
GC-ECD	EPA 8082/8082A	PCB-1260
GC-ECD	EPA 8082/8082A	PCB-1262
GC-ECD	EPA 8082/8082A	PCB-1268
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDD
GC/MS	EPA 8280A/8280B	OCDD
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDF
GC/MS	EPA 8280A/8280B	2,3,4,7,8-PeCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDF

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDF
GC/MS	EPA 8280A/8280B	2,3,4,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8,9-HpCDF
GC/MS	EPA 8280A/8280B	OCDF
GC/MS	EPA 8280A/8280B	Total TCDD
GC/MS	EPA 8280A/8280B	Total PeCDD
GC/MS	EPA 8280A/8280B	Total HxCDD
GC/MS	EPA 8280A/8280B	Total HeptaCDD
GC/MS	EPA 8280A/8280B	Total TCDF
GC/MS	EPA 8280A/8280B	Total PeCDF
GC/MS	EPA 8280A/8280B	Total HxCDF
GC/MS	EPA 8280A/8280B	Total HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,7,8-TeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8-PeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,6,7,8-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8,9-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,6,7,8-HpCDD
GC/HRMS	EPA 8290/ 8290A/1613B	OCDD
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,7,8-TeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8-PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,4,7,8-PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,6,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8,9-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,4,6,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,6,7,8-HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8,9-HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	OCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total TCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total PeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total HpCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total TCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total HpCDF

Solid and Chemical Materials		
Technology	Method	Analyte
HPLC/UV	EPA 8330A/8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	Glycerol trinitrate (Nitroglycerin)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitro- 1,3,5-triazine (Hexogen)
HPLC/UV	EPA 8330A/8330B	Methyl-2,4,6- trinitrophenylnitramine
HPLC/UV	EPA 8330A/8330B	Nitrobenzene
HPLC/UV	EPA 8330A/8330B	2-Nitrotoluene (o-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	3-Nitrotoluene (m-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	4-Nitrotoluene (p-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	Octahydro-1,3,5,7- tetranitro 1,3,5,7-tetracine (Octogen)
HPLC/UV	EPA 8330A/8330B	Picric acid
HPLC/UV	EPA 8330A/8330B	Pentaerythritol Tetranitrate
HPLC/UV	EPA 8330A/8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4,6-Trinitrotoluene
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3-dinitroso-5- nitro-1,3,5, triazine (DNX)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitroso- 1,3,5-triazine (TNX)
HPLC/UV	EPA 8330A/8330B	1-Nitroso-3,5-dinitro-1,3,5- triazacyclohexane (MNX)
HPLC/UV	EPA 8330A Modified / WS-LC-0010	Nitroguanidine
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-ethylperfluoro-1- octanesulfonamido)-ethanol [N-Et-FOSE]
GC-HRMS	EPA 8290 Modified / WS-ID-0021	2-(N-Methylperfluoro-1- octanesulfonamido)-ethanol [N-Me-FOSE]
LC/MS/MS	EPA 537 Modified / WS-LC-0025	6:2 Fluorotelomer sulfonate (6:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	8:2 Fluorotelomer sulfonate (8:2 FTS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctane sulfonamide (EtFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Ethyl perfluorooctanesulfon amidacetic acid (EtFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctane sulfonamide (MeFOSA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	N-Methyl perfluorooctanesulfon amidoacetic acid (MeFOSAA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctanoic acid (PFOA)

Solid and Chemical Materials		
Technology	Method	Analyte
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctane Sulfonic Acid (PFOS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutyric acid (PFBA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoropentanoic acid (PFPA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexanoic acid (PFHxA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorononanoic acid (PFNA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecanoic acid (PFDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroundecanoic acid (PFUDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorododecanoic acid (PFDoDA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotridecanoic acid (PFTriA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorotetradecanoic acid (PDTeA)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluoroheptane Sulfonic Acid (PFHpS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorodecane Sulfonic Acid (PFDS)
LC/MS/MS	EPA 537 Modified / WS-LC-0025	Perfluorooctane Sulfonamide (FOSA)
GC/HRMS	EPA 1668A/1668C	PCB 1
GC/HRMS	EPA 1668A/1668C	PCB 2
GC/HRMS	EPA 1668A/1668C	PCB 3
GC/HRMS	EPA 1668A/1668C	PCB 4
GC/HRMS	EPA 1668A/1668C	PCB 5
GC/HRMS	EPA 1668A/1668C	PCB 6
GC/HRMS	EPA 1668A/1668C	PCB 7
GC/HRMS	EPA 1668A/1668C	PCB 8
GC/HRMS	EPA 1668A/1668C	PCB 9
GC/HRMS	EPA 1668A/1668C	PCB 10
GC/HRMS	EPA 1668A/1668C	PCB 11
GC/HRMS	EPA 1668A/1668C	PCB 12

Solid and Chemical Materials		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 13
GC/HRMS	EPA 1668A/1668C	PCB 14
GC/HRMS	EPA 1668A/1668C	PCB 15
GC/HRMS	EPA 1668A/1668C	PCB 16
GC/HRMS	EPA 1668A/1668C	PCB 17
GC/HRMS	EPA 1668A/1668C	PCB 18
GC/HRMS	EPA 1668A/1668C	PCB 19
GC/HRMS	EPA 1668A/1668C	PCB 20
GC/HRMS	EPA 1668A/1668C	PCB 21
GC/HRMS	EPA 1668A/1668C	PCB 22
GC/HRMS	EPA 1668A/1668C	PCB 23
GC/HRMS	EPA 1668A/1668C	PCB 24
GC/HRMS	EPA 1668A/1668C	PCB 25
GC/HRMS	EPA 1668A/1668C	PCB 26
GC/HRMS	EPA 1668A/1668C	PCB 27
GC/HRMS	EPA 1668A/1668C	PCB 28
GC/HRMS	EPA 1668A/1668C	PCB 29
GC/HRMS	EPA 1668A/1668C	PCB 30
GC/HRMS	EPA 1668A/1668C	PCB 32
GC/HRMS	EPA 1668A/1668C	PCB 31
GC/HRMS	EPA 1668A/1668C	PCB 33
GC/HRMS	EPA 1668A/1668C	PCB 34
GC/HRMS	EPA 1668A/1668C	PCB 35
GC/HRMS	EPA 1668A/1668C	PCB 36
GC/HRMS	EPA 1668A/1668C	PCB 37
GC/HRMS	EPA 1668A/1668C	PCB 38
GC/HRMS	EPA 1668A/1668C	PCB 39
GC/HRMS	EPA 1668A/1668C	PCB 40
GC/HRMS	EPA 1668A/1668C	PCB 41
GC/HRMS	EPA 1668A/1668C	PCB 42
GC/HRMS	EPA 1668A/1668C	PCB 43
GC/HRMS	EPA 1668A/1668C	PCB 44
GC/HRMS	EPA 1668A/1668C	PCB 45
GC/HRMS	EPA 1668A/1668C	PCB 46
GC/HRMS	EPA 1668A/1668C	PCB 47
GC/HRMS	EPA 1668A/1668C	PCB 48
GC/HRMS	EPA 1668A/1668C	PCB 49
GC/HRMS	EPA 1668A/1668C	PCB 50
GC/HRMS	EPA 1668A/1668C	PCB 51



Solid and Chemical Materials		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 52
GC/HRMS	EPA 1668A/1668C	PCB 53
GC/HRMS	EPA 1668A/1668C	PCB 54
GC/HRMS	EPA 1668A/1668C	PCB 55
GC/HRMS	EPA 1668A/1668C	PCB 56
GC/HRMS	EPA 1668A/1668C	PCB 57
GC/HRMS	EPA 1668A/1668C	PCB 58
GC/HRMS	EPA 1668A/1668C	PCB 59
GC/HRMS	EPA 1668A/1668C	PCB 60
GC/HRMS	EPA 1668A/1668C	PCB 61
GC/HRMS	EPA 1668A/1668C	PCB 62
GC/HRMS	EPA 1668A/1668C	PCB 63
GC/HRMS	EPA 1668A/1668C	PCB 64
GC/HRMS	EPA 1668A/1668C	PCB 65
GC/HRMS	EPA 1668A/1668C	PCB 66
GC/HRMS	EPA 1668A/1668C	PCB 67
GC/HRMS	EPA 1668A/1668C	PCB 68
GC/HRMS	EPA 1668A/1668C	PCB 69
GC/HRMS	EPA 1668A/1668C	PCB 70
GC/HRMS	EPA 1668A/1668C	PCB 71
GC/HRMS	EPA 1668A/1668C	PCB 72
GC/HRMS	EPA 1668A/1668C	PCB 73
GC/HRMS	EPA 1668A/1668C	PCB 74
GC/HRMS	EPA 1668A/1668C	PCB 75
GC/HRMS	EPA 1668A/1668C	PCB 76
GC/HRMS	EPA 1668A/1668C	PCB 77
GC/HRMS	EPA 1668A/1668C	PCB 78
GC/HRMS	EPA 1668A/1668C	PCB 79
GC/HRMS	EPA 1668A/1668C	PCB 80
GC/HRMS	EPA 1668A/1668C	PCB 81
GC/HRMS	EPA 1668A/1668C	PCB 82
GC/HRMS	EPA 1668A/1668C	PCB 83
GC/HRMS	EPA 1668A/1668C	PCB 84
GC/HRMS	EPA 1668A/1668C	PCB 85
GC/HRMS	EPA 1668A/1668C	PCB 86
GC/HRMS	EPA 1668A/1668C	PCB 87
GC/HRMS	EPA 1668A/1668C	PCB 88
GC/HRMS	EPA 1668A/1668C	PCB 89
GC/HRMS	EPA 1668A/1668C	PCB 90



Solid and Chemical Materials		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 91
GC/HRMS	EPA 1668A/1668C	PCB 92
GC/HRMS	EPA 1668A/1668C	PCB 93
GC/HRMS	EPA 1668A/1668C	PCB 94
GC/HRMS	EPA 1668A/1668C	PCB 95
GC/HRMS	EPA 1668A/1668C	PCB 96
GC/HRMS	EPA 1668A/1668C	PCB 97
GC/HRMS	EPA 1668A/1668C	PCB 98
GC/HRMS	EPA 1668A/1668C	PCB 99
GC/HRMS	EPA 1668A/1668C	PCB 100
GC/HRMS	EPA 1668A/1668C	PCB 101
GC/HRMS	EPA 1668A/1668C	PCB 102
GC/HRMS	EPA 1668A/1668C	PCB 103
GC/HRMS	EPA 1668A/1668C	PCB 104
GC/HRMS	EPA 1668A/1668C	PCB 105
GC/HRMS	EPA 1668A/1668C	PCB 106
GC/HRMS	EPA 1668A/1668C	PCB 107
GC/HRMS	EPA 1668A/1668C	PCB 108
GC/HRMS	EPA 1668A/1668C	PCB 109
GC/HRMS	EPA 1668A/1668C	PCB 110
GC/HRMS	EPA 1668A/1668C	PCB 111
GC/HRMS	EPA 1668A/1668C	PCB 112
GC/HRMS	EPA 1668A/1668C	PCB 113
GC/HRMS	EPA 1668A/1668C	PCB 114
GC/HRMS	EPA 1668A/1668C	PCB 115
GC/HRMS	EPA 1668A/1668C	PCB 116
GC/HRMS	EPA 1668A/1668C	PCB 117
GC/HRMS	EPA 1668A/1668C	PCB 118
GC/HRMS	EPA 1668A/1668C	PCB 119
GC/HRMS	EPA 1668A/1668C	PCB 120
GC/HRMS	EPA 1668A/1668C	PCB 121
GC/HRMS	EPA 1668A/1668C	PCB 122
GC/HRMS	EPA 1668A/1668C	PCB 123
GC/HRMS	EPA 1668A/1668C	PCB 124
GC/HRMS	EPA 1668A/1668C	PCB 125
GC/HRMS	EPA 1668A/1668C	PCB 126
GC/HRMS	EPA 1668A/1668C	PCB 127
GC/HRMS	EPA 1668A/1668C	PCB 128
GC/HRMS	EPA 1668A/1668C	PCB 129

Solid and Chemical Materials		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 130
GC/HRMS	EPA 1668A/1668C	PCB 131
GC/HRMS	EPA 1668A/1668C	PCB 132
GC/HRMS	EPA 1668A/1668C	PCB 133
GC/HRMS	EPA 1668A/1668C	PCB 134
GC/HRMS	EPA 1668A/1668C	PCB 135
GC/HRMS	EPA 1668A/1668C	PCB 136
GC/HRMS	EPA 1668A/1668C	PCB 137
GC/HRMS	EPA 1668A/1668C	PCB 138
GC/HRMS	EPA 1668A/1668C	PCB 139
GC/HRMS	EPA 1668A/1668C	PCB 140
GC/HRMS	EPA 1668A/1668C	PCB 141
GC/HRMS	EPA 1668A/1668C	PCB 142
GC/HRMS	EPA 1668A/1668C	PCB 143
GC/HRMS	EPA 1668A/1668C	PCB 144
GC/HRMS	EPA 1668A/1668C	PCB 145
GC/HRMS	EPA 1668A/1668C	PCB 146
GC/HRMS	EPA 1668A/1668C	PCB 147
GC/HRMS	EPA 1668A/1668C	PCB 148
GC/HRMS	EPA 1668A/1668C	PCB 149
GC/HRMS	EPA 1668A/1668C	PCB 150
GC/HRMS	EPA 1668A/1668C	PCB 151
GC/HRMS	EPA 1668A/1668C	PCB 152
GC/HRMS	EPA 1668A/1668C	PCB 153
GC/HRMS	EPA 1668A/1668C	PCB 154
GC/HRMS	EPA 1668A/1668C	PCB 155
GC/HRMS	EPA 1668A/1668C	PCB 156
GC/HRMS	EPA 1668A/1668C	PCB 157
GC/HRMS	EPA 1668A/1668C	PCB 158
GC/HRMS	EPA 1668A/1668C	PCB 159
GC/HRMS	EPA 1668A/1668C	PCB 160
GC/HRMS	EPA 1668A/1668C	PCB 161
GC/HRMS	EPA 1668A/1668C	PCB 162
GC/HRMS	EPA 1668A/1668C	PCB 163
GC/HRMS	EPA 1668A/1668C	PCB 164
GC/HRMS	EPA 1668A/1668C	PCB 165
GC/HRMS	EPA 1668A/1668C	PCB 166
GC/HRMS	EPA 1668A/1668C	PCB 167
GC/HRMS	EPA 1668A/1668C	PCB 168



Solid and Chemical Materials		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 169
GC/HRMS	EPA 1668A/1668C	PCB 170
GC/HRMS	EPA 1668A/1668C	PCB 171
GC/HRMS	EPA 1668A/1668C	PCB 172
GC/HRMS	EPA 1668A/1668C	PCB 173
GC/HRMS	EPA 1668A/1668C	PCB 174
GC/HRMS	EPA 1668A/1668C	PCB 175
GC/HRMS	EPA 1668A/1668C	PCB 176
GC/HRMS	EPA 1668A/1668C	PCB 177
GC/HRMS	EPA 1668A/1668C	PCB 178
GC/HRMS	EPA 1668A/1668C	PCB 179
GC/HRMS	EPA 1668A/1668C	PCB 180
GC/HRMS	EPA 1668A/1668C	PCB 181
GC/HRMS	EPA 1668A/1668C	PCB 182
GC/HRMS	EPA 1668A/1668C	PCB 183
GC/HRMS	EPA 1668A/1668C	PCB 184
GC/HRMS	EPA 1668A/1668C	PCB 185
GC/HRMS	EPA 1668A/1668C	PCB 186
GC/HRMS	EPA 1668A/1668C	PCB 187
GC/HRMS	EPA 1668A/1668C	PCB 188
GC/HRMS	EPA 1668A/1668C	PCB 189
GC/HRMS	EPA 1668A/1668C	PCB 190
GC/HRMS	EPA 1668A/1668C	PCB 191
GC/HRMS	EPA 1668A/1668C	PCB 192
GC/HRMS	EPA 1668A/1668C	PCB 193
GC/HRMS	EPA 1668A/1668C	PCB 194
GC/HRMS	EPA 1668A/1668C	PCB 195
GC/HRMS	EPA 1668A/1668C	PCB 196
GC/HRMS	EPA 1668A/1668C	PCB 197
GC/HRMS	EPA 1668A/1668C	PCB 198
GC/HRMS	EPA 1668A/1668C	PCB 199
GC/HRMS	EPA 1668A/1668C	PCB 200
GC/HRMS	EPA 1668A/1668C	PCB 201
GC/HRMS	EPA 1668A/1668C	PCB 202
GC/HRMS	EPA 1668A/1668C	PCB 203
GC/HRMS	EPA 1668A/1668C	PCB 204
GC/HRMS	EPA 1668A/1668C	PCB 205
GC/HRMS	EPA 1668A/1668C	PCB 206
GC/HRMS	EPA 1668A/1668C	PCB 207

Solid and Chemical Materials		
Technology	Method	Analyte
GC/HRMS	EPA 1668A/1668C	PCB 208
GC/HRMS	EPA 1668A/1668C	PCB 209
Preparation	Method	Type
Acid Digestion (Aqueous)	EPA 3005A/3010A	Inorganics
Acid Digestion (Solid)	EPA 3050B	Inorganics
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Non-Volatile Organics
Ultrasonic Extraction	EPA 3550B/3550C	Semivolatile and Non-Volatile Organics
Solvent Dilution	EPA 3580A	Semivolatile and Non-Volatile Organics
Purge and Trap	EPA 5030B	Volatile Organic Compounds
Purge and Trap	EPA 5035/5035A	Volatile Organic Compounds
Table / Jar Shake	WS-OP-0005	Chemical Warfare Degradates (in solid)
Microwave Extraction	EPA 3546	Semivolatile and Non-Volatile Organics
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs
Silica Gel Cleanup	EPA 3630C	Column Cleanup
TCLP Extraction	EPA 1311	Toxicity Characteristic Leaching Procedure

Air and Emissions		
Technology	Method	Analyte
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel

Air and Emissions		
Technology	Method	Analyte
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
Gravimetric	40CFR Part 50 App B	TSP (Total Suspended Particulate)
Gravimetric	40CFR Part 50 App J	PM10
GC/MS	EPA TO14A/TO15	1,1,1-Trichloroethane
GC/MS	EPA TO14A/TO15	1,1,2,2-Tetrachloroethane
GC/MS	EPA TO14A/TO15	1,1,2-Trichloroethane
GC/MS	EPA TO14A/TO15	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA TO14A/TO15	1,1-Dichloroethane
GC/MS	EPA TO14A/TO15	1,1-Dichloroethene
GC/MS	EPA TO14A/TO15	1,2,3-Trichlorobenzene
GC/MS	EPA TO14A/TO15	1,2,3-Trichloropropane
GC/MS	EPA TO14A/TO15	1,2,4-Trichlorobenzene
GC/MS	EPA TO14A/TO15	1,2,4-Trimethylbenzene
GC/MS	EPA TO14A/TO15	1,2-Dibromoethane
GC/MS	EPA TO14A/TO15	1,2-Dichlorobenzene
GC/MS	EPA TO14A/TO15	1,2-Dichloroethane
GC/MS	EPA TO14A/TO15	1,2-Dichloropropane
GC/MS	EPA TO14A/TO15	1,3,5-Trimethylbenzene
GC/MS	EPA TO14A/TO15	1,3-Dichlorobenzene
GC/MS	EPA TO14A/TO15	1,4-Dichlorobenzene
GC/MS	EPA TO14A/TO15	1,4-Dioxane
GC/MS	EPA TO14A/TO15	2-Butanone (MEK)
GC/MS	EPA TO14A/TO15	2-Chlorotoluene
GC/MS	EPA TO14A/TO15	2-Hexanone (MBK)
GC/MS	EPA TO14A/TO15	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA TO14A/TO15	4-Ethyltoluene
GC/MS	EPA TO14A/TO15	4-Isopropyltoluene
GC/MS	EPA TO14A/TO15	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA TO14A/TO15	Acetone
GC/MS	EPA TO14A/TO15	Acrolein
GC/MS	EPA TO14A/TO15	Allyl Chloride
GC/MS	EPA TO14A/TO15	Alpha Methyl Styrene
GC/MS	EPA TO14A/TO15	Benzene

Air and Emissions		
Technology	Method	Analyte
GC/MS	EPA TO14A/TO15	Benzyl Chloride
GC/MS	EPA TO14A/TO15	Bromodichloromethane
GC/MS	EPA TO14A/TO15	Bromoform
GC/MS	EPA TO14A/TO15	Bromomethane
GC/MS	EPA TO14A/TO15	Butadiene (1,3-Butadiene)
GC/MS	EPA TO14A/TO15	Butane
GC/MS	EPA TO14A/TO15	Carbon Disulfide
GC/MS	EPA TO14A/TO15	Carbon Tetrachloride
GC/MS	EPA TO14A/TO15	Chlorobenzene
GC/MS	EPA TO14A/TO15	Chlorodifluoromethane
GC/MS	EPA TO14A/TO15	Chloroethane
GC/MS	EPA TO14A/TO15	Chloroform
GC/MS	EPA TO14A/TO15	Chloromethane
GC/MS	EPA TO14A/TO15	cis-1,2-Dichloroethene
GC/MS	EPA TO14A/TO15	cis-1,3-Dichloropropene
GC/MS	EPA TO14A/TO15	Cyclohexane
GC/MS	EPA TO14A/TO15	Dibromochloromethane
GC/MS	EPA TO14A/TO15	Dibromomethane
GC/MS	EPA TO14A/TO15	Dichlorodifluoromethane
GC/MS	EPA TO14A/TO15	Ethyl Acetate
GC/MS	EPA TO14A/TO15	Ethylbenzene
GC/MS	EPA TO14A/TO15	Hexachlorobutadiene
GC/MS	EPA TO14A/TO15	Hexane
GC/MS	EPA TO14A/TO15	Isooctane (2,2,4- Trimethylpentane)
GC/MS	EPA TO14A/TO15	Isopropyl Alcohol
GC/MS	EPA TO14A/TO15	Isopropylbenzene
GC/MS	EPA TO14A/TO15	m & p Xylene
GC/MS	EPA TO14A/TO15	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA TO14A/TO15	Methylene Chloride
GC/MS	EPA TO14A/TO15	Naphthalene
GC/MS	EPA TO14A/TO15	n-Butanol
GC/MS	EPA TO14A/TO15	n-Butylbenzene
GC/MS	EPA TO14A/TO15	n-Heptane
GC/MS	EPA TO14A/TO15	n-Nonane
GC/MS	EPA TO14A/TO15	n-Octane
GC/MS	EPA TO14A/TO15	n-Propylbenzene
GC/MS	EPA TO14A/TO15	o-Xylene
GC/MS	EPA TO14A/TO15	Pentane
GC/MS	EPA TO14A/TO15	Propene

Air and Emissions		
Technology	Method	Analyte
GC/MS	EPA TO14A/TO15	sec-Butylbenzene
GC/MS	EPA TO14A/TO15	Styrene
GC/MS	EPA TO14A/TO15	tert-Butylbenzene
GC/MS	EPA TO14A/TO15	Tetrachloroethene
GC/MS	EPA TO14A/TO15	Tetrahydrofuran
GC/MS	EPA TO14A/TO15	Toluene
GC/MS	EPA TO14A/TO15	trans-1,2-Dichloroethene
GC/MS	EPA TO14A/TO15	trans-1,3-Dichloropropene
GC/MS	EPA TO14A/TO15	Trichloroethene
GC/MS	EPA TO14A/TO15	Trichlorofluoromethane
GC/MS	EPA TO14A/TO15	Vinyl Acetate
GC/MS	EPA TO14A/TO15	Vinyl Bromide
GC/MS	EPA TO14A/TO15	Vinyl Chloride
GC/MS	EPA TO14A/TO15	Xylenes, Total
GC-FID/TCD	ASTM1946D / EPA 3C	Carbon Dioxide
GC-FID/TCD	ASTM1946D / EPA 3C	Nitrogen
GC-FID/TCD	ASTM1946D / EPA 3C	Oxygen
GC-FID/TCD	ASTM1946D / EPA 3C	Helium
GC-FID/TCD	ASTM1946D / EPA 3C	Hydrogen
GC-FID/TCD	ASTM1946D / EPA 3C	Methane
GC-FID/TCD	ASTM1946D / EPA 3C	Carbon Monoxide[A1]
GC/MS	EPA TO14A/TO15	Gasoline Range Organics (GRO)
GC/MS	EPA TO14A/TO15	TPH as Gasoline
GC/MS SIM	EPA TO15 SIM	1,1,1-Trichloroethane
GC/MS SIM	EPA TO15 SIM	1,1,2,2-Tetrachloroethane
GC/MS SIM	EPA TO15 SIM	1,1,2-Trichloroethane
GC/MS SIM	EPA TO15 SIM	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS SIM	EPA TO15 SIM	1,1-Dichloroethane
GC/MS SIM	EPA TO15 SIM	1,1-Dichloroethene
GC/MS SIM	EPA TO15 SIM	1,2,3-Trichloropropane
GC/MS SIM	EPA TO15 SIM	1,2,4-Trichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,2-Dibromoethane
GC/MS SIM	EPA TO15 SIM	1,2-Dichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,2-Dichloroethane
GC/MS SIM	EPA TO15 SIM	1,2-Dichloropropane
GC/MS SIM	EPA TO15 SIM	1,3-Dichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,4-Dichlorobenzene
GC/MS SIM	EPA TO15 SIM	1,4-Dioxane
GC/MS SIM	EPA TO15 SIM	Acrolein

Air and Emissions		
Technology	Method	Analyte
GC/MS SIM	EPA TO15 SIM	Benzene
GC/MS SIM	EPA TO15 SIM	Benzyl Chloride
GC/MS SIM	EPA TO15 SIM	Bromodichloromethane
GC/MS SIM	EPA TO15 SIM	Butadiene (1,3-Butadiene)
GC/MS SIM	EPA TO15 SIM	Carbon Tetrachloride
GC/MS SIM	EPA TO15 SIM	Chlorobenzene
GC/MS SIM	EPA TO15 SIM	Chloroethane
GC/MS SIM	EPA TO15 SIM	Chloroform
GC/MS SIM	EPA TO15 SIM	Chloromethane
GC/MS SIM	EPA TO15 SIM	cis-1,2-Dichloroethene
GC/MS SIM	EPA TO15 SIM	cis-1,3-Dichloropropene
GC/MS SIM	EPA TO15 SIM	Dibromochloromethane
GC/MS SIM	EPA TO15 SIM	Dichlorodifluoromethane
GC/MS SIM	EPA TO15 SIM	Ethylbenzene
GC/MS SIM	EPA TO15 SIM	Hexachlorobutadiene
GC/MS SIM	EPA TO15 SIM	m & p Xylene
GC/MS SIM	EPA TO15 SIM	Methyl tert-butyl Ether (MTBE)
GC/MS SIM	EPA TO15 SIM	Methylene Chloride
GC/MS SIM	EPA TO15 SIM	Naphthalene
GC/MS SIM	EPA TO15 SIM	o-Xylene
GC/MS SIM	EPA TO15 SIM	Styrene
GC/MS SIM	EPA TO15 SIM	Tetrachloroethene
GC/MS SIM	EPA TO15 SIM	Toluene
GC/MS SIM	EPA TO15 SIM	trans-1,2-Dichloroethene
GC/MS SIM	EPA TO15 SIM	trans-1,3-Dichloropropene
GC/MS SIM	EPA TO15 SIM	Trichloroethene
GC/MS SIM	EPA TO15 SIM	Trichlorofluoromethane
GC/MS SIM	EPA TO15 SIM	Vinyl Chloride
GC/MS SIM	EPA TO15 SIM	Xylenes, Total
GC/MS	EPA TO-13A	1,2,4-Trichlorobenzene
GC/MS	EPA TO-13A	1,2-Dichlorobenzene
GC/MS	EPA TO-13A	1,3-Dichlorobenzene
GC/MS	EPA TO-13A	1,3-Dinitrobenzene
GC/MS	EPA TO-13A	1,4-Dichlorobenzene
GC/MS	EPA TO-13A	1-Methylnaphthalene
GC/MS	EPA TO-13A	2,3,4,6-Tetrachlorophenol
GC/MS	EPA TO-13A	2,4,5-Trichlorophenol
GC/MS	EPA TO-13A	2,4,6-Trichlorophenol
GC/MS	EPA TO-13A	2,4-Dichlorophenol

Air and Emissions		
Technology	Method	Analyte
GC/MS	EPA TO-13A	2,4-Dimethylphenol
GC/MS	EPA TO-13A	2,4-Dinitrophenol
GC/MS	EPA TO-13A	2,4-Dinitrotoluene
GC/MS	EPA TO-13A	2,6-Dichlorophenol
GC/MS	EPA TO-13A	2,6-Dinitrotoluene
GC/MS	EPA TO-13A	2-Chloronaphthalene
GC/MS	EPA TO-13A	2-Chlorophenol
GC/MS	EPA TO-13A	2-Methylnaphthalene
GC/MS	EPA TO-13A	2-Methylphenol
GC/MS	EPA TO-13A	2-Nitroaniline
GC/MS	EPA TO-13A	2-Nitrophenol
GC/MS	EPA TO-13A	3&4-Methylphenol
GC/MS	EPA TO-13A	3,3'-Dichlorobenzidine
GC/MS	EPA TO-13A	3-Nitroaniline
GC/MS	EPA TO-13A	4,6-Dinitro-2-methylphenol
GC/MS	EPA TO-13A	4-Bromophenyl phenyl ether
GC/MS	EPA TO-13A	4-Chloro-3-methylphenol
GC/MS	EPA TO-13A	4-Chloroaniline
GC/MS	EPA TO-13A	4-Chlorophenyl phenyl ether
GC/MS	EPA TO-13A	4-Nitroaniline
GC/MS	EPA TO-13A	4-Nitrophenol
GC/MS	EPA TO-13A	Acenaphthene
GC/MS	EPA TO-13A	Acenaphthylene
GC/MS	EPA TO-13A	Aniline
GC/MS	EPA TO-13A	Anthracene
GC/MS	EPA TO-13A	Benzo(a)anthracene
GC/MS	EPA TO-13A	Benzo(a)pyrene
GC/MS	EPA TO-13A	Benzo(b)fluoranthene
GC/MS	EPA TO-13A	Benzo(g,h,i)perylene
GC/MS	EPA TO-13A	Benzo(k)fluoranthene
GC/MS	EPA TO-13A	Benzoic Acid
GC/MS	EPA TO-13A	Benzyl Alcohol
GC/MS	EPA TO-13A	Benzyl butyl Phthalate
GC/MS	EPA TO-13A	Biphenyl
GC/MS	EPA TO-13A	Bis(2-chloroethoxy) Methane
GC/MS	EPA TO-13A	Bis(2-chloroethyl) Ether
GC/MS	EPA TO-13A	Bis(2-chloroisopropyl) Ether
GC/MS	EPA TO-13A	Carbazole
GC/MS	EPA TO-13A	Chrysene

Air and Emissions		
Technology	Method	Analyte
GC/MS	EPA TO-13A	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA TO-13A	Dibenz(a,h)anthracene
GC/MS	EPA TO-13A	Dibenzofuran
GC/MS	EPA TO-13A	Diethyl Phthalate
GC/MS	EPA TO-13A	Dimethyl Phthalate
GC/MS	EPA TO-13A	Di-n-butyl Phthalate
GC/MS	EPA TO-13A	Di-n-octyl Phthalate
GC/MS	EPA TO-13A	Fluoranthene
GC/MS	EPA TO-13A	Fluorene
GC/MS	EPA TO-13A	Hexachlorobenzene
GC/MS	EPA TO-13A	Hexachlorobutadiene
GC/MS	EPA TO-13A	Hexachlorocyclopentadiene
GC/MS	EPA TO-13A	Hexachloroethane
GC/MS	EPA TO-13A	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA TO-13A	Isophorone
GC/MS	EPA TO-13A	Naphthalene
GC/MS	EPA TO-13A	Nitrobenzene
GC/MS	EPA TO-13A	n-Nitrosodimethylamine
GC/MS	EPA TO-13A	n-Nitrosodi-n-propylamine
GC/MS	EPA TO-13A	n-Nitrosodiphenylamine
GC/MS	EPA TO-13A	Pentachlorophenol
GC/MS	EPA TO-13A	Phenanthrene
GC/MS	EPA TO-13A	Phenol
GC/MS	EPA TO-13A	Pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	1-Methylnaphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	2-Methylnaphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Acenaphthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Acenaphthylene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Anthracene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(a)anthracene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(a)pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(b)fluoranthene

Air and Emissions		
Technology	Method	Analyte
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(g,h,i)perylene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(k)fluoranthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Chrysene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Fluoranthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Fluorene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Naphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Phenanthrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Pyrene
GC-ECD	EPA TO-4A/TO-10A	PCB-1016
GC-ECD	EPA TO-4A/TO-10A	PCB-1221
GC-ECD	EPA TO-4A/TO-10A	PCB-1232
GC-ECD	EPA TO-4A/TO-10A	PCB-1242
GC-ECD	EPA TO-4A/TO-10A	PCB-1248
GC-ECD	EPA TO-4A/TO-10A	PCB-1254
GC-ECD	EPA TO-4A/TO-10A	PCB-1260
GC-ECD	EPA TO-4A/TO-10A	PCB-1262
GC-ECD	EPA TO-4A/TO-10A	PCB-1268
Preparation	Method	Type
Acid Digestion (Filters, Solid)	EPA 3050B	Inorganics
Soxhlet extraction of PUF	TO-4A/TO-10A	PCBs in Air
Soxhlet extraction of PUF/XAD	TO-13	Semivolatiles in Air
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs

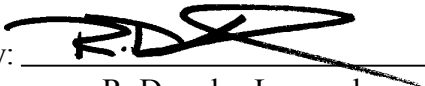


Certificate # L2468

Notes:

- 1) This laboratory offers commercial testing service.

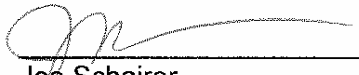
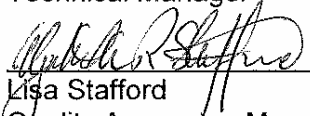
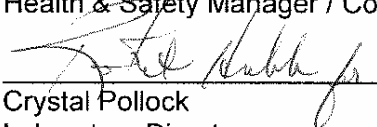


Approved by: 
R. Douglas Leonard
Chief Technical Officer

Date: January 20, 2017

Issued: 1/20/17

Title: Waste Disposal

Approvals (Signature/Date):	
 Richard Kester Technical Manager	 Joe Schairer Health & Safety Manager / Coordinator
 Lisa Stafford Quality Assurance Manager	 Crystal Pollock Laboratory Director
9-9-15 Date	9/9/15 Date
9/11/2015 Date	9/11/15 Date

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Facility Distribution No. <u>Uncontrolled</u>	Sacramento Distributed To: <u>Bids Folder</u>

1. PURPOSE

- 1.1. The purpose of this procedure is to provide details for waste management procedures established in Corporate Environmental Health and Safety Manual (CW-E-M-001) Section 13 that apply to TestAmerica Sacramento.

2. SCOPE

- 2.1. TestAmerica Sacramento is a Large Quantity Generator (LQG), which creates over 1000 kg/month of hazardous waste (CW-E-M-001 Section 13.3). Consequently, TestAmerica Sacramento maintains an Emergency Response Team (ERT) for spills.
- 2.2. Drums must be shipped within 90 days from the date waste is first placed in them. Only non-hazardous soapy water from glassware cleaning is disposed via laboratory sinks to the sewer (CW-E-M-001).

3. SAFETY

- 3.1. Procedures shall be carried out in a manner that protects the health and safety of all associates. When moving a waste drum, always use a drum dolly, and a ramp for palletizing. Pipettes, broken glass and VOAs will be collected and transported in cardboard boxes to prevent lacerations. Drums will be closed and liquid removed from the top to prevent spills in transport. Secondary containment will be used in liquid transport where possible to prevent spills. Doors will be kept closed as much as possible to discourage intrusion. Lab trash buckets should be carried to the drum and the tied bag placed inside with the opening facing up to minimize lacerations or spills. OSHA mandates the use of spring loaded vent bungs on solvent drums to prevent pressurization explosions.
- 3.2. The sump in the waste processing area where lab wastewater is collected in a holding tank is a confined space that requires special training equipment and preparation before entry. Contact the EHSC before entering the sump. The West Sacramento Fire Department may be able to provide the needed fall protection/extraction harness.
- 3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported **immediately** to a laboratory supervisor.
- 3.4. In addition to lab coat and protective safety glasses, the following personal protective equipment is required for specific tasks:
 - 3.4.1. When moving 55-gallon drums, the waste processor will wear steel toe boots and work gloves.

- 3.4.2. When adding bottles to glass drums, crushing glass and lab trash, or cleaning the Vyleater, puncture resistant gloves are required.
- 3.4.3. When operating either the Rampactor or Vyleater, hearing protection is required.
- 3.4.4. Wear a faceshield when pouring HF, concentrated acids or bases, or solvents.
- 3.5. Pollution Prevention
 - 3.5.1. Drain Protectors (magnetized sheets) are used to cover/close the affected storm drain during load out for waste shipment.
 - 3.5.2. The use of sumps and secondary containment throughout the building helps to minimize the potential for contaminants to escape if they are spilled.
 - 3.5.3. Routine testing and monitoring of POTW discharge ensures that we remain in compliance with regulatory limits.
 - 3.5.4. The presence of specially treated hard surfaces in building sumps and work areas prevents leakage of contaminants into the ground.
 - 3.5.5. The use of closed containers and spring loaded vent bungs minimizes air pollution.
 - 3.5.6. Earthquake cables in H-3's and banding pallets minimizes risk of drums falling over.
- 3.6. Waste Management
 - 3.6.1. Contaminated tools and materials (that cannot be cleaned) become hazardous waste, and must be disposed of as the original contaminant.
 - 3.6.2. Special spill response equipment includes drum lifter lever, and UN rated 85-gallon salvage drum. Leaky drums should be placed with the top towards the top of the salvage drum to facilitate final treatment and disposal.

4. DEFINITIONS

- 4.1. Waste: Material no longer useful in the laboratory requiring disposal.
- 4.2. Drum: Cylindrical container for holding waste. Usually 55-gallon, may range from 5- to 85-gallon.
- 4.3. Satellite container: Smaller container near the point of waste generation that is emptied at least once a year.

- 4.4. H-3 Room: Purpose built room for bulk waste storage. They feature fire doors, explosion proof wiring/lighting, separate ventilation, waxed sprinkler heads, and a grated sump to contain waste spills.

5. PROCEDURE

Wastes are either excess sample or analytical wastes. Analytical waste may include reagents (excess, expired, off-specification, or spilled), expired standards and curves, sample extracts, extracted solid or liquid samples, and machine fluids such as coolant and vacuum pump oil. Samples may accumulate in laboratories (filters in Air Tox, VOAs and core tubes in VOA instrument) or may be returned to Sample Control.

- 5.1. Waste Collection: Waste is collected at multiple locations around the facility, then consolidated at either H-3 Waste Accumulation Rooms or the main waste collection area.
- 5.1.1. Waste is collected in the lab as outlined in Section 5.3 through 5.7. Labels are applied to all containers to identify the type of waste, hazardous constituents, collection start date or last emptied date, and the relative hazard of the waste. The National Fire Protection Association (NFPA) diamond system is our preferred method. Pre-made labels for satellite collection containers are available through EH&S, and are maintained on the safety drive, in the one of the "labels" folders.
- 5.1.2. Satellite collection areas and containers are found in all prep and instrument labs. These must be emptied when they are full, or no more than one year after the first waste is put into the container, whichever comes first.
- 5.1.2.1. Instrument satellite waste containers are typically 2.5-liter to 20-liter plastic or plastic-coated glass carboys. When full, these are either dumped into a 55-gallon drum in an H-3 closet or moved directly to the main waste area, depending on the type of waste. Refer to the specific operations SOP.
- 5.1.2.2. Lab satellite trash cans are 5-gallon plastic buckets, with lids. When emptied, these are dumped into a 55-gallon drum in an H-3 closet or the main waste area.
- 5.1.2.3. Glassware satellite collection containers are cardboard boxes that are moved to the main waste area for disposal.
- 5.1.2.4. Storage cabinets in the main waste area.
- 5.1.3. 90-day collection areas are the "H-3" closets located between the metals prep lab and the air toxics prep lab; between sample administration and the organic prep lab; and between the organic prep lab and the low-resolution dioxin prep

lab. There is a 55-gallon drum for incinerable lab trash in general chemistry.

- 5.1.3.1. At the air toxics/metals prep H-3; there are drums for the accumulation of landfill lab trash and acid waste.
- 5.1.3.2. At the sample administration/organic prep H-3, there are drums for the accumulation of LLE waste, landfill and high solvent lab trash.
- 5.1.3.3. At the organic prep/low resolution dioxin prep H-3, there are solvent waste, HPLC waste, high solvent and landfill lab trash, and LLE waste water.

5.2. Sample Disposal Procedure

5.2.1. Create a Container Disposal Report

- 5.2.1.1. In TALS go to Sample Management and click on Internal Chain of Custody.
- 5.2.1.2. Change the Search By box to Current Location
- 5.2.1.3. Click the Current Loc box and select the location you would like to search. Then select OK.

NOTE: Only one location can be searched at a time.

- 5.2.1.4. Select the Search – Append box on the top far right to load all containers on that shelf.
- 5.2.1.5. Once all shelves have been searched, right click on grid and Select Disposable Containers.
- 5.2.1.6. TALS will ask you if you want to bypass the disposal days in the project, select no.
- 5.2.1.7. TALS will check and highlight the containers ready for disposal (may take a few minutes depending on quantity).
- 5.2.1.8. Right click on the grid and click Clear Unselected (may take a few minutes depending on quantity.).
- 5.2.1.9. Select the Export button on the bottom right of the screen.
- 5.2.1.10. Save the spreadsheet to a location naming it appropriately.
- 5.2.1.11. Remove any unwanted columns for easy printing (leave the columns titled Container ID, Lab Sample ID, Client Sample ID, Sample Matrix, Container Type, Storage Location and Current Location).

5.2.1.12. Print this list and store in a known location for employees to pull samples for disposal.

NOTE: These containers have not been disposed of and are only a list of containers ready for disposal.

5.2.2. Remove containers from storage location.

5.2.2.1. The Containers Disposal Report lists the individual containers for disposal, not samples nor entire jobs. Only remove the containers outlined on the disposal report. Ask the Sample Custodian about the status of remnant containers in a job being disposed.

5.2.2.2. Remove the containers to a cart and check them off on the Container Disposal Report. Use “NF” or “0” to designate containers not found. Using only the top shelf of the cart and arranging in neat rows will facilitate scanning out.

5.2.3. Scan the containers into TALS for disposal.

5.2.3.1. Open TALS (menu 40) in your own username and password. Click Sample Management, then select Internal Chain of Custody.

5.2.3.2. Change the Search By box to read Lab Sample ID.

5.2.3.3. Scan every container into TALS.

5.2.3.3.1. Right click on the grid and choose Select Disposable Containers.

5.2.3.3.2. TALS will ask you if you want to Bypass the disposal days in the project. Select ‘no’.

5.2.3.3.3. TALS will generate a list recommending disposal drums for each container. LLE (liquid-liquid extraction water) is the default for containers with the least hazard, regardless of matrix. RCRA Soil is for solids having higher levels of metals or organics. HPLC (high performance liquid chromatography waste) is for aqueous samples with high organic contamination. Acid Drum is for aqueous samples high in metals. PCB (polychlorinated biphenyls) and Dioxins will go to lab packs.

5.3. Liquids: refer to Section 5.14, waste list.

Laboratory analysts are responsible for disposing liquid waste as listed in the specific operations SOPs. These will provide the following direction:

- 5.3.1. Dispose waste liquids to the appropriate container in the lab area.
- 5.3.2. Acid, Liquid-Liquid Extraction (LLE), Solvent, and High Performance Liquid Chromatography (HPLC) wastes:
 - 5.3.2.1. When the satellite collection container is ready to dump, transfer the contents to the appropriate drum in the H-3 Room.
 - 5.3.2.2. When the solvent drum is full (two to six inches from the top), bung the drum and transfer it to the waste collection area in the warehouse. Determine “fullness” with a wooden dowel or dipstick.

Note: There is a \$5 reward for turning in solvent drums that are properly filled.
 - 5.3.2.3. Return with an empty drum to H-3, attach and date a proper in-house label.

WARNING: When using a drum dolly to move either a full or empty 55-gallon drum, you should always face the drum.

- 5.3.3. Deliver other wastes and expired liquids (VOA vials, standards, curves, and extract vials) directly to the main waste room.
- 5.4. Processing Liquid wastes
 - 5.4.1. VOA liquid waste: The VOA analysts collect analytical waste in 4-L plastic jugs. These are transferred to the processor and treated as follows:
 - 5.4.1.1. Add two tablespoons of sodium bicarbonate (baking soda) to each jug to assure pH 6-8.
 - 5.4.1.2. Pour into a blue plastic drum.
 - 5.4.2. VOA vials: VOA analysts collect 40 ml vials of unused sample in cardboard boxes lined with plastic bags. Once they are transferred to the main waste room, the processor performs the following:
 - 5.4.2.1. Place boxes of VOA vials on the narrow table next to the blue Vyleater. Check all fingerscrews on the Vyleater for tightness. Power up by throwing the main power switch to ON. Place a 20-L plastic carboy with 250 g of sodium bicarbonate under the Vyleater with the hose down its throat. Place the rubber mat in front to reduce breakage of fallen VOA vials. Situate a lab trash drum under the right side of the Vyleater. Prepare the drum by powdering the bottom of the drum with sodium bicarbonate to neutralize any hydrochloric acid, followed by a half inch of vermiculite to absorb any liquid.

- 5.4.2.2. Press the left and right green buttons to start the Vyleater. Pull the adjustment wheel from under the Vyleater and insert just below the hopper. Adjust the rollers to the point where they are almost touching. Load about 10 VOA vials into the hopper and raise it to the top, where it dumps into the Vyleater. Readjust the rollers so that they crush the vials being processed. Pull the adjustment wheel from under the Vyleater and insert below hopper to adjust width between grinder wheels to match vial size. The Vyleater will chew down the vials rapidly when the correct width is achieved. Observe that the liquid flows into the carboy and the broken vials are delivered to the lab trash drum. Use the plastic scoop to add vials to the hopper. This drops fewer on the floor. Repeat process until carboy is full or VOAs are all crushed. Wait until liquid stops flowing before changing carboy. Dump full carboys to the blue plastic drum designated for incineration.
- 5.4.2.3. When done, clean the Vyleater: Use the sodium bicarbonate squirt bottle to neutralize any HCL in the hopper and grinder area.

WARNING: DO NOT stick any body parts into the Vyleater. Wear heavy rubber coated gloves when cleaning the Vyleater.

Stop the Vyleater by pressing the Red button. Then switch OFF the power on the wall. Open the panel on the right end. Open the inner panel. Use the long handle scraper to clear most of the glass off the top screen. Pull out the screen while brushing remaining glass into the Vyleater. Lean the top screen against the Vyleater front and repeat for the lower screen. Scrape glass on the bottom towards yourself with the scraper. Use the wide putty knife to pick up and dispose to lab trash drum. Replace screens, close Vyleater and remove lab trash drum to Rampactor for more waste.

- 5.4.3. Process solvent vials in the Vyleater observing the following differences: A 20-L steel drum is used to collect the vials, then pour the waste solvent into a 4-liter polyethylene jug. The 4-liter jug is then emptied into the flammable waste drum in the Advanced Technology H-3. Five gallon open-top drums delivered by analysts may contain bottles (60-250 mls). These are set aside and poured to the collection drum. Neat materials and standards with hazards other than flammability are set aside for lab packs. Wait overnight for vapors to clear before cleaning the Vyleater.
- 5.4.4. Enter the drum in logbook using a sequential number for the year. Document the start date, full date, shipping date and manifest number when shipped. Check the LLE and HPLC drums for pH (6-8 is acceptable).
- 5.4.5. Torque the bungs.

- 5.4.5.1. For waste packaged in blue poly drums (LLE, HPLC, and Acid), use the yellow preset torque wrench to apply 20 foot-pounds to each bung.
- 5.4.5.2. For waste packaged in steel drums, (solvents), replace the bungs with a 2 inch self-venting bung to prevent pressure buildup until shipped. Ship with the original bung torqued with a red pre-set torque wrench to 60 foot-pounds.
- 5.4.6. Apply a preprinted paper in-house label and write in drum number and start date. Make sure that the label is applied to upper third of the drum.
- 5.4.7. Next to the waste label, apply a 100 mm square DOT diamond indicating hazard class: 3 for solvent and HPLC, and 8 for Acid drum.
- 5.4.8. Mark the drum on its top and sides with the drum number (from logbook) and profile number (from waste list or logbook). Place the drum number on the side between RCRA and DOT labels, with the side profile number placed immediately below labels. Write the numbers on the top so that they can be read from the label side. Use a black Sanford "Magnum 44" marker on poly drums and the tops of steel drums. Use a yellow Sanford "Mean Streak" marker to mark steel drum sides.
- 5.4.9. Strap drums with the same waste profile together on pallets. When less than four drums are shipped, they may be strapped with other compatible drums going to the same TSDF (Treatment Storage Disposal Facility). Solvent drums are kept in the small shed in the warehouse parking lot until shipment.
- 5.5. Solids: See high VOA lab trash, land fill lab trash, soil and RCRA Soil on waste list, Section 5.14.
 - 5.5.1. Analysts dispose hazardous lab trash to the appropriate container in the lab area (lab trash bucket, glass box).
 - 5.5.2. When the container is full, the analyst transfers it to the waste collection area in the warehouse or to the lab trash drum located in each H-3. Analyst replaces box or liner in lab trash bucket and marks new start date. Lab trash drums in H-3s are transported to main waste room when full, or not more than 90 days from start date.
 - 5.5.3. The Processor uses orange Rampactor to consolidate lab trash. Rampacting the waste saves on the number of drums used, transport costs, and brings the drums up to the weight listed on the profile.

WARNING: In addition to lab coat and safety glasses, cut and puncture resistant gloves and earmuffs are required when operating the Rampactor. Before operation,

read, follow, and replace the instruction sheets located in the blue capped steel tube welded on the right side below the control levers.

- 5.5.4. Sweep any broken glass out of the Rampactor. Seat drums fully over the bottom metal disk in the chamber. A shard of glass can tilt the drum. When the top disk crushes the edge of the drum, it can become wedged in place.
- 5.5.5. Mark the position for the bottom of the drum. Place a perfect empty open-top drum in the Rampactor chamber. Center it as well as possible over the bottom disk. Close the chamber door and latch. Turn on the electric power switch on the wall to the left of the Rampactor. Push both levers back until the top disk lowers to the level of the drum top. Observe through the tiny slit at the top of the door. Use a flashlight to help see. Pull both handles forward (towards yourself) to stop the ram and disk. Turn off the electric power.
- Open the chamber door. Center the top of the drum under the top disk. Draw a circle around the drum bottom on the Rampactor using a yellow Sanford Meanstreak marker. Allow to dry. This allows you to place the drum more accurately. Remove the empty drum
- 5.5.6. Select an open top steel lab trash drum. Drums from lab areas will have a white paper in-house waste label with start date. Use oldest start date data.
- 5.5.7. Log in the drum. Find the drum logbook under the radio. Turn to the current year and contents page. Turn to the Lab Trash pages. Enter the next drum number in the logbook, on the drum and on the waste label. Mark the profile number on the top of the drum. Use the current date when starting a new drum.
- Note: Do not apply start dates from glass boxes. They are satellite containers and may accumulate for one year. Lab trash drums must be shipped within 90 days of the start date.*
- 5.5.8. Place the drum in the chamber and center on the disk. Close the chamber door, latching top and bottom. Turn on the electric power. Push both levers away from you and release. Carefully watch the disk descend to make sure it clears the edge of the drum. If it appears that the ram will not clear the edge of the drum properly, jerk back the levers. Then follow the procedures in paragraph 5.5.5 and reposition the drum so that it is centered under the ram and disk. The ram is bottomed when the dial reaches the line at 4 o'clock. Pull the bottom lever towards you, if it does not pop out automatically. The dial will indicate full pressure again when the ram and disk reach the top. Pull both levers towards yourself. Turn off the electric power. Open the chamber door.

- 5.5.9. Add more bags of lab trash or boxes of glass, and crush until no more can be added, or it is too heavy to move. Mix light plastic with glass to achieve an average weight. Pull bags from drums by the knot. Do not inhale while your head is in or over the drum.
- 5.5.10. Top off the full drum with vermiculite. It is stored in bags under the pallet racks.
- 5.5.11. Put ring and lid on drum. If the drum top is out of round, open the ring further and drop it 1/3 down the drum. Use the Rampactor carefully to seat the lid, then bring up the ring and fasten with a 5/8-inch bolt (from the box under the radio).
- 5.5.12. Torque drum bolt to 60 foot-pounds.
- 5.5.13. Enter "Full" date in logbook. Mark drum top as in Section 5.4.8.
- 5.6. Soil: Bring excess soil samples from Sample Control to the main waste room 30 days after invoice date, so clients have time to review and request additional tests. Soil must be thermally treated to comply with the California and US Department of Agriculture permits to import soils from quarantine areas. Soils with high metals may be drummed under the RCRA soil profile: (CH94287-RCRA)
VOA analysts bring soil samples after testing. Once soil is received, the processor handles the material as follows:
 - 5.6.1. Log, label and mark a drum for soil (profile CH94287). Line the drum with a 55 gallon plastic liner (kept on top of Tank 1 in the pump room).
 - 5.6.2. Place soil samples in the drum. Glass containers and acetate tubes may be placed in whole. Do not open containers marked as high results for lead. Otherwise pour samples of loose, dry consistency over the other containers to fill the space, and reduce the number of waste drums required. Re-cap empty glass containers and place in high VOA lab trash for thermal treatment.
 - 5.6.3. Remove the contents of metal core tubes with a soil sampling gouge furnished with a wooden handle. Reserve the tubes (segregated by metal) for salvage. Soak the core tubes (sleeves) in 10% Chlorine bleach solution to kill agricultural pests.
 - 5.6.4. Attach copies of soil permits for TSDF and PPQ Form 550 to soil drum.
- 5.7. Non Hazardous solids: Empty solvent jugs, and sample bottles.
Analysts drain empty solvent bottles to appropriate waste drum and air overnight in hood to vent vapors. Keep bottles inverted so heavier than air vapors fall out.

In the Organic Prep and Dioxin Prep laboratories, place bottles in a drum and transport to the main waste room. Clean client sample bottles, with identification removed, are collected in the same drum. In other areas, collect and transport empty bottles on tub carts and place into the open-top drums just to the right of the double yellow doors.

Once the empty solvent jugs and sample bottles are received in the main waste room, the processor handles them as described below:

- 5.7.1. Remove empty bottles from the glass collection drum(s) until even with the top of the drum. Otherwise they will fall and splinter. Place a metal lid over the glass to discourage jumpers. Using a drum truck, move the full drum to the Rampactor.
- 5.7.2. Crush the solvent bottles in the Rampactor and dump the glass into the dumpster using the forklift. The brown glass is borosilicate and melts at a higher temperature than soda ash glass, and is not recycled in California. Glass is crushed the same as lab trash (refer to Section 5.5) with the following exceptions:
 - 5.7.2.1. When client identification can not be removed, sample bottles are crushed with high VOA lab trash to protect client confidentiality.
- 5.7.3. Place the glass drum in the chamber over the bottom disk. Remove the metal lid. Close the chamber door, latching top and bottom. Turn on the electric power. Push both levers away from you and release. Carefully observe the top disk descend to make sure it clears the edge of the drum. If it does not, jerk back the levers, and adjust as in Section 5.5.5.
- 5.7.4. The ram is bottomed when the dial stops advancing. Pull the bottom lever towards you, if it does not pop out automatically. The dial will indicate full pressure again when the ram and disk reach the top. If plastic bottles have crawled around the edge, lower the disk a tad to release them. Pull both levers towards yourself. Turn off the electric power. Open the chamber door. Cover the drum to contain dust. Remove the drum or add more glass and repeat. When the crushed glass reaches the chime one-third from the bottom, stop and place the drum in the drum carrier. It is heavy enough.
- 5.7.5. When the container is one-third full, contents are transferred to a dumpster, and eventually are disposed in the local municipal landfill.
- 5.8. Dumping glass to the dumpster:
 - 5.8.1. Open the rollup door in the main waste room all the way up. Position the drum carrier a few feet back from the doorway. There is a berm at the door so the forks will bounce up and down as your front forklift tires go over.

- 5.8.2. Open the warehouse rollup door by hitting the top button. Let it go all the way up. Back out the forklift and close the rollup door to prevent unauthorized entry.
- 5.8.3. Adjust the forks to maximum width to fit the drum carrier. Bring the forks up to your middle thigh. Brake on. Neutral gear. Stand between the forks, facing the right fork you will adjust first. Disengage the lock at the top of the fork. Place your left thumb at the top of the fork. Pull up the fork with your right hand just enough to relieve the pressure at the elbow of the fork. Shift your body weight to your right foot. Shove the fork out with even pressure at top, fork elbow (with left leg), and mid fork with right hand. Shove all the way out. Turn 180° to face the other fork and repeat. Remember to push fork with inside leg, or it will bind.
- 5.8.4. Drive forklift to main waste door. Raise forks so bottom is at big crossbar (about knee height) to clear bumps. Tilt full forward and lower to meet drum carrier and shove in. Tilt the forks back to retain drum carrier.
- 5.8.5. Open both bat wing arms of drum carrier. The small one on the left will stay open if you drape the locking chain back towards the forklift. The tail of the chain should dip into the chain storage cup for the light spinner chain. Roll the drum in with the drum truck. Push in as far as possible. Bring the locking chain around the front of the drum. Turn the long black latch handle counterclockwise as far as you can without dropping the pawl. Pull the chain taut and place a link between the lugs. Turn the latch handle strongly clockwise to pull in the drum and tighten the chain. The ratchet will hold. (Pull the handle tight and lift the pawl to release when done.)
- 5.8.6. Lift the forks to the crossbar again. Ensure that the top of the mast clears the bottom of the door. Check for traffic and blow horn. Back out. Drive along west edge of basketball court until you can see the street entrance of the parking lot. This ensures you are visible to approaching drivers. Raise the bottom of the forks to the big bolts on the mast to clear the dumpster.
- 5.8.7. Drive in slowly until your side of the drum is just inside the dumpster and a few inches higher.
- 5.8.8. Park the forklift and dismount to the right.
- 5.8.9. Pull the tilt-chain with both hands to swing the drum forward 180 degrees and dump the glass. Do not breathe the glass dust. Beat the bottom of the drum to dislodge plastic bottles that may have wedged. Tilt the drum back so open side with the third chime is up. Repeat as needed.

WARNING: If the wind is blowing dust from the dumping process back towards

you, use a dust filter/nuisance mask, available from the EH&S Coordinator or Hazardous Waste Specialist.

- 5.8.10. Lower the drum carrier all the way to the main waste room floor. Take the forklift back to the front of the warehouse. Close all the doors.
- 5.8.11. Leave the last empty drum in the carrier so you can move it from a standing posture.
- 5.9. Palletizing Drums:
 - 5.9.1. Place a four-way pallet, in good condition, in a parking space next to the roll up door about two inches from the curb. Place the yellow plastic ramp at the other end of the pallet. Using the curb and ramp reduces the strain required to load the drums on the pallets. Use the drum truck to move the pallets and place them so the labels face out at a 45° angle. This orientation allows the labels to be read from the front (in the waste truck) or from the side (in rows awaiting shipment). Place four drums of the same profile together to facilitate counting for the manifest and transfer to treatment facility. Next best is to group drums going to the same treatment facility. Never strap together drums whose contents are not compatible (see Corporate Safety Manual).
 - 5.9.2. Band the drums with half-inch plastic tape so they do not jump off the pallet while loading the waste truck or during shipment. Pull about seventeen feet of tape out of the banding machine and loop clockwise around the four drums on the pallet. The band should be supported by the neck of a poly drum or the chime of a steel drum. Fold ten inches of the loose end back along the length of the band. The steel buckle has two prongs that should be located points up and out. Push the loop of tape through the back of the buckle and over the prong on the same side of the buckle. Fold the running end of the tape back on itself and push it through the buckle and over the prong on its side. Position the tensioner about ten inches left of the buckle with the slots facing up. Grip the red and black handles together to lift the foot, and place the drum side band between the frame and the foot. Place the running band through both the front cutting slot and one of the middle winding slots. Lift the red handle away from the black handle to ratchet the winding slots until the band is tight. Squeeze the red and black handles again to cut the running band, and release the tensioner from the band around the drums.
 - 5.9.3. Using the forklift, stack the pallets two high in the main waste collection area, leaving aisles that extend from the roll-up door to the back of the area between the stacked pallets.
 - 5.9.4. Note: Flammable drums such as Solvent or HPLC may not be stacked.

5.10. Shipping waste:

- 5.10.1. Waste material must be shipped before the oldest drum is 90 days from start date. Ship every drum that is older than 14 days, so that shipments are about 75 days apart. Roughly 4 weeks before shipping, estimate the number of drums and cost of the shipment. Print the “Drum Tally” form (FS02/Public/aaSacSafe/Waste 2/ Drumtally) and count the number of drums for each profile. Estimate the number to be shipped by dividing current number by weeks since last shipment, and multiplying by total weeks from last shipment to next shipment (usually ten).
- 5.10.2. Use the Excel spreadsheet at FS02/Public/aaSacsafe/waste/counts/shipments to estimate weights and disposal costs for the shipment. Request a Purchase Order (PO) through JD Edwards. Use the vendor number 667711 for Clean Harbors, or 544420 for Nexeo if applicable, from the safety “Purchases” folder. The cost center for waste disposal is 320.23000. Enter item one, one shipment, and the full cost of the shipment on one line. Notify the controller, lab director, and regional manager so the large purchase order may be approved as quickly as possible if not approved following day.
- 5.10.3. Contact the waste vendor and request a waste pickup for the week preceding the 90 day date. Send your lab pack inventories and list of lab pack drums for approval. Lab packs must be approved for shipment, which requires at least two weeks. Enter the disposal codes (see Lab Pack Guidelines) on the lab pack inventories, and tops of drums. Photocopy the lab pack inventories double sided to reduce bulk in records, and place the originals on the drums. Use clear packing list envelopes so the drum number is visible.
- 5.10.4. Send an e-mail to Sacramento - All, Sacramento-Corporate Staff and EMLab-Sacramento explaining when the waste truck will block egress by automobile from the back parking lot. On the day of shipment, install the drain mat to prevent the contents of a leaking drum from entering the storm drain. The drain is behind the parking bumper so no one will park right on top of it. The two foot square magnetic mat is kept on the personnel door in the main waste room. Place the spill cart near the drain and familiarize all participants with the blue drum uprighting lever. Use the parking slots next to the ramp as staging area. Replace the vent bungs on the solvent drums with solid shipping bungs. Pull the flammable drums from the small shed and place on pallets. Arrange the pallets on the asphalt in a checkerboard so drums may be placed from both sides. Do a final “as shipped” count.
- 5.10.5. Have the driver back his trailer even with the cryogen fill station closest to the waste ramp. Stand where the driver can see you in the mirror and, when the trailer is within six feet close your hands together to indicate remaining distance, then place palms towards the driver to say “stop here”. Verify the

driver's identity. Give the driver a broom to sweep out any wood chips. Use the forklift to place the pallet jack in the trailer if needed. One fork should be under the handle near the bigger wheels, where the weight is. Pump the pallet jack up a bit so the forks will slide out after delivery. Lift the pallets to the lower crossbar to get them out of the building without hitting the ground. Lift to the second crossbar to fit onto trailer. When only eight feet of trailer remains, take out the pallet jack. Push the remaining pallets in place using the heavy pallet stored by the safety shower. Offer the driver five (5) DOT Placards located in the shelves to the right of the sink.

- 5.10.6. Sign and date the DOT manifest and land ban forms. Check DTSC requirements regularly. Current regulations require only one land ban form per year per profile, but you may get one per shipment. Have the driver sign. Continuation sheets are not signed. Pull your Generator copy. Make two photocopies of the front pages so that we have a completely legible copy for TestAmerica records and one to send to DTSC, PO Box 400, Sacramento, CA 95812-0400 within thirty days of shipment.
- 5.10.7. Stand at the end of the wall when the waste truck leaves, and flag down any traffic that might run into the truck. Send e-mail notification of quarantine soil shipment to USDA. Update drum logbook and waste shipment logbook. Sweep floor, and mop if needed. Vacuum spider webs from flammable solvent shed. Order steel drums, vent caps, vermiculite, yellow bags, placards, clear envelopes, drum labels, smaller drums/pails and blue drums as needed for the next cycle.
- 5.11. Soapy Wash Water: Pipes carry soapy water from laboratory sinks to the 500-gallon tank under the grate in the wash water treatment room. A pneumatic pump propels the water through a bag filter, two carbon filters, and a flow meter to plastic tank three (T-3). Daily procedures are posted on the inside door of the treatment room. The logbook is kept on T-2 next to the broken flow meter. Check that pH in T-3 is between 5 and 11 before discharging.
 - 5.11.1. Fill tank two (T-2) at the beginning of each month. Take VOA and metals samples to Sample Control. Hold the water in T-2 until testing confirms contaminants are within allowed levels. David Alltucker is the TestAmerica Sacramento Project Manager for quote 3203545. Results are kept in the file folder titled "POTW Data" in the waste coordinators desk.
 - 5.11.2. Notify Joe Schairer (EH&S/Facilities) when non-conforming materials are found in the wash water sump. Note: organic materials such as latex paint, propylene glycol, or ethylene glycol may clog the carbon filters and require immediate replacement. The slug may be stored in polyethylene drums or tank one (T-1) until a determination is made.

- 5.11.3. Clean the foot valve and in-line strainer every three months. Replace the bag filter with a new one from the cabinet. Cut a slit in the dirty bag filter and remove the water with a dry/wet vacuum. Force the plastic mouth into an oval to slip off the retention disk. Cut the internal ears off the plastic mouth of the new bag. Follow the directions on the carbon drums when replacing. Attach a plastic standpipe to the outflow stub. Backfill with water overnight. Backflush one day before connecting as final drum. Use the stinger and tap water carboy to re-prime the pneumatic pump. Do not enter the sump.
- 5.12. Waste Area Inspections: The waste processor inspects the main waste storage area, the H-3 waste storage closets, the sample archive room and the waste sheds weekly using the standard inspection forms (aaSacsafe/Waste/Weekly Main Checklist). Results are recorded in the safety logbook for the area.
- 5.13. Records Retention: Records are retained in accordance with the Quality Assurance Manual (QAM), maintained on the Purple Q. Several specific types of records are addressed here for convenience.
 - 5.13.1. Inspection Records: General inspection records are retained for two years. OSHA inspection records, when present, are retained for six years.
 - 5.13.2. Tracking logs, as part of the waste manifest process, are retained indefinitely.
 - 5.13.3. Test data from internal samples is retained per the records retention policy.
 - 5.13.4. Manifests and supporting/associated documentation are retained indefinitely.
 - 5.13.5. Land Disposal Restrictions, as part of the waste manifest process, are filed with and retained with the manifests.
 - 5.13.6. State or Federal reports are retained for seven years.
 - 5.13.7. Other documents. Waste profiles, as part of the manifest process, are retained indefinitely.
- 5.14. TestAmerica Sacramento Waste Profile List
 - 5.14.1. Flammable Solvent (Clean Harbors profile AP336072_143, collected in and shipped to in 55-gallon steel drums): Toluene, Hexane, Acetone, Methanol, Isooctane, and Methylene chloride. Note that Benzene and Diethyl ether wastes are lab packed. These drums weigh more than 350 pounds. OSHA mandated vent caps are used while in shed.
 - 5.14.2. HPLC Solvents & Water (Nexeo profile 44-11304 / Clean Harbors profile AP336073_165) collected in and shipped in 55-gallon blue plastic drums:

Water with acetonitrile, methanol. This waste stream is incinerated, so aqueous samples unsuitable for disposal to LLE Water due to organic contaminants may go here. Examples include “Dark liquor”. The HPLC profiles may be altered in the future to accommodate Ethylene glycol and Propylene glycol coolant solutions, if required.

- 5.14.3. Acid Drum (Nexeo profile 44-11305) shipped in 55-gallon blue plastic drums): Water with nitric acid, hydrochloric acid and sulfuric acid. The Nexeo profile may have RCRA levels of Chromium or Lead. The Clean Harbors profile CH94280 may also contain Arsenic. Nexeo has a \$750 shipment minimum circa 2012.
- 5.14.4. Soil Samples (Clean Harbors profile CH94287 collected and shipped in 55-gallon steel drums): Soil samples, ash, and sludge. This may contain USDA quarantine soils so it must be thermally treated to eliminate crop pests. USDA permit for the receiving facility must be attached to each drum.
- 5.14.5. Laboratory Trash Land Fill (Clean Harbors profile CH94284-LF collected and shipped in 55-gallon steel drums): Contaminated personal protective equipment, used filters, aluminum oxide, calcium silicate, silica gel, absorbed acid spills, and dirty glassware. These drums get Class 8 “Corrosive” DOT label and receive acid vials from the metals lab.
- 5.14.6. Laboratory Trash High VOC (Clean Harbors profile CH94284-HV collected and shipped in 55-gallon steel drums): Contaminated personal protective equipment, used filters, sodium sulfate with Methylene chloride, aluminum oxide, calcium silicate, silica gel, absorbed solvent spills, trash with Chrome 6 contamination and glassware from soil samples or those with ineradicable client identification. These drums carry a Class 9 DOT label and are incinerated.
- 5.14.7. LLE Water: This is clean, unused aqueous samples or aqueous samples that have been extracted with methylene chloride and retain about 2% DCM. Nexeo profile 44-22167 is sent to US Ecology, Beatty, Nevada, for solidification and land fill. (Clean Harbors profile CH94286 for incineration is kept active as backup)
- 5.14.8. All Lab packs: Custom pack guidelines supplied by Clean Harbors will be followed. Packed and shipped in either fiber or steel drums, as appropriate, size depends on volume of waste to be shipped). Required for samples (oil or soil), which contain over 50 ppm polychlorinated biphenyls. TestAmerica typically includes PCB standards even though our source standards are less than 50 ppm. TestAmerica also disposes the vials from advanced technology. The TCDD/TCDF spiking standards are used formulations that may be incinerated as PCB waste. Unused formulations carry “F” codes, which

would preclude treatment in the United States. PCBs must be incinerated at a higher temperature and longer dwell time than other hazardous wastes to prevent dioxin formation. Hydrofluoric acid drums use “pig mat pulp” absorbent to avoid any risk of reaction. Potassium permanganate/nitric acid samples are packed in a poly drum.

- 5.14.9. Mercury Lab packs: Chemical oxygen demand (COD) waste and metallic mercury contaminated with sulfur is packed separately in plastic drums, size depends on volume of waste to be shipped, but typically are 5-gallon pails and sent to Clean Harbors.
- 5.14.10. Standard Lab packs are composed of small containers containing process wastes, samples, or excess chemicals not including mercury or PCBs. They are packed in fiber, plastic or steel drums; size depends on the volume, and shipped to Clean Harbors.
- 5.14.11. Carbon Filters (Clean Harbors profile CH94282) are 55-gallon steel drums filled with activated carbon. They are Siemens ASC-200-2 filters used to remove acetone and dichloromethane from our soapy wash water. The filter is spent when either contaminant nears 1.0 PPM or the drum bulges. Residual water is drained to the lab sink before shipment. Place the drum on the heavy pallet reserved as a pusher and rotate the standpipe counterclockwise to collect the water in a bucket or pan. Recap the 2-inch outlet stub for shipment. Each drum may last one year before replacement in June or September.
- 5.14.12. Empty steel drums (Clean Harbors profile CH101820). These are drums that were damaged during the rampacking process. State law prohibits discarding empty 55-gallon drums in the dumpster.
- 5.14.13. Medical waste (Clean Harbors profile MWCH220382). This includes extracted blood and tissue samples, or first aid supplies contaminated with blood. These are 30 gallon or smaller fiber drums as specified by Clean Harbors.

6. RESPONSIBILITIES

- 6.1. Waste Coordinator (EH&S Manual 13.4.1). The Waste Coordinator is required to take eight-hour initial Hazardous Waste Management training, four-hour annual refreshers and an eight-hour initial Hazardous Material Management training on Department of Transportation regulations and four-hour annual refreshers. The waste coordinator performs weekly audits of the 90 day storage areas. The coordinator maintains and provides the correct in-house waste labels for drums, carboys, and all other waste containers. The coordinator reviews the compatibility of waste containers, establishes

waste stream profiles, and ships hazardous, and biohazard waste. The waste coordinator prepares the following:

- Biennial Report to California Department of Toxic Substances Control,
- Provide information to California tax preparer (Bill Nash) as required.
- Environmental Protection Agency (EPA) generator ID as required.
- Mail copy of manifest to California Department of Toxic Substances Control within thirty days of waste shipment.

6.2. Waste Processor (CSM 13.4.2): Responsibilities of the waste processor include:

- o Prepare dump reports (see 5.2).
Pull containers from Sample Control.
- o Crush extract and VOA vials in the Vyleater,
- o Dump excess samples to the appropriate drum for shipping.
- o Use the Rampactor to crush clean glass and hazardous Laboratory Trash,
- o Pump the soapy water from laboratory sinks daily, and
- o Log, inspect, seal, label, mark and stack the drums on pallets for shipment.

6.3. Analyst: Labels and dates waste containers per the operational SOPs. Ensures that waste containers are emptied and the labels are updated appropriately. Follows the procedures in the operational SOPs, and as outlined here. Remove client identification from bottles. Analysts should report unusually high results to Waste Processor and Waste Coordinator (Greater than RCRA levels, greater than 50 ppm of PCB's or greater than 25 ppb TCDD/TCDF TEQ (toxic equivalent)).

6.4. Project Managers: E-mail the waste processor and sample administration staff within 30 days after invoicing the project if samples or extracts are to be retained longer than 30 days. PM's should enter in TALS non-standard disposal requirements.

7. REFERENCES/CROSS REFERENCES

- 7.1. Operational SOP's.
- 7.2. TestAmerica Environmental Health and Safety Manual (CW-E-H-001).
- 7.3. TestAmerica Sacramento Addendum to the EHSM (WS-PEHS-0002)
- 7.4. Sacramento Quality Assurance Manual, for current facility map (WS-QAM).
- 7.5. TestAmerica Records Retention Policy, (CW-L-P-001).
- 7.6. Manufacturer's instruction manuals for the Rampactor and Vyleater.

7.7. Department of Transportation security plan

7.8. Clean Harbors Self-Pack Guidelines

8. ATTACHMENTS

8.1. No attachments are present.

9. REVISION HISTORY

9.1. WS-EHS-0001 Revision 4.5, Effective 09/18/2015

9.1.1. Updated copyright statement on cover page.

9.1.2. Section 5.14.9 – Added chemical oxygen demand waste to sentence.

9.1.3. Section 5.14.9 – Deleted sentence referencing Heritage recycling for COD waste.

9.1.4. Editorial changes.

9.2. WS-EHS-0001, Revision 4.3, Effective 09/05/2014

9.2.1. Appended to Section 3.1 – “Pipettes, broken glass and VOAs will be collected and transported in cardboard boxes to prevent lacerations. Drums will be closed and liquid removed from the top to prevent spills in transport. Secondary containment will be used in liquid transport where possible to prevent spills. Doors will be kept closed as much as possible to discourage intrusion. Lab trash buckets should be carried to the drum and the tied bag place inside with the opening facing up to minimize lacerations or spills. OSHA mandates the use of spring loaded vent bungs on solvent drums to prevent pressurization explosions.”

9.2.2. Appended to Section 3.2 – “The West Sacramento Fire Department may be able to provide the needed fall protection/extraction harness.”

9.2.3. Changed Section 3.6.2 to – “Special spill response equipment includes drum lifter lever, and UN rated 85-gallon salvage drum. Leaky drums should be placed with the top towards the top of the salvage drum to facilitate final treatment and disposal.”

9.2.4. Replaced previous Section 5.2. with the current Section 5.2 detailing criteria for disposing of sample waste.

9.2.5. Changed first sentence in Section 6.1 – “Waste Coordinator (EH&S Manual

13.4.1). The Waste Coordinator is required to take eight-hour initial Hazardous Waste Management training and four-hour annual refreshers.”

9.3. WS-EHS-0001, Revision 4.3, Effective 9/12/2013

9.3.1. Updated disposal procedures – edits to sections 2.1, 5.2, 5.4.3, 5.10.1, 5.10.2, 5.11.2, 5.12, 5.14.10, and 5.14.

9.3.2. Removed the following items from Responsibilities, Section 6.1, as they were marked “delete” in the text:

- Waste minimization plan, (Delete as we have a waiver).
- Tax return for (Delete as this is done by Bill Nash in Irvine)
 - o Environmental Fee,
 - o Generator Fee,
 - o EPA ID verification and manifest fee,
 - o Yolo County hazardous material fee, and
 - o Sacramento city hazardous materials fees,
- Quarterly reports to the Sacramento Publicly Owned Treatment Works (POTW). Delete as the Sacramento municipal POTW does not require reports.

9.3.3. Removed Revision history items older than 2010. They may be found in previous versions of this SOP.

9.3.4. Editorial changes

9.4. WS-EHS-0001, Revision 4.2, Effective 9/07/2012

9.4.1. Updated disposal procedures

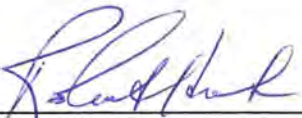
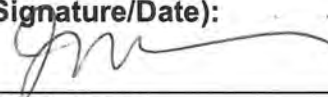
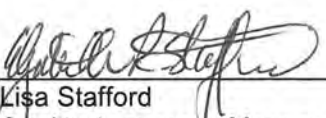
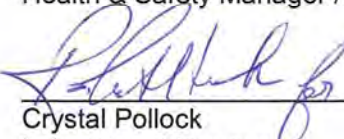
9.4.2. Editorial changes

9.5. WS-EHS-0001, Revision 4.1, Effective 03/18/2011

9.5.1. Editorial changes.

**Title: Per- and Polyfluorinated Substances (PFAS) in Water, Soils,
Sediments and Tissue**

[Method 537 Modified]

Approvals (Signature/Date):			
			
Robert Hrabak Technical Manager	Joe Schairer Health & Safety Manager / Coordinator	Lisa Stafford Quality Assurance Manager	Crystal Pollock Laboratory Manager
1/30/17	1/30/17	1/27/17	1/31/17
Date	Date	Date	Date

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1. SCOPE AND APPLICATION

- 1.1. This procedure describes the analysis of water, soil, sediment and tissue samples for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS).

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
Perfluoro-n-hexanoic acid	PFHxA	307-24-4
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluoro-n-decanoic acid	PFDA	335-76-2
Perfluoro-n-undecanoic acid	PFUdA (PFUnA)	2058-94-8
Perfluoro-n-dodecanoic acid	PFDoA	307-55-1
Perfluoro-n-tridecanoic acid	PFTTrDA	72629-94-8
Perfluoro-n-tetradecanoic acid	PFTeDA (PFTA)	376-06-7
Perfluoro-n-hexadecanoic acid	PFHxDA	67905-19-5
Perfluoro-n-octadecanoic acid	PFODA	16517-11-6
Perfluorinated sulfonic acids (PFSAAs)		
Perfluoro-1-butanedisulfonic acid	PFBS	375-73-5
Perfluoro-1-hexanedisulfonic acid	PFHxS	355-46-4
Perfluoro-1-heptanedisulfonic acid	PFHpS	375-92-8
Perfluoro-1-octanedisulfonic acid	PFOS	1763-23-1
Perfluoro-1-decanedisulfonic acid	PFDS	335-77-3
Perfluorinated sulfonamides (FOSA)		
Perfluoro-1-octanesulfonamide	FOSA	754-91-6
N-ethylperfluoro-1-octanesulfonamide	EtFOSA	4151-50-2
N-methylperfluoro-1-octanesulfonamide	MeFOSA	31506-32-8
Perfluorinated sulfonamidoacetic acids (FOSAA)		
N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2991-50-6
N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA	2355-31-9
Fluorotelomer sulfonates (FTS)		
1H,1H,2H,2H-perfluorooctane sulfonate (6:2)	6:2 FTS	27619-97-2
1H,1H,2H,2H-perfluorodecane sulfonate (8:2)	8:2 FTS	39108-34-4

Abbreviations in parenthesis are the abbreviations listed in Method 537, where they differ from the abbreviation used by the laboratory's LIMS.

Sample results for PFOA may also be reported as APFO, at the request of the client. (See Section 12.7)

- 1.2. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	250 mL	2.0 ng/L – 100 ug/L	2.0 ng/L to 400 ug/L
Soil/Sediment/Tissue	5 g	0.2 ug/kg – 20 ug/kg	0.2 to 100 ug/kg

- 1.3. Due to poor chromatographic peak shape which degraded with repeated injections for Perfluoro-1-octanesulfonamidoamide (FOSSA), this analyte is no longer included in the method.
- 1.4. The procedure for the analysis of water samples via direct aqueous injection (DAI) for a subset of the list in Section 1.1 using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a SCIEX 5500 is described in the Appendix to this SOP.
- 1.5. When undertaking projects for the Department of Defense (DoD) and/or the Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, “Federal Program Requirements” must be checked and incorporated.

2. SUMMARY OF METHOD

- 2.1. Water samples are extracted using a solid phase extraction (SPE) cartridge, unless EtFOSA and MeFOSA are requested. PFAS are eluted from the cartridge with an ammonium hydroxide/methanol solution.
- 2.2. Soil samples are extracted with a KOH/methanol solution using an orbital shaker for 3 hours followed by sonication for 12 hours. The mixture is centrifuged and the solvent filtered.
- 2.2.1. Optional cleanups may include sample freezing and/or cleanup by SPE cartridge, unless EtFOSA and MeFOSA are requested.
- 2.3. The final 80:20 methanol:water extracts are analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using 20 mM ammonium acetate/water and methanol. The mass spectrometer detector is operated in the electrospray (ESI) negative ion mode for the analysis of PFAS.
- 2.4. An isotope dilution technique is employed with this method for most compounds of interest. The isotope dilution analytes (IDA) consist of carbon-13 labeled analogs, oxygen-18 labeled analogs or deuterated analogs of the compounds of interest, and

they are spiked into the samples at the time of extraction. This technique allows for the correction for analytical bias encountered when analyzing more chemically complex environmental samples. The isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have a labeled analog are quantitated by the IDA method using a closely related labeled analog.

- 2.5. Quantitation by the external standard method is employed for the IDA analytes and assumes a proportional relationship between the initial calibration and the analyte in the extract. The ratio of the peak response to mass or concentration injected is used to prepare a calibration curve. Peak response is measured as the area of the peak.

3. DEFINITIONS

- 3.1. PFCAs: Perfluorocarboxylic acids
- 3.2. PFSA: Perfluorinated sulfonates
- 3.3. FOSA: Perfluorinated sulfonamides
- 3.4. PFOA: Perfluorooctanoic acid (may also be written PHOA).
- 3.5. APFO: Ammonium perfluorooctanoate
- 3.6. PFOS: Perfluorooctane sulfonate (may also be written PHOS)
- 3.7. MPFOA: Perfluoro-n-[1,2,3,4-¹³C₄]octanoic acid. Carbon-13 labeled PFOA
- 3.8. MPFOS: Perfluoro-1-[1,2,3,4-¹³C₄]octanesulfonate. Carbon-13 labeled PFOS
- 3.9. PTFE: Polytetrafluoroethylene (e.g., Teflon®)
- 3.10. SPE: Solid phase extraction.
- 3.11. PP: Polypropylene
- 3.12. PE: Polyethylene
- 3.13. HDPE: High density polyethylene
- 3.14. IDA: Isotope dilution analytes
- 3.15. Further definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

4. INTERFERENCES

- 4.1. PFAS have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed below in Section 6.
- 4.2. To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.
- 4.3. PTFE products can be a source of PFOA contamination. The use of PTFE in the procedure should be avoided or at least thoroughly tested before use. Polypropylene (PP) or polyethylene (PE, HDPE) products may be used in place of PTFE products to minimize PFOA contamination.
 - 4.3.1. Standards and samples are injected from polypropylene autosampler vials with polypropylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.
 - 4.3.2. Random evaporation losses have been observed with the polypropylene caps causing high IDA recovery after the vial was punctured and sample re-injected. For this reason, it is best to inject standards and samples once in the analytical sequence.
 - 4.3.3. Teflon-lined screw caps have detected PFAS at low concentrations. Repeated injection from the same teflon-lined screw cap have detected PFNA at increasing concentration as each repeated injection was performed, therefore, it is best to use polypropylene screw caps.
- 4.4. Volumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFOA. These items should be labeled for use only with similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.
- 4.5. Commercial sources of PFOS, PFHxS, PFOA, and other PFAS may produce several peaks in the chromatogram. These adjacent peaks are either completely resolved or not resolved but with a profound deflection that can be resolved during peak integration. The later of the peaks matches the retention time of the single labeled PFAS peak. In general, earlier peaks are branched isomers and are not a result of peak splitting. When reference standards of technical mixtures of specific PFAS are available, they should be used to ensure that all appropriate peaks are included during peak integration. Refer to Section 7, Reagents, for the available technical mixtures utilized by this SOP.

- 4.6. The phenomenon of the linear and branched isomers of PFOS exists for other PFAS, such as PFHxS and PFBS. Thus, in an attempt to reduce PFOS bias, it is required that m/z 449>80 transition be used as the quantitation transition.
- 4.7. Both branched and linear PFAS can potentially be found in the environment. For the compounds that give rise to more than one peak, all the chromatographic peaks observed in the standard and/or sample must be integrated and the areas included.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns

- 5.1.1. Preliminary toxicity studies indicate that PFAS could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFAS must be handled in the laboratory as hazardous and toxic chemicals.
- 5.1.2. Exercise caution when using syringes with attached filter disc assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.
- 5.1.3. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.
- 5.1.4. Eye protection that satisfies ANSI Z87.1 (as per the TestAmerica Corporate Safety Manual), laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.
- 5.1.5. Perfluorocarboxylic acids are acids and are not compatible with strong bases.
- 5.1.6. The use of vacuum systems presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior

to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.7. Glass containers are not to be used for “tumbling” soil samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material ⁽¹⁾	Hazards	Exposure Limit ⁽²⁾	Signs and Symptoms of Exposure
Acetic Acid (3-2-1)	Corrosive Poison Flammable	10 ppm-TWA 15 ppm-STEL	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage to the upper respiratory tract. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent damage, including blindness. Brief exposure to 5000 PPM can be fatal.
Hexane (2-3-0)	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Can cause pain and severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause deep ulcerations to skin, permanent eye damage, circulatory failure and swallowing may be fatal.

Material ⁽¹⁾	Hazards	Exposure Limit ⁽²⁾	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Potassium Hydroxide	Corrosive Poison		Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
Sodium Hydroxide	Corrosive Poison	2 mg/cm ³ (Ceiling)	Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

- 6.1. 15 mL polypropylene test tubes with polypropylene screw caps.
- 6.2. 50 mL graduated plastic centrifuge tubes.
- 6.3. 125 mL HDPE wide-mouth bottles.
- 6.4. 16 oz or 500 mL HDPE bottles with HDPE screw caps.
- 6.5. Analytical balance capable of accurately weighing to the nearest 0.0001g, and checked for accuracy each day it is used in accordance with WS-QA-0041.
- 6.6. Syringe filter, Millipore Millex-HV 0.45 um, or equivalent. Do not use PTFE type filters.
- 6.7. 300 µL autosampler vials, polypropylene, with polypropylene screw caps, Waters PN 1860004112, or equivalent.
- 6.8. SPE columns
 - 6.8.1. Phenomenex Strata SPE C18, 6 mL, 500 mg, part number 8B-S002-HCH,

Waters SepPak C18, 1 to 10g, or equivalent.

- 6.8.2. Waters Oasis WAX 150 mg/6 cc (PN 186002493) for the cleanup of solids.
- 6.8.3. Waters Oasis WAX 500 mg/6 cc (PN 186004647) for extraction of PFAS from aqueous sample.
- 6.8.4. Phenomonex Gemini 3 μ m C18 110Å, 50 X 2 mm, Part No. 00B-4439-B0.
- 6.8.5. Phenomonex Luna 5 μ m C18(2) 100Å, 30 X 3 mm, Part No. 00A-4252-Y0.
- 6.9. Granulated carbon.
- 6.10. Vacuum manifold for Solid Phase Extraction (SPE).
- 6.11. Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc.). These should be disposable where possible, or marked and segregated for high-level versus low-level use.
- 6.12. Bottle rotating apparatus for soil extractions.
- 6.13. Glass fiber filter, Whatman GF/F, catalog number 1825 090 or equivalent.
- 6.14. Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS) – Either of the instruments described below, or equivalent, may be used for this method. Both HPLC are equipped with a refrigerated autosampler, an injection valve, and a pump capable of variable flow rate. The use of a column heater is required to maintain a stable temperature throughout the analytical run. Data is processed using Chrom Peak Review, version 2.1 or equivalent.
 - 6.14.1. Waters LC/MS/MS

This consists of a Waters Acquity UPLC system interfaced with a Waters Quattro Premiere tandem mass spectrometer. The instrument control and data acquisition software is MassLynx version 4.1, or equivalent.

 - 6.14.1.1. Analytical column: Waters Acquity UPLC BEH C18 1.7 μ m, 3.0 mm x 150 mm, Part No. 186004690,
 - 6.14.1.2. PFAS Isolator column, Waters Acquity UPLC BEH Shield RP-18, 1.7 μ m, 2.1 mm x 50 mm, PN 186004476, or equivalent. This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.

6.14.2. SCIEX LC/MS/MS

This system consists of a Shimadzu HPLC interfaced with a SCIEX 5500 Triple Quad MS. The instrument control and data acquisition software is SCIEX Analyst, version 1.6.3 or equivalent.

6.14.2.1. Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.

6.14.2.2. Phenomenex Gemini C₁₈ 3 um, 3.0 mm x 100 mm, Part No. 00D-4439-Y0, or equivalent.

6.14.2.3. PFAS Isolator column, Phenomenex Luna C₁₈ 5 um, 50 mm x 4.6 mm, part no. 00B-4252-E0 or equivalent. This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.

6.15. Preventive and routine maintenance is described in the table below

HPLC/MS/MS Preventative Maintenance
<u>As Needed:</u> Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes.
<u>Daily (When in use)</u> Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that corona and multiplier are functioning.
<u>Semi-Annually</u> Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable.
<u>Annually</u> Vacuum system components including fans and fan covers.

HPLC/MS/MS Preventative Maintenance
Clean/replace fan filters, if applicable.

7. REAGENTS AND STANDARDS

- 7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 7.1.1. Acetic acid, glacial
 - 7.1.2. Ammonium acetate (20 mM in water)
 - 7.1.3. Ammonium hydroxide (NH₄OH), 0.3% in methanol
 - 7.1.4. Hexane
 - 7.1.5. Hydrochloric acid (HCl), 2.0 M solution in water
 - 7.1.6. Methanol
 - 7.1.7. Potassium hydroxide (KOH), 0.4% in methanol
 - 7.1.8. Ottawa Sand
 - 7.1.9. Sodium hydroxide (NaOH), 0.1N, in water
 - 7.1.10. Water, Nanopure or Millipore, must be free of interference and target analytes
- 7.2. Standards
- 7.2.1. PFAS are purchased as high purity solids (96% or greater) or as certified solutions. Standard materials are verified compared to a second source material at the time of initial calibration. The solid stock material is stored at room temperature or as specified by the manufacturer or vendor.
 - 7.2.2. If solid material is used for preparing a standard, stock standard solutions are prepared from the solids and are stored at $4 \pm 2^{\circ}\text{C}$. Stock standard solutions should be brought to room temperature before using. Standards are monitored for signs of degradation or evaporation. Standard solutions must be replaced at least annually from the date of preparation.

- 7.2.3. PFBS, PFHxS, PFHpS, PFOS, PFDS, MPFOS, and many other PFAS are not available in the acid form, but rather as their corresponding salts, such as sodium or potassium. The standards are prepared and corrected for their salt content according to the equation below.

$$\text{Mass}_{\text{acid}} = \text{Measured Mass}_{\text{salt}} \times \text{MW}_{\text{acid}} / \text{MW}_{\text{salt}}$$

Where: MW_{acid} is the molecular weight of PFAA

MW_{salt} is the molecular weight of the purchased salt.

- 7.2.4. For example, the molecular weight of PFOS is 500.1295 and the molecular weight of NaPFOS is 523.1193. Therefore, the amount of NaPFOS used must be adjusted by a factor of 1.046

7.3. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of PFOA and PFOS stock solutions in 80% methanol/water. The calibration stock solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.4. Initial Calibration (ICAL) Levels (ng/mL)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
Perfluoroalkylcarboxylic acids (PFCAs)							
PFBA	0.5	1.0	5.0	20	50	200	400
PFPeA	0.5	1.0	5.0	20	50	200	400
PFHxA	0.5	1.0	5.0	20	50	200	400
PFHpA	0.5	1.0	5.0	20	50	200	400
PFOA	0.5	1.0	5.0	20	50	200	400
PFNA	0.5	1.0	5.0	20	50	200	400
PFDA	0.5	1.0	5.0	20	50	200	400
PFUdA	0.5	1.0	5.0	20	50	200	400
PFDoA	0.5	1.0	5.0	20	50	200	400
PFTTrDA	0.5	1.0	5.0	20	50	200	400
PFTeDA	0.5	1.0	5.0	20	50	200	400
PFHxDA	0.5	1.0	5.0	20	50	200	400
PFODA	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonic acids (PFSAs)							
PFBS	0.5	1.0	5.0	20	50	200	400
PFHxS *	0.5	1.0	5.0	20	50	200	400
PFHpS	0.5	1.0	5.0	20	50	200	400
PFOS *	0.5	1.0	5.0	20	50	200	400
PFDS	0.5	1.0	5.0	20	50	200	400

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
Perfluorinated sulfonamides (FOSA)							
FOSA	0.5	1.0	5.0	20	50	200	400
EtFOSA	0.5	1.0	5.0	20	50	200	400
MeFOSA	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonamidoacetic acids (FOSAA)							
EtFOSAA	0.5	1.0	5.0	20	50	200	400
MeFOSAA	0.5	1.0	5.0	20	50	200	400
Fluorotelomer sulfonates (FTS)							
6:2 FTS	0.5	1.0	5.0	20	50	200	400
8:2 FTS	0.5	1.0	5.0	20	50	200	400
Labeled Isotope Dilution Analytes (IDA)							
MPFBA	50	50	50	50	50	50	50
M5PFPeA	50	50	50	50	50	50	50
MPFHxA	50	50	50	50	50	50	50
MPFHpA	50	50	50	50	50	50	50
M4PFOA	50	50	50	50	50	50	50
MPFNA	50	50	50	50	50	50	50
MPFDA	50	50	50	50	50	50	50
MPFUdA	50	50	50	50	50	50	50
MPFDoA	50	50	50	50	50	50	50
MPFHxS	50	50	50	50	50	50	50
MPFOS	50	50	50	50	50	50	50
M8FOSA	50	50	50	50	50	50	50
D5-EtFOSA	50	50	50	50	50	50	50
D3-MeFOSA	50	50	50	50	50	50	50
D5-EtFOSAA	50	50	50	50	50	50	50
D3-MeFOSAA	50	50	50	50	50	50	50
M2-6:2FTS	50	50	50	50	50	50	50
M2-8:2FTS	50	50	50	50	50	50	50

* both branched and linear isomers are used.

Note: Sample extracts are in 80% MeOH/H₂O.

FOSAA may be added to the mix and are added at the same concentration as FOSA.

Note- The above calibration limits are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.

- 7.4.1. A technical (qualitative) grade PFOA standard is analyzed initially, then after initial calibration when a new column is installed or when significant changes are made to the HPLC parameters. This solution is used as a reference for the PFOA isomers (branched and linear) retention times.

7.5. Initial Calibration Verification Standard (ICV).

A second source solution for PFAS is purchased from the same vendor; the PFC-MXB contains most of the target analytes in this mixture and is used as an ICV. A few compounds are not available in this mixture, may not be available as another lot, and are not available from another vendor. For these analytes only, a second analyst may prepare a second source standard from the same source as the ICAL to produce an ICV. The recommended concentration of the ICV standard should be in the mid-range of the calibration curve. The concentration may be adjusted if the initial calibration levels are changed or altered. The IS is added at a fixed concentration of 50 ng/mL.

7.6. LCS/Matrix PFC Spike Solution, 500 ng/mL.

The PFC spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at a concentration of 500 ng/mL in methanol.

7.7. PFC Isotope Dilution Analyte Solution, 1000 ng/mL.

The PFC-IS solution is prepared by diluting all labeled PFAS to produce a solution containing each compound at a concentration of 1000 ng/mL in methanol.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Soil samples are collected in pre-cleaned 250 mL HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6°C for shipment to the laboratory.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6°C. Water samples must be extracted within 14 days of collection. Soil samples must also be extracted within 14 days of collection. Tissue samples must be extracted within 1 year of collection if stored at -20°C. Extracts must be refrigerated at 0 - 6°C, and analyzed within 40 days from extraction.

NOTE: As of this writing, Method 537 provides for a 14 day holding time for water samples preserved with Trizma buffer. The scientific literature indicates that perfluorinated substances are highly persistent in the environment. TestAmerica Sacramento has conducted holding time studies that support a 14 day holding time for aqueous samples with and without Trizma preservation. TestAmerica Denver has conducted stability studies indicating that medium- and low-level solutions of PFOA are stable for at least three months in polystyrene and polypropylene plastics at 0-6°C. The 14 /40 day holding times given above are based on the holding time study and general EPA convention for the holding time of extractable organic compounds in water and soil.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability (IDOC)

The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

- 9.2. Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC program document (WS-PQA-003) for further details of the batch definition.
 - 9.2.1. The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a matrix spike/matrix spike duplicate (MS/MSD), a laboratory control sample (LCS) and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count toward the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD, an LCSD may be substituted if batch precision is required by the program or client. In the event that multiple MS/MSDs are run with a batch due to client requirements, the additional MS/MSDs do not count toward the maximum 20 samples in a batch.
- 9.3. One method blank (MB, laboratory reagent blank) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. For solid samples, the method blank is an aliquot of Ottawa sand. The method blank is processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside of the control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria.
 - 9.3.1. If the MB produces a peak within the retention time window of any of the analytes, determine the source of the contamination and eliminate the interference before processing samples.
 - 9.3.2. The method blank must not contain any analyte at or above the reporting limit, or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.
 - 9.3.3. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.
 - 9.3.4. Re-extraction and reanalysis of samples associated with an unacceptable

method blank is required when reportable concentrations are determined in the samples.

- 9.3.5. Refer to WS-PQA-003 for further details of the corrective actions.
- 9.3.6. Projects performed under the auspices of the DOD/DOE must meet QSM specific criteria for method blanks. Results are acceptable if the blank contamination is less than $\frac{1}{2}$ of the reporting limit for each analyte, or less than $\frac{1}{10}$ of the regulatory limit, or less than $\frac{1}{10}$ of the sample result for the same analyte, whichever is greater. If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. If contamination remains, the contaminated samples should be re-prepared and reanalyzed with a new MB and batch-specific QC samples.
- 9.4. A laboratory control sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water for aqueous samples and Ottawa sand for solids) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside of the control limits. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See WS-PQA-0003 for specific acceptance criteria. The control limits for the LCS are stored in TALS.
- 9.5. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside of the control limits must be within the control limits in the LCS. Corrective actions must be documented on a nonconformance memo, then implemented when recoveries of any spiked analyte are outside of the control limits provided by TALS or by the client.
- 9.6. A duplicate control sample (LCSD or DCS) may be added when insufficient sample volume is provided to process an MS/MSD pair, or is requested by the client. The LCSD is evaluated in the same manner as the LCS. See WS-PQA-003 for specific acceptance criteria.
- 9.7. Initial calibration verification (ICV) – When available, a second source standard is analyzed with the initial calibration curve. The concentration should be at the mid range of the curve.

Corrective actions for the ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards.

9.8. Isotope Dilution Analytes

9.8.1. The IDA solution is added to each field and QC sample at the time of extraction, as described in Section 11. As described in Section 7, this solution consists of isotopically labeled analogs of the analytes of interest.

9.8.2. IDA recoveries are flagged if they are outside of the acceptance limits (25–150%). Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-noise ratio is greater than 10:1.

9.8.2.1. Evaluate data quality for usability, flag and submit a non-conformance memo for any analytes outside of the recovery criteria, and report if data is deemed not adversely effected.

9.8.2.2. Re-extraction of samples should be performed if the signal-to-noise for any IDA is less than 10:1 or if the IDA recoveries fall below 10%.

9.8.2.2.1. Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-P-003 “Calibration Curves and Selection of Calibration Points”.

10.2. Routine instrument operating conditions are listed in the table in Section 11.6.1.

10.3. Instrument Tuning

Instrument tuning is done initially when the method is first developed and thereafter as needed to maintain the sensitivity and selectivity of the method. Tuning is done by infusing each individual compound (native and IDA) into the mobile phase using a tee fitting at a point just before the entrance to the electrospray probe. The responses for the parent and daughter ions for each compound are observed and optimized for sensitivity and resolution. Mass assignments are reviewed and calibrated if necessary.

The mass assignments must be within ± 0.5 amu of the values shown in the table in Section 11.5.1.

- 10.4. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include, but are not limited to new columns or pump seals. A new calibration is not required after minor maintenance.
- 10.5. With the exception of the circumstances delineated in policy CA-Q-P-003, it is not acceptable to remove points from a calibration curve. In any event, at least five points must be included in the calibration curve. Average Response Factor and linear fit calibrations require five points, whereas Quadratic (second order) calibrations require six points.
- 10.6. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.
- 10.7. All units used in the calculations must be consistently uniform, such as concentration in ng/mL.
- 10.8. Initial Calibration
 - 10.8.1. A number of analytical standards of different analyte concentrations are used to generate the curve. Each standard is injected once to obtain the peak response for each analyte at each concentration. These standards define the working range of the analysis.
 - 10.8.1.1. A minimum of five analytical standards is used when using average response factor and/or linear calibration fits.
 - 10.8.1.2. A minimum of six analytical standards is used when a quadratic fit is used to generate the curve.
 - 10.8.2. Calibration is by average response factor, linear fit, or by quadratic fit. Quadratic fit is used for the analyte if the response is non-linear.
 - 10.8.2.1. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated by isotope dilution must be $< 35\%$ for the curve to be valid.
 - 10.8.2.2. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated by IDA must be $< 50\%$ for the curve to be valid.
 - 10.8.2.3. For linear fit, the intercept of the line must be less than $\frac{1}{2}$ the reporting limit, and the coefficient of determination (r^2) must be

greater than or equal to 0.990 for the curve to be considered valid (or the correlation coefficient (r) > 0.995).

10.9. Calibration Curve Fits

10.9.1. Linear regression or quadratic curves may be used to fit the data to a calibration function. Detailed descriptions and formulas for each fitting type can be found in SOP CA-Q-P-003, "Calibration Curves and Selection of Calibration Points".

10.9.2. The linear curve uses the following function:

Equation 1

$$y = bx + c$$

Where:

$$\begin{aligned} y &= \frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)} \\ x &= \text{concentration} \\ b &= \text{slope} \\ c &= \text{intercept} \end{aligned}$$

10.9.3. The quadratic curve uses the following function:

Equation 2

$$y = ax^2 + bx + c$$

Where y, x, b, and c are the same as above, and a = curvature.

10.9.4. The external standard method uses the following equation:

Equation 3

$$\text{Response Factor} = \frac{\text{Peak Area}}{\text{Concentration of Solution (ng / mL)}}$$

10.9.5. Evaluation of Calibration Curves

The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- The absolute value of the intercept of a regression line (linear or non-linear) at zero response must be less than the reporting limit.
- There should be no carryover at or above 1/2 MRL after a high CAL standard.

If these criteria are not met, instrument conditions and standards will be checked, and the ICAL successfully repeated before continuing.

10.9.6. Weighting of Calibration Points

In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at

the low end of the calibration. Because accuracy at the low end of the curve is very important for this analysis, it is preferable to increase the weighting of the lower concentration points. 1/concentration or 1/x weighting is encouraged. Visual inspection of the line fitted to the data is important in selecting the best fit.

10.10. Initial Calibration Blank (ICB)

10.10.1. Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of 80:20 methanol:water blank.

10.10.2. The result for the calibration blank must be less than the reporting limit.

10.10.3. If the ICB is greater than the reporting limit then the source of contamination must be identified and any necessary cleaning completed, and then the instrument should be recalibrated.

10.11. Initial Calibration Verification (ICV)

10.11.1. Following the ICAL and the ICB, an ICV standard obtained from a different source or vendor than the ICAL standards is analyzed. This ICV standard is a mid-range standard.

10.11.2. The recovery for the ICV must meet the appropriate following criteria;

10.11.2.1. The native analyte must be within or equal to 60-140% for all native analytes quantitated by isotope dilution.

10.11.2.2. The native must be within or equal to 50-150% for all native analytes quantitated by internal standard (ie, those compounds that do not have corresponding isotopically labelled analogs).

10.11.2.3. The IDA must be within or equal to 50-150%.

10.11.3. See Section 9.7 for corrective actions in the event that the ICV does not meet the criteria above.

10.12. Continuing Calibration Verification (CCV)

Analyze a CCV at the beginning of a run, the end of a run, and after every 10 samples to determine if the calibration is still valid. The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required. The CCVs are usually at the mid-level range of the curve and should vary throughout the run. The curve and ICV do not need to be run every day. To start an analytical run a CCV can be analyzed and if it meets acceptance criteria a run can be started. In addition the low standard in the curve must be analyzed and must be within $\pm 50\%$ of the expected value.

- 10.12.1. The recovery for the CCV standards must be equal to or within 60-140% for all natives quantitated by isotope dilution and equal to or within 50% to 150% for all natives quantitated by internal standard. The recovery for the IDA must be within or equal to 50-150%.
- 10.12.2. If this is not achieved, the instrument has drifted outside the calibration limits. The instrument must be recalibrated.

11. PROCEDURE

- 11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Non-Conformance Memo (NCM). The NCM process is described in more detail in SOP WS-QA-0023. The NCM shall be filed in the project file and addressed in the case narrative.
- Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.2. Water Sample Preparation

- 11.2.1. Visually inspect samples for the presence of settled and or suspended sediment. If sediment is apparent, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration. File an NCM noting the need for filtration.

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

- 11.2.2. Measure 250 mL of each sample using a graduated cylinder and pour into a labeled 16 oz polyethylene (HDPE) bottle. *Prepare separate aliquots of 1.0 mL if EtFOSA and/or MeFOSA are requested.*
- 11.2.3. Prepare additional aliquots of a field sample for the MS/MSD, if requested.
- 11.2.4. Prepare two 250 mL aliquots of HPLC-grade water for the method blank and LCS (or 1.0 mL if EtFOSA and/or MeFOSA are requested.)
- 11.2.5. Spike the LCS and MS/MSD (if requested) with 0.020 mL (20 uL) of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 40 ng/L. If EtFOSA and/or MeFOSA are required, increase the amount of LCS Matrix PFC spike solution added to 200 uL.

- 11.2.6. Add 0.025 mL (25 uL) of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial. If EtFOSA and/or MeFOSA are requested increase the amount of IDA added to 125 uL.
- 11.2.7. If EtFOSA and/or MeFOSA are requested adjust the final volume (FV) of these aliquots to 5.0 mL with MeOH. Vortex each sample. Then transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.
- 11.3. Solid Phase Extraction (SPE) of Aqueous Samples
(Do not perform SPE clean up if EtFOSA and/or MeFOSA are requested.)
The automated Zymark Auto-Trace Workstation can be used as long as the program follows these conditions and passes the background check.
- 11.3.1. Condition the SPE cartridges (Waters WAX, 500 mg/6 cc) by passing the following without drying the column.
NOTE: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*
- WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.**
- 11.3.2. Wash with 5.0 mL of 0.3% NH₄OH/methanol.
- 11.3.3. Wash with 5.0 mL of 0.1N NaOH/water. Close valve when ~ 200 uL remains on top to keep column wet. After this step, the columns cannot go dry until the completion of loading and rinsing samples.
- 11.3.4. Appropriately label the columns and add the reservoir to the column.
- 11.3.5. Add samples to the columns and with vacuum, pull the entire 250 mL aliquot of the sample through the cartridge at rate of approximately 2 to 5 drops per second.
- 11.3.6. After the final loading of the sample but before completely passed through the column, rinse the SPE column with 1 mL of water.
- 11.3.7. After the sample and water rinse has completely passed through the cartridge, allow the column to dry well with vacuum for 15 minutes.

11.4. SPE Column Wash of Aqueous Samples with Hexane

- 11.4.1. Load the first 5 mL of hexane to soak for five minutes, then elute to waste.
- 11.4.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).
- 11.4.3. Allow the column to dry with vacuum for 5 to 10 minutes. Columns must be dried before continuing.

11.5. SPE Elution of Aqueous Samples – using 15 mL polypropylene test tubes as receiving tubes in the SPE manifold.

- 11.5.1. Rinse samples bottles with 5 mL of 0.3% NH₄OH/methanol and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.
- 11.5.2. Repeat sample bottle to column reservoir rinse and cartridge elution with a second 5 mL aliquot of 0.3% NH₄OH/methanol. The total collection should be approximately 10 mL.

11.6. Extract Concentration for Aqueous Samples

- 11.6.1. Concentrate each sample under a gentle stream of nitrogen to near dryness.
- 11.6.2. Add 400 uL of methanol to each extract, soak and vortex to mix well to reconstitute extract.
- 11.6.3. Add 100 uL of water to each sample for a final solvent composition of 80:20 methanol:water and vortex to mix the mixture well.
- 11.6.4. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.
- 11.6.5. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.

11.7. Soil, Sediment and Tissue Sample Preparation and Extraction

- 11.7.1. Visually inspect soil samples for homogeneity.
- 11.7.2. Weigh a representative 5 g aliquot of soil, sediment or tissue sample into a 50 mL HDPE wide-mouth bottle. Weigh additional sample amounts for the matrix spike and matrix spike duplicate analyses if they are requested. *(Prepare separate aliquots if EtFOSA and/or MeFOSA are requested.)*

- 11.7.3. For the method blank and LCS matrix, use 5 g each of Ottawa sand.
- 11.7.4. Spike the LCS and MS/MSD (if requested) with 0.040 mL (40 uL) of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 4.0 ng/g.
- 11.7.5. Add 0.05 mL (50 uL) of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.
- 11.7.6. Cap the bottles and allow the spike to settle into the sample matrix. Gently shake the bottles to mix the spike into the matrix.
- 11.7.7. Add 20 mL of 0.4% KOH/methanol to each sample.
- 11.7.8. Shake each sample on an orbital shaker at room temperature for 3 hours.
- 11.7.9. Following the shaking, extract the samples in an ultrasonic water bath for an additional 12 hours.
- 11.7.10. After the completion of extraction, centrifuge each sample at 3500 rpm for 15 minutes.
- 11.7.11. Collect and decant the KOH/methanol extract to a new 50 mL centrifuge tube.
- 11.7.12. Add another 2 mL of 0.4% KOH/methanol solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.
- 11.7.13. Combine the rinsate to the first corresponding tubes.
- 11.7.14. To the final KOH/methanol extract, add 2 mL of water to each. (*Omit this step if EtFOSA and/or MeFOSA are requested.*)
- 11.7.15. Concentrate the KOH/methanol/water extract under nitrogen to less than 2 mL, and dilute with water to 15 mL final volume. (*Omit this step if EtFOSA and/or MeFOSA are requested.*)
- 11.7.16. Acidify with 80 uL of glacial acetic acid, and mix the contents well with vortex mixer. Check the pH to ensure pH is between 6 to 8.
- 11.7.17. Centrifuge at 3500 rpm for 15 minutes.
- 11.8. Solid Cleanup by SPE
(Do not perform SPE clean up if EtFOSA and/or MeFOSA are requested. Proceed directly to Section 11.11)
- 11.8.1. Set up WAX 150 mg/6 cc SPE columns for sample cleanup using vacuum

manifold.

- 11.8.2. Condition the SPE cartridges by passing the following without drying the column.

***NOTE:** The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

- 11.8.3. Wash with 5.0 mL of 0.3% NH₄OH/methanol.

- 11.8.4. Wash with 10 mL of 0.1N NaOH/water. Close valve when ~ 500uL remains on top of column to keep column wet. *After this step, the columns cannot go dry until the completion of loading and rinsing samples.*

- 11.8.5. Add extracts to the columns and with vacuum, pull the entire extracts through the cartridge at rate of approximately 3 to 5 drops per second.

- 11.8.6. Rinse the sample tube with 5 mL of water and add to the SPE column.

- 11.8.7. Dry the columns with vacuum for 15 minutes.

11.9. SPE Column Wash of Solid Samples with Hexane

- 11.9.1. Load the first 5 mL of hexane to soak for five minutes, and elute to waste.

- 11.9.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

- 11.9.3. Allow the column to dry with vacuum for 10 minutes. Columns must be dried before continuing.

11.10. SPE Elution of Solid Samples – using 15 mL polypropylene test tube as receiving tube in the SPE manifold.

- 11.10.1. Elute the analytes from the cartridge with 5.0 mL of 0.3% NH₄OH/methanol, first allow the solution to soak for 5 minutes, and then elute into the 15 mL collection tube.

- 11.10.2. Add a second 5 mL of 0.3% NH₄OH/methanol and collect the eluant into the collection tube. The total collection should be approximately 10 mL.

11.11. Extract Concentration for Solid Samples

- 11.11.1. Concentrate each sample under a gentle stream of nitrogen to near dryness.
- 11.11.2. Add 800 uL of methanol to each extract, soak and vortex to mix well to reconstitute extract.
- 11.11.3. Add 200 uL of water to each sample for a final solvent composition of 80:20 methanol:water and vortex to mix the mixture well.
- 11.11.4. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.
- 11.11.5. Seal the vial with a polypropylene screw cap. *Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.*

11.12. Other types of Sample Cleanup

- 11.12.1. Freezing technique to remove lipids.
If samples contain lipids then freeze the methanolic extract and QC extracts at -20°C for at least 1 hour. Collect the solvent layer.
- 11.12.2. Cleanup with graphitized carbon which may also be used to remove organic interferences.
 - 11.12.2.1. Add 100 mg of graphitized carbon to each sample extract and QC extracts.
 - 11.12.2.2. Shake vigorously and then let sit for 10 minutes.
 - 11.12.2.3. Centrifuge each sample for 2 minutes at 1000 rpm.
 - 11.12.2.4. Decant the solvent layer
- 11.12.3. Concentrate each sample under a gentle stream of nitrogen to approximately 0.5 mL.
- 11.12.4. Add 200 uL of Millipore water to each sample.
- 11.12.5. Bring the final volume to 1.0 mL with methanol (80% methanol/20% water).

11.12.6. Filter through a 0.45 µm syringe filter as necessary or centrifuge the extracts to obtain a clear supernant. *Note: Syringe filter should be checked for PFAS background before using.*

WARNING: Application of excessive pressure has caused disc filters to rupture and burst. Exercise discretion when filtering.

11.13. Instrument Analysis

Suggested operating conditions are listed below for the Waters LCMS system:

Recommended Instrument Operating Conditions					
HPLC Conditions (Waters Acquity UPLC)					
Column (Column temp = 50°C)	Waters Acquity BEH 1.7µm C18, 3.0 x 150 mm				
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol				
Gradient Program	Time	%A	%B	Curve	Flow Rate mL/min.
	0	98	2	6	0.30
	1	98	2	6	0.30
	2	50	50	6	0.30
	12	10	90	6	0.30
	12.5	0	100	6	0.30
	16	0	100	6	0.30
	16.2	98	2	6	0.30
	Maximum pressure limit = 15,000 psi				
Injection Size	10 µL (fixed amount throughout the sequence)				
Run Time	~20 minutes				
Mass Spectrometer Interface Settings (Quattro Premier XE)					
MS Interface Mode			ESI Negative Ion		
Capillary (kV)			2.8		
Cone (V)			Varies from 8.0 to 65		
Extractor (V)			3		
Source Temp			135°C		
Desolvation Temp			350°C		
Cone Gas (nitrogen) Flow			25 L/hour		
Desolvation Gas (nitrogen) Flow			1100 L/hour		

Recommended Instrument Operating Conditions						
Mass Spectrometer Scan Settings (Quattro Premier XE)						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
PFBA	Native analyte	213 > 169	0.02	8	10	1
13C4-PFBA	IDA	217 > 172	0.02	12	10	1
PFPeA	Native analyte	263 > 219	0.02	10	10	2

Recommended Instrument Operating Conditions						
<i>Mass Spectrometer Scan Settings (Quattro Premier XE)</i>						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
13C5PFPeA	IDA	268 > 223	0.02	11	9	2
PFBS	Native analyte	299 > 80	0.02	45	35	2
PFHxA	Native analyte	313 > 269	0.02	10	10	3
13C2-PFHxA	IDA	315 > 270	0.02	12	9	3
PFHpA	Native analyte	363 > 319	0.02	10	10	4
13C4-PFHpA	IDA	367 > 322	0.02	12	10	4
PFHxS	Native analyte	399 > 80	0.02	55	35	4
18O2-PFHxS	IDA	403 > 84	0.02	50	40	4
PFOA	Native analyte	413 > 369	0.02	12	10	5
13C4PFOA	IDA	417 > 372	0.02	12	12	5
PFHpS	Native analyte	449 > 80	0.02	60	38	5
PFNA	Native analyte	463 > 419	0.02	16	10	7
13C5-PFNA	IDA	468 > 423	0.02	12	12	7
PFOS	Native analyte	499 > 80	0.02	60	40	6
13C4-PFOS	IDA	503 > 80	0.02	35	48	6
PFDA	Native analyte	513 > 469	0.02	16	12	8
813C2-PFDA	IDA	515 > 470	0.02	14	12	8
PFUdA	Native analyte	563 > 519	0.02	15	12	10
13C2-PFUdA	IDA	565 > 520	0.02	14	12	10
PFDS	Native analyte	599 > 80	0.02	74	48	10
FOSA	Native analyte	498 > 78	0.02	40	32	9
13C8-FOSA	IDA	506 > 78	0.02	48	32	9
PFDoA	Native analyte	613 > 569	0.02	15	14	11
13C2-PFDoA	IDA	615 > 570	0.02	16	12	11
PFTrDA	Native analyte	663 > 619	0.02	12	12	11
PFTeDA	Native analyte	713 > 669	0.02	12	18	11
PFHxDA	Native analyte	813 > 769	0.02	18	15	12
PFODA	Native analyte	913 > 869	0.02	20	16	12
13C2-PFTeDA	IDA	715 > 670	0.02	15	15	11
13C2-PFHxDA	IDA	815 > 770	0.02	18	15	12
EtFOSA	Native analyte	526 > 169	0.02	45	36	11
d5EtFOSA	IDA	531 > 169	0.02	40	30	11
MeFOSA	Native analyte	512 > 169	0.02	45	25	11
d5MeFOSA	IDA	515 > 169	0.02	40	30	11
EtFOSAA	Native analyte	584 > 419	0.02	35	20	9
d5-EtFOSAA	IDA	589 > 419	0.02	30	25	9
MeFOSAA	Native analyte	570 > 419	0.02	30	28	9
d5-MeFOSAA	IDA	573 > 419	0.02	30	25	9
6:2FTS	Native analyte	427 > 407	0.02	40	30	5
M2-6:2FTS	IDA	429 > 409	0.02	40	28	5

Recommended Instrument Operating Conditions						
<i>Mass Spectrometer Scan Settings (Quattro Premier XE)</i>						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
8:2FTS	Native analyte	527 > 507	0.02	40	28	8
M2-8:2FTS	IDA	529 > 509	0.02	40	28	8

Recommended Instrument Operating Conditions				
<i>Retention Times & Quantitation (Quattro Premier XE)</i>				
Native Compounds	Native RT (minutes)	IS analog	IS RT (minutes)	Quantitation Method
PFBA	4.77	13C4-PFBA	4.79	Isotope Dilution
PFPeA	5.90	13C5-PFPeA	5.92	Isotope Dilution
PFBS	6.01	18O2-PFHxS	8.64	IS calculation
PFHxA	7.22	13C2-PFHxA	7.25	Isotope Dilution
PFHpA	8.57	13C4-PFHpA	8.59	Isotope Dilution
PFHxS	8.60	18O2-PFHxS	8.64	Isotope Dilution
PFOA	9.80	13C4-PFOA	9.83	Isotope Dilution
PFHpS	9.80	13C4-PFOS	10.90	IS calculation
PFNA	10.88	13C5-PFNA	10.92	Isotope Dilution
PFOS	10.87	13C4-PFOS	10.90	Isotope Dilution
PFDA	11.82	13C2-PFDA	11.86	Isotope Dilution
FOSA	12.41	13C8-FOSA	12.46	Isotope Dilution
PFDS	12.57	13C4-PFOS	10.90	IS calculation
PFUdA	12.62	13C2-PFUdA	12.66	Isotope Dilution
PFDoA	13.32	13C2-PFDoA	13.34	Isotope Dilution
PFTTrDA	13.91	13C2-PFDoA	13.34	IS calculation
PFTeDA	14.39	13C2-PFDoA	13.34	IS calculation
PFHxDA	15.16	13C2-PFDoA	13.34	IS calculation
PFODA	15.57	13C2-PFDoA	13.34	IS calculation
EtFOSA	14.13	d-EtFOSA	14.11	Isotope Dilution
MeFOSA	13.73	d-MeFOSA	13.73	Isotope Dilution
EtFOSAA	12.63	D5-EtFOSAA	12.62	Isotope Dilution
MeFOSAA	12.3	D3-MeFOSAA	12.28	Isotope Dilution
6:2FTS	10.08	M2-6:FTS	10.08	Isotope Dilution
8:2FTS	11.95	M2-8:FTS	11.95	Isotope Dilution

Recommended Instrument Operating Conditions				
HPLC Conditions (Shimadzu HPLC)				
Column (Column temp = 45°C)	Phenomenex Gemini 3 µm C18 110Å, 50 X 2 mm			
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol			
Gradient Program	Time	%A	%B	Flow Rate mL/min.
	0	90	10	0.60
	0.1	45	55	0.60
	4.5	1	99	0.60
	4.95	1	99	0.60
	5	90	10	0.60
	Maximum pressure limit = 5,000 psi			
Injection Size	2 µL (fixed amount throughout the sequence)			
Run Time	~6.6 minutes			
Mass Spectrometer Interface Settings (SCIEX 5500)				
MS Interface Mode			ESI Negative Ion	
Ion Spray Voltage (kV)			4.5	
Entrance Potential (V)			5	
Declustering Potential (V)			25	
Desolvation Temp			600°C	
Curtain Gas			35 psi	
Collision Gas			8 psi	

Recommended Instrument Operating Conditions								
<i>Mass Spectrometer Scan Settings (SCIEX 5500)</i>								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	RT (Min)
PFBA	Native analyte	212.9 > 169	0.011	-5	-12	-25	-31	1.74
13C4 PFBA	IDA	217 > 172	0.011	-5	-12	-25	-31	1.74
PFBS	Native analyte	298.9 > 80	0.011	-6	-58	-55	-37	1.76
PFBS_2	Native analyte	298.9 > 99	0.011	-5	-40	-55	-12	1.76
PFPeA	Native analyte	262.9 > 219	0.011	-7	-12	-20	-34	1.99
13C5 PFPeA	IDA	267.9 > 223	0.011	-7	-12	-20	-35	1.99
PFHxA	Native analyte	313 > 269	0.011	-5	-12	-25	-37	2.25
13C2 PFHxA	IDA	315 > 270	0.011	-5	-12	-25	-38	2.25
PFHpA	Native analyte	363 > 319	0.011	-6	-12	-25	-41	2.57
13C4 PFHpA	IDA	367 > 322	0.011	-6	-12	-25	-41	2.57
PFHxS	Native analyte	399 > 80	0.011	-12	-74	-60	-43	2.59
18O2 PFHxS	IDA	403 > 84	0.011	-12	-74	-60	-43	2.59
6:2 FTS	Native analyte	427 > 407	0.011	-7	-32	-50	-10	2.91
M2-6:2FTS	IDA	429 > 409	0.011	-7	-32	-50	-10	2.91
PFOA	Native analyte	413 > 369	0.011	-6	-14	-25	-44	2.93
PFOA_2	Native analyte	413 > 169	0.011	-5	-22	-25	-12	2.93

Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings (SCIEX 5500)								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	RT (Min)
13C4_PFOA	IDA	417 > 372	0.011	-6	-14	-25	-44	2.93
PFHpS	Native analyte	449 > 80	0.011	-11	-88	-65	-46	2.94
PFNA	Native analyte	463 > 419	0.011	-6	-14	-25	-47	3.29
13C5_PFNA	IDA	468 > 423	0.011	-6	-14	-25	-48	3.29
PFOS	Native analyte	499 > 80	0.011	-9	-108	-65	-50	3.29
PFOS_2	Native analyte	499 > 99	0.011	-5	-58	-65	-12	3.29
13C4_PFOS	IDA	503 > 80	0.011	-9	-108	-65	-50	3.29
PFDA	Native analyte	513 > 469	0.011	-6	-16	-25	-51	3.65
13C2_PFDA	IDA	515 > 470	0.011	-6	-16	-25	-51	3.65
8:2_FTS	Native analyte	527 > 507	0.011	-7	-40	-50	-15	3.65
M2-8:2_FTS	IDA	529 > 509	0.011	-7	-40	-50	-15	3.65
PFOSA	Native analyte	498 > 78	0.011	-8	-85	-60	-50	3.7
13C8_PFOSA	IDA	506 > 78	0.011	-8	-85	-60	-50	3.7
N-MeFOSAA	Native analyte	570 > 419	0.011	-7	-36	-40	-15	3.82
d3-MeFOSAA	IDA	573 > 419	0.011	-7	-36	-40	-15	3.82
PFDS	Native analyte	599 > 80	0.011	-11	-118	-85	-54	3.96
PFUdA	Native analyte	563 > 519	0.011	-7	-18	-25	-54	3.97
13C2_PFUdA	IDA	565 > 520	0.011	-7	-18	-25	-54	3.97
N-EtFOSAA	Native analyte	584 > 419	0.011	-7	-36	-50	-15	3.99
d5-EtFOSAA	IDA	589 > 419	0.011	-7	-36	-50	-15	3.99
MeFOSA	Native analyte	512 > 169	0.011	-7	-37	-75	-15	4.21
d3MeFOSA	IDA	515 > 169	0.011	-7	-37	-75	-15	4.21
PFDoA	Native analyte	613 > 569	0.011	-5	-18	-25	-54	4.3
13C2_PFDoA	IDA	615 > 570	0.011	-5	-18	-25	-54	4.3
EtFOSA	Native analyte	526 > 169	0.011	-7	-37	-75	-15	4.39
d5EtFOSA	IDA	531 > 169	0.011	-7	-37	-75	-15	4.39
PFTrDA	Native analyte	663 > 619	0.011	-7	-20	-25	-54	4.56
PFTeDA	Native analyte	713 > 669	0.011	-2	-22	-25	-10	4.79
PFTeDA_2	Native analyte	713 > 169	0.011	-7	-36	-25	-30	4.79
13C2_PFTeDA	IDA	715 > 670	0.011	-2	-22	-25	-10	4.79
PFHxDA	Native analyte	813 > 769	0.011	-7	-24	-25	-54	5.25
13C2_PFHxDA	IDA	815 > 770	0.011	-7	-24	-25	-54	5.25
PFODA	Native analyte	913 > 869	0.011	-7	-26	-25	-54	5.55

11.13.1. Tune and calibrate the instrument as described in Section 10.

11.13.2. A typical run sequence is as follows:

- Primer (A number of primers are injected for conditioning of the instrument before analysis, especially when the instrument was idled or changed from a different analysis).
- Blank
- Calibration Curve

- ICB
- ICV
- MB
- LCS
- LCSD (if applicable)
- Sample 1
- Sample 1 MS (if applicable)
- Sample 1 MSD (if applicable)
- Sample 2 (up to sample 10 before next CCV)
- CCV
- Up to 10 samples.
- End sequence with CCV

12. CALCULATIONS

12.1. If the concentration of the analyte ions exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. It may be necessary to dilute samples due to matrix.

12.2. Qualitative Identification

12.2.1. The retention times of PFAS with labeled standards must be the same as that of the labeled IDA's to within 0.05 min. For PFAS with no labeled standards, the RT must be within ± 0.3 minutes of the ICV and CCV standards. *Note: The IS RT and native RT may be offset by 0.02 to 0.04 minutes.*

12.3. The ICAL established in Section 10 is used to calculate concentrations for the extracts.

12.4. Extract concentrations are calculated as below. The first equation applies to the linear fit, the second to the quadratic line fit.

Equation 4 Concentration, ng/mL = $\frac{y - c}{b}$

Equation 5 Concentration, ng/mL = $\frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$

Where:

$$y = \frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$$

$$x = \text{concentration}$$

a = curvature
b = slope
c = intercept

12.5. Water Sample Result Calculation:

Equation 6 Concentration, ng/L = $\frac{C_{ex} V_t}{V_o}$

Where:

C_{ex} = Concentration measured in sample extract (ng/mL)
 V_t = Volume of total extract (mL)
 V_o = Volume of water extracted (L)

12.6. Soil Sample Result Calculation:

Equation 7 Concentration, ng / g = $\frac{C_{ex} V_t}{W_s D}$

Where ng/g = µg/kg and:

C_{ex} = Concentration measured in sample extract (ng/mL)
 V_t = Volume of total extract (mL)
 W_s = Weight of sample extracted (g)
 D = Fraction of dry solids, which is calculated as follows:

$$\frac{100 - \% \text{ moisture in sample}}{100} \quad (\text{for dry weight result})$$

12.7. IDA Recovery Calculation:

Equation 8 % Recovery = $\frac{RF_{ex} A_t}{Amt}$

Where ng/g = µg/kg and:

RF_{ex} = Response Factor for IDA compound
 A_t = Area response for IDA compound
 Amt = Amount spike of IDA

12.8. If results are to be reported as ammonium perfluorooctanoate (APFO), instead of PFOA, apply a multiplier of 1.0406 to the sample results to correct for the molecular weight differences between PFOA and APFO or this adjustment can be made during the preparation of the standards used for calibration. (Use one, not both.)

12.9. Raw data, calibration summaries, QC data, and sample results are reviewed by the analyst. These must also be reviewed thoroughly by a second qualified person. See

the Data Review Policy (WS-PQA-0012). These reviews are documented on the Data Review Checklist.

13. METHOD PERFORMANCE

- 13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.
- 13.2. Method Detection Limit
The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.
- 13.3. Initial Demonstration of Capability (IDOC)
Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.
- 13.4. The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-0006 and policy WS-PQA-003.

14. POLLUTION PREVENTION

- 14.1. All waste will be disposed of in accordance with Federal, State and Local regulations.
- 14.2. Solid phase extraction used for water samples greatly reduces the amount of solvent used compared to liquid-liquid extraction.
- 14.3. Standards and reagents are purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.
- 14.4. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 14.5. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless waste is being transferred.

- 14.6. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out:

- 15.1. Assorted test tubes, autovials, syringes, filter discs and cartridges. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the hazardous waste – landfill steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.
- 15.2. Extracted soil samples, used sodium sulfate, paper funnel filters, glass wool, thimbles, and extracted solids contaminated with solvents. Dump these materials into an orange contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the incineration steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.
- 15.3. Waste Methanol. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to no less than six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.
- 15.4. Mixed water/methanol waste from soil extraction. Collect the waste in the HPLC waste carboy. When full, or after no more than one year, dump into the blue plastic HPLC collection drum in the H3 closet. When the drum is full, to no less than six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.
- 15.5. Aqueous acidic waste from the LCMS instrument contaminated with methanol. This is collected in a 1-gallon carboy at the instrument. When the carboy is full, or after no more than one year, it is emptied into the blue plastic HPLC collection drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.
- 15.6. Autovials contaminated with methanol. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After all autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methanol and other contaminants to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES

- 16.1. Cheryl Moody, Wai Chi Kwan, Johnathan W. Martin, Derek C. G. Muir, Scott A. Mabury, "Determination of Perfluorinated Surfactants in Surface Water Samples by Two Independent Analytical Techniques: Liquid Chromatography/Tandem Mass Spectrometry and ¹⁹F NMR," Analytical Chemistry 2001, 73, 2200-2206.
- 16.2. John Giesy et al., "Accumulation of Perfluorooctane Sulfonate in Marine Mammals", Environmental Science & Technology, 2001 Vol. 35, No. 8, pages 1593-1598.
- 16.3. U.S. EPA, "Residue Chemistry Test Guidelines, OPPTS 860.1340, Residue Analytical Method", EPA 712-C-95-174, August 1995.
- 16.4. STL Denver White Paper DEN-W-LC-002, "Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, September 5, 2003.
- 16.5. STL Denver White Paper DEN-W-LC-003, "Addendum A to Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, August 6, 2003.
- 16.6. STL Denver White Paper DEN-W-LC-004, "Method Validation Study for Analysis of Perfluorooctanoic Acid in Waters by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, January 26, 2005.
- 16.7. Waters application note; "Acquity UPLC System for Quantifying Trace Levels of Perfluorinated Compounds with an Acquity PFC Analysis Kit", Peter J. Lee, Evan T. Bernier, Gordon T. Fujimoto, Jeremy Shia, Michael S. Young, and Alice J. Di Gloia, Waters Corporation, Milford, MA. USA.
- 16.8. US EPA, "Method 537 - Determination of Selected Perfluorinated alkyl acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)", Version 1.1, September 2009, J.A. Shoemaker, P.E. Grimmett, B.K. Boutin, EPA Document #: EPA/600/R-08/092

17. METHOD MODIFICATIONS

Modifications from Method 537 are detailed below:

- 17.1. Water sample containers are not preserved with Trizma.
- 17.2. The method has been modified to address soil/solid matrices. The extraction holding time is set at 14 days.

- 17.3. The analyte list has been expanded. The number of labeled analytes has been expanded as well to improve quantitation.
- 17.4. The reporting limits differ as they are all set at one consistent value.
- 17.5. Calibration levels differ from the referenced method.
- 17.6. More labeled analytes are fortified into the samples prior to the extraction process. Most target analytes are quantitated against a labeled analyte.
- 17.7. There is no symmetry requirement.
- 17.8. Calibration, both initial and continuing, has different acceptance criteria due to the longer list of analytes, and the use of IDA/external standard quantitation.
- 17.9. The eluents and HPLC configuration differs. As a result the final extract is in 80:20 methanol: water.
- 17.10. The LCS and MS/MSD are spiked at one concentration and do not rotate between a low to high levels.
- 17.11. Samples are not checked for residual chlorine or pH.
- 17.12. A different SPE cartridge (Waters OASIS WAX) is used for the extraction process. As a result solvents and elution procedures are different.

18. ATTACHMENTS

- 18.1. Attachment 1 - Analysis of Perfluorinated Compounds (PFAS) in Water via Direct Aqueous Injection (DAI).

19. REVISION HISTORY

Revisions to Attachment 1 are documented in the attachment.

- 19.1. WS-LC-0025, Revision 2.2, Effective 02/02/2017
 - 19.1.1. Title, revised to read, "Per- and Polyfluorinated Substances (PFAS) in Water, Soils, Sediments and Tissue"
 - 19.1.2. Many occurrences of the acronym "PFC" have been replaced with "PFAS". If the acronym "PFC" is used within the laboratory as an identifier, it has been kept in the SOP in that context.
 - 19.1.3. References to polypropylene (PP) containers for use within the laboratory have been changed to polyethylene (HDPE), as these are the containers in use.

- 19.1.4. Section 2.5, deleted the following (redundant): “Isotope dilution technique is employed with this method for most compounds of interest. The IDA’s consist of carbon-13 labeled analogs or oxygen-18 labeled analogs of the compounds of interest, and they are spiked to the samples at the time of extraction. This technique allows correction for analytical bias encountered when analyzing more chemically complex environmental samples, because the isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have a labeled analog are quantitated by IDA method using a closely related labeled analog.”
- 19.1.5. Section 4.5, revised to read, “Commercial sources of PFOS, PFHxS, PFOA, and other PFAS may produce several peaks in the chromatogram. These adjacent peaks are either completely resolved or not resolved but with a profound deflection that can be resolved during peak integration. The later of the peaks matches the retention time of the single labeled PFAS peak. In general, earlier peaks are branched isomers and are not a result of peak splitting. When reference standards of technical mixtures of specific PFAS are available, they should be used to ensure that all appropriate peaks are included during peak integration. Refer to Section 7, Reagents, for the available technical mixtures utilized by this SOP.”
- 19.1.6. Section 6.1, removed: “8 mL test tubes, screw thread, with caps.”
- 19.1.7. Sections 6.10 (Isolator Column), 6.13 (Waters UPLC system), 6.14 (Columns + Shimadzu HPLC) reorganized as Section 6.14 and subsections.
- 19.1.8. Section 6.14 (after reorganization) changed to read, “Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS) – Either of the instruments described below, or equivalent, may be used for this method. Both HPLC are equipped with a refrigerated autosampler, an injection valve, and a pump capable of variable flow rate. The use of a column heater is required to maintain a stable temperature throughout the analytical run. Data is processed using Chrom Peak Review, version 2.1 or equivalent.
- 19.1.9. Section 6.14.1, inserted to read, “Waters LC/MS/MS
This consists of a Waters Acquity UPLC system interfaced with a Waters Quattro Premiere tandem mass spectrometer. The instrument control and data acquisition software is MassLynx version 4.1, or equivalent.”
- 19.1.10. Section 6.14.1.1, inserted to read, “Analytical column: Waters Acquity UPLC BEH C18 1.7 μ m, 3.0 mm x 150 mm, Part No. 186004690”
- 19.1.11. Section 6.14.1.1, inserted to read, “PFAS Isolator column, Waters Acquity

UPLC BEH Shield RP-18, 1.7 μ m, 2.1 mm x 50 mm, PN 186004476, or equivalent. This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.”

19.1.12. Section 6.14.2, inserted to read, “SCIEX LC/MS/MS

This system consists of a Shimadzu HPLC interfaced with a SCIEX 5500 Triple Quad MS. The instrument control and data acquisition software is SCIEX Analyst, version 1.6.3 or equivalent.”

19.1.13. Section 6.14.2.1, inserted to read, “Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.”

19.1.14. Section 6.14.2.2, inserted to read, “Phenomenex Gemini C₁₈ 3 μ m, 3.0 mm x 100 mm, Part No. 00D-4439-Y0, or equivalent.”

19.1.15. Section 6.14.2.3, inserted to read, “PFAS Isolator column, Phenomenex Luna C₁₈ 5 μ m, 50 mm x 4.6 mm, part no. 00B-4252-E0 or equivalent. This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.”

19.1.16. Section 10.11.2.2, changed to read, “The native must be within or equal to 50-150% for all native analytes quantitated by internal standard (ie, those compounds that do not have corresponding isotopically labelled analogs).”

19.1.17. Section 11.2, moved as paragraph under Section 11.1. Renumbered Section 11.

19.1.18. Section 11.2.1, appended the following, “File an NCM noting the need for filtration.”

19.1.19. Section 11.13, MS Scan Settings Tables – under the comments section, replaced compound names with “native analyte”. Correlation between compound names and abbreviations is at the beginning of the SOP.

19.1.20. Editorial changes.

19.2. WS-LC-0025, Revision 2.1, Effective 12/09/2016

19.2.1. Section 8.2, second sentence, changed 7 days to 14 days.

19.2.2. Note following Section 8.2, changed to read: “NOTE: As of this writing, Method 537 provides for a 14 day holding time for water samples preserved with Trizma buffer. The scientific literature indicates that perfluorinated substances are highly persistent in the environment. TestAmerica Sacramento has conducted holding time studies that support a 14 day holding time for

aqueous samples with and without Trizma preservation. TestAmerica Denver has conducted stability studies indicating that medium- and low-level solutions of PFOA are stable for at least three months in polystyrene and polypropylene plastics at 0-6C. The 14 day/40 day holding times given above are based on the holding time study and general EPA convention for the holding time of extractable organic compounds in water and soil.”

19.2.3. Section 17.1, removed the second sentence “Holding time has been changed to 7 days for extraction.”

19.2.4. Editorial Changes

19.3. WS-LC-0025, Revision 2.0, Effective 11/18/2016

19.3.1. Replace “internal standard” with “IDA” throughout SOP.

19.3.2. Section 4.7, changed last sentence of paragraph to include “...in the standard **and/or sample** must...”.

19.3.3. Section 6.9.4, added - “Phenomenex Gemini 3 µm C18 110Å, 50 X 2 mm, Part No. 00B-4439-B0.”

19.3.4. Section 6.9.5, added – “Phenomenex Luna 5 µm C18(2) 100Å, 30 X 3 mm, Part No. 00A-4252-Y0.”

19.3.5. Section 6.14, added – “SCIEX 5500 Triple Quad MS. The system utilizes Chrom Peak Review, version 2.1 or equivalent.”

19.3.6. Section 7.4.1, added - “A technical (qualitative) grade PFOA standard is analyzed after an initial calibration initially, when a new column is installed or when significant changes are made to the HPLC parameters. This solution is used as a reference for the PFOA isomers (branched and linear) retention times.”

19.3.7. Section 11.14.1, HPLC settings, gradient time 1, corrected flow rate from 0330 mL/min to 0.30 mL/min.

19.3.8. Section 11.14.1, MS Settings, removed the following lines for Waters instrument and added additional Table for Shimadzu HPLC.

PFPeS	Perfluoropentanesulfonate	3749 > 80	0.02	55	32	3
PFNS	Perfluorononanesulfonate	549 > 80	0.02	65	54	8
PFDoS	Perfluorododecanesulfonate	699 > 80	0.02	80	55	11

19.3.9. Section 15, added – “Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.”

- 19.3.10. Editorial changes.
- 19.4. WS-LC-0025, Revision 1.9, Effective 05/27/2016
 - 19.4.1. Editorial Changes.
 - 19.4.2. Added Attachment 1.
- 19.5. WS-LC-0025, Rev 1.8, Effective 05/19/2016
 - 19.5.1. Section 1.2, changed water sample volume from 500 to 250 mL.
 - 19.5.2. Section 7.6, change the LCS solution from 1000 ng/mL to 500 ng/mL
 - 19.5.3. Section 8.1, changed the sample container from 500 mL volume to 250 mL volume for water samples.
 - 19.5.4. Section 11.3.2, 11.3.4, and 11.4.5, changed water sample volume from 500 to 250 mL.
 - 19.5.5. Section 11.3.5, change the volume of spike added for EtFOSA/MeFOSA from 100 uL to 200 uL.
 - 19.5.6. Section 11.3.6, change the volume of solution added from 0.050mL (50 uL) to 0.025 ml (25 ul), and from 250 to 125 uL if EtFOSA/MeFOSA is requested.
 - 19.5.7. Section 11.7.2, change the volume of methanol from 800 uL to 400 uL.
 - 19.5.8. Section 11.7.3, change the volume of water added from 200 uL to 100 uL.
 - 19.5.9. Section 11.8.4, changed the volume of spike added from 20 uL to 40 uL.
 - 19.5.10. Editorial Changes
- 19.6. WS-LC-0025 Rev. 1.7, Effective 03/18/2016
 - 19.6.1. Section 4.5 – Deleted the last sentence in this section: “Until more information is available” and changed “excluded” to “included”.
 - 19.6.2. Section 4.7 - Deleted the last sentence. “Chromatographic peaks in a sample must be integrated in the same way as the CAL standard.”
 - 19.6.3. Section 7.4 – Changed upper calibration limit (CS-7) for all analytes from 500 ng/mL to 400 ng/mL
 - 19.6.4. Section 9.8.2 – Revised 1st sentence to “IDA recoveries are flagged if they are

outside of the acceptance limits (25–150%)”

19.6.5. Section 11.3.5 – Added to end of Section, “If EtFOSA and/or MeFOSA are required, increase the amount of LCS Matrix PFC spike solution added to 100 uL.”

19.6.6. Editorial changes.

19.7. WS-LC-0025 Rev. 1.6, Effective January 22, 2016

19.7.1. Section 11.6.1 – Revised to include rinse of sample container

19.7.2. Section 11.6.2 – Revised to include rinse of sample container.

19.7.3. Editorial changes

19.8. WS-LC-0025 Rev. 1.5, Effective November 1, 2015

19.8.1. Added the analytes EtFOSA, MeFOSA, EtFOSAA, MeFOSAA, 6:2FTS and 8:2FTS to Sections 1.1, 7.4 and 11.14.

19.8.2. Added Sections 2.5, 10.9.4 and 12.7 to address external standard quantitation.

19.8.3. Section 9.8 was updated and Section 12.7 added to address the calibration and quantitation of IDA compounds.

19.8.4. Added verbiage to Section 11 to address specifics if EtFOSA and/or MeFOSA are requested.

19.8.5. Added Section 11.3.7 for the specific processing of aqueous samples for EtFOSA and/or MeFOSA.

19.8.6. Added analytes and pertinent information to Section 11.14.1.

19.8.7. Editorial changes

19.9. WS-LC-0025 Rev. 1.4, Effective August 28, 2015

19.9.1. Updated copyright statement on cover page.

19.9.2. Section 1.1 – Renamed sulfonates to sulfonic acids and corrected the CAS# for PFHpS and PFOS. Removed FOSSA from table.

19.9.3. Section 7.4 – Renamed sulfonates to sulfonic acids and removed FOSSA remark.

- 19.9.4. Section 6.18 – Routine and Preventative maintenance table added.
- 19.9.5. Added Section(s) 17.1 thru 17.13 - Method modifications to Method 537.
- 19.9.6. Editorial changes.
- 19.10. WS-LC-0025 Rev. 1.3, Effective Oct. 31, 2014
 - 19.10.1. Removed references to glass containers in Section 8.1, 8.2 and Notes following Section 8.
 - 19.10.2. Editorial changes
- 19.11. WS-LC-0025 Rev. 1.2, Effective July 5, 2013
 - 19.11.1. Updated Tables in Section 11.14 with current specifications.
 - 19.11.2. Editorial changes.
- 19.12. WS-LC-0025 Rev. 1.1, Effective May 25, 2012
 - 19.12.1. Editorial revisions.
- 19.13. WS-LC-0025 revision 1.0, Effective May 3, 2011
 - 19.13.1. This is the original SOP. SOP was created from WS-LC-0020.

Analysis of Per- and Polyfluorinated Compounds (PFAS) in Water via Direct Aqueous Injection (DAI)**1. SCOPE AND APPLICATION**

- 1.1. This procedure describes the analysis of water samples via direct aqueous injection (DAI) for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a SCIEX 5500.

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluorinated sulfonic acids (PFSA's)		
Perfluoro-1-butanedisulfonic acid	PFBS	375-73-5
Perfluoro-1-hexanedisulfonic acid	PFHxS	355-46-4
Perfluoro-1-octanedisulfonic acid	PFOS	1763-23-1

- 1.2. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	1.0 mL	2.0 ng/L	2 to 200 ng/L

2. SUMMARY OF METHOD

- 2.1. A 1 mL aliquot of sample is diluted to a 40:60 methanol:water extract and analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using 20mM ammonium acetate/water and methanol.

3. DEFINITIONS

Refer to Section 3 of the main body of this SOP for a summary of definitions.

4. INTERFERENCES

Refer to Section 4 of the main body of this SOP for interferences.

5. SAFETY

Refer to Section 5 of the main body of this SOP for safety information.

6. EQUIPMENT AND SUPPLIES

Refer to Section 6 of the main body of this SOP for supplies, other than those listed below specific to the DAI analysis.

- 6.1. 2 mL auto sampler vials, clear glass, Thermo Scientific Nucleon surestop vial, part no. C5000-1, or equivalent.

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- 6.2. Vial caps, Thermo Scientific National AVCS blue cap, pre slit TEF/STL septa, part no. C5000-55B or equivalent.
- 6.3. Eppendorf 1000 uL epTIPS, part no. 022491954 or equivalent.
- 6.4. Eppendorf 200 uL epTIPS, part no. 022491938 or equivalent.
- 6.5. 50 mL graduated plastic centrifuge tubes, SCP Science DigiTUBES part no. 010-500-263 or equivalent

The 5 items above are to be maintained in the drawer labeled “Segregated Supplies for DAI Analysis” in the LC/MS instrument room

- 6.6. 1000 uL Pipette: Eppendorf Research Plus
- 6.7. 100 uL Pipette: Rainin EDP3-Plus
- 6.8. 250 mL HDPE bottles with PPE screw caps, ESS part no. 0250-1902-QC or equivalent.
- 6.9. Analytical columns
 - 6.9.1. Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm, Part No. 00D-4439-Y0, or equivalent.
 - 6.9.2. PFAS Isolator column, Phenomenex Luna C18 5 um, 50 mm x 4.6 mm, part no. 00B-4252-E 0 or equivalent.
- 6.10. SCIEX 5500 Triple Quad MS. The system utilizes Chrom Peak Review, version 2.1 or equivalent.
- 6.11. Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.

7. REAGENTS AND STANDARDS

Refer to Section 7 of the main body of this SOP for reagents and standards, other than those listed below specific to the DAI analysis.

- 7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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- 7.1.1. Ammonium acetate, Fisher Optima LCMS grade (20 mM in water), part no. A114-50, or equivalent.
- 7.1.2. Methanol, Baker HPLC grade, part no. 9093-03.
- 7.1.3. Water, Nanopure or Millipore or Fisher Optima LCMS grade, part no. W6-4, must be free of interference and target analytes.

7.2. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of the stock solutions (Section 7.2 of the main body of this SOP) in 40:60 methanol:water. The calibration stock solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.3. Initial Calibration (ICAL) Levels (ng/L)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7	CS-8
Perfluoroalkylcarboxylic acids (PFCAs)								
PFHpA	1.0	2.0	5.0	10	20	50	100	200
PFOA	1.0	2.0	5.0	10	20	50	100	200
PFNA	1.0	2.0	5.0	10	20	50	100	200
Perfluorinated sulfonic acids (PFSAs)								
PFBS	1.0	2.0	5.0	10	20	50	100	200
PFHxS	1.0	2.0	5.0	10	20	50	100	200
PFOS	1.0	2.0	5.0	10	20	50	100	200
Labeled Isotope Dilution Analytes (IDA)								
13C4-PFHpA	50	50	50	50	50	50	50	50
13C4-PFOA	50	50	50	50	50	50	50	50
13C5-PFNA	50	50	50	50	50	50	50	50
18O2-PFHxS	50	50	50	50	50	50	50	50
13C4-PFOS	50	50	50	50	50	50	50	50

Note- The above calibration levels are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.

7.4. LCS/Matrix PFC Spike Solution, 100 ng/mL.

The PFC spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at 100 ng/mL in methanol.

7.5. PFC Isotope Dilution Analyte (IDA) Spike Solution, 1 ng/mL.

The PFC-IDA solution is prepared by diluting all labeled PFAS to produce a solution containing each at 1 ng/mL in methanol.

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8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

- 8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6 °C for shipment to the laboratory.
- 8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6 °C. Water samples must be analyzed within 28 days of collection.

9. QUALITY CONTROL

Refer to Section 9 of the main body of this SOP for Quality Control information.

10. CALIBRATION

Refer to Section 10 of the main body of the SOP for calibration information.

11. PROCEDURE

Refer to Section 11 of the main body of this SOP for procedures, other than those listed below specific to the DAI analysis.

11.1. Water Sample Preparation

- 11.1.1. Visually inspect samples for the presence of settled and or suspended sediment. If sediment is apparent, remove the aliquot for testing from the aqueous layer. If the sediment concentration is too high centrifuge the sample first or filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration. File an NCM noting the need for filtration.

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

- 11.1.2. Prepare an LCS and method blank by adding 250 mL of HPLC grade water into a 250 mL HDPE bottle.
- 11.1.3. If requested, find the client assigned sample for MS/MSD.
- 11.1.4. Spike directly into the sample bottles for the LCS and MS/MSD (if requested) with 0.050 mL (50 uL) of the LCS/Matrix PFC Spike solution (Section 7.4). This will result in a sample concentration of 20 ng/L. Shake well to disperse spike.
- 11.1.5. Measure 1 mL of each sample using an Eppendorf pipette and pour into a

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labeled 2.0 mL injection vial. This includes the LCS and method blank samples as well.

- 11.1.6. Be sure to “prepare” the pipette by collecting two 1 mL aliquots and disposing of them, and then collect the aliquot for testing.
- 11.1.7. Add 83 uL of surrogate solution (PFC IDA Spike Solution, Section 7.5) into each vial for each sample and QC sample. This will result in an extract concentration of 50 ng/L for the surrogate.
- 11.1.8. Add 577 uL of methanol to each sample for a final solvent composition of 40:60 methanol:water.
- 11.1.9. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.
- 11.1.10. Vortex to mix the mixture well.

11.2. Instrument Analysis

11.2.1. Suggested operation conditions are listed below:

Routine Instrument Operating Conditions					
HPLC Conditions (Shimadzu HPLC)					
Column (Column temp = 35°C)	Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm				
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol				
Gradient Program	Time (min)	%A	%B	Curve	Flow Rate (mL/min)
	0	90	10	6	0.60
	1	90	10	6	0.60
	1.5	35	65	6	0.60
	8	5	95	6	0.60
	8.1	1	99	6	0.60
	12	1	99	6	0.60
	12.5	90	10	6	0.60
Maximum Pressure limit = 5,000 psi					
Injection Size	950 uL (fixed amount throughout the sequence)				
Run Time	17.1 minutes				

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Routine Instrument Operating Conditions	
Mass Spectrometer Interface Settings (SCIEX 5500)	
MS Interface Mode	ESI Negative Ion
Ion Spray Voltage (kV)	4.5
Entrance Potential (V)	5
Declustering Potential (V)	25
Desolvation Temp	550 °C
Curtain Gas (nitrogen) Flow	35 psi
Collision Gas (nitrogen) Flow	8 psi

Routine Instrument Operating Conditions						
Mass Spectrometer Scan Settings (SCIEX 5500)						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)
PFBS	Perfluorobutanesulfonate	299 > 80	0.02	6	58	55
18O2-PFHxS	IS	403 > 84	0.02	12	74	60
PFHpA	Perfluoroheptanoic acid	363 > 319	0.02	6	12	25
13C4-PFHpA	IS	367 > 322	0.02	6	12	25
PFHxS	Perfluorohexanesulfonate	399 > 80	0.02	12	74	60
18O2-PFHxS	IS	403 > 84	0.02	12	74	60
PFOA	Perfluorooctanoic acid	413 > 369	0.02	6	14	25
13C4PFOA	IS	417 > 372	0.02	6	14	25
PFNA	Perfluorononanoic acid	463 > 419	0.02	6	14	25
13C5-PFNA	IS	468 > 423	0.02	6	14	25
PFOS	Perfluorooctanesulfonate	499 > 80	0.02	9	108	65
13C4-PFOS	IS	503 > 80	0.02	9	108	65

Native Compounds	Native RT (minutes)	IS analog	IS RT (minutes)	Quantitation Method
PFBS	6.68	18O2-PFHxS	7.76	IS calculation
PFHpA	7.77	13C4-PFHpA	7.77	Isotope Dilution
PFHxS	7.76	18O2-PFHxS	7.76	Isotope Dilution
PFOA	8.44	13C4-PFOA	8.44	Isotope Dilution
PFNA	9.10	13C5-PFNA	9.10	Isotope Dilution
PFOS	9.06	13C4-PFOS	9.06	Isotope Dilution

11.2.2. Tune and calibrate the instrument as described in Section 10.

12. CALCULATIONS

Refer to Section 12 of the main body of this SOP for calculation information.

13. METHOD PERFORMANCE

Refer to Section 13 of the main body of this SOP for method performance information.

**Analysis of Per- and Polyfluorinated
Compounds (PFAS) in Water via Direct
Aqueous Injection (DAI)**

14. POLLUTION PREVENTION

Refer to Section 14 of the main body of this SOP for pollution prevention information.

15. WASTE MANAGEMENT

Refer to Section 15 of the main body of this SOP for waste management information.

16. REFERENCES

Refer to Section 16 of the main body of this SOP for reference information.

17. METHOD MODIFICATIONS

Refer to Section 17 of the main body of this SOP for modifications from Method 537, except as detailed below:

17.1. Water samples are prepared at 1.0 mL, not 250 mL.

17.2. Water sample containers are not preserved with Trizma. Holding time has been changed to 28 days for analysis.

17.3. The eluents and HPLC configuration differs. As a result the final extract is in 40:60 methanol:water.

18. ATTACHMENTS

There are no attachments to this Appendix.

19. REVISION HISTORY

19.1. WS-LC-0025 Attachment 1, Revision 2.2, Effective 01/31/2017

19.1.1. Title, changed to read, "Analysis of Per- and Polyfluorinated Compounds (PFAS) in Water via Direct Aqueous Injection (DAI)"

19.1.2. Many occurrences of the acronym "PFC" have been replaced with "PFAS". If the acronym "PFC" is used within the laboratory as an identifier, it has been kept in the SOP in that context.

19.1.3. Section 11.1, appended the following, "File an NCM noting the need for filtration."

19.1.4. Section 18 – Changed SOP to Appendix.

19.1.2 Editorial revisions including revision history (19.2 below) and header.

**Analysis of Per- and Polyfluorinated
Compounds (PFAS) in Water via Direct
Aqueous Injection (DAI)**

19.2. WS-LC-0025 Attachment 1, Revision 2.1, Effective 12/09/2016

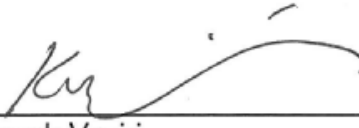
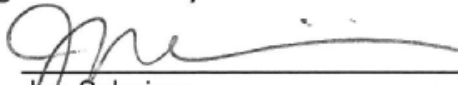
19.2.1. No revisions to this Appendix.

19.3. WS-LC-0025 Attachment 1, Revision 1.9, Effective 05/27/2016

19.3.1. This is the first version of this Appendix.

Title: Sample Receipt and Procedures

[Quality Assurance Procedure]

Approvals (Signature/Date):	
 Koroush Vaziri Department Supervisor	3/20/2017 Date
 Joe Schairer Health & Safety Manager / Coordinator	3/21/17 Date
 Lisa Stafford Quality Assurance Manager	3/21/2017 Date
 Crystal Pollock Laboratory Director	3.22.17 Date

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1. SCOPE AND APPLICATION

- 1.1. It is the responsibility of the employee to perform the procedure described here in full compliance with this SOP.
- 1.2. It is the responsibility of the Laboratory Director, QA Manager, and Departmental Supervisors of this facility to ensure that the analysis is performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this SOP correctly.

2. SUMMARY OF METHOD

- 2.1. This SOP describes the procedures for laboratory chain-of-custody, including receipt and acceptance of sample shipments, storage requirements, generation of computer records, and corrective actions for sample receipt anomalies.

3. DEFINITIONS

- 3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

4. INTERFERENCES

- 4.1. Any checks on samples or storage of samples should be done to eliminate any cross contamination.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002), and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

- 5.1.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide sufficient protection when handling closed sample containers.

- 5.1.2. Ice chests and shipping containers may be heavy. Always use safe lifting procedures. Whenever possible, use mechanical devices to lift or move containers from the floor to countertop/fume hood level to be unpacked.
- 5.1.3. Full ice chests larger than “lunchbox size” are not to be carried any further than ten feet. Instead, use a cart or scissors lift to move them. Repeated short trips can also present the same lifting/carrying hazard.
- 5.1.4. Any time a broken sample bottle is found inside an ice chest, immediately close the ice chest and move it into a fume hood before continuing to unpack the ice chest.
- 5.1.5. Associates opening any ice chests or shipping container must wear approved cut-resistant gloves while removing the samples. If any glass containers are found to be broken, cut resistant gloves must also be worn while discarding packing material and cleaning the shipping container. These can be Hyflex CR + (nitrile coated Kevlar) or MAPA Blue-Grip latex. Chemical protective gloves must be worn under or over these gloves, as needed. All packing materials are to be discarded in this situation.
- 5.1.6. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred, sub-sampled and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.1.7. Laboratory procedures such as repetitive use of pipettes, repetitive subsampling, moving heavy shipping containers, unloading shipping containers, and manipulation of glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.
- 5.1.8. Safety policies apply to ALL sample administration visitors, including auditors, employees, couriers or clients who deliver samples.
- 5.1.9. Some types of biological samples may present special hazards. Refer to Appendix 8 of this document for more information.
- 5.1.10. Samples containing or potentially containing chemical warfare agents or degradates present a special hazard. Review Appendix 7 of this document

before opening any coolers containing these types of samples.

5.2. Primary Material Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid (1)	Corrosive Oxidizer Dehydrat or	1 mg/m ³	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m ³	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Hydrochloric Acid (1)	Corrosive Poison	5 ppm- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid (1)	Corrosive Oxidizer Poison	2 ppm- TWA 4 ppm- STEL	Nitric acid is extremely hazardous. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Bisulfate	Corrosive	None listed	Contact may cause skin/eye burns. Inhalation can cause irritation of the respiratory tract with burning pain in the nose and throat, coughing, wheezing and shortness of breath. Causes chemical burns to the respiratory tract. May cause fatal spasms, inflammation or pulmonary/respiratory edema.
Zinc Acetate	Irritant	None Listed	Symptoms of skin or eye contact include redness, itching and pain.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

- 6.1. IR thermometer calibrated at a minimum of once per quarter against an NIST reference thermometer.
- 6.2. Filament thermometer calibrated at a minimum of once annually against an NIST reference thermometer.
- 6.3. Probe thermometer capable of reading to 0.1°C calibrated at a minimum of once annually against a NIST reference thermometer.
- 6.4. pH paper (Range pH 2 to pH 12 or equivalent).
- 6.5. Pipette.

7. REAGENTS AND STANDARDS

- 7.1. Not applicable. All preservatives are applied in other departments. Associates must be aware of the hazards associated with those preservatives in Section 5.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. This SOP does not address sample collection.
- 8.2. Preservation and storage of samples is determined by each method. See method SOPs.

9. QUALITY CONTROL

- 9.1. Not applicable.

10. CALIBRATION

- 10.1. Thermometers are calibrated according to WS-QA-0016.
- 10.2. All electronically operated thermometers (including IR thermometers) must have their calibration verified each day of use. IR thermometers will be checked against mercury thermometer ID 4881 and the corrected temperature must be within 1 degree. If the corrected temperature is greater than 1 degree then QA must be notified and that thermometer will not be used until it is recalibrated. IR thermometers are to be calibrated quarterly against a NIST reference thermometer.

11. PROCEDURE

- 11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a non-conformance memo (NCM) and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The non-conformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A non-conformance memo shall be used for this documentation.

11.2. Sample Receiving Acceptance Policy

Before samples are received, the laboratory should provide the client's sample collection personnel the TestAmerica Sacramento Sample Receiving Acceptance Policy (see Appendix 1). It is sent along with the bottle order.

11.3. Receiving Shipment

For samples received "over the counter" rather than from a commercial carrier, see also section 11.13.

11.3.1. Shipments are received from a variety of commercial carriers. When shipments are received, record the time that the carrier arrived and the number of packages (including coolers) received on the daily shipment log. Refer to this time when entering the receipt time onto the temporary record form and into the TALS system.

11.3.2. Verify accuracy of each shipping container's delivery address. Note the condition of the seal and if the custody seal number is present. Seals with evidence of tampering require that the client be notified and an NCM generated.

11.4. Opening the Shipment

11.4.1. Following safety policies, open the shipping container and remove paper work.

11.4.2. All shipping containers should be opened in a fume hood. After opening, the shipping container may be moved to a counter for further processing as long as it is determined that none of the following conditions apply. If any of the following conditions apply, the shipping container must remain under the fume hood during processing.

- The shipment is accompanied by MSD documentation,
- The container contains dry ice,

- Sample containers within the shipping container are broken or leaking,
- The shipping container exhibits a strong, noxious odor,
- The shipment contains concentrated product samples, or

11.4.3. If a shipment contains broken or leaking samples, notify the project manager via e-mail. Continue processing the shipment under the hood with cut-resistant gloves. Digital pictures may be taken of the broken samples and the files attached to the project manager's e-mail. If there are no intact samples for a given sample point and analysis, call the project manager so that the PM can notify the client as soon as possible. Dispose of broken samples according to the facility hazardous waste procedures. The Hazardous Waste Specialist or Environmental Health and Safety Coordinator should be contacted if additional information is required.

11.5. Processing the shipment

- 11.5.1. Assume custody of the samples by signing and dating the COC in the section marked "Received for Lab By" or "Received By". Use the date and time that the cooler was received from the commercial carrier which is recorded on the daily shipment log. This is also required for legal chain of custody. If the COC is not relinquished then file an NCM.
- 11.5.2. Record the tracking number from the air bill on the temporary record sheet and record whether or not the shipment came with a seal (if TestAmerica seal, record the red seal number). If the air bill indicates that the shipment is one out of several shipment containers, record this on the Cooler ID line on the temporary record sheet.
- 11.5.3. Immediately after opening, determine and record whether ice or artificial coolants are present. Measure the temperature and record the results on the temporary record form, noting both the observed and corrected sample temperatures. Record the corrected temperature on the COC as well. Make sure to fill out the rest of the temporary record form when necessary and initial and date the bottom.
- 11.5.3.1. Use the infrared thermometer by directing the thermometer at the temperature blank container (or a random sample container if no temperature blank is provided), making sure that no labels or packaging materials are interfering with the direct contact of the infrared beam and the container.
- 11.5.3.2. Use the AK thermometer by removing the thermometer from its casing, turning it on, and placing the thermometer probe into the

temperature blank. If no temperature blank is provided, place the probe in between two of the sample containers, making sure to hold the containers together so that they are touching. Make sure to let the thermometer equilibrate before recording the temperature. Note any correction factors to be applied.

- 11.5.3.3. If a random sample(s) is used to measure the temperature, make sure the sample(s) are placed back into the cooler until the cooler is being unpacked.
- 11.5.3.4. A non-conformance memo must be entered if the temperature reading is below 0°C or above 6°C. This notifies the project manager via email and is stored in TALS with the sample data. In the non-conformance memo software, be sure to include the client ID for all samples that are associated with a temperature exceedance.
- 11.5.3.5. Some sample matrices do not require cooling during transit. Situations where acceptable temperature range exceedance may be expected include, but are not limited to:
 - Samples delivered within six hours of close of daily sampling event (Note: Cooling agent must be present).
 - Paperboard and dry pulp samples; non-conformance memo not necessary.
 - Dry incinerator ashes; non-conformance memo not necessary.
 - Samples for metals analysis only; non-conformance memo not necessary. Note: This does not apply to samples for mercury analysis. Such samples must be shipped on ice at 4 °C, and narrated with a non-conformance memo if not on ice.
 - Dry product samples; non-conformance memo not necessary.
 - Volatile air samples in bags or canisters.
- 11.5.4. Start a folder for the login. Use a manila folder unless the samples are part of a rush order, in which case use a red folder, or if the samples require subcontracted tests and will be sent elsewhere, in which case use a blue folder. Rush samples will usually be labeled with a "RUSH" sticker. Typically for subcontracted tests, the project manager will send out an e-mail before receiving the samples so that they can be caught or it will be clear based on the COC that the samples are for subcontracted tests.
- 11.5.5. Place the COC in the folder along with the temporary record form.

- 11.5.5.1. Certain clients may require that the shipping manifest/air bill be included in the documentation. The list of clients who require this can be found on the corkboard in the back of the lab. If the shipping manifest/air bill is not in good shape, a photocopy is acceptable to include in the documentation. The shipping manifest is an integral part of the project documentation and is included with the finalized project when required by the client or regulatory agency.
- 11.5.5.2. Certain clients (such as those in Alaska) may require that the custody seals or a photocopy of the seals be kept for inclusion in the permanent record.
- 11.5.6. Examine accompanying documentation. If documentation is absent and the shipment arrival was unexpected, contact the lead project manager for assistance.
- 11.5.7. Unpack the samples. If the client/site requires association of samples to specific coolers, keep the samples separated based on the cooler in which they arrived, and label each group accordingly. Otherwise, arrange the samples by the order on the COC.
 - 11.5.7.1. If samples will be exposed to ambient temperatures for more than 20 minutes after they are removed from the cooler, the samples requiring temperature preservation should be placed in a sample storage refrigerator (volatile samples stored separately) until the sample login process can begin.
- 11.5.8. Complete the bottle inventory (see Appendix 3), if it is necessary to aid in logging in. As a part of this, examine any VOA vials for bubbles, and note the number of containers with bubbles in the upper left of the box, with the total number of VOA vials in the lower right of the box. Compare the containers and their state of preservation with the list of analyses requested. Although some exceptions are permitted regarding particular clients, container types must compare to those listed in the Sample Receiving Reference Guide, CA-Q-WI-025SAC. Discrepancies of this nature must also be annotated on the COC and a non-conformance memo filed.
- 11.5.9. If Encore® sample containers are present in the shipment, take the following action:
 - Send e-mail notification to the distribution list: Sacramento-Rush GCMS and include the associated PM.
 - Send the e-mail as priority. This will help the VOA department with prioritization.

- Use a subject line of “Encores to preserve”, and file the SDG job number.
- Include the date and time the first sample will expire. This will also assist the VOA department in prioritization. If the sample expires in less than 2 hours, notify all analysts in the department as well.
- Store the encores in “V” fridge and post the red “ENCORES” sign on the fridge. This sign will alert the analyst that there are samples in the fridge for pickup. Once the samples have been picked up, the red sign on the fridge will be changed to a green colored “NO ENCORES”.

11.5.10. Based on workload, samples may now be stored with their folder, chain of custody, and complete inventory of samples received until the remaining login steps are performed.

11.6. Prioritizing Workload for Login

Frequently, the shipping containers from a given carrier will have samples from multiple clients with diverse needs. To ensure prompt and timely service while meeting the needs of the clients, it is necessary to prioritize the order in which sample deliveries are logged in.

- 11.6.1. Prioritize the shipment according to the Turn Around Time (TAT), while ensuring extraction holding times have not been exceeded. "RUSH" projects are placed in a red folder, projects requiring a project manager's immediate attention to add tests are placed in blue folders, and all other projects are placed in manila folders. The affected department is to be notified by email or page when the samples are available.
- 11.6.2. For each client/site, examine the analyses requested and the sampling dates/times. Be aware of any notation for expected TAT, or a due date that is less than 10 days, or hold times that will expire within 72 hours. Most aqueous organic analyses require extraction within seven days of sampling. If three days or less are remaining, consider the lot to be a priority and file in a manila folder (Methods 8280 and 8290 dioxin/furan analyses are an exception, requiring extraction within thirty days).
- 11.6.3. Any analysis that has a standard holding time of forty-eight hours or less is considered a "Short-Holding Time" (SHT) analysis. These are reported to the affected department immediately with the Short Holding Time Test notification (see Appendix 2). The receiving chemist initials and records the date and time on the SHT form. Any requested TAT of 10 days or less is also considered a priority.

11.7. Sample Login

- 11.7.1. Select the project with the highest priority:
 - Short holds are processed immediately.
 - Blue folder (requires a project manager's immediate attention to add test and/or arrangements for sending samples to subcontract laboratories ("send outs"))
 - Red folder (rush)
 - Manila folder (normal TAT)
 - Grey folder (Air)
- 11.7.2. Line the samples up on a counter. Organize them by the order on the COC. If the client requires association of containers to coolers, make sure to keep that association when lining up the samples. Keep in mind that samples requiring thermal preservation should not be exposed to ambient temperatures for more than 20 minutes during the login process.
- 11.7.3. Read shipment documentation. Ideally, samples are to have possession documented on a COC form. The COC will identify samples individually by alphanumeric designators, list sampling dates/times for each sample, requested analyses and document possession. Signatures of possession qualify samples as having been "received under Chain-of-Custody". A Letter-of-Transmittal is also accepted as definitive documentation. Other forms of documentation include Request for Analysis, Shipping Order, Purchase Order, and various computer listings of sample information. If no documentation that lists sample identifications exists, complete a COC when accepting the samples.
- 11.7.4. Check the samples against the COC for accuracy (e.g., sample ID, collection date/time, etc.). If the samples have not been received in good condition, it must be noted on the COC and an NCM must be filed. All discrepancies must be noted as well, including the lack of a relinquishing signature from the shipper.

NOTE: *Good condition is loosely defined as all containers intact with no obvious discrepancies present. The shipment is estimated at this time to be viable; what is being requested coincides with what has been received. Temperature exceedances and minor discrepancies become issues when so specified by contract or client instruction. The presence of bubbles in volatile containers is documented. All such observations must be documented on custody chains, and by using the non-conformance memo software.*

- 11.7.5. For all work sampled at or concerning government property, any federal projects, or as specified in client QAPPs, the pH of the preserved aqueous samples must be checked and recorded on Sacramento Sample Receiving Preservation Check Form QA-611 (Appendix 4). VOA vials are exceptions

from this requirement. The pH is notated with a check mark or X per container type if it meets specifications. If the pH is out of specification, record the actual pH.

- 11.7.5.1. After placing the samples in the hood, invert the sample container three times, remove the cap and pour a small aliquot of the sample into the lid. Pour the aliquot from the lid onto a fresh piece of pH indicator paper. Compare against the pH color grid table located on the pH strip container. Recheck readings that indicate samples were unpreserved when they should have been. Note discrepancies on the COC. Notify the project manager and file a non-conformance memo for the concerned sample.
- 11.7.5.2. For metals samples that should be preserved but are received unpreserved, page the metals department. A technician will preserve the samples and document the preservation. An NCM must be filed for this anomaly.
- 11.7.5.3. Store the completed preservation check form in the folder.
- 11.7.6. Check the walk-in fridge and other storage areas to determine the storage location(s) for the samples. Designate a storage location using the following guide:
 - 11.7.6.1. R-14 (V) - volatile containers awaiting transfer to the VOA group refrigerators.
 - 11.7.6.2. F10 - samples requiring freezing, including plant/animal tissues, and soil samples for method 1668 and 1699. "Samples stored in glass jars which are preserved by freezing must be placed in a Ziploc bag for secondary containment in case of glass breakage."
 - 11.7.6.3. R22 - any samples where secondary containment in Ziploc bags is deemed necessary due to smells, spillage, degradation of container exterior or suspected high concentration of analytes.
 - 11.7.6.4. C1 - dry pulp, paper samples or cassettes.
 - 11.7.6.5. EPA1 - soil samples received from the Environmental Protection Agency, solvents, air toxics projects for method 29, or clients requiring dioxin work under SOW DFLM01.1.
 - 11.7.6.6. Walk-In – Main sample storage area. If samples do not meet any of the above requirements, they may be stored in the Walk-In cooler.

Note: Location selections specify shelf by letter, example: W4A.

Note: Dioxin soil samples received in clear glass jars must be stored in boxes to protect from light.

- 11.7.7. Open TALS. Enter the Sample Management tab. Navigate to the login tab, and use information from the COC and the project manager to obtain a project number to associate with the login. Enter the login number on the top section of the bottle lot inventory and record on the COC.
 - 11.7.7.1. Enter information into the Receipt/Info and Receipt/Containers tabs. If the client requires association of containers to coolers, be sure to enter the specific cooler for each container in the cooler field for the container.
 - 11.7.7.2. Complete the receipt/checklist tab. Complete anomalies if prompted.
 - 11.7.7.3. Complete the information on the Login/Samples and Login/Samples tabs.
 - 11.7.7.4. File any holding-time violation reports (HTV), if necessary, by clicking the “NCM” button, and entering the appropriate information. Answer the queries, and be certain to mail the HTV to the department supervisor and project manager involved. The HTV is filed by sample administration when a holding time is expired upon receipt. If the holding times have not completely expired for a project, the department handling the analysis in question files the HTV.
 - 11.7.7.5. When all information has been entered, print the sample labels.
- 11.7.8. Label sample containers. Be certain the labels adhere to containers. When possible, place the large labels (Appendix 5.3a) along the long axis of the bottle, so that the barcode is not wrapping around a curve. Unless the container is a VOA vial, place the small label (Appendix 5.3b) on the container lid. Attempt to leave all client label information exposed. When this is not feasible, affix label so that the client ID, sampling date/time, and preservative are showing. Preprinted laboratory names may be covered with no consequence. If label adhesive is insufficient, use cellophane tape to secure label. Place sample in Ziploc bag when storage location is R22. Labeling may be peer reviewed whenever the complexity of the project warrants a second check to ensure accuracy. A peer compares the order of the samples to the documentation and ensures that all containers of a sample have the same sample number and are sequential.

11.7.9. Circumstances where a review is warranted may include:

- A large number of samples are present.
- Many containers per sample are present.
- VOA containers are present.
- Client identifications are illegible or confusing.
- A review is requested by a TestAmerica Sacramento employee.

11.7.10. Scan the COC and attach to the job in TALS.

11.7.11. When login is complete, place the folder in the project manager's tray in sample administration. At this time, the folder should contain:

- Original COC.
- Air bill and secondary documentation, if necessary.
- Bottle lot inventory.
- Temp record form.

11.8. Shipping Container Return

11.8.1. Broken samples and packing material contaminated with spilled samples **MUST** be disposed of as hazardous lab trash. Shipping containers must be decontaminated before being put back into use. Decontamination procedures will depend on what was spilled. See EH&S staff for specific instructions.

11.8.2. Except when discarded, shipping containers are cleaned in bottle prep or returned to the client. Containers belonging to TestAmerica Sacramento are marked with a permanent marker. If the container belongs to a client and they request a return, either a return label will be included in the shipment or the client will make a note requesting a return. Make sure that the return address is saved in this case. All hazardous materials labeling must **be removed or defaced** in some way. Dry the interior of the container if wet, replace packing and return to bottle prep.

11.8.3. Packing and artificial ice that are deemed reusable may be returned to the client. Packing that resembles trash or is ruined during unpacking is disposed as trash. Drain any water from the container before sealing it closed. Due to partially frozen artificial ice, client containers may not be perfectly dry when returned. Complete a return-mailing label if necessary. Secure it with cellophane tape and seal the container.

11.8.4. Temperature blanks are kept and reused.

- 11.9. Per USDA permit regulations, ice chests that have been used to ship soil samples must be decontaminated with 10% bleach solution during the cleaning process before being re-used or shipped empty to another location.
- 11.9.1. Ice chests that have had soil samples are marked with a label that reads, "Soil Sample – Clean with Bleach" and moved to the bottle prep area where they are cleaned with 10% bleach.
- 11.10. Disposal of Ice
- 11.10.1. When ice is present, it is often enclosed in plastic bags. A basin used for dumping ice is located in sample administration. Open the bags and dump ice into the basin. Water should not be left running in the basin as the basin drains into a closed system. When excessive amounts of ice are received, collect the ice in an ice chest and dump it over the storm drains outside the building. **This is permissible only if the ice and coolers are uncontaminated. Additionally, ice used to ship any soil samples may not be disposed down the storm drain. It may only be disposed of down the basin in sample administration.**
- 11.11. Refrigerator and Freezer Temperatures
- 11.11.1. Refrigerator and freezer temperatures will be monitored and recorded once daily Monday through Saturday. Any temperature below 0°C or above 6°C for the refrigerators and above -10°C for the freezer will need corrective action. Refer to WS-QA-0005, Temperature Monitoring and Corrective Action for Refrigerators and Freezers. Temperatures will be recorded on the appropriate charts. Temperature charts will then be stored in the temperature logbook. All charts for a particular refrigeration unit will be stored together in chronological order.
- 11.12. Internal Sample Tracking
- Samples are tracked within the facility using the Internal Chain of Custody (ICOC) feature of TALS.
- 11.12.1. In TALS, the analyst selects Internal Chain of Custody and ascertains storage location. Then the analyst removes appropriate containers from storage unit and organizes them by lot and sample number. Analyst completes checkout portion of internal COC in TALS using the barcode scanner.
- 11.12.2. If a sample is completely used up in the extraction laboratory, then the disposition of the container must be documented. All empty containers not returned to sample administration must be recorded as destroyed in testing on

the ICOC. If the containers are not to be returned and will enter the waste stream through the extraction lab, the analyst will also enter this information during checkout.

- 11.12.3. Analyst completes the checkout process before leaving sample control. At return, the analyst completes the check-in portion of the ICOC. Analyst returns samples to their storage location. If subsampling or aliquoting is performed, whether outside the area or not, containers must be checked out by the analyst. The original containers' whereabouts and those of any other containers generated must be documented. Transfer of volatile containers from VOA to RD and RF do not require verification.

11.13. Sending Out Subcontracted Work

- 11.13.1. In the event samples require being subcontracted to another lab, the samples will be processed in the very same manner as all other samples. Samples should be received and processed as described in Sections 11.1 to 11.7 of this SOP. The project manager should notify the sample receiving staff as to all samples they require to be shipped out.
- 11.13.2. Using TALS, generate a new TestAmerica Sacramento chain of custody for the subcontracted samples by scanning out the containers to the desired lab. Contact the project manager for assistance if any difficulties are encountered. Project Managers must verify samples to be sent out and relinquish the COC.
- 11.13.3. Samples should be packaged in a manner in accordance with the TestAmerica Sacramento Receiving Acceptance Policy. Additional packing may be used to ensure samples have adequate protection from breakage. Samples are subsequently packaged in a bag containing copious amounts of uncontaminated wet ice. There should be sufficient ice to maintain the samples within the temperature range of 0°C to 6°C throughout transit. The ice packed samples are stored in the appropriate sized insulated container.
- 11.13.4. Ensure the COC is packed in the shipping container and that it is protected from moisture from ice and/or samples (in the event any containers become compromised).
- 11.13.5. The packed container is then sealed with a signed and dated Custody Seal and a generous amount of shipping tape to ensure the contents remain secured. Ensure they are labeled with the appropriate shipping labels and delivered to the courier. Add the tracking number to TALS.

11.14. Client Deliveries ("Over the Counter")

11.14.1. Samples received “over the counter” are samples hand delivered by the client, sampler or courier directly employed by the client's company. The pivotal question in such deliveries is whether the person possessing the shipment is in custodial possession of the samples or merely transporting the shipment. If in custodial possession, the deliverer will need to relinquish custody to you before departing and will probably retain a copy of the documentation. To determine the matter, simply ask the deliverer if they have signed any of the sample documents.

11.14.2. Mention any discrepancies and allow corrections to be made by the client, sampler or courier. Also ask for any special instructions or important aspects of the project (i.e. rush turn-around times, impending hold-time violations, sample matrix specifics, etc.). Solicit the client's needs such as shipping containers, bottle orders, to speak with a project manager or any other requests. After the client's departure, complete any aspects of Sections 11.1 through 11.15 left unfinished. Prioritize the new project within the existing workload and proceed with the highest priority.

11.15. For after hours sample receipt instructions for non-sample administrative personnel, refer to Appendix 9.

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable.

13. METHOD PERFORMANCE

13.1. The Department Manager has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

14. POLLUTION PREVENTION

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for “Waste Management and Pollution Prevention.”

All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

- 14.1. All ice, or melted ice, that has been used to store or ship any soil samples, or in any container with soil samples, must be allowed to melt through a 100 mesh screen, in order to comply with our USDA soil permit.
- 14.2. Per USDA permit regulations, ice chests that have been used to ship soil samples must be decontaminated with 10% bleach solution during the cleaning process before being re-used or shipped empty to another location.

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to WS-EHS-0001. The following waste streams are produced when this method is carried out.

- 15.1. Uncontaminated packing materials such as vermiculite, bubble wrap, plastic bags, paperwork, etc. These are collected in the uncontaminated lab trash cans, and are disposed of to the dumpster at the end of the day.
- 15.2. Contaminated solid packing materials, including broken glass, caused by the breakage of sample containers during shipment. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full, tie the plastic bag liner shut and put the lab trash into the applicable steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.
- 15.3. Contaminated melted ice and aqueous samples of unknown hazards, spilled when their sample container breaks during shipment. These materials are collected and disposed of in accordance with instructions from the Hazardous Waste Specialist, depending on the type of sample that was spilled.
- 15.4. Contaminated melted ice and solid or soil samples of unknown hazards, spilled when their sample container breaks during shipment. These materials are collected and disposed of in accordance with instructions from the Hazardous Waste Specialist, depending on the type of sample that was spilled.

16. REFERENCES/CROSS REFERENCES

- 16.1. Manual for the Certification of Laboratories Analyzing Drinking Water, Fifth edition, January 2005, Appendix A.
- 16.2. Sacramento Glass Safety SOP, WS-QA-0025, current revision.

17. METHOD MODIFICATIONS

- 17.1. There are no deviations from the method.

18. ATTACHMENTS

- 18.1. Appendix 1 - TestAmerica Sacramento Sample Receiving Acceptance Policy
- 18.2. Appendix 2 – Short Hold Test Notification
- 18.3. Appendix 3 – Bottle Lot Inventory
- 18.4. Appendix 4 – Sample Receiving Preservation Log
- 18.5. Appendix 5 – Example Bottle and other Labels
- 18.6. Appendix 6 – Flow Chart
- 18.7. Appendix 7 – Chemical Warfare Degradates – Potential Hazards in Sample Receipt
- 18.8. Appendix 8 – Handling of Blood or Other Potential Infectious Materials
- 18.9. Appendix 9 – After Hours Sample Receipt Procedures for Non-Sample Administrative Personnel
- 18.10. Appendix 10 – Sample Receipt Notes Form
- 18.11. Appendix 11 – Canister Receipt Notes Form
- 18.12. Appendix 12 – Revenue Source Tracking Procedure

19. REVISION HISTORY

- 19.1. WS-QA-0003, Revision 12.3, Effective 03/22/2017
 - 19.1.1. Section 11.4.2, removed “Under the following conditions, the shipping container must be opened in a fume hood, or, if the condition is discovered upon opening, it must be moved to a fume hood before further processing.
 - 19.1.2. Section 11.4.2, added “All shipping containers should be opened in a fume hood. After opening, the shipping container may be moved to a counter for further processing as long as it is determined that none of the following conditions apply. If any of the following conditions apply, the shipping container must remain under the fume hood during processing.”

- 19.1.3. Section 11.4.3, removed “The client program requires opening within a fume hood.”
- 19.1.4. Section 11.5, split up into sections.
- 19.1.5. Section 11.5.1, moved “If the COC is not relinquished then file an NCM,” into this section from Section 11.5.3.
- 19.1.6. Added Section 11.5.2, “Record the tracking number from the air bill on the temporary record sheet and record whether or not the shipment came with a seal (if TestAmerica seal, record the red seal number). If the air bill indicates that the shipment is one out of several shipment containers, record this on the Cooler ID line on the temporary record sheet.”
- 19.1.7. Section 11.5.3, removed “...ascertain if ice or artificial coolants are present. Use the date and time received by the commercial carrier. If the COC is not relinquished then file an NCM. Measure temperature and record the results on the temporary record form.”
- 19.1.8. Section 11.5.3, added “...determine and record whether ice or artificial coolants are present. Measure the temperature and record the results on the temporary record form, noting both the observed and corrected sample temperatures. Record the corrected temperature on the COC as well. Make sure to fill out the rest of the temporary record form when necessary and initial and date the bottom.”
- 19.1.9. Section 11.5.3.1, in place of “sample containers” used “the temperature blank container (or a random sample container if no temperature blank is provided).”
- 19.1.10. Section 11.5.3.1, removed “Note the uncorrected and corrected sample temperature on the sample receiving notes page and write the corrected temperature on the COC. If the COC lists a temperature blank, or one is present in the shipping container, locate the blank and measure the temperature. If a temperature blank is not present, take the temperature of a random sample in the cooler to determine the temperature.”
- 19.1.11. Added Section 11.5.3.2 to read, “Use the AK thermometer by removing the thermometer from its casing, turning it on, and placing the thermometer probe into the temperature blank. If no temperature blank is provided, place the probe in between two of the sample containers, making sure to hold the containers together so that they are touching. Make sure to let the thermometer equilibrate before recording the temperature. Note any correction factors to be applied.”

- 19.1.12. Added Section 11.5.3.3 to read “If a random sample(s) is used to measure the temperature, make sure the sample(s) are placed back into the cooler until the cooler is being unpacked.”
- 19.1.13. Section 11.5.3.5, added “Volatile air samples in bags or canisters.”
- 19.1.14. Section 11.5.4, added “Use a manila folder unless the samples are part of a rush order, in which case use a red folder, or if the samples require subcontracted tests and will be sent elsewhere, in which case use a blue folder. Rush samples will usually be labeled with a “RUSH” sticker. Typically for subcontracted tests, the project manager will send out an e-mail before receiving the samples so that they can be caught or it will be clear based on the COC that the samples are for subcontracted tests.”
- 19.1.15. Section 11.5.5, removed “Photocopy the shipping manifest/airbill and add to the sample receiving notes page and include the 8 ½” x 11” facsimile in the folder.
- 19.1.16. Added Section 11.5.5.1 to read “Certain clients may require that the shipping manifest/air bill be included in the documentation. The list of clients who require this can be found on the corkboard in the back of the lab. If the shipping manifest/air bill is not in good shape, a photocopy is acceptable to include in the documentation.”
- 19.1.17. Added Section 11.5.7.1 to read “If samples will be exposed to ambient temperatures for more than 20 minutes after they are removed from the cooler, the samples requiring temperature preservation should be placed in a sample storage refrigerator (volatile samples stored separately) until the sample login process can begin.”
- 19.1.18. Section 11.7.2, added “Keep in mind that samples requiring thermal preservation should not be exposed to ambient temperatures for more than 20 minutes during the login process.”
- 19.1.19. Section 11.5.8, changed “TestAmerica Sacramento Quality Assurance Manual Tables 22.1 through 22.8” to “Sample Receiving Reference Guide, CA-Q-WI-025SAC.”
- 19.1.20. Section 11.10.1, removed “...and the amounts disposed in this way are recorded.”
- 19.1.21. Appendix 1, updated Sample Acceptance Policy document to latest version (02/02/2015)

- 19.1.22. Appendix 10, updated Sample Receiving Notes document to latest version (Rev. 1.3)
- 19.1.23. Editorial changes.
- 19.2. WS-QA-0003, Revisions 12.2, Effective 11/11/2016
 - 19.2.1. Section 11.4.4.2 – Replaced LIMS with TALS.
 - 19.2.2. Section 11.7.8 – Replaced ‘login function’ with ‘Sample Management tab’.
 - 19.2.3. Updated Appendix 2 and Appendix 11 forms.
 - 19.2.4. Editorial changes.
- 19.3. WS-QA-0003, Revision 12.1, Effective 04/11/2016
 - 19.3.1. Edited paragraphs 5.1.2 and 5.1.5 to reflect practices on opening sample shipping containers and cleaning containers that are found to have broken glass sample containers inside of them.
 - 19.3.2. Added paragraph 5.1.3 regarding lifting and movement of full ice chests.
 - 19.3.3. Edited paragraphs 11.8 and 14.2 regarding cleaning of ice chests that had soil samples in them.
 - 19.3.4. Added paragraph 16.2, Glass Safety SOP.
 - 19.3.5. Editorial changes
- 19.4. WS-QA-0003, Revision 12.0, Effective 12/11/2015
 - 19.4.1. Inserted Appendix 11, Canister Receipt Notes Form
 - 19.4.2. Updated Appendix 10 – Sample Receipt Notes Form
 - 19.4.3. Editorial comments
- 19.5. WS-QA-0003, Revision 11.9, Effective 09/30/2014
 - 19.5.1. Added to Section 10.2. “IR thermometers will be checked against mercury thermometer ID 4881 and the corrected temperature must be with 1 degree. If the corrected temperature is greater than 1 degree then QA must be notified and that thermometer will not be used until it is recalibrated. IR thermometers are to be calibrated quarterly against an NIST reference thermometer.”

- 19.5.2. Editorial comments.
- 19.6. WS-QA-0003, Revision 11.8, Effective 07/12/2013
 - 19.6.1. Deleted Section 11.3.3 – computer entry SRL (Sample Receipt Login) form is not used.
 - 19.6.2. Modified Section 11.44.1 to include - Note the uncorrected and corrected sample temperature on the sample receiving notes page and write the corrected temperature on the COC.
 - 19.6.3. Editorial changes.
- 19.7. WS-QA-0003, Revision 11.7, Effective 5/3/2013
 - 19.7.1. Inserted new Sample Acceptance Policy (Appendix 1)
 - 19.7.2. Editorial revisions.
- 19.8. WS-QA-0003, Revision 11.6, Effective 12/15/2012
 - 19.8.1. Updated forms to TALS specifications.
 - 19.8.2. Editorial revisions.
- 19.9. WS-QA-0003, Revision 11.5, Effective 11/18/2011
 - 19.9.1. Deleted “purple folders” form Sections 11.5.3 and 11.6.1.4.
 - 19.9.2. Section 11.4.2.2 - Changed temperature range from 2 - 6°C to 0 - 6°C.
 - 19.9.3. Updated LRC forms in Appendices 3 – 7.
 - 19.9.4. Editorial revisions.
- 19.10. WS-QA-0003, Revision 11.4, Effective 06/17/2011.
 - 19.10.1. Added Section 11.4.3 – Photocopy the shipping manifest/airbill and include the 8½” x 11” facsimile in the folder with the Chain of Custody and LRC documentation. The shipping manifest is an integral part of the project documentation and will be scanned and included with the finalized project.

APPENDIX 1

The TNI Standard and TestAmerica Sacramento have specific requirements under which all samples will be received by the laboratory for analysis. TestAmerica Sacramento will review your sample shipment against those requirements as listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

VOA vials should be stored in controlled conditions. Exposure of trip blanks to temperature fluctuations is likely to cause development of bubbles in the trip blanks.

When completing the chain of custody form, please note that you must sign your name in the "relinquished by" box.

Requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples, shall be provided.
- Samples must be accompanied by written disclosure of the known or suspected presence of any hazardous substances, as defined by applicable federal or state law.
- Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/ATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).
- Each sample shall be collected in the appropriate sample container and labeled with unique, durable and indelible identification.
- Drinking water samples for Method 1613B that may have residual chlorine must be checked and treated in the field, or collected in sodium thiosulfate preserved containers.
- Containers of water meant for perchlorate analysis should have adequate headspace to prevent anaerobic microbial degradation. A void approximately 1/3 of the container volume is sufficient.
- The samples shall arrive at the laboratory with adequate remaining holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.
- Received samples must not exhibit obvious signs of damage, contamination or inadequate preservation.
- Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C and above freezing (0° C). For methods with other temperature criteria (e.g. some bacteriological methods require ≤ 10° C), the samples must arrive within ± 2° C of the required temperature or within the method specified range.
 1. Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements above. In these cases, the samples shall be considered acceptable if the samples were received on ice.
 2. If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.
 3. Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.
- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

- For samples undergoing chemical warfare degradate analysis, the sample must be screened for agent prior to shipment in accordance with appendix 10 of our Sample Receipt Procedure (WS-QA-0003).
- Samples containing mammalian tissue will not be accepted without prior coordination with a project manager. Additional conditions for receipt and handling of tissue are outlined in appendix 11 of our Sample Receipt Procedure (WS-QA-0003).
- Air canisters (SUMMA® and other brands) have additional requirements:
 - Never write or affix a label directly on a canister. A special tag is attached to each canister for this purpose.
 - Complete the Canister Field Data Record with the initial and final vacuum/pressure reading for each canister during sampling.
 - Close all valves completely prior to shipping or transporting.
 - Return canisters, filters, flow controllers, vacuum flow regulators, and any other supplied equipment must be returned even if they were not used. Pack equipment carefully to minimize in-transit damage. Sampling equipment that is damaged, lost or not returned will be invoiced to the client at the replacement cost. Delayed return of equipment to the laboratory may result in additional rental charges.
 - Do not attempt to adjust or alter any equipment, as it may result in loss of sample integrity as well as equipment damage that may be invoiced to the client.

The laboratory will notify the client/Project Manager upon sample receipt if the samples fail to meet any of the above requirements.

APPENDIX 2



**TestAmerica West Sacramento
SHORT HOLDING TEST NOTIFICATION**

Client _____ ☐ SHT Analysis
 Lot ID _____ ☐ Pending HTV (Routine Tests)
 Date & time received by Sample Control _____ ☐ RUSH (24 - 48 - 72 HR TAT)
 Date & time received in Gen Chem _____ ☐ See Attached
 Chemist Notified _____
 Holding time already violated? ☐ Yes ☐ No
 If HTV, has NCM been filed? ☐ Yes ☐ No
☐ MS/MSD required on sample # _____

General Analysis (select those that apply)	Sample #	Expiration Date & Time
☐ pH ☐ Alkalinity	_____	_____
☐ Cr6+ ☐ EC	_____	_____
☐ pH-Soils ☐ TDS/TSS	_____	_____
Method 353.2		
☐ NO ₂ +NO ₃	_____	_____
☐ NO ₂	_____	_____
☐ NO ₃ asO ₃	_____	_____
Method 300.0		
☐ NO ₃ ☐ NO ₂ ☐ OPO ₄	_____	_____
☐ Br ☐ Cl ☐ SO ₄ ☐ F	_____	_____
☐ Other _____	_____	_____
_____	_____	_____
_____	_____	_____
☐ Chain of Custody attached		

Comments: _____

APPENDIX 5

Example Labels

1. Label for the Folder

	Loc: 320 1197
Login: 320-1197	
PM: Sadler, Jeremy	
Company: TestAmerica Laboratories, Inc.	

2. Label for the COC

	Loc: 320 1197
320-1197 Chain of	

3. Labels for the Samples

a. For the side of the container


320-1197-A-1
0114301X/51122/112812
Location: R14 shelf A VOA storage in Sample Contro
Bottle: Voa Vial 40ml - Hydrochloric Acid
Sampled: 11/28/2012 12:00 AM 320-47099 COC

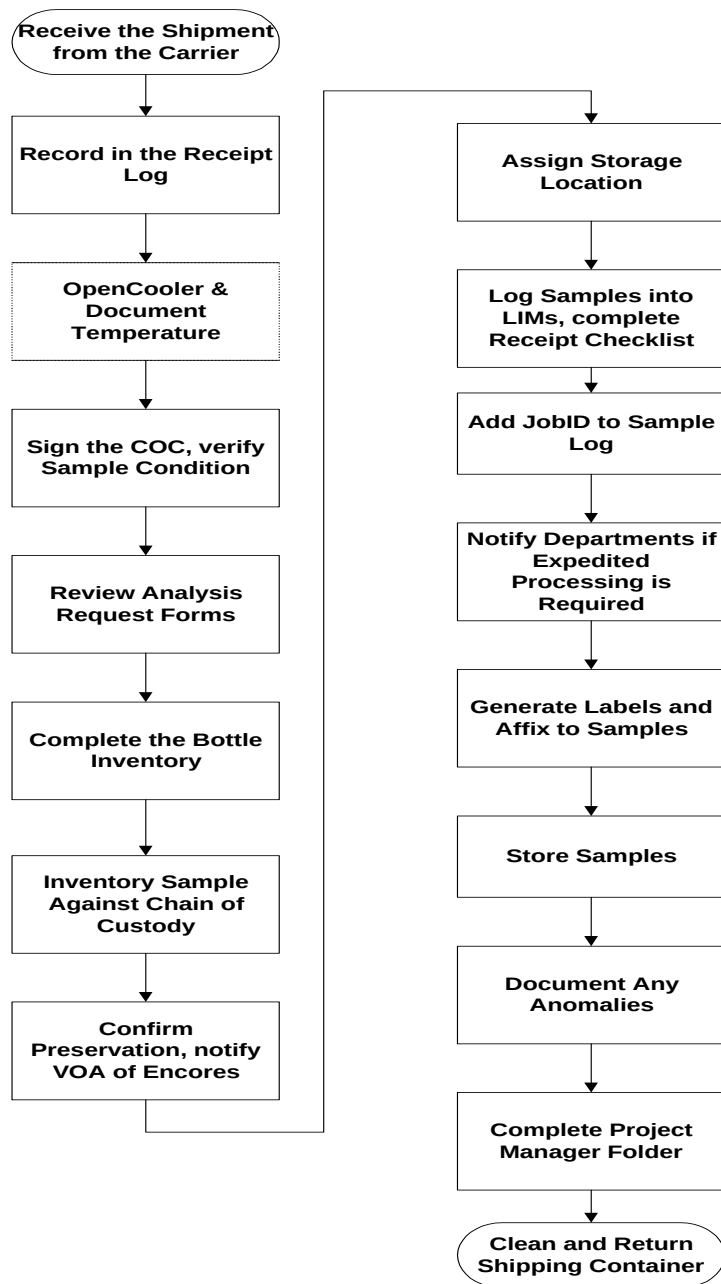
b. For the lid of the container

Loc: 320
1197
#1
A

4. Label for archive paperwork

	
550-61072 Field Sheet	550-61072 Field

APPENDIX 6 Flow Diagram



APPENDIX 7

Chemical Warfare Degradates - Potential Hazards in Sample Receipt

Background

TestAmerica Sacramento regularly receives samples to be analyzed for degradates of chemical warfare agents. These degradates generally are no more toxic than most of the compounds we deal with every day. The fact that these compounds are degradates of chemical warfare agents does, however, present a different type of potential hazard for us. We have developed policies regarding the handling of such samples. The purpose of this document is to discuss these compounds and the potential hazards involved in handling them.

Please note that TestAmerica Sacramento does NOT analyze samples for actual chemical warfare agents such as mustard, lewisite, Sarin, GD, VX, phosgene and tear gas. Parent compounds are analyzed by laboratories that have specialized personnel training, security, and handling procedures.

The toxicity of the by-products of some chemical warfare materials are more than the parent compound. An example of this is VX and EA 2192. Testing protocol for degradates is not necessarily specific for the analyte being screened for and the scientific community has not come to consensus on what "positive" test results actually mean. For this reason, in all cases where positive screening data is received for the parent compounds, Corporate EH&S, the Project Manager, local EH&S staff and senior management must be consulted before deciding to accept and proceed with handling such samples.

Review of Agent Compounds

Chemical warfare agents fall into a wide variety of categories, ranging from relatively mild chemicals such as tear gas to lethal nerve agents such as Sarin. The two types of agents we are most concerned with are **nerve agents** and **blistering agents**. Other lethal agents have been developed and tested for use in chemical warfare; however, these other compounds are either extremely volatile and reactive (and therefore highly unlikely to be present in an environmental sample) or were never produced in significant quantities in the U.S.

Nerve agents

These are members of the organophosphate class of compounds. They are similar to many common household pesticides such as diazinon. The difference is that nerve agents are far more toxic to humans. The first nerve agent, Tabun, was discovered prior to World War I by a German pesticide company during the process of screening new compounds for use as pesticides. Once the toxicity of Tabun was determined, various governments began screening many related compounds. Out of this effort have come five established (non-classified, i.e. public domain) agents. --- **Tabun (or GA), Sarin (or GB), GD, GF, and VX.**

All of these compounds are toxic via inhalation, ingestion, skin contact, or just about any other route of entry into the body.

A characteristic of these compounds is their volatility. A more volatile nerve agent will disperse in air more effectively than a less volatile one.

Less volatile agents, on the other hand, will remain around on soil, vegetation, clothing, etc. and will therefore last longer. Out of these various agents, the only two produced in significant quantities in the U.S. are Sarin and VX. **Sarin is the most volatile** of the above agents while VX is the least volatile. Both are extremely toxic - a drop of pure VX barely visible to the naked eye is enough to kill a person through skin contact.

Blistering agents

These were the first of the modern agents developed specifically for military purposes. Unlike nerve agents, blistering agents are different mixtures of one compound, sulfur mustard, with other non-toxic chemicals which affect its dispersion characteristics.

The primary hazard with sulfur mustard involves skin contact. The term “blistering agents” is somewhat of a misnomer - it will kill you if you inhale enough of it, but this is not likely to occur as it is not particularly volatile under normal conditions. Significant quantities of sulfur mustard (a.k.a. HD or HT) have been produced in the U.S.

Chemical Agents in the Environment

It is unlikely (but never impossible) that we at TestAmerica Sacramento will receive a sample that contains a dangerous concentration of active agent. We **should not** receive samples from any areas known to be contaminated with active chemical agent (areas with buried drums, old munitions, etc.) because the Army policy for such sites is to destroy the agent onsite.

Additionally, most of the areas in which degradate analysis is required are areas in which agent was used decades ago - all active agent is likely to have degraded. Nonetheless, it is imperative that we know as much as possible regarding the compounds and the samples in order to protect ourselves against any potential hazard.

The behavior of these compounds in the environment has been extensively studied. Most of the information we have is from a study titled **Environmental Chemistry and Fate of Chemical Warfare Agents**. This study was prepared for the Army Corps of Engineers by Southwest Research Institute in 1994.

Sites containing chemical warfare related material or chemical warfare materials are divided into **“stockpile” and “non-stockpile” sites**. Stockpile sites are where the vast majority of CWM’s are stored. Non-stockpile sites are where smaller amounts of CWM’s are located. Non-stockpile sites may contain: buried CWM, chemical weapon production facilities, binary chemical weapons and miscellaneous CWM.

What is important to realize is that based on the **Survey and Analysis Report** prepared by the US Army Chemical Material Destruction Agency (11/93), there are “potential burials at 82 locations in 33 states, the US Virgin Islands and the District of Columbia.....Some of the 82 locations have multiple burial sites.” Given this wide span of impacted areas, for every shipment received by TestAmerica Sacramento for CWM analysis, adherence to procedures listed in this document and the

Environmental Health and Safety Manual is strictly required.

Nerve Agents

Both of the compounds we are concerned with (Sarin and VX) undergo **hydrolysis in the presence of water**. This hydrolysis proceeds at different rates, depending upon the compound. At worst (cool temperature, normal pH, no dissolved ions, no microbes), either of these compounds getting into water would be degraded to extremely low levels ($< 1 \times 10^{-6}$ of the original concentration) within a couple of years. It is more likely that this level of degradation would occur much more quickly.

Degradation in soil is a far more complex issue. The rate of hydrolysis will depend upon a variety of factors, including soil moisture, pH, mineral content, microbes, temperature, etc. Most available studies show that these compounds last no more than a few days in the tested soil types.

Sulfur Mustard

In some ways, sulfur mustard behaves in a fashion similar to the nerve agents - i.e. it hydrolyzes rapidly in the presence of water. There is, however, an important difference. Under the right conditions, **the hydrolysis products of mustard can polymerize and form bubbles containing active mustard**. The mustard inside of these bubbles is shielded from further hydrolysis by the hydrolysis products. This has turned out to be a problem in areas where large amounts of mustard were dumped at sea. Fishermen in such areas have been injured when pulling up nets contaminated with blobs of active mustard. An indication that this may have occurred would be a biphasic sample. This situation can also occur in soil.

Potential Hazards to TestAmerica Sacramento Personnel

Following steps listed in this appendix and other safety policies will help reduce hazards to the greatest degree possible. However, such policies are no substitute for educated, observant personnel.

As always, you must think about what you are doing when you handle these materials. No policy can account for every potential situation. Staff members are expected to follow all sample handling policies identified in the Corporate Safety Manual and steps following sample receipt listed in this appendix.

Soil and water samples will be screened for agent prior to their shipment to TestAmerica Sacramento, unless an exception has been granted by the Corporate Director of EH&S. Data should be reviewed at the project management and EH&S staff level to ensure samples are “safe” for handling. If the screening status is unknown (i.e. no data is available), project management personnel should be consulted. Samples will **not** be handled. If staff are unavailable, the cooler will be left in cold storage until the situation is resolved. The expiration of analytical holding times will not be considered as sufficient reason to handle/process CWM samples prior to receipt of screening data.

Positive “hits” on samples containing by-products of agents (like EA 2192) must also be reviewed at the project management and EH&S staff level to ensure samples are “safe” for staff to handle.

When in doubt, seek assistance from project management, operations manager, or EH&S staff.

Corporate EH&S staff are also a resource which must be consulted before deciding to proceed with any “questionable” samples received.

Sample Receipt Procedures

⇒ Follow WS-QA-003

⇒ Review screening data BEFORE opening the cooler if soil samples.

Note: Double gloves are required when handling chemical warfare degradate samples.

⇒ Following established safety policies, open the cooler, remove any paperwork, and check the interior condition.

⇒ All coolers from CWM sites will be initially opened in a fume hood. Once you have determined that there are no broken or leaking sample containers, the cooler may be moved to a bench top for further processing.

⇒ If a cooler contains a broken or leaking sample, isolate the cooler in the hood and IMMEDIATELY contact the project manager, operation manager and/or EH&S staff.

⇒ Any sample which appears to be biphasic in appearance must be isolated. Immediate notification to project management, operation manager and/or EH&S staff is required.

⇒ When in doubt, get help regarding sample receipt. Worker health and safety is **paramount** to sample analysis.

⇒ Regardless of screening data, any cooler containing samples for degradate analysis (or any other samples from an area suspected of potential agent contamination) **should be inspected carefully** upon receipt. Anyone inspecting the samples, logging them in, or handling them for any other reason should observe all of TestAmerica Sacramento’s regular safety procedures.

In addition, **two pairs of gloves** will be worn in order to minimize any potential for skin contact with toxic compounds. Please note that **skin contact appears to be the most likely potential route of exposure**. This is based on the fact that nerve agents are likely to have degraded leaving sulfur mustard as the most likely potential contaminant and due to the likelihood that the samples will be cold. The temperature in the cooler is important from the standpoint of safety as well as sample integrity - cold samples mean a significantly lower potential for any kind of toxic vapor formation. Coolers containing broken jars or bottles should be placed in a hood immediately and left there until the client has been contacted. EH&S staff and/or the project managers will give instructions regarding return to client or disposal based on the screening data.

In conclusion, it must be emphasized that these samples must be handled with the appropriate level of care. Observant, educated personnel are our best defense against exposure to any kind of toxic materials found in our samples.

APPENDIX 8

Handling of Blood or Other Potentially Infectious Material

Background

TestAmerica Sacramento has, upon occasion, received a variety of biological samples for various environmental analyses. Biological samples present a very different type of hazard than “typical” environmental samples. Depending on the type of sample delivered for analysis, and the types of analyses requested, a variety of additional precautions and protective measures may be required when receiving, processing and storing these samples.

Types of Biohazard Samples

There are many types of biohazard samples. Not all biological samples are necessarily biohazard samples. Some of the types of biohazard samples that have been received at TestAmerica Sacramento in the past include:

- Human blood
- Human tissue
- Human breast milk
- Rodent or other mammalian tissue
- Human waste products, usually samples from municipal sewage treatment plants

While fish, crawfish, clams, plant tissue, grasses, and such are all biological samples, they are not generally considered to be a biohazard threat.

Specific Hazards Associated with Biohazard Samples

The unique threat associated with biohazard samples is infectious diseases. Typically, these are Human Immuno Virus (HIV) and Hepatitis B. Other potential hazards include (but are not limited to) rabies, bubonic plague and the Hanta virus. Some of these hazards we are prepared to work with effectively, and others we are not.

Samples potentially infected with bubonic plague or the Hanta virus requires engineering controls that are not in place at TestAmerica Sacramento. Accordingly, we will not accept samples potentially infected by these diseases. These samples include whole rodents or other mammals, mammal parts, or homogenized mammal tissue. Mammal tissue samples that are known NOT to be infected with these diseases may be accepted for analysis under certain conditions. These samples must be homogenized and the sample tissue must be “fixed” in a 4% or higher formalin solution. The outside of the sample container must have been disinfected with a Centers for Disease Control (CDC) approved disinfectant after the sample was placed in the container but before it was shipped to us. Examples of this disinfectant solution are a 10% bleach solution or a 5% Lysol solution.

General Procedures

Universal precautions: All human and mammal blood, fluid and tissue samples are assumed to be infectious. All staff members will wear two pairs of protective gloves when handling or working with biohazard samples. Safety glasses and a face shield are required. Fume hood sashes will be closed as far as possible, consistent with safe work practices. Lab coats will always be worn and buttoned up. Lab coats worn when handling biohazard samples will not be worn outside of the laboratory. When work is finished with biohazard samples, lab coats worn during the process will be sent out for cleaning. If they have been splashed or contaminated with any infectious sample, they will be disposed of as biohazardous waste. Workers will exercise caution to avoid injury with tools possibly infected from biohazard work, such as glass pipettes, metal spatulas, broken glass, etc. All waste material will be disposed of in appropriately marked containers as biohazardous waste. Workers with open wounds, sores or broken skin shall not handle biohazardous samples. Pregnant workers shall be especially familiar with and adhere to precautions to minimize the risk of transmission. Employees

involved with handling human blood or tissue samples will be offered the opportunity to receive the Hepatitis B vaccination series. This may be accepted, declined, or accepted at a later date.

Engineering and administrative controls: Signs will be posted on all doorways leading into areas where biohazardous samples are being handled. These signs will be clearly visible and will identify that biohazard work is in progress. When these signs are posted, personnel not involved in the work will stay out of the work area. If this is impractical, the biohazard work will be performed in an isolated area that is clearly marked. No one may enter this area without permission from the sample administration technician or chemist doing the work. Personal protective equipment will be removed immediately upon leaving the area and disposed of or cleaned properly. Eating, drinking, use of tobacco products, gum, hard candy, applying cosmetics or lip balm and use of contact lenses are all prohibited in any areas where biohazard samples are being handled. Employees working with biohazard samples will thoroughly wash their hands with disinfectant soap when finished work and before leaving the lab. Work areas will be thoroughly cleaned and disinfected when biohazard work is complete. This includes properly disposing of bench paper and used equipment such as pipets, disinfecting all reusable equipment such as glassware, metal spatulas, and disinfecting work surfaces. Broken glassware that is potentially infected must not be picked up directly with the hands. Any trash cans or containers that may have been contaminated will be inspected, cleaned, and disinfected with an appropriate disinfecting solution.

Sample Receipt Procedures

Follow WS-QA-0003

If advance notification is provided of incoming biohazard samples, contact EH&S, review this appendix and ensure that you are familiar with the safety procedures involved. Ensure that you have a clear workspace, that you know in advance where the samples will be stored, and that there is space available to store them.

Note: All potential biohazard samples must be kept in locked storage - either WR1 or WF1.

Note: Double gloves and a face shield are required when handling biohazard samples.

Biohazard samples shall be opened in a fume hood.

Following established procedures, open the cooler, remove any paperwork and check the interior condition.

If a shipping container has a broken or leaking sample, isolate the cooler in a fume hood and IMMEDIATELY contact the project manager and EH&S staff.

Note any comments or warnings on sample containers (including shipment paperwork) regarding specific threats or hazards.

When in doubt, get help regarding sample receipt. Your health and safety is of paramount importance.

The most likely methods of transmission of disease when handling biohazard samples are splashing infected blood or tissue onto an open cut or sore or into your eyes, mucous membranes or mouth. The likelihood of transmission via these routes can be almost completely eliminated by following proper procedures.

Exercise care when handling samples so that they do not drop or get knocked over.

Wear two pair of protective gloves – latex, vinyl or nitrile.

Don't work around biohazard samples with open cuts or sores.

Wear your safety glasses with a face shield.

Ensure that all skin is covered, such as your wrists and forearms

Wear your lab coat, properly fastened.

APPENDIX 9

After Hours Sample Receipt Procedures for Non-Sample Administrative Personnel

Normal business hours for receiving samples are Monday through Friday, 8:00 am to 6:00 pm, and Saturday, 8:00 am to 12:00 pm. In the event that samples are delivered outside of normal operating hours, and only non-Sample Administrative personnel are available to accept the delivery, the following procedures should be followed:

- If samples are delivered directly by a client, have him/her relinquish the COC, sign your name on the "Received By" line, note the time received, and make a copy of the signed COC for the client. Keep the original COC with the samples.
- If samples are delivered by a courier, and the COC is taped inside a cooler, note who delivered the samples and what time they arrived on a "Notes Form" (QA-812, an example is in Appendix 11 of this SOP).
- Record the date/time received and the custody seal IDs on the "Notes Form". Open the cooler/container and measure the temperature of the samples and/or temperature blank (if easily accessible) using the IR or AK thermometer. Do not take the temperature of ice or packing material. Record the temperature(s) on the form.
- If samples are not contained in a cooler, document what kind of cooling agents were used, if any, on the Notes Form. Put the form with the COC.
- Place cooler/samples on a cart and store in the walk-in refrigerator.
- Send an email to "SACSC" to notify them that samples were received outside of normal hours, and include any pertinent information (i.e. when received, who delivered the samples, where they are located, sample receipt temperatures, etc.) to assist them in processing the samples when they return.

APPENDIX 11

Canister Receipt Notes

Airbill Here



Sacramento

Sample Receiving Notes - Job _____

Use this form to record Sample Custody Seal, Cooler Custody Seal, Temperature & corrected Temperature & other observations. File in the job folder with the COC.

Notes:	Cooler Custody Seal:
	# Bags: _____ 1L, _____ 2L, _____ 10L
	# Canisters: _____ 1L, _____ 6L, TA ☐ Non TA ☐
	Transferred by Sacramento - Yes ☐ No ☐
	# Canisters Unused: _____ 1L, _____ 6L
	# Flow Regulators: _____, # Gauges: _____
	Co-locator _____
	Initial & Date _____

APPENDIX 12

Revenue Source Tracking



Corporate Quality

Work Instruction for
Laboratory / Service Center Operations

This Work Instruction is to be attached as an Appendix to Location-Specific Sample Receiving and / or Shipping Standard Operating Procedures (SOPs).

This Work Instruction is in effect as of 7/1/2012 in all Locations.

The purpose of this Work Instruction is to provide instructions to be followed in the gathering of data to track revenue sources from service centers and transitioning from re-missioned locations. This information is collected from the processes employed in sample shipment and receiving.

Label Generation

Labels will be printed centrally and supplied to each location.

Labels will be generated with location name and a bar code.

Labels will be generated for all service center locations, including those labs that house service center functions.

Some locations will have multiple labels to allow for multiple aspects of tracking. For instance, Boston will have two label types: Boston and Boston / Westfield.

When a location runs low on labels, they need to contact Janet.McAfee@testamericainc.com to request additional labels. Two week's notice should be provided to obtain additional labels.

Service Center Responsibility (Beginning 7/1/2012)

Each service center will be responsible for affixing a label on the inside of the cooler lid in the upper left corner.

Service Center personnel will label every cooler that they ship out, drop off, pick up, or that is delivered to them from a client, every time.

Service Center personnel need to ensure that any old labels are removed prior to affixing their labels.

NOTE: Labels are to be removed by lab personnel, but in order to ensure integrity of the system, the service center needs to check before applying their label!

In cases where the service center is tracking multiple sources of revenue (such as Boston) the staff at the service center needs to distinguish between clients and / or jobs that were previously sent to a re-mission location such as Westfield. If work for a particular client or job was previously shipped to Westfield, the alternate sticker of "Boston/Westfield" is to be used on the cooler.

Where two locations are involved in providing support, the following is the order of priority for which service center labels should be on the cooler:

1. Location that picked up the samples from the client (provided logistical support)
2. Location that dropped off the bottle kit / cooler to the client (provided logistical support)
3. Location that shipped bottles to the client (in lieu of a lab shipping bottles)

While it isn't common for one service center to provide bottle kits, and a second service center to pick up samples, it can happen. The service center picking up the samples should ensure their label is the only label on the cooler when shipping it to a lab.



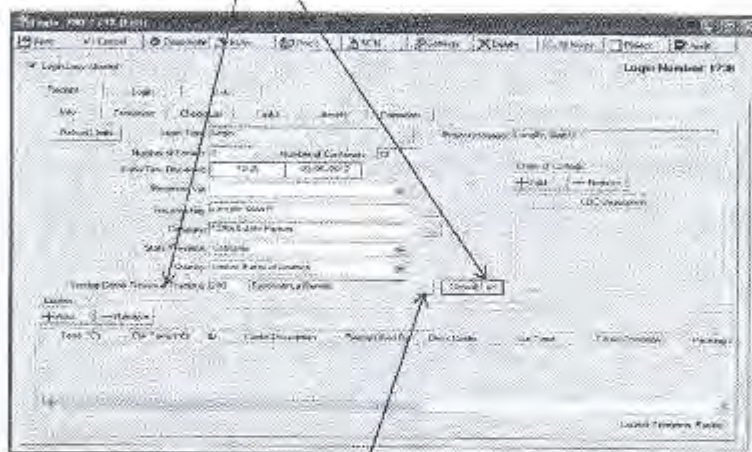
Corporate Quality

Work Instruction for
Laboratory / Service Center Operations

Lab / Sample Receiving Responsibility (Beginning 7/1/2012)

For every log in, sample receiving personnel are responsible to scan the label from the inside of the cooler lid, which will enter the "Service Center Revenue Tracking" into TALs.

If there is no label present on the inside of the cooler lid, sample receiving will select their home location by using the "Default Loc" button.



If the scanning process is not successful (scanning failure, or other reason), the service center location must be selected using the list feature in TALs.

As of July 1, 2012 this date will be considered mandatory in TALs. The login cannot be saved successfully until the Service Center Tracking field information has been completed.

Once the label from the cooler is scanned, the label must be removed from the cooler and thrown away.

THE LEADER IN ENVIRONMENTAL TESTING

Laboratory / Service Center Operations

Project Manager (PM) Responsibility (Beginning 1/1/2012)

The PM's should endeavor to identify the "source" of revenue on the project, or job. For example, if a project was previously handled in Westfield but has transitioned to Buffalo, the PM should select the appropriate **Service Location** as Westfield on the Project screen.

[illegible]



Corporate Quality

**Work Instruction for
Laboratory / Service Center Operations**

The PM may become aware of the "source" of revenue sometime after the project build is complete. The information can also be updated on the Job tab of a login, but the same data field is referred to as **Work Origin** on the Job tab of a login.

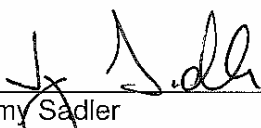
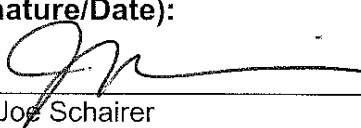
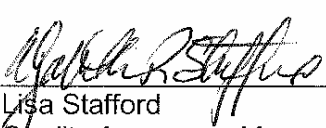
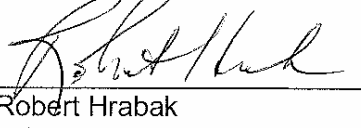
The screenshot shows the 'Job' tab of a login in the TestAmerica system. The 'Work Origin' field is highlighted with a red arrow. The interface includes a menu bar, a toolbar, and a main data entry area with various fields for job details.

Field	Value
Job Description	Pyrite Steel Project
Job ID	113700-1
Job Status	113700-1
Job Date	11/30/2012
Job Time	11:59 PM
Job From	To: 11/30/2012 11:59 PM
Job To	11/30/2012 11:59 PM
Job From	11/30/2012 11:59 PM
Job To	11/30/2012 11:59 PM
Work Origin	Sacramento Workfield

Invoices associated with projects / jobs / logins that are tagged with this information will be tracked as being sourced from a service center and / or re-mission location. It is the responsibility of PM, Service Center and Sample Login staff to ensure every effort is made to properly identify the "Source Location" for all revenues.

Title: Sample Transfer within TALS

[Quality Assurance Procedure]

Approvals (Signature/Date):			
	9/10/14		9/10/14
Jeremy Sadler	Date	Joe Schairer	Date
Technical Manager		Health & Safety Manager / Coordinator	
	9/10/2014		9/10/14
Lisa Stafford	Date	Robert Hrabak	Date
Quality Assurance Manager		Laboratory Manager	

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Sacramento
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1. SCOPE AND APPLICATION

- 1.1. The purpose of this procedure is to provide details for handling transfer of samples, extracts and digestates within the laboratory in accordance with DoD and NELAC requirements.

2. SCOPE

- 2.1. This procedure applies to the transfer of all sample types within the laboratory.

3. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

- 3.1. For hazards associated with the products of a specific analysis or preparation procedure, please refer to the appropriate analytical or preparation SOP.
- 3.2. Specific Safety Concerns or Requirements
 - 3.2.1. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, vinyl and nitrile gloves all provide sufficient protection when handling close sample containers.
 - 3.2.2. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore all samples must be opened, transferred, sub-sampled and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.
 - 3.2.3. Laboratory procedures such as repetitive use of pipettes, repetitive sub-sampling, moving heavy shipping containers, unloading shipping containers, and manipulation of glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

- 3.2.4. Safety policies apply to ALL sample administration visitors, including auditors, employees, couriers or clients who deliver samples.

- 3.3. Primary Material Used

As this procedure is concerned only with the transfer of samples within closed containers within the laboratory, there are no specific chemical materials used.

4. DEFINITIONS

- 4.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

5. PROCEDURE

- 5.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

- 5.2. Running a backlog report

- 5.2.1. At your work station, open TALS.

- 5.2.2. Click “Analyst” and open “Back Log Report”.

- 5.2.3. Select desired department in “Backlog to Run” field.

- 5.2.4. Click “Run”.

- 5.2.5. Filter results based on “Method”, “Sample Matrix”, “Status”, etc.

- 5.2.6. Select containers for sample preparation.

- 5.2.7. Right-click and select “Create SRF” (Sample Request Form).

Note: Some departments can determine container location without creating the SRF.

- 5.2.8. If you selected samples in Scheduled status, select “no” in order to only load samples selected and not all samples in a previous SRF.

- 5.2.9. For each requested sample, select an appropriate container to check out.
- 5.2.10. After each sample has one container selected, click “Save”.
- 5.2.11. Print the SRF.
 - 5.2.11.1. Seldom a dialog box will ask whether you want to print the SRF with the container barcode (generally no), sample notes (generally yes), method notes (generally yes), and login notes (generally yes).

5.3. Transferring samples to a new location.

- 5.3.1. Grab selected containers from the appropriate storage location listed on the SRF.
 - 5.3.1.1. For summa canisters or tedlar bags, the storage location may not be in Sample Control therefore the containers can be checked out of TALS using a nearby computer.
- 5.3.2. Open TALS; under “Sample Management” select “Internal Chain of Custody”.

Note: All containers must be checked out/in in Sample Control.

- 5.3.3. In the drop-down menu for “Search By” select “Lab Sample ID”, click on the blank field, and scan the barcode on each container taken.
 - 5.3.4. If the barcode does not scan, enter the container ID manually.
 - 5.3.5. Once every container is scanned into TALS, right-click on the grid and click “Select All”.
 - 5.3.6. Verify your name is in the “User” box.
 - 5.3.7. In the “Container Location” field, search and select the appropriate department where the containers will be moved.
 - 5.3.8. Once done, click “Transfer Custody/Change Location”.
 - 5.3.9. Take the containers to the appropriate department marked in TALS for sample preparation.
- 5.4. Returning samples to the original storage location.
- Most original sample containers must be returned to Sample Control after aliquots have been removed for analysis. Exceptions are volatile air samples in summa canisters or air sample bags, which are returned to their assigned storage shelf, and samples expended during the analysis. The exceptions are discussed in the procedure below.

- 5.4.1. Bring the containers to Sample Control.
 - 5.4.1.1. For summa canisters or tedlar bags, the storage location may not be in Sample Control. Therefore, the containers can be returned to their storage location in TALS using a nearby computer.
 - 5.4.1.2. If samples were expended during preparation/testing, containers do not need to be returned to sample control, and the rest of the steps may be performed using a nearby computer.
- 5.4.2. Open TALS; under “Sample Management” select “Internal Chain of Custody”.

Note: All containers removed from or returned to Sample Control must be checked out/in in Sample Control.

- 5.4.3. In the drop-down menu for “Search By” select “Lab Sample ID”, click on the blank field, and scan the barcode on each container to be returned.
- 5.4.4. Once every container is scanned into TALS, right-click on the grid and click “Select All”.
- 5.4.5. Verify your name is in the “User” box.
- 5.4.6. Click “Return to Storage Location”.
- 5.4.7. If the entire contents of the container were used, change the container location to “Destroyed in Testing”.
- 5.4.8. If another Prep lab needs the container, change the container location to that lab.
- 5.4.9. Store containers as marked in TALS.

6. RESPONSIBILITIES

- 6.1. Each associate is responsible for using the ICOC function in TALS whenever samples, extracts or digestates are transferred from one storage location to another. Associates are also responsible for marking samples as “Destroyed in Testing” when a sample container is emptied.
- 6.2. The Laboratory Director, Operations Managers, QA Manager, and Departmental Supervisors/Managers of this facility are responsible for ensuring that associates are using the ICOC function in TALS appropriately. It is also their responsibility to supply adequate training, materials, and equipment to enable the employee to perform this procedure correctly.

7. REFERENCES/CROSS REFERENCES

- 7.1. “Internal Chain of Custody”, TALS Training Brief, CW-I-T-090, 12/07/2011
- 7.2. Quality Systems Manual, DoD/DOE QSM, Revision 5, July 2013, Module 2, Section 5.8.8.
- 7.3. 2009 TNI Standard, EL-V1M2-ISO-2009, Section 5.8.8.

8. ATTACHMENTS

- 8.1. Not applicable

9. REVISION HISTORY

- 9.1. WS-QA-0034, Revision 0, Effective 09/19/2014
 - 9.1.1. This is the first version of this policy.



**LABORATORY
ACCREDITATION
BUREAU** a division of A-S-B



Certificate of Accreditation

ISO/IEC 17025:2005

Certificate Number L2305

TestAmerica Laboratories

St. Louis Facility
13715 Rider Trail North
Earth City Missouri 63045

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation valid through: April 6, 2019

R. Douglas Leonard, Jr., President, COO
Laboratory Accreditation Bureau
Presented the 6th of April 2016

*See the laboratory's Scope of Accreditation for details of accredited parameters

**Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

Scope of Accreditation For TestAmerica Laboratories

St. Louis Facility
13715 Rider Trail North
Earth City, Missouri 63045
Marti Ward
314-298-8566

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (LABPR 403 DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM V5) based on the TNI Standard - Environmental Laboratory Sector, Volume 1 – Management and Technical Requirements for Laboratories Performing Environmental Analysis, Sept 2009 (EL-V1-2009); accreditation is granted to **TestAmerica Laboratories** to perform the following tests:

Accreditation granted through: **April 6, 2019**

Testing - Environmental

Non-Potable Water		
Technology	Method	Analyte
ICP-AES	EPA 6010C	Aluminum
ICP-AES	EPA 6010C	Antimony
ICP-AES	EPA 6010C	Arsenic
ICP-AES	EPA 6010C	Barium
ICP-AES	EPA 6010C	Beryllium
ICP-AES	EPA 6010C	Bismuth
ICP-AES	EPA 6010C	Boron
ICP-AES	EPA 6010C	Cadmium
ICP-AES	EPA 6010C	Calcium
ICP-AES	EPA 6010C	Chromium
ICP-AES	EPA 6010C	Cobalt
ICP-AES	EPA 6010C	Copper
ICP-AES	EPA 6010C	Iron
ICP-AES	EPA 6010C	Lead
ICP-AES	EPA 6010C	Lithium
ICP-AES	EPA 6010C	Magnesium

Non-Potable Water		
Technology	Method	Analyte
ICP-AES	EPA 6010C	Manganese
ICP-AES	EPA 6010C	Molybdenum
ICP-AES	EPA 6010C	Nickel
ICP-AES	EPA 6010C	Phosphorus
ICP-AES	EPA 6010C	Potassium
ICP-AES	EPA 6010C	Selenium
ICP-AES	EPA 6010C	Silicon
ICP-AES	EPA 6010C	Silver
ICP-AES	EPA 6010C	Sodium
ICP-AES	EPA 6010C	Strontium
ICP-AES	EPA 6010C	Sulfur
ICP-AES	EPA 6010C	Thallium
ICP-AES	EPA 6010C	Thorium
ICP-AES	EPA 6010C	Tin
ICP-AES	EPA 6010C	Titanium
ICP-AES	EPA 6010C	Vanadium
ICP-AES	EPA 6010C	Zinc
GC/MS	EPA 8260C	Acetone
GC/MS	EPA 8260C	Acetonitrile
GC/MS	EPA 8260C	Acrolein
GC/MS	EPA 8260C	Acrylonitrile
GC/MS	EPA 8260C	Benzene
GC/MS	EPA 8260C	Benzyl chloride
GC/MS	EPA 8260C	Bromobenzene
GC/MS	EPA 8260C	Bromochloromethane
GC/MS	EPA 8260C	Bromodichloromethane
GC/MS	EPA 8260C	Bromoform
GC/MS	EPA 8260C	Bromomethane
GC/MS	EPA 8260C	n-Butanol
GC/MS	EPA 8260C	2-Butanone
GC/MS	EPA 8260C	n-Butylbenzene
GC/MS	EPA 8260C	sec-Butylbenzene
GC/MS	EPA 8260C	tert-Butylbenzene
GC/MS	EPA 8260C	Carbon disulfide
GC/MS	EPA 8260C	Carbon tetrachloride

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260C	Chlorobenzene
GC/MS	EPA 8260C	Chlorobromomethane
GC/MS	EPA 8260C	2-Chloro-1,3-butadiene
GC/MS	EPA 8260C	Chlorodibromomethane
GC/MS	EPA 8260C	Dibromochloromethane
GC/MS	EPA 8260C	Chloroethane
GC/MS	EPA 8260C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260C	Chloroform
GC/MS	EPA 8260C	Chloromethane
GC/MS	EPA 8260C	Allyl chloride
GC/MS	EPA 8260C	2-Chlorotoluene
GC/MS	EPA 8260C	4-Chlorotoluene
GC/MS	EPA 8260C	Cyclohexane
GC/MS	EPA 8260C	Cyclohexanone
GC/MS	EPA 8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260C	1,2-Dibromoethane
GC/MS	EPA 8260C	Dibromomethane
GC/MS	EPA 8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260C	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260C	Dichlorodifluoromethane
GC/MS	EPA 8260C	1,1-Dichloroethane
GC/MS	EPA 8260C	1,2-Dichloroethane
GC/MS	EPA 8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260C	1,1-Dichloroethene
GC/MS	EPA 8260C	1,2-Dichloroethene (total)
GC/MS	EPA 8260C	1,2-Dichloropropane
GC/MS	EPA 8260C	1,3-Dichloropropane
GC/MS	EPA 8260C	2,2-Dichloropropane
GC/MS	EPA 8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260C	1,1-Dichloropropene
GC/MS	EPA 8260C	1,2-Dichloro-1,1,2,2-tetrafluoroethane

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260C	Dimethyl disulfide
GC/MS	EPA 8260C	1,4-Dioxane
GC/MS	EPA 8260C	Ethyl acetate
GC/MS	EPA 8260C	Ethylbenzene
GC/MS	EPA 8260C	Ethyl ether
GC/MS	EPA 8260C	Diethyl ether
GC/MS	EPA 8260C	Ethyl methacrylate
GC/MS	EPA 8260C	Freon 113
GC/MS	EPA 8260C	Hexachlorobutadiene
GC/MS	EPA 8260C	n-Hexane
GC/MS	EPA 8260C	2-Hexanone
GC/MS	EPA 8260C	Iodomethane
GC/MS	EPA 8260C	Isobutanol
GC/MS	EPA 8260C	Isopropylbenzene
GC/MS	EPA 8260C	p-Isopropyltoluene
GC/MS	EPA 8260C	Methacrylonitrile
GC/MS	EPA 8260C	Methyl acetate
GC/MS	EPA 8260C	Methyl butyl ketone
GC/MS	EPA 8260C	Methylcyclohexane
GC/MS	EPA 8260C	Dichloromethane
GC/MS	EPA 8260C	Methylene chloride
GC/MS	EPA 8260C	Methyl methacrylate
GC/MS	EPA 8260C	4-Methyl-2-pentanone
GC/MS	EPA 8260C	MTBE
GC/MS	EPA 8260C	Naphthalene
GC/MS	EPA 8260C	2-Nitropropane
GC/MS	EPA 8260C	Nonanal
GC/MS	EPA 8260C	Pentachloroethane
GC/MS	EPA 8260C	Propionitrile
GC/MS	EPA 8260C	n-Propylbenzene
GC/MS	EPA 8260C	Styrene
GC/MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260C	Tetrachloroethene
GC/MS	EPA 8260C	Tetrahydrofuran

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260C	Toluene
GC/MS	EPA 8260C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260C	Trichloroethene
GC/MS	EPA 8260C	Trichlorofluoromethane
GC/MS	EPA 8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260C	Trichlorotrifluoroethane
GC/MS	EPA 8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C	Vinyl acetate
GC/MS	EPA 8260C	Vinyl chloride
GC/MS	EPA 8260C	m-Xylene & p-Xylene
GC/MS	EPA 8260C	o-Xylene
GC/MS	EPA 8260C	Xylenes (total)
GC/MS	EPA 8260C SIM	1,4-Dioxane
GC/MS	EPA 624	Acetone
GC/MS	EPA 624	Acetonitrile
GC/MS	EPA 624	Acrolein
GC/MS	EPA 624	Acrylonitrile
GC/MS	EPA 624	Benzene
GC/MS	EPA 624	Benzyl chloride
GC/MS	EPA 624	Bromobenzene
GC/MS	EPA 624	Bromochloromethane
GC/MS	EPA 624	Bromodichloromethane
GC/MS	EPA 624	Bromoform
GC/MS	EPA 624	Bromomethane
GC/MS	EPA 624	n-Butanol
GC/MS	EPA 624	2-Butanone
GC/MS	EPA 624	n-Butylbenzene
GC/MS	EPA 624	sec-Butylbenzene
GC/MS	EPA 624	tert-Butylbenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 624	Carbon disulfide
GC/MS	EPA 624	Carbon tetrachloride
GC/MS	EPA 624	Chlorobenzene
GC/MS	EPA 624	Chlorobromomethane
GC/MS	EPA 624	2-Chloro-1,3-butadiene
GC/MS	EPA 624	Chlorodibromomethane
GC/MS	EPA 624	Dibromochloromethane
GC/MS	EPA 624	Chloroethane
GC/MS	EPA 624	2-Chloroethyl vinyl ether
GC/MS	EPA 624	Chloroform
GC/MS	EPA 624	Chloromethane
GC/MS	EPA 624	Allyl chloride
GC/MS	EPA 624	2-Chlorotoluene
GC/MS	EPA 624	4-Chlorotoluene
GC/MS	EPA 624	Cyclohexane
GC/MS	EPA 624	Cyclohexanone
GC/MS	EPA 624	1,2-Dibromo-3-chloropropane
GC/MS	EPA 624	1,2-Dibromoethane
GC/MS	EPA 624	Dibromomethane
GC/MS	EPA 624	1,2-Dichlorobenzene
GC/MS	EPA 624	1,3-Dichlorobenzene
GC/MS	EPA 624	1,4-Dichlorobenzene
GC/MS	EPA 624	trans-1,4-Dichloro-2-butene
GC/MS	EPA 624	Dichlorodifluoromethane
GC/MS	EPA 624	1,1-Dichloroethane
GC/MS	EPA 624	1,2-Dichloroethane
GC/MS	EPA 624	cis-1,2-Dichloroethene
GC/MS	EPA 624	trans-1,2-Dichloroethene
GC/MS	EPA 624	1,1-Dichloroethene
GC/MS	EPA 624	1,2-Dichloroethene (total)
GC/MS	EPA 624	1,2-Dichloropropane
GC/MS	EPA 624	1,3-Dichloropropane
GC/MS	EPA 624	2,2-Dichloropropane
GC/MS	EPA 624	cis-1,3-Dichloropropene
GC/MS	EPA 624	trans-1,3-Dichloropropene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 624	1,1-Dichloropropene
GC/MS	EPA 624	1,2-Dichloro-1,1,2,2-tetrafluoroethane
GC/MS	EPA 624	Dimethyl disulfide
GC/MS	EPA 624	1,4-Dioxane
GC/MS	EPA 624	Ethyl acetate
GC/MS	EPA 624	Ethylbenzene
GC/MS	EPA 624	Ethyl ether
GC/MS	EPA 624	Diethyl ether
GC/MS	EPA 624	Ethyl methacrylate
GC/MS	EPA 624	Freon 113
GC/MS	EPA 624	Hexachlorobutadiene
GC/MS	EPA 624	n-Hexane
GC/MS	EPA 624	2-Hexanone
GC/MS	EPA 624	Iodomethane
GC/MS	EPA 624	Isobutanol
GC/MS	EPA 624	Isopropylbenzene
GC/MS	EPA 624	p-Isopropyltoluene
GC/MS	EPA 624	Methacrylonitrile
GC/MS	EPA 624	Methyl acetate
GC/MS	EPA 624	Methyl butyl ketone
GC/MS	EPA 624	Methylcyclohexane
GC/MS	EPA 624	Dichloromethane
GC/MS	EPA 624	Methylene chloride
GC/MS	EPA 624	Methyl methacrylate
GC/MS	EPA 624	4-Methyl-2-pentanone
GC/MS	EPA 624	MTBE
GC/MS	EPA 624	Naphthalene
GC/MS	EPA 624	2-Nitropropane
GC/MS	EPA 624	Nonanal
GC/MS	EPA 624	Pentachloroethane
GC/MS	EPA 624	Propionitrile
GC/MS	EPA 624	n-Propylbenzene
GC/MS	EPA 624	Styrene
GC/MS	EPA 624	1,1,1,2-Tetrachloroethane
GC/MS	EPA 624	1,1,2,2-Tetrachloroethane

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 624	Tetrachloroethene
GC/MS	EPA 624	Tetrahydrofuran
GC/MS	EPA 624	Toluene
GC/MS	EPA 624	1,3,5-Trichlorobenzene
GC/MS	EPA 624	1,2,3-Trichlorobenzene
GC/MS	EPA 624	1,2,4-Trichlorobenzene
GC/MS	EPA 624	1,1,1-Trichloroethane
GC/MS	EPA 624	1,1,2-Trichloroethane
GC/MS	EPA 624	Trichloroethene
GC/MS	EPA 624	Trichlorofluoromethane
GC/MS	EPA 624	1,2,3-Trichloropropane
GC/MS	EPA 624	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 624	Trichlorotrifluoroethane
GC/MS	EPA 624	1,2,4-Trimethylbenzene
GC/MS	EPA 624	1,3,5-Trimethylbenzene
GC/MS	EPA 624	Vinyl acetate
GC/MS	EPA 624	Vinyl chloride
GC/MS	EPA 624	m-Xylene & p-Xylene
GC/MS	EPA 624	o-Xylene
GC/MS	EPA 624	Xylenes (total)
GC/MS	EPA 8270D	Acenaphthene
GC/MS	EPA 8270D	Acenaphthylene
GC/MS	EPA 8270D	Acetophenone
GC/MS	EPA 8270D	2-Acetylaminofluorene
GC/MS	EPA 8270D	4-Aminobiphenyl
GC/MS	EPA 8270D	Aniline
GC/MS	EPA 8270D	Anthracene
GC/MS	EPA 8270D	Aramite (total)
GC/MS	EPA 8270D	Atrazine
GC/MS	EPA 8270D	Azobenzene
GC/MS	EPA 8270D	Benzaldehyde
GC/MS	EPA 8270D	Benzidine
GC/MS	EPA 8270D	Benzo(a)anthracene
GC/MS	EPA 8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270D	Benzo(k)fluoranthene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270D	Benzoic acid
GC/MS	EPA 8270D	Benzo(ghi)perylene
GC/MS	EPA 8270D	Benzo(a)pyrene
GC/MS	EPA 8270D	Benzyl alcohol
GC/MS	EPA 8270D	1,1'-Biphenyl
GC/MS	EPA 8270D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270D	bis(2-Chloroisopropyl) ether
GC/MS	EPA 8270D	bis(2-Ethylhexyl) phthalate
GC/MS	EPA 8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270D	n-Butylbenzenesulfonamide
GC/MS	EPA 8270D	Butyl benzyl phthalate
GC/MS	EPA 8270D	Caprolactam
GC/MS	EPA 8270D	Carbazole
GC/MS	EPA 8270D	4-Chloroaniline
GC/MS	EPA 8270D	Chlorobenzilate
GC/MS	EPA 8270D	p-Chlorobenzilate
GC/MS	EPA 8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270D	2-Chloronaphthalene
GC/MS	EPA 8270D	2-Chlorophenol
GC/MS	EPA 8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270D	Chrysene
GC/MS	EPA 8270D	Cresols (total)
GC/MS	EPA 8270D	Cyclohexanol
GC/MS	EPA 8270D	Diallate
GC/MS	EPA 8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270D	Dibenzo(a,h)anthracene
GC/MS	EPA 8270D	Dibenzofuran
GC/MS	EPA 8270D	Di-n-butyl phthalate
GC/MS	EPA 8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270D	2,4-Dichlorophenol
GC/MS	EPA 8270D	2,6-Dichlorophenol
GC/MS	EPA 8270D	Diethyl phthalate

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270D	O,O-Diethyl-O-(2-pyrazinyl) phosphorothioate
GC/MS	EPA 8270D	Dimethoate
GC/MS	EPA 8270D	p-Dimethylaminoazobenzene
GC/MS	EPA 8270D	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270D	3,3'-Dimethylbenzidine
GC/MS	EPA 8270D	Dimethylformamide
GC/MS	EPA 8270D	alpha,alpha-Dimethylphenethylamine
GC/MS	EPA 8270D	2,4-Dimethylphenol
GC/MS	EPA 8270D	Dimethyl phthalate
GC/MS	EPA 8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270D	1,4-Dinitrobenzene
GC/MS	EPA 8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270D	2,4-Dinitrophenol
GC/MS	EPA 8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270D	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270D	Dinoseb
GC/MS	EPA 8270D	Di-n-octyl phthalate
GC/MS	EPA 8270D	1,4-Dioxane
GC/MS	EPA 8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270D	Disulfoton
GC/MS	EPA 8270D	Ethyl methacrylate
GC/MS	EPA 8270D	Ethyl methanesulfonate
GC/MS	EPA 8270D	Famphur
GC/MS	EPA 8270D	Fluoranthene
GC/MS	EPA 8270D	Fluorene
GC/MS	EPA 8270D	Hexachlorobenzene
GC/MS	EPA 8270D	Hexachlorobutadiene
GC/MS	EPA 8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270D	Hexachloro-1,3-cyclopentadiene
GC/MS	EPA 8270D	Hexachloroethane
GC/MS	EPA 8270D	Hexachlorophene
GC/MS	EPA 8270D	Hexachloropropene
GC/MS	EPA 8270D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D	Isodrin
GC/MS	EPA 8270D	Isophorone

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270D	Isosafrole
GC/MS	EPA 8270D	Kepone
GC/MS	EPA 8270D	Methapyrilene
GC/MS	EPA 8270D	2-Methylbenzenamine
GC/MS	EPA 8270D	3-Methylcholanthrene
GC/MS	EPA 8270D	4,4'-Methylenebis(2-chloroaniline)
GC/MS	EPA 8270D	Methyl methacrylate
GC/MS	EPA 8270D	Methyl methanesulfonate
GC/MS	EPA 8270D	2-Methylnaphthalene
GC/MS	EPA 8270D	Methyl parathion
GC/MS	EPA 8270D	2-Methylphenol
GC/MS	EPA 8270D	3-Methylphenol & 4-Methylphenol
GC/MS	EPA 8270D	2-Methylphenol, 3-methylphenol and 4-methylphenol
GC/MS	EPA 8270D	Methylphenols (total)
GC/MS	EPA 8270D	Naphthalene
GC/MS	EPA 8270D	1,4-Naphthoquinone
GC/MS	EPA 8270D	1-Naphthylamine
GC/MS	EPA 8270D	2-Naphthylamine
GC/MS	EPA 8270D	2-Nitroaniline
GC/MS	EPA 8270D	3-Nitroaniline
GC/MS	EPA 8270D	4-Nitroaniline
GC/MS	EPA 8270D	Nitrobenzene
GC/MS	EPA 8270D	2-Nitrophenol
GC/MS	EPA 8270D	4-Nitrophenol
GC/MS	EPA 8270D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270D	N-Nitrosodi-n-butylamine
GC/MS	EPA 8270D	N-Nitrosodiethylamine
GC/MS	EPA 8270D	N-Nitrosodimethylamine
GC/MS	EPA 8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270D	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270D	N-Nitrosomethylethylamine
GC/MS	EPA 8270D	N-Nitrosomorpholine
GC/MS	EPA 8270D	N-Nitrosopiperidine
GC/MS	EPA 8270D	N-Nitrosopyrrolidine
GC/MS	EPA 8270D	5-Nitro-o-toluidine
GC/MS	EPA 8270D	2,2'-oxybis(1-Chloropropane)

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270D	Parathion
GC/MS	EPA 8270D	Pentachlorobenzene
GC/MS	EPA 8270D	Pentachloroethane
GC/MS	EPA 8270D	Pentachloronitrobenzene
GC/MS	EPA 8270D	Pentachlorophenol
GC/MS	EPA 8270D	Phenacetin
GC/MS	EPA 8270D	Phenanthrene
GC/MS	EPA 8270D	Phenol
GC/MS	EPA 8270D	p-Phenylene diamine
GC/MS	EPA 8270D	Phorate
GC/MS	EPA 8270D	2-Picoline
GC/MS	EPA 8270D	Pronamide
GC/MS	EPA 8270D	Pyrene
GC/MS	EPA 8270D	Pyridine
GC/MS	EPA 8270D	Safrole
GC/MS	EPA 8270D	Sulfotepp
GC/MS	EPA 8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270D	Tetraethyldithiopyrophosphate (Sulfotepp)
GC/MS	EPA 8270D	Thionazin
GC/MS	EPA 8270D	o-Toluidine
GC/MS	EPA 8270D	Tributyl phosphate
GC/MS	EPA 8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270D	O,O,O-Triethyl phosphorothioate
GC/MS	EPA 8270D	1,3,5-Trinitrobenzene
GC/MS	EPA 8270D	Tris(2-chloroethyl)phosphate
GC/MS	EPA 8270D	1-Methyl naphthalene
GC/MS	EPA 625	Acenaphthene
GC/MS	EPA 625	Acenaphthylene
GC/MS	EPA 625	Acetophenone
GC/MS	EPA 625	2-Acetylaminofluorene
GC/MS	EPA 625	4-Aminobiphenyl
GC/MS	EPA 625	Aniline
GC/MS	EPA 625	Anthracene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 625	Aramite (total)
GC/MS	EPA 625	Atrazine
GC/MS	EPA 625	Azobenzene
GC/MS	EPA 625	Benzaldehyde
GC/MS	EPA 625	Benzidine
GC/MS	EPA 625	Benzo(a)anthracene
GC/MS	EPA 625	Benzo(b)fluoranthene
GC/MS	EPA 625	Benzo(k)fluoranthene
GC/MS	EPA 625	Benzoic acid
GC/MS	EPA 625	Benzo(ghi)perylene
GC/MS	EPA 625	Benzo(a)pyrene
GC/MS	EPA 625	Benzyl alcohol
GC/MS	EPA 625	1,1'-Biphenyl
GC/MS	EPA 625	bis(2-Chloroethoxy)methane
GC/MS	EPA 625	bis(2-Chloroethyl) ether
GC/MS	EPA 625	bis(2-Chloroisopropyl) ether
GC/MS	EPA 625	bis(2-Ethylhexyl) phthalate
GC/MS	EPA 625	4-Bromophenyl phenyl ether
GC/MS	EPA 625	n-Butylbenzenesulfonamide
GC/MS	EPA 625	Butyl benzyl phthalate
GC/MS	EPA 625	Caprolactam
GC/MS	EPA 625	Carbazole
GC/MS	EPA 625	4-Chloroaniline
GC/MS	EPA 625	Chlorobenzilate
GC/MS	EPA 625	p-Chlorobenzilate
GC/MS	EPA 625	4-Chloro-3-methylphenol
GC/MS	EPA 625	2-Chloronaphthalene
GC/MS	EPA 625	2-Chlorophenol
GC/MS	EPA 625	4-Chlorophenyl phenyl ether
GC/MS	EPA 625	Chrysene
GC/MS	EPA 625	Cresols (total)
GC/MS	EPA 625	Cyclohexanol
GC/MS	EPA 625	Diallate
GC/MS	EPA 625	Dibenz(a,h)anthracene
GC/MS	EPA 625	Dibenzo(a,h)anthracene
GC/MS	EPA 625	Dibenzofuran
GC/MS	EPA 625	Di-n-butyl phthalate
GC/MS	EPA 625	1,2-Dichlorobenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 625	1,3-Dichlorobenzene
GC/MS	EPA 625	1,4-Dichlorobenzene
GC/MS	EPA 625	3,3'-Dichlorobenzidine
GC/MS	EPA 625	2,4-Dichlorophenol
GC/MS	EPA 625	2,6-Dichlorophenol
GC/MS	EPA 625	Diethyl phthalate
GC/MS	EPA 625	O,O-Diethyl-O-(2-pyrazinyl) phosphorothioate
GC/MS	EPA 625	Dimethoate
GC/MS	EPA 625	p-Dimethylaminoazobenzene
GC/MS	EPA 625	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 625	3,3'-Dimethylbenzidine
GC/MS	EPA 625	Dimethylformamide
GC/MS	EPA 625	alpha,alpha-Dimethylphenethylamine
GC/MS	EPA 625	2,4-Dimethylphenol
GC/MS	EPA 625	Dimethyl phthalate
GC/MS	EPA 625	1,3-Dinitrobenzene
GC/MS	EPA 625	1,4-Dinitrobenzene
GC/MS	EPA 625	4,6-Dinitro-2-methylphenol
GC/MS	EPA 625	2,4-Dinitrophenol
GC/MS	EPA 625	2,4-Dinitrotoluene
GC/MS	EPA 625	2,6-Dinitrotoluene
GC/MS	EPA 625	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 625	Dinoseb
GC/MS	EPA 625	Di-n-octyl phthalate
GC/MS	EPA 625	1,4-Dioxane
GC/MS	EPA 625	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 625	Disulfoton
GC/MS	EPA 625	Ethyl methacrylate
GC/MS	EPA 625	Ethyl methanesulfonate
GC/MS	EPA 625	Famphur
GC/MS	EPA 625	Fluoranthene
GC/MS	EPA 625	Fluorene
GC/MS	EPA 625	Hexachlorobenzene
GC/MS	EPA 625	Hexachlorobutadiene
GC/MS	EPA 625	Hexachlorocyclopentadiene
GC/MS	EPA 625	Hexachloro-1,3-cyclopentadiene
GC/MS	EPA 625	Hexachloroethane
GC/MS	EPA 625	Hexachlorophene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 625	Hexachloropropene
GC/MS	EPA 625	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 625	Isodrin
GC/MS	EPA 625	Isophorone
GC/MS	EPA 625	Isosafrole
GC/MS	EPA 625	Kepone
GC/MS	EPA 625	Methapyrilene
GC/MS	EPA 625	2-Methylbenzenamine
GC/MS	EPA 625	3-Methylcholanthrene
GC/MS	EPA 625	4,4'-Methylenebis(2-chloroaniline)
GC/MS	EPA 625	Methyl methacrylate
GC/MS	EPA 625	Methyl methanesulfonate
GC/MS	EPA 625	2-Methylnaphthalene
GC/MS	EPA 625	Methyl parathion
GC/MS	EPA 625	2-Methylphenol
GC/MS	EPA 625	3-Methylphenol & 4-Methylphenol
GC/MS	EPA 625	2-Methylphenol, 3-methylphenol and 4-methylphenol
GC/MS	EPA 625	Methylphenols (total)
GC/MS	EPA 625	Naphthalene
GC/MS	EPA 625	1,4-Naphthoquinone
GC/MS	EPA 625	1-Naphthylamine
GC/MS	EPA 625	2-Naphthylamine
GC/MS	EPA 625	2-Nitroaniline
GC/MS	EPA 625	3-Nitroaniline
GC/MS	EPA 625	4-Nitroaniline
GC/MS	EPA 625	Nitrobenzene
GC/MS	EPA 625	2-Nitrophenol
GC/MS	EPA 625	4-Nitrophenol
GC/MS	EPA 625	4-Nitroquinoline-1-oxide
GC/MS	EPA 625	N-Nitrosodi-n-butylamine
GC/MS	EPA 625	N-Nitrosodiethylamine
GC/MS	EPA 625	N-Nitrosodimethylamine
GC/MS	EPA 625	N-Nitrosodiphenylamine
GC/MS	EPA 625	N-Nitrosodi-n-propylamine
GC/MS	EPA 625	N-Nitrosomethylethylamine
GC/MS	EPA 625	N-Nitrosomorpholine
GC/MS	EPA 625	N-Nitrosopiperidine
GC/MS	EPA 625	N-Nitrosopyrrolidine

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 625	5-Nitro-o-toluidine
GC/MS	EPA 625	2,2'-oxybis(1-Chloropropane)
GC/MS	EPA 625	Parathion
GC/MS	EPA 625	Pentachlorobenzene
GC/MS	EPA 625	Pentachloroethane
GC/MS	EPA 625	Pentachloronitrobenzene
GC/MS	EPA 625	Pentachlorophenol
GC/MS	EPA 625	Phenacetin
GC/MS	EPA 625	Phenanthrene
GC/MS	EPA 625	Phenol
GC/MS	EPA 625	p-Phenylene diamine
GC/MS	EPA 625	Phorate
GC/MS	EPA 625	2-Picoline
GC/MS	EPA 625	Pronamide
GC/MS	EPA 625	Pyrene
GC/MS	EPA 625	Pyridine
GC/MS	EPA 625	Safrole
GC/MS	EPA 625	Sulfotepp
GC/MS	EPA 625	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 625	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 625	Tetraethyldithiopyrophosphate (Sulfotepp)
GC/MS	EPA 625	Thionazin
GC/MS	EPA 625	o-Toluidine
GC/MS	EPA 625	Tributyl phosphate
GC/MS	EPA 625	1,2,4-Trichlorobenzene
GC/MS	EPA 625	2,4,5-Trichlorophenol
GC/MS	EPA 625	2,4,6-Trichlorophenol
GC/MS	EPA 625	O,O,O-Triethyl phosphorothioate
GC/MS	EPA 625	1,3,5-Trinitrobenzene
GC/MS	EPA 625	Tris(2-chloroethyl)phosphate
GC/MS	EPA 625	1-Methyl naphthalene
GC-ECD	EPA 8081B	Aldrin
GC-ECD	EPA 8081B	alpha-BHC
GC-ECD	EPA 8081B	beta-BHC
GC-ECD	EPA 8081B	delta-BHC
GC-ECD	EPA 8081B	gamma-BHC (Lindane)
GC-ECD	EPA 8081B	alpha-Chlordane
GC-ECD	EPA 8081B	gamma-Chlordane

Non-Potable Water		
Technology	Method	Analyte
GC-ECD	EPA 8081B	Chlordane (technical)
GC-ECD	EPA 8081B	4,4'-DDD
GC-ECD	EPA 8081B	2,4'-DDD
GC-ECD	EPA 8081B	4,4'-DDE
GC-ECD	EPA 8081B	2,4'-DDE
GC-ECD	EPA 8081B	4,4'-DDT
GC-ECD	EPA 8081B	2,4'-DDT
GC-ECD	EPA 8081B	Dieldrin
GC-ECD	EPA 8081B	Endosulfan I
GC-ECD	EPA 8081B	Endosulfan II
GC-ECD	EPA 8081B	Endosulfan sulfate
GC-ECD	EPA 8081B	Endrin
GC-ECD	EPA 8081B	Endrin aldehyde
GC-ECD	EPA 8081B	Endrin ketone
GC-ECD	EPA 8081B	Heptachlor
GC-ECD	EPA 8081B	Heptachlor epoxide
GC-ECD	EPA 8081B	Methoxychlor
GC-ECD	EPA 8081B	Toxaphene
GC-ECD	EPA 608	Aldrin
GC-ECD	EPA 608	alpha-BHC
GC-ECD	EPA 608	beta-BHC
GC-ECD	EPA 608	delta-BHC
GC-ECD	EPA 608	gamma-BHC (Lindane)
GC-ECD	EPA 608	alpha-Chlordane
GC-ECD	EPA 608	gamma-Chlordane
GC-ECD	EPA 608	Chlordane (technical)
GC-ECD	EPA 608	4,4'-DDD
GC-ECD	EPA 608	2,4'-DDD
GC-ECD	EPA 608	4,4'-DDE
GC-ECD	EPA 608	2,4'-DDE
GC-ECD	EPA 608	4,4'-DDT
GC-ECD	EPA 608	2,4'-DDT
GC-ECD	EPA 608	Dieldrin
GC-ECD	EPA 608	Endosulfan I
GC-ECD	EPA 608	Endosulfan II
GC-ECD	EPA 608	Endosulfan sulfate

Non-Potable Water		
Technology	Method	Analyte
GC-ECD	EPA 608	Endrin
GC-ECD	EPA 608	Endrin aldehyde
GC-ECD	EPA 608	Endrin ketone
GC-ECD	EPA 608	Heptachlor
GC-ECD	EPA 608	Heptachlor epoxide
GC-ECD	EPA 608	Methoxychlor
GC-ECD	EPA 608	Toxaphene
GC-ECD	EPA 608	Aroclor 1016
GC-ECD	EPA 608	Aroclor 1221
GC-ECD	EPA 608	Aroclor 1232
GC-ECD	EPA 608	Aroclor 1242
GC-ECD	EPA 608	Aroclor 1248
GC-ECD	EPA 608	Aroclor 1254
GC-ECD	EPA 608	Aroclor 1260
GC-ECD	EPA 608	Aroclor 1262
GC-ECD	EPA 608	Aroclor 1268
GC-ECD	EPA 8082A	Aroclor 1016
GC-ECD	EPA 8082A	Aroclor 1221
GC-ECD	EPA 8082A	Aroclor 1232
GC-ECD	EPA 8082A	Aroclor 1242
GC-ECD	EPA 8082A	Aroclor 1248
GC-ECD	EPA 8082A	Aroclor 1254
GC-ECD	EPA 8082A	Aroclor 1260
GC-ECD	EPA 8082A	Aroclor 1262
GC-ECD	EPA 8082A	Aroclor 1268
GC-ECD	EPA 8151A	2,4-D
GC-ECD	EPA 8151A	Dalapon
GC-ECD	EPA 8151A	2,4-DB
GC-ECD	EPA 8151A	Dicamba
GC-ECD	EPA 8151A	Dichlorprop
GC-ECD	EPA 8151A	Dinoseb
GC-ECD	EPA 8151A	2,4,5-TP (Silvex)
GC-ECD	EPA 8151A	2,4,5-T
GC-FID	RSK-175	Methane
GC-FID	RSK-175	Ethane
GC-FID	RSK-175	Ethene

Non-Potable Water		
Technology	Method	Analyte
GC-FID	RSK-175	Acetylene
GC-FID	EPA 8015B	Ethanol
GC-FID	EPA 8015B	Methanol
GC-FID	EPA 8015B	Ethylene glycol
GC-FID	EPA 8015B	Propylene glycol
GC-FID	EPA 8015B	Diesel Range Organics
GC-FID	EPA 8015B	Motor Oil Range Organics
GC-FID	EPA 8015B	TPH (as Diesel)
GC-FID	EPA 8015B	Gasoline Range Organics
HPLC	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC	EPA 8330B	4-Amino-2,6-dinitrotoluene
HPLC	EPA 8330B	1,3-Dinitrobenzene
HPLC	EPA 8330B	2,4-Dinitrotoluene
HPLC	EPA 8330B	2,6-Dinitrotoluene
HPLC	EPA 8330B	HMX
HPLC	EPA 8330B	HNAB
HPLC	EPA 8330B	HNS
HPLC	EPA 8330B	Nitrobenzene
HPLC	EPA 8330B	Nitroglycerin
HPLC	EPA 8330B	2-Nitrotoluene
HPLC	EPA 8330B	3-Nitrotoluene
HPLC	EPA 8330B	4-Nitrotoluene
HPLC	EPA 8330B	PETN
HPLC	EPA 8330B	RDX
HPLC	EPA 8330B	TATB
HPLC	EPA 8330B	Tetryl
HPLC	EPA 8330B	MX
HPLC	EPA 8330B	DNX
HPLC	EPA 8330B	TNX
HPLC	EPA 8330B	1,3,5-Trinitrobenzene
HPLC	EPA 8330B	2,4,6-Trinitrotoluene
GC/MS	EPA 8270D SIM	Acenaphthene
GC/MS	EPA 8270D SIM	Acenaphthylene
GC/MS	EPA 8270D SIM	Anthracene
GC/MS	EPA 8270D SIM	Benzo(a)anthracene
GC/MS	EPA 8270D SIM	Benzo(b)fluoranthene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270D SIM	Benzo(ghi)perylene
GC/MS	EPA 8270D SIM	Benzo(a)pyrene
GC/MS	EPA 8270D SIM	Chrysene
GC/MS	EPA 8270D SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270D SIM	Fluoranthene
GC/MS	EPA 8270D SIM	Fluorene
GC/MS	EPA 8270D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D SIM	Naphthalene
GC/MS	EPA 8270D SIM	Phenanthrene
GC/MS	EPA 8270D SIM	Pyrene
ICP-MS	EPA 6020A	Aluminum
ICP-MS	EPA 6020A	Antimony
ICP-MS	EPA 6020A	Arsenic
ICP-MS	EPA 6020A	Barium
ICP-MS	EPA 6020A	Beryllium
ICP-MS	EPA 6020A	Bismuth
ICP-MS	EPA 6020A	Boron
ICP-MS	EPA 6020A	Cadmium
ICP-MS	EPA 6020A	Calcium
ICP-MS	EPA 6020A	Cerium
ICP-MS	EPA 6020A	Cesium
ICP-MS	EPA 6020A	Chromium
ICP-MS	EPA 6020A	Cobalt
ICP-MS	EPA 6020A	Copper
ICP-MS	EPA 6020A	Gold
ICP-MS	EPA 6020A	Hafnium
ICP-MS	EPA 6020A	Iron
ICP-MS	EPA 6020A	Lanthanum
ICP-MS	EPA 6020A	Lead
ICP-MS	EPA 6020A	Lithium
ICP-MS	EPA 6020A	Magnesium
ICP-MS	EPA 6020A	Manganese
ICP-MS	EPA 6020A	Molybdenum
ICP-MS	EPA 6020A	Neodymium
ICP-MS	EPA 6020A	Nickel

Non-Potable Water		
Technology	Method	Analyte
ICP-MS	EPA 6020A	Niobium
ICP-MS	EPA 6020A	Palladium
ICP-MS	EPA 6020A	Phosphorus
ICP-MS	EPA 6020A	Platinum
ICP-MS	EPA 6020A	Potassium
ICP-MS	EPA 6020A	Praseodymium
ICP-MS	EPA 6020A	Rhenium
ICP-MS	EPA 6020A	Rhodium
ICP-MS	EPA 6020A	Ruthenium
ICP-MS	EPA 6020A	Samarium
ICP-MS	EPA 6020A	Selenium
ICP-MS	EPA 6020A	Silicon
ICP-MS	EPA 6020A	Silver
ICP-MS	EPA 6020A	Sodium
ICP-MS	EPA 6020A	Strontium
ICP-MS	EPA 6020A	Tantalum
ICP-MS	EPA 6020A	Tellurium
ICP-MS	EPA 6020A	Thallium
ICP-MS	EPA 6020A	Thorium
ICP-MS	EPA 6020A	Tin
ICP-MS	EPA 6020A	Titanium
ICP-MS	EPA 6020A	Tungsten
ICP-MS	EPA 6020A	Uranium
ICP-MS	EPA 6020A	Uranium 233
ICP-MS	EPA 6020A	Uranium 234
ICP-MS	EPA 6020A	Uranium 235
ICP-MS	EPA 6020A	Uranium 236
ICP-MS	EPA 6020A	Uranium 238
ICP-MS	EPA 6020A	Vanadium
ICP-MS	EPA 6020A	Yttrium
ICP-MS	EPA 6020A	Zinc
ICP-MS	EPA 6020A	Zirconium
ICP-MS	EPA 200.8	Aluminum
ICP-MS	EPA 200.8	Antimony
ICP-MS	EPA 200.8	Arsenic
ICP-MS	EPA 200.8	Barium

Non-Potable Water		
Technology	Method	Analyte
ICP-MS	EPA 200.8	Beryllium
ICP-MS	EPA 200.8	Bismuth
ICP-MS	EPA 200.8	Boron
ICP-MS	EPA 200.8	Cadmium
ICP-MS	EPA 200.8	Calcium
ICP-MS	EPA 200.8	Cerium
ICP-MS	EPA 200.8	Cesium
ICP-MS	EPA 200.8	Chromium
ICP-MS	EPA 200.8	Cobalt
ICP-MS	EPA 200.8	Copper
ICP-MS	EPA 200.8	Gold
ICP-MS	EPA 200.8	Hafnium
ICP-MS	EPA 200.8	Iron
ICP-MS	EPA 200.8	Lanthanum
ICP-MS	EPA 200.8	Lead
ICP-MS	EPA 200.8	Lithium
ICP-MS	EPA 200.8	Magnesium
ICP-MS	EPA 200.8	Manganese
ICP-MS	EPA 200.8	Molybdenum
ICP-MS	EPA 200.8	Neodymium
ICP-MS	EPA 200.8	Nickel
ICP-MS	EPA 200.8	Niobium
ICP-MS	EPA 200.8	Palladium
ICP-MS	EPA 200.8	Phosphorus
ICP-MS	EPA 200.8	Platinum
ICP-MS	EPA 200.8	Potassium
ICP-MS	EPA 200.8	Praseodymium
ICP-MS	EPA 200.8	Rhenium
ICP-MS	EPA 200.8	Rhodium
ICP-MS	EPA 200.8	Ruthenium
ICP-MS	EPA 200.8	Samarium
ICP-MS	EPA 200.8	Selenium
ICP-MS	EPA 200.8	Silicon
ICP-MS	EPA 200.8	Silver
ICP-MS	EPA 200.8	Sodium
ICP-MS	EPA 200.8	Strontium

Non-Potable Water		
Technology	Method	Analyte
ICP-MS	EPA 200.8	Tantalum
ICP-MS	EPA 200.8	Tellurium
ICP-MS	EPA 200.8	Thallium
ICP-MS	EPA 200.8	Thorium
ICP-MS	EPA 200.8	Tin
ICP-MS	EPA 200.8	Titanium
ICP-MS	EPA 200.8	Tungsten
ICP-MS	EPA 200.8	Uranium
ICP-MS	EPA 200.8	Vanadium
ICP-MS	EPA 200.8	Yttrium
ICP-MS	EPA 200.8	Zinc
ICP-MS	EPA 200.8	Zirconium
ICP-AES	EPA 200.7	Aluminum
ICP-AES	EPA 200.7	Antimony
ICP-AES	EPA 200.7	Arsenic
ICP-AES	EPA 200.7	Barium
ICP-AES	EPA 200.7	Beryllium
ICP-AES	EPA 200.7	Bismuth
ICP-AES	EPA 200.7	Boron
ICP-AES	EPA 200.7	Cadmium
ICP-AES	EPA 200.7	Calcium
ICP-AES	EPA 200.7	Chromium
ICP-AES	EPA 200.7	Cobalt
ICP-AES	EPA 200.7	Copper
ICP-AES	EPA 200.7	Iron
ICP-AES	EPA 200.7	Lead
ICP-AES	EPA 200.7	Lithium
ICP-AES	EPA 200.7	Magnesium
ICP-AES	EPA 200.7	Manganese
ICP-AES	EPA 200.7	Molybdenum
ICP-AES	EPA 200.7	Nickel
ICP-AES	EPA 200.7	Phosphorus
ICP-AES	EPA 200.7	Potassium
ICP-AES	EPA 200.7	Selenium
ICP-AES	EPA 200.7	Silicon
ICP-AES	EPA 200.7	Silver

Non-Potable Water		
Technology	Method	Analyte
ICP-AES	EPA 200.7	Sodium
ICP-AES	EPA 200.7	Strontium
ICP-AES	EPA 200.7	Sulfur
ICP-AES	EPA 200.7	Thallium
ICP-AES	EPA 200.7	Thorium
ICP-AES	EPA 200.7	Tin
ICP-AES	EPA 200.7	Titanium
ICP-AES	EPA 200.7	Vanadium
ICP-AES	EPA 200.7	Zinc
CVAA	EPA 7470A	Mercury
Colorimetric	EPA 9010C EPA 9012B	Cyanide
Ion Chromatography	EPA 300.0/9056A	Bromide
Ion Chromatography	EPA 300.0/9056A	Chloride
Ion Chromatography	EPA 300.0/9056A	Fluoride
Ion Chromatography	EPA 300.0/9056A	Nitrate
Ion Chromatography	EPA 300.0/9056A	Nitrite
Ion Chromatography	EPA 300.0/9056A	Sulfate
Ion Chromatography	EPA 300.0/9056A	Ortho-phosphate
Ion Chromatography	EPA 300.0/9056A	Iodide
Ion Chromatography	EPA 314.0	Perchlorate
Gravimetric	SM 2540B SM 2540C SM 2540D	Solids
Probe	EPA 9040C EPA 9045D EPA 150.1	pH
Titration	SM 2320B EPA 310.1	Alkalinity
Titration	EPA 9030	Sulfide
Penske-Martin	EPA 1010A	Ignitability
Colormetric	EPA 353.1	nitrate/Nitrite
Colormetric	EPA 350.1	Ammonia
TOC Analyzer	EPA 9060A	TOC
Titrimetric	EPA 9020B	TOX
Colormetric	EPA 7196A	Hex Chromium

Non-Potable Water		
Technology	Method	Analyte
Gravimetric	EPA 1664A	Oil & Grease
Gravimetric	EPA 1664A	TPH
Probe	EPA 9050A	Conductivity
Gas Flow Proportional Counter	EPA 900.0 EPA 9310	gross alpha/beta
Gas Flow Proportional Counter	ST-RC-0036 ST-RD-0403	Chlorine-36
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	total radium
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 / DOE HASL 300 Sr-02	Strontium-90
Liquid Scintillation Counter	EPA 906.0	Tritium
Liquid Scintillation Counter	Eichrom Technologies TCW01/TCS01	Tecnetium-99
Liquid Scintillation Counter	EERF C-01-C14	Carbon-14
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium-137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140

Non-Potable Water		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Calcium-45
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192

Non-Potable Water		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese-56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 83
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)

Non-Potable Water		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95

Non-Potable Water		
Technology	Method	Analyte
Alpha Spectroscopy	DOE HASL 300 A-01-R	Alpha spec analysis:
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Uranium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Thorium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Americium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Plutonium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Neptunium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Curium
Liquid Scintillation Counter	Eichrom Technologies OTW01, OTS01	Lead-210
Alpha Spectroscopy	Laboratory SOP ST-RC-0210	Polonium-210
Liquid Scintillation Counter	Eichrom Technologies FEW01	Iron-55
Liquid Scintillation Counter	DOE RP-300	Nickel 59/63
Liquid Scintillation Counter	SM 7500-IB	Iodine-129
Preparation	Method	Type
Organic Extraction & Sample Prep	EPA 3500C	Organic Extraction & Sample Prep
Volatile Prep	EPA 5000	Sample Preparation for Volatile Organic Compounds
Organic Cleanup	EPA 3600A	Cleanup for Organic extracts
Organic prep/analysis	EPA 8000C	Determinative Chromatographic Separations
Acid Digestion (Aqueous samples)	EPA 3010A	Acid Digestion for Metals (Aqueous samples)
Purge & Trap	EPA 5030C	Purge & Trap for Aqueous Volatile
Sep Funnel Liquid-Liquid Extraction	EPA 3510C	Sep Funnel Liquid-Liquid Extraction
Organic Cleanup	EPA 3600A	Cleanup for Organic extracts
Florisil Cleanup	EPA 3620C	Florisil Cleanup

Non-Potable Water		
Technology	Method	Analyte
Sulfur Cleanup	EPA 3660B	Sulfur Cleanup
Acid Clean Up	EPA 3665A	Acid Clean Up for PCBs
TCLP Extraction	EPA 1311	TCLP Extraction
SPLP Extraction	EPA 1312	SPLP Extraction
CWET Extraction	CA Title 22	CWET Extraction
Solid Phase Extraction	EPA 3535A	Solid Phase Extraction

Drinking Water		
Technology	Method	Analyte
Gas Flow Proportional Counter	EPA 900.0 EPA 9310	gross alpha/beta
Gas Flow Proportional Counter	ST-RC-0036 ST-RD-0403	Chlorine-36
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 / DOE HASL 300 Sr-02	Strontium-90
Liquid Scintillation Counter	EPA 906.0	Tritium
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124

Drinking Water		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium-137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Calcium-45
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58

Drinking Water		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese-56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 83
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94

Drinking Water		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228

Drinking Water		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95

Solid and Chemical Materials		
Technology	Method	Analyte
ICP-AES	EPA 6010C	Aluminum
ICP-AES	EPA 6010C	Antimony
ICP-AES	EPA 6010C	Arsenic
ICP-AES	EPA 6010C	Barium
ICP-AES	EPA 6010C	Beryllium
ICP-AES	EPA 6010C	Bismuth
ICP-AES	EPA 6010C	Boron
ICP-AES	EPA 6010C	Cadmium
ICP-AES	EPA 6010C	Calcium
ICP-AES	EPA 6010C	Chromium
ICP-AES	EPA 6010C	Cobalt

Solid and Chemical Materials		
Technology	Method	Analyte
ICP-AES	EPA 6010C	Copper
ICP-AES	EPA 6010C	Iron
ICP-AES	EPA 6010C	Lead
ICP-AES	EPA 6010C	Lithium
ICP-AES	EPA 6010C	Magnesium
ICP-AES	EPA 6010C	Manganese
ICP-AES	EPA 6010C	Molybdenum
ICP-AES	EPA 6010C	Nickel
ICP-AES	EPA 6010C	Phosphorus
ICP-AES	EPA 6010C	Potassium
ICP-AES	EPA 6010C	Selenium
ICP-AES	EPA 6010C	Silicon
ICP-AES	EPA 6010C	Silver
ICP-AES	EPA 6010C	Sodium
ICP-AES	EPA 6010C	Strontium
ICP-AES	EPA 6010C	Sulfur
ICP-AES	EPA 6010C	Thallium
ICP-AES	EPA 6010C	Thorium
ICP-AES	EPA 6010C	Tin
ICP-AES	EPA 6010C	Titanium
ICP-AES	EPA 6010C	Vanadium
ICP-AES	EPA 6010C	Zinc
GC/MS	EPA 8260C	Acetone
GC/MS	EPA 8260C	Acetonitrile
GC/MS	EPA 8260C	Acrolein
GC/MS	EPA 8260C	Acrylonitrile
GC/MS	EPA 8260C	Benzene
GC/MS	EPA 8260C	Benzyl chloride
GC/MS	EPA 8260C	Bromobenzene
GC/MS	EPA 8260C	Bromochloromethane
GC/MS	EPA 8260C	Bromodichloromethane
GC/MS	EPA 8260C	Bromoform
GC/MS	EPA 8260C	Bromomethane
GC/MS	EPA 8260C	n-Butanol
GC/MS	EPA 8260C	2-Butanone
GC/MS	EPA 8260C	n-Butylbenzene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260C	sec-Butylbenzene
GC/MS	EPA 8260C	tert-Butylbenzene
GC/MS	EPA 8260C	Carbon disulfide
GC/MS	EPA 8260C	Carbon tetrachloride
GC/MS	EPA 8260C	Chlorobenzene
GC/MS	EPA 8260C	Chlorobromomethane
GC/MS	EPA 8260C	2-Chloro-1,3-butadiene
GC/MS	EPA 8260C	Chlorodibromomethane
GC/MS	EPA 8260C	Dibromochloromethane
GC/MS	EPA 8260C	Chloroethane
GC/MS	EPA 8260C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260C	Chloroform
GC/MS	EPA 8260C	Chloromethane
GC/MS	EPA 8260C	Allyl chloride
GC/MS	EPA 8260C	2-Chlorotoluene
GC/MS	EPA 8260C	4-Chlorotoluene
GC/MS	EPA 8260C	Cyclohexane
GC/MS	EPA 8260C	Cyclohexanone
GC/MS	EPA 8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260C	1,2-Dibromoethane
GC/MS	EPA 8260C	Dibromomethane
GC/MS	EPA 8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260C	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260C	Dichlorodifluoromethane
GC/MS	EPA 8260C	1,1-Dichloroethane
GC/MS	EPA 8260C	1,2-Dichloroethane
GC/MS	EPA 8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260C	1,1-Dichloroethene
GC/MS	EPA 8260C	1,2-Dichloroethene (total)
GC/MS	EPA 8260C	1,2-Dichloropropane
GC/MS	EPA 8260C	1,3-Dichloropropane
GC/MS	EPA 8260C	2,2-Dichloropropane
GC/MS	EPA 8260C	cis-1,3-Dichloropropene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260C	1,1-Dichloropropene
GC/MS	EPA 8260C	1,2-Dichloro-1,1,2,2-tetrafluoroethane
GC/MS	EPA 8260C	Dimethyl disulfide
GC/MS	EPA 8260C	1,4-Dioxane
GC/MS	EPA 8260C	Ethyl acetate
GC/MS	EPA 8260C	Ethylbenzene
GC/MS	EPA 8260C	Ethyl ether
GC/MS	EPA 8260C	Diethyl ether
GC/MS	EPA 8260C	Ethyl methacrylate
GC/MS	EPA 8260C	Freon 113
GC/MS	EPA 8260C	Hexachlorobutadiene
GC/MS	EPA 8260C	n-Hexane
GC/MS	EPA 8260C	2-Hexanone
GC/MS	EPA 8260C	Iodomethane
GC/MS	EPA 8260C	Isobutanol
GC/MS	EPA 8260C	Isopropylbenzene
GC/MS	EPA 8260C	p-Isopropyltoluene
GC/MS	EPA 8260C	Methacrylonitrile
GC/MS	EPA 8260C	Methyl acetate
GC/MS	EPA 8260C	Methyl butyl ketone
GC/MS	EPA 8260C	Methylcyclohexane
GC/MS	EPA 8260C	Dichloromethane
GC/MS	EPA 8260C	Methylene chloride
GC/MS	EPA 8260C	Methyl methacrylate
GC/MS	EPA 8260C	4-Methyl-2-pentanone
GC/MS	EPA 8260C	MTBE
GC/MS	EPA 8260C	Naphthalene
GC/MS	EPA 8260C	2-Nitropropane
GC/MS	EPA 8260C	Nonanal
GC/MS	EPA 8260C	Pentachloroethane
GC/MS	EPA 8260C	Propionitrile
GC/MS	EPA 8260C	n-Propylbenzene
GC/MS	EPA 8260C	Styrene
GC/MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C	1,1,2,2-Tetrachloroethane

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260C	Tetrachloroethene
GC/MS	EPA 8260C	Tetrahydrofuran
GC/MS	EPA 8260C	Toluene
GC/MS	EPA 8260C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260C	Trichloroethene
GC/MS	EPA 8260C	Trichlorofluoromethane
GC/MS	EPA 8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260C	Trichlorotrifluoroethane
GC/MS	EPA 8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C	Vinyl acetate
GC/MS	EPA 8260C	Vinyl chloride
GC/MS	EPA 8260C	m-Xylene & p-Xylene
GC/MS	EPA 8260C	o-Xylene
GC/MS	EPA 8260C	Xylenes (total)
GC/MS	EPA 8270D	Acenaphthene
GC/MS	EPA 8270D	Acenaphthylene
GC/MS	EPA 8270D	Acetophenone
GC/MS	EPA 8270D	2-Acetylaminofluorene
GC/MS	EPA 8270D	4-Aminobiphenyl
GC/MS	EPA 8270D	Aniline
GC/MS	EPA 8270D	Anthracene
GC/MS	EPA 8270D	Aramite (total)
GC/MS	EPA 8270D	Atrazine
GC/MS	EPA 8270D	Azobenzene
GC/MS	EPA 8270D	Benzaldehyde
GC/MS	EPA 8270D	Benzidine
GC/MS	EPA 8270D	Benzo(a)anthracene
GC/MS	EPA 8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270D	Benzoic acid

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270D	Benzo(ghi)perylene
GC/MS	EPA 8270D	Benzo(a)pyrene
GC/MS	EPA 8270D	Benzyl alcohol
GC/MS	EPA 8270D	1,1'-Biphenyl
GC/MS	EPA 8270D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270D	bis(2-Chloroisopropyl) ether
GC/MS	EPA 8270D	bis(2-Ethylhexyl) phthalate
GC/MS	EPA 8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270D	n-Butylbenzenesulfonamide
GC/MS	EPA 8270D	Butyl benzyl phthalate
GC/MS	EPA 8270D	Caprolactam
GC/MS	EPA 8270D	Carbazole
GC/MS	EPA 8270D	4-Chloroaniline
GC/MS	EPA 8270D	Chlorobenzilate
GC/MS	EPA 8270D	p-Chlorobenzilate
GC/MS	EPA 8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270D	2-Chloronaphthalene
GC/MS	EPA 8270D	2-Chlorophenol
GC/MS	EPA 8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270D	Chrysene
GC/MS	EPA 8270D	Cresols (total)
GC/MS	EPA 8270D	Cyclohexanol
GC/MS	EPA 8270D	Diallate
GC/MS	EPA 8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270D	Dibenzo(a,h)anthracene
GC/MS	EPA 8270D	Dibenzofuran
GC/MS	EPA 8270D	Di-n-butyl phthalate
GC/MS	EPA 8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270D	2,4-Dichlorophenol
GC/MS	EPA 8270D	2,6-Dichlorophenol
GC/MS	EPA 8270D	Diethyl phthalate
GC/MS	EPA 8270D	O,O-Diethyl-O-(2-pyrazinyl) phosphorothioate

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270D	Dimethoate
GC/MS	EPA 8270D	p-Dimethylaminoazobenzene
GC/MS	EPA 8270D	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270D	3,3'-Dimethylbenzidine
GC/MS	EPA 8270D	Dimethylformamide
GC/MS	EPA 8270D	alpha,alpha-Dimethylphenethylamine
GC/MS	EPA 8270D	2,4-Dimethylphenol
GC/MS	EPA 8270D	Dimethyl phthalate
GC/MS	EPA 8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270D	1,4-Dinitrobenzene
GC/MS	EPA 8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270D	2,4-Dinitrophenol
GC/MS	EPA 8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270D	2-sec-Butyl-4,6-dinitrophenol
GC/MS	EPA 8270D	Dinoseb
GC/MS	EPA 8270D	Di-n-octyl phthalate
GC/MS	EPA 8270D	1,4-Dioxane
GC/MS	EPA 8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270D	Disulfoton
GC/MS	EPA 8270D	Ethyl methacrylate
GC/MS	EPA 8270D	Ethyl methanesulfonate
GC/MS	EPA 8270D	Famphur
GC/MS	EPA 8270D	Fluoranthene
GC/MS	EPA 8270D	Fluorene
GC/MS	EPA 8270D	Hexachlorobenzene
GC/MS	EPA 8270D	Hexachlorobutadiene
GC/MS	EPA 8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270D	Hexachloro-1,3-cyclopentadiene
GC/MS	EPA 8270D	Hexachloroethane
GC/MS	EPA 8270D	Hexachlorophene
GC/MS	EPA 8270D	Hexachloropropene
GC/MS	EPA 8270D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D	Isodrin
GC/MS	EPA 8270D	Isophorone
GC/MS	EPA 8270D	Isosafrole

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270D	Kepone
GC/MS	EPA 8270D	Methapyrilene
GC/MS	EPA 8270D	2-Methylbenzenamine
GC/MS	EPA 8270D	3-Methylcholanthrene
GC/MS	EPA 8270D	4,4'-Methylenebis(2-chloroaniline)
GC/MS	EPA 8270D	Methyl methacrylate
GC/MS	EPA 8270D	Methyl methanesulfonate
GC/MS	EPA 8270D	2-Methylnaphthalene
GC/MS	EPA 8270D	Methyl parathion
GC/MS	EPA 8270D	2-Methylphenol
GC/MS	EPA 8270D	3-Methylphenol & 4-Methylphenol
GC/MS	EPA 8270D	2-Methylphenol, 3-methylphenol and 4-methylphenol
GC/MS	EPA 8270D	Methylphenols (total)
GC/MS	EPA 8270D	Naphthalene
GC/MS	EPA 8270D	1,4-Naphthoquinone
GC/MS	EPA 8270D	1-Naphthylamine
GC/MS	EPA 8270D	2-Naphthylamine
GC/MS	EPA 8270D	2-Nitroaniline
GC/MS	EPA 8270D	3-Nitroaniline
GC/MS	EPA 8270D	4-Nitroaniline
GC/MS	EPA 8270D	Nitrobenzene
GC/MS	EPA 8270D	2-Nitrophenol
GC/MS	EPA 8270D	4-Nitrophenol
GC/MS	EPA 8270D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270D	N-Nitrosodi-n-butylamine
GC/MS	EPA 8270D	N-Nitrosodiethylamine
GC/MS	EPA 8270D	N-Nitrosodimethylamine
GC/MS	EPA 8270D	N-Nitrosodiphenylamine
GC/MS	EPA 8270D	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270D	N-Nitrosomethylethylamine
GC/MS	EPA 8270D	N-Nitrosomorpholine
GC/MS	EPA 8270D	N-Nitrosopiperidine
GC/MS	EPA 8270D	N-Nitrosopyrrolidine
GC/MS	EPA 8270D	5-Nitro-o-toluidine
GC/MS	EPA 8270D	2,2'-oxybis(1-Chloropropane)
GC/MS	EPA 8270D	Parathion

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270D	Pentachlorobenzene
GC/MS	EPA 8270D	Pentachloroethane
GC/MS	EPA 8270D	Pentachloronitrobenzene
GC/MS	EPA 8270D	Pentachlorophenol
GC/MS	EPA 8270D	Phenacetin
GC/MS	EPA 8270D	Phenanthrene
GC/MS	EPA 8270D	Phenol
GC/MS	EPA 8270D	p-Phenylene diamine
GC/MS	EPA 8270D	Phorate
GC/MS	EPA 8270D	2-Picoline
GC/MS	EPA 8270D	Pronamide
GC/MS	EPA 8270D	Pyrene
GC/MS	EPA 8270D	Pyridine
GC/MS	EPA 8270D	Safrole
GC/MS	EPA 8270D	Sulfotepp
GC/MS	EPA 8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270D	Tetraethyldithiopyrophosphate (Sulfotepp)
GC/MS	EPA 8270D	Thionazin
GC/MS	EPA 8270D	o-Toluidine
GC/MS	EPA 8270D	Tributyl phosphate
GC/MS	EPA 8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270D	O,O,O-Triethyl phosphorothioate
GC/MS	EPA 8270D	1,3,5-Trinitrobenzene
GC/MS	EPA 8270D	Tris(2-chloroethyl)phosphate
GC/MS	EPA 8270D	1-Methyl naphthalene
GC-ECD	EPA 8081B	Aldrin
GC-ECD	EPA 8081B	alpha-BHC
GC-ECD	EPA 8081B	beta-BHC
GC-ECD	EPA 8081B	delta-BHC
GC-ECD	EPA 8081B	gamma-BHC (Lindane)
GC-ECD	EPA 8081B	alpha-Chlordane
GC-ECD	EPA 8081B	gamma-Chlordane
GC-ECD	EPA 8081B	Chlordane (technical)

Solid and Chemical Materials		
Technology	Method	Analyte
GC-ECD	EPA 8081B	4,4'-DDD
GC-ECD	EPA 8081B	2,4'-DDD
GC-ECD	EPA 8081B	4,4'-DDE
GC-ECD	EPA 8081B	2,4'-DDE
GC-ECD	EPA 8081B	4,4'-DDT
GC-ECD	EPA 8081B	2,4'-DDT
GC-ECD	EPA 8081B	Dieldrin
GC-ECD	EPA 8081B	Endosulfan I
GC-ECD	EPA 8081B	Endosulfan II
GC-ECD	EPA 8081B	Endosulfan sulfate
GC-ECD	EPA 8081B	Endrin
GC-ECD	EPA 8081B	Endrin aldehyde
GC-ECD	EPA 8081B	Endrin ketone
GC-ECD	EPA 8081B	Heptachlor
GC-ECD	EPA 8081B	Heptachlor epoxide
GC-ECD	EPA 8081B	Methoxychlor
GC-ECD	EPA 8081B	Toxaphene
GC-ECD	EPA 8082A	Aroclor 1016
GC-ECD	EPA 8082A	Aroclor 1221
GC-ECD	EPA 8082A	Aroclor 1232
GC-ECD	EPA 8082A	Aroclor 1242
GC-ECD	EPA 8082A	Aroclor 1248
GC-ECD	EPA 8082A	Aroclor 1254
GC-ECD	EPA 8082A	Aroclor 1260
GC-ECD	EPA 8082A	Aroclor 1262
GC-ECD	EPA 8082A	Aroclor 1268
GC-ECD	EPA 8151A	2,4-D
GC-ECD	EPA 8151A	Dalapon
GC-ECD	EPA 8151A	2,4-DB
GC-ECD	EPA 8151A	Dicamba
GC-ECD	EPA 8151A	Dichlorprop
GC-ECD	EPA 8151A	Dinoseb
GC-ECD	EPA 8151A	2,4,5-TP (Silvex)
GC-ECD	EPA 8151A	2,4,5-T
HPLC	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC	EPA 8330B	4-Amino-2,6-dinitrotoluene

Solid and Chemical Materials		
Technology	Method	Analyte
HPLC	EPA 8330B	1,3-Dinitrobenzene
HPLC	EPA 8330B	2,4-Dinitrotoluene
HPLC	EPA 8330B	2,6-Dinitrotoluene
HPLC	EPA 8330B	HMX
HPLC	EPA 8330B	HNAB
HPLC	EPA 8330B	HNS
HPLC	EPA 8330B	Nitrobenzene
HPLC	EPA 8330B	Nitroglycerin
HPLC	EPA 8330B	2-Nitrotoluene
HPLC	EPA 8330B	3-Nitrotoluene
HPLC	EPA 8330B	4-Nitrotoluene
HPLC	EPA 8330B	PETN
HPLC	EPA 8330B	RDX
HPLC	EPA 8330B	TATB
HPLC	EPA 8330B	Tetryl
HPLC	EPA 8330B	MXN
HPLC	EPA 8330B	DNX
HPLC	EPA 8330B	TNX
HPLC	EPA 8330B	1,3,5-Trinitrobenzene
HPLC	EPA 8330B	2,4,6-Trinitrotoluene
GC/MS	EPA 8270D SIM	Acenaphthene
GC/MS	EPA 8270D SIM	Acenaphthylene
GC/MS	EPA 8270D SIM	Anthracene
GC/MS	EPA 8270D SIM	Benzo(a)anthracene
GC/MS	EPA 8270D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270D SIM	Benzo(ghi)perylene
GC/MS	EPA 8270D SIM	Benzo(a)pyrene
GC/MS	EPA 8270D SIM	Chrysene
GC/MS	EPA 8270D SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270D SIM	Fluoranthene
GC/MS	EPA 8270D SIM	Fluorene
GC/MS	EPA 8270D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D SIM	Naphthalene
GC/MS	EPA 8270D SIM	Phenanthrene
GC/MS	EPA 8270D SIM	Pyrene

Solid and Chemical Materials		
Technology	Method	Analyte
GC-FID	EPA 8015B	Diesel Range Organics
GC-FID	EPA 8015B	Motor Oil Range Organics
GC-FID	EPA 8015B	TPH (as Diesel)
GC-FID	EPA 8015B	Gasoline Range Organics
GC-FID	EPA 8015B	Ethanol
GC-FID	EPA 8015B	Methanol
GC-FID	EPA 8015B	Ethylene glycol
GC-FID	EPA 8015B	Propylene glycol
ICP-MS	EPA 6020A	Aluminum
ICP-MS	EPA 6020A	Antimony
ICP-MS	EPA 6020A	Arsenic
ICP-MS	EPA 6020A	Barium
ICP-MS	EPA 6020A	Beryllium
ICP-MS	EPA 6020A	Bismuth
ICP-MS	EPA 6020A	Boron
ICP-MS	EPA 6020A	Cadmium
ICP-MS	EPA 6020A	Calcium
ICP-MS	EPA 6020A	Cerium
ICP-MS	EPA 6020A	Cesium
ICP-MS	EPA 6020A	Chromium
ICP-MS	EPA 6020A	Cobalt
ICP-MS	EPA 6020A	Copper
ICP-MS	EPA 6020A	Gold
ICP-MS	EPA 6020A	Hafnium
ICP-MS	EPA 6020A	Iron
ICP-MS	EPA 6020A	Lanthanum
ICP-MS	EPA 6020A	Lead
ICP-MS	EPA 6020A	Lithium
ICP-MS	EPA 6020A	Magnesium
ICP-MS	EPA 6020A	Manganese
ICP-MS	EPA 6020A	Molybdenum
ICP-MS	EPA 6020A	Neodymium
ICP-MS	EPA 6020A	Nickel
ICP-MS	EPA 6020A	Niobium
ICP-MS	EPA 6020A	Palladium
ICP-MS	EPA 6020A	Phosphorus

Solid and Chemical Materials		
Technology	Method	Analyte
ICP-MS	EPA 6020A	Platinum
ICP-MS	EPA 6020A	Potassium
ICP-MS	EPA 6020A	Praseodymium
ICP-MS	EPA 6020A	Rhenium
ICP-MS	EPA 6020A	Rhodium
ICP-MS	EPA 6020A	Ruthenium
ICP-MS	EPA 6020A	Samarium
ICP-MS	EPA 6020A	Selenium
ICP-MS	EPA 6020A	Silicon
ICP-MS	EPA 6020A	Silver
ICP-MS	EPA 6020A	Sodium
ICP-MS	EPA 6020A	Strontium
ICP-MS	EPA 6020A	Tantalum
ICP-MS	EPA 6020A	Technetium-99
ICP-MS	EPA 6020A	Tellurium
ICP-MS	EPA 6020A	Thallium
ICP-MS	EPA 6020A	Thorium
ICP-MS	EPA 6020A	Tin
ICP-MS	EPA 6020A	Titanium
ICP-MS	EPA 6020A	Tungsten
ICP-MS	EPA 6020A	Uranium
ICP-MS	EPA 6020A	Uranium 233
ICP-MS	EPA 6020A	Uranium 234
ICP-MS	EPA 6020A	Uranium 235
ICP-MS	EPA 6020A	Uranium 236
ICP-MS	EPA 6020A	Uranium 238
ICP-MS	EPA 6020A	Vanadium
ICP-MS	EPA 6020A	Yttrium
ICP-MS	EPA 6020A	Zinc
ICP-MS	EPA 6020A	Zirconium
CVAA	EPA 7471B	Mercury
Colormetric	EPA 9010C EPA 9012B	Cyanide
Ion Chromatrography	EPA 300.0 EPA 9056A	Bromide
Ion Chromatrography	EPA 300.0 EPA 9056A	Chloride

Solid and Chemical Materials		
Technology	Method	Analyte
Ion Chromatography	EPA 300.0 EPA 9056A	Fluoride
Ion Chromatography	EPA 300.0 EPA 9056A	Nitrate
Ion Chromatography	EPA 300.0 EPA 9056A	Nitrite
Ion Chromatography	EPA 300.0 EPA 9056A	Sulfate
Ion Chromatography	EPA 300.0 EPA 9056A	Ortho-phosph
Ion Chromatography	EPA 300.0 EPA 9056A	Iodide
Ion Chromatography	EPA 314.0	Perchlorate
Probe	EPA 9045	pH
Titration	SM 2320B EPA 310.1	Alkalinity
Titration	EPA 9030	Sulfide
Penske-Martin	EPA 1010A	Ignitability
Colormetric	EPA 353.1	nitrate/Nitrite
Colormetric	EPA 350.1	Ammonia
TOC Analyzer	EPA 9060A	TOC
Colormetric	EPA 7196A	Hex Chromium
Gravimetric	EPA 1664A	Oil & Grease
Gravimetric	EPA 1664A	TPH
Probe	EPA 9050A	Conductivity
Gas Flow Proportional Counter	EPA 900.0 EPA 9310	gross alpha/beta
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	total radium
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 / DOE HASL 300 Sr-02	Strontium-90
Liquid Scintillation Counter	EPA 906.0	Tritium
Liquid Scintillation Counter	Eichrom Technologies TCW01/TCS01	Tecnetium-99
Liquid Scintillation Counter	EERF C-01-C14	Carbon-14

Solid and Chemical Materials		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium-137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Calcium-45
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137

Solid and Chemical Materials		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese-56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 83

Solid and Chemical Materials		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230

Solid and Chemical Materials		
Technology	Method	Analyte
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95
Alpha Spectroscopy	DOE HASL 300 A-01-R	Alpha spec analysis:
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Uranium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Thorium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Americium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Plutonium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Neptunium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Curium
Liquid Scintillation Counter	Eichrom Technologies OTW01, OTS01	Lead-210
Alpha Spectroscopy	Laboratory SOP ST-RC-0210	Polonium-210
Liquid Scintillation Counter	DOE RP-300	Nickel 59/63
Liquid Scintillation Counter	SM 7500-IB	Iodine-129

Solid and Chemical Materials		
Preparation	Method	Type
Organic Extraction & Sample Prep	EPA 3500C	Organic Extraction & Sample Prep
Volatile Prep	EPA 5000	Sample Preparation for Volatile Organic Compounds
Organic Cleanup	EPA 3600A	Cleanup for Organic extracts
Organic prep/analysis	EPA 8000C	Determinative Chromatographic Separations
Acid Digestion (Aqueous samples)	EPA 3010A	Acid Digestion for Metals (Aqueous samples)
Acid Digestion (solids)	EPA 3050B	Acid Digestion for Metals of Sediment/Soils
Purge & Trap	EPA 5030C	Purge & Trap for Aqueous Volatile Samples
Closed System Purge & Trap and Extraction for Volatiles	EPA 5035A	Closed System Purge & Trap and Extraction for Volatiles
Sep Funnel Liquid-Liquid Extraction	EPA 3510C	Sep Funnel Liquid-Liquid Extraction
Ultrasonic Extraction	EPA 3550C	Ultrasonic Extraction Organic Soils
Solid Phase Extraction	EPA 3535A	Solid Phase Extraction
Acid Clean-up	EPA 3665A	Acid Clean Up for PCBs
Florisil Cleanup	EPA 3620C	Florisil Cleanup
Sulfur Cleanup	EPA 3660B	Sulfur Cleanup
Waste Dilution	EPA 3585	Waste Dilution Volatile Organics
Waste Dilution	EPA 3580A	Waste Dilution SemiVolatile Organics
TCLP Extraction	EPA 1311	TCLP Extraction
SPLP Extraction	EPA 1312	SPLP Extraction
CWET Extraction	CA Title 22	CWET Extraction
Alkaline Digestion	EPA 3060A	Alkaline Digestion for Hexavalent Chromium

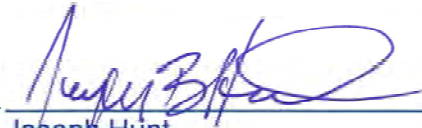
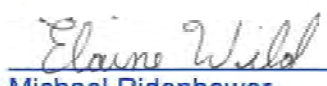
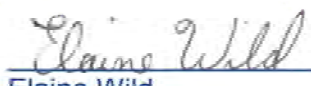
Notes:

- 1) This laboratory offers commercial testing service.

Approved by: 
R. Douglas Leonard
 Chief Technical Officer

Date: March 2, 2017

**Title: EXTRACTABLE TOTAL PETROLEUM HYDROCARBONS BY
GC-FID
[SW-846 8015B/D; IOWA OA-2]**

Approvals (Signature/Date):	
 Joseph Hunt GC Department Lead	6/22/15 Date
 Michael Ridenhower Health & Safety Manager / Coordinator	6/22/15 Date
 Marti Ward Quality Assurance Manager	06/22/2015 Date
 Elaine Wild Laboratory Director	6/22/15 Date

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP documents the procedure used to determine extractable petroleum hydrocarbons (EPH), also known as total recoverable petroleum hydrocarbons (TRPH), or TPH in environmental samples by Gas Chromatograph with a Flame Ionization Detector (GC/FID).
- 1.2 Matrices to which this procedure is applicable are water, water miscible wastes, soil, sludges, oil and miscellaneous solids.
- 1.3 This procedure is amenable to the determination of common hydrocarbon mixtures which are soluble in methylene chloride, are amenable to gas chromatography, and have boiling points between approximately 150 and 450 °C.
- 1.4 Carbon ranges from C10 to C40 are detected. Typical hydrocarbons are Diesel, Motor Oil, Kerosene, Jet fuel, Mineral Spirits and Hydraulic Oil.
- 1.5 Sample preparation techniques are described in SOP ST-OP-0002.
- 1.6 This SOP is based on SW-846 methods 8000C and 8015B, 8015D and the IOWA method OA-2.
- 1.7 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMs).
 - 1.7.1 Additional compounds may be amenable to this procedure. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument. When an MDL study can not be conducted, the MDL is set equal to the reporting limit.

2.0 SUMMARY OF METHOD

- 2.1 Organic compounds are extracted from neutral pH environmental samples with methylene chloride. The volume of methylene chloride is concentrated to give an extract of known volume.
- 2.2 A portion of the extract is analyzed by GC/FID. The GC must be previously calibrated using multi-level calibration standards. The petroleum hydrocarbons are quantified as Diesel Range Organics (DRO) or Oil Range Organics (ORO) unless a specific hydrocarbon product is requested by the client.

3.0 DEFINITIONS

- 3.1 See the Quality Assurance Manual (QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Diesel is defined as hydrocarbon peaks eluting in the carbon range C10-C28.
- 3.3 Motor oil is defined as hydrocarbon peaks eluting in the carbon range C28-C40.
- 3.4 For method OA-2, hydrocarbons are determined by their elution pattern.

4.0 INTERFERENCES

- 4.1 Any GC chromatographable compound, organic or inorganic, which gives a flame detector response in the retention time window selected, will be included in the quantification of total petroleum hydrocarbons. The method is non-specific and all co-extracted compounds are detected.

- 4.2 Solvents, reagents, glassware and other sample processing hardware may yield artifacts and interferences to sample extracts. Strict attention to glassware cleaning and handling and demonstration of solvent purity will lead to minimization of these interferences.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
- 5.2.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.2.2 There are areas of high voltage in both the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- 5.3 PRIMARY MATERIALS USED
- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time weighted average			
STEL – Short term exposure limit			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 GC/FID system: The lab utilizes an Agilent 6890 with dual flame ionization detector (FID) system with autosampler.
- 6.1.1 The GC column type and instrument run conditions are posted in the front of the maintenance calendars and reside in the Chemstation method.
- 6.2 Data System – Chemstation for acquisition and Chrom for data processing.
- 6.3 Amber vials. Crimp top seals.
- 6.4 Pipettes, disposable

- 6.5 Micro syringes: 10µL, 250µL, 500µL, 1000µL. Hamilton 1700 Series.
- 6.6 Analytical Balance, capable of weighing ± 0.01 grams.
- 6.7 Volumetric flasks, Class A, various volumes

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision. "Recipes" for standards are found in the reagent module of LIMS.
- 7.2 Primary standards
 - 7.2.1 Standards are either purchased from a commercial vendor or prepared by dilution of neat liquid mixtures in methylene chloride.
 - 7.2.2 Storage: The standards are stored at -20 to 0° C in amber glass vials with Teflon-lined caps.
 - 7.2.3 Shelf life:
 - 7.2.3.1 Unopened ampoules: manufacturer's expiration date.
 - 7.2.3.2 Opened ampoules and Intermediate standards: Manufacturer's expiration date, or 1 year, whichever is sooner.
 - 7.2.3.3 Working standards: Primary standard's expiration date or 6 months, whichever is sooner.
 - 7.2.4 Degradation:
 - 7.2.4.1 If standard degradation is indicated, the standard is discarded, irrespective of its' shelf life.
- 7.3 o-Terphenyl surrogate
- 7.4 Calibration Standards
 - 7.4.1 Calibration standards for total petroleum hydrocarbon analysis may vary according to the requests of clients. A diesel #2 standard and motor oil (a mid-weight oil) are used for initial calibrations. Other hydrocarbons, such as Kerosene, Jet Fuel A, Mineral Spirits, and Fuel Oil #6 can be analyzed upon request.
 - 7.4.2 Secondary Standards - Secondary standards for calibration are prepared by dilution in methylene chloride of the primary standards mentioned. The secondary standards are subject to the same storage requirements as the primary standards.
 - 7.4.3 Working Calibration Standards - The working calibration standards are prepared in methylene chloride: diesel and motor oil at a minimum of five concentration levels. One of the concentration levels should be $\leq$ to the concentration of the reporting limit, and the remaining levels should define the expected range of concentrations in the samples or the linear range of the instrument. All working calibration standards expire after 6 months or at the expiration date of their primary standards, whichever is shorter. Working standards are stored in the freezer.
 - 7.4.4 Second Source for calibration verification (ICV). Purchased from a different vendor than the calibration standards.
- 7.5 Carbon range retention time marker standard
 - 7.5.1 This marker standard includes C10 (decane), C12 (dodecane), C24 (tetracosane), C28 (octacosane) and C40 (tetracontane).
- 7.6 Gases for carrier and make-up: Hydrogen carrier, Nitrogen make-up

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Water samples are unpreserved and refrigerated at 4 ± 2 °C.
- 8.3 Soil samples are refrigerated at 4 ± 2 °C.
- 8.4 The extraction holding time for water samples is 7 days.
- 8.5 The extraction holding time for soil samples is 14 days.
- 8.6 The extract analysis holding time is 40 days from the date of extraction.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
 - 9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.2 Method Blank (MB)

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 DI water is used as the blank matrix for water batches.
- 9.2.4 Sodium sulfate is used as the blank matrix for solid batches.

9.3 Laboratory Control Sample (LCS)

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.
- 9.3.3 DI water, spiked with the analytes of interest is used as the LCS for water batches.
- 9.3.4 Sodium sulfate, spiked with the analytes of interest is used as the LCS for solid batches.

9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5 Procedural Variations/ Nonconformance and Corrective Action

- 9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

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- 9.5.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 External standard calibration is used.
- 10.2 Marker Standard
- 10.2.1 Analyze a carbon range retention time marker standard daily.
- 10.2.2 This marker standard includes C10 (decane), C12 (dodecane), C24 (tetracosane), C28 (octacosane) and C40 (tetracontane). Additional markers may be analyzed upon client request.
- 10.3 Initial Calibration
- 10.3.1 Inject an aliquot of each level of the calibration standard, minimum 5 points, for the target compounds being analyzed (i.e., diesel and/or motor oil). The low level standard should be at or below the reporting limit. The highest level standard defines the working range of the detector. Quadratic (Second order curves) require 6 calibration points.
- 10.3.1.1 If additional hydrocarbons are requested (e.g. kerosene), a minimum 5 point calibration for SW 8015 or a minimum 3 point calibration for OA-2 is performed, with the low level standard at or below the reporting limit.
- 10.3.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., FID replacement).
- 10.3.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica corporate policy, "Selection of Calibration Points", CA-T-P-002.
- 10.3.4 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 20\%$.
- 10.3.5 If the %RSD in the initial calibration is $> 20\%$, then linear regression may be used to evaluate the curve or recalibration may be done.
- 10.3.5.1 If a linear regression curve is used, the intercept of the curve at zero response must be less than the reporting limit for the analyte.
- 10.3.5.2 If a linear regression curve is used, r^2 must be ≥ 0.990
- 10.3.5.3 Use of $1/\text{Concentration}^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve.
- 10.4 Initial Calibration Verification (ICV)
- 10.4.1 The initial calibration verification standard should be from a different standard source than the one used for the initial calibration.
- 10.4.2 An ICV for the target compounds being analyzed (diesel and/or motor oil) must be performed with every initial calibration.
- 10.4.2.1 If additional hydrocarbons were requested, an ICV is analyzed for those hydrocarbons.
- 10.4.3 The ICV performance for each analyte must be within: $\pm 20\%$ D criteria for 8015D, and $\pm 15\%$ D for 8015B.
- 10.4.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard.
- 10.4.3.2 Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).
- 10.4.3.3 Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the

laboratory director. Any such action must be documented in an NCM.

- 10.5 Continuing Calibration Verification (CCV)
 - 10.5.1 A CCV may be from the same source as the calibration or a second source.
 - 10.5.2 Analyze a CCV for each target hydrocarbon being analyzed. It is not necessary to analyze a CCV for every hydrocarbon having an initial calibration, **only** those being analyzed.
 - 10.5.3 **8015 requirements**
 - 10.5.3.1 Analyte response factors must be verified at the beginning of each analytical run, (by either an ICV or a CCV), after every 10 samples and at the end of the analytical run through the analysis of a mid-level calibration standard.
 - 10.5.3.2 A CCV %D must be within +/- 20% D for each analyte for 8015D
 - 10.5.3.3 A CCV %D must be within +/-15%D for each analyte for 8015B.
 - 10.5.4 **OA-2 requirements**
 - 10.5.4.1 Analyte response factors must be verified on each working day by the measurement of one or more calibration standards. The laboratory runs a calibration standard at the beginning of each analytical run, (by either an ICV or a CCV), after every 10 samples and at the end of the analytical run through the analysis of a mid-level calibration standard.
 - 10.5.4.2 The ending CCV may be omitted for OA-2.
 - 10.5.4.3 A CCV %D must be within +/- 20% D for each analyte.
 - 10.5.5 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument.
 - 10.5.5.1 If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed.
 - 10.5.5.2 If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require calibration and all samples run after the last passing CCV must be reanalyzed.
- 10.6 Retention Time Windows
 - 10.6.1 Retention Time Windows: analytes and surrogate(s):
 - 10.6.1.1 Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.
 - 10.6.1.2 The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the midpoint standard of the initial calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.
 - 10.6.1.3 If the retention time window as calculated above is less than +/- 0.015 minutes, use +/- 0.015 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.
 - 10.6.1.4 A new retention time window study is performed annually or when the analytical column from a new vendor or different stationary phase is used. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.
 - 10.6.2 Retention Time Window for Target Analytes

- 10.6.2.1 Retention Time windows for hydrocarbons are established daily. The retention time window is based on the retention time of the applicable carbon marker standard.
- 10.6.3 Corrective Action for Retention Times
- 10.6.3.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed.

11.0 PROCEDURE

- 11.1 Allow standards, samples and sample extracts to reach ambient temperature before analysis.
- 11.2 All analysis conditions and injection volumes for samples must be the same as for the calibration standards.
- 11.3 Sample introduction:
- 11.3.1 The samples are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.
- 11.4 Perform all qualitative and quantitative measurements. When the standards and extracts are not being used, refrigerate them at -20 to 0°C and protected from light in screw cap vials equipped with unpierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 External Standard Calculations
- 12.2.1 Analyte Concentration (µg/L) in sample

Concentration (µg/L):

$$[C] = \frac{A_x * V_t * D}{\overline{CF} * V_i * V_s}$$

Where:

$[C]$	=	Analyte Concentration in sample (µg/L)
A_x	=	Area of peak (response)
V_t	=	Total volume of extract (µL)
D	=	Dilution factor
$\overline{CF}$	=	Calibration factor ($\overline{RF}$ in target) – Response factor
V_i	=	Volume of extract injected (µL)
V_s	=	Volume of sample extracted

- 12.2.2 On column concentration (ug/mL):

$$[OC] = \frac{A_x}{\overline{CF}}$$

Where:

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$$[OC] = \text{On Column Concentration [typically expressed in } \mu\text{g/mL (ppm)]}$$

Then substitute/derive

$$[C] = [OC] \left(\frac{V_t * D}{V_i * V_s} \right)$$

When *on column concentration* $[OC]$ is equal to the *CAL-AMT (calibration amount)* of the low level standard needed to support the *reporting limit* ($\mu\text{g/L}$) and we solve the equation for *concentration* ($\mu\text{g/L}$)

Then

$$[C] = RL = [OC] \left(\frac{V_t * D}{V_i * V_s} \right)$$

Where:

$RL = \text{Reporting Limit}$

12.3 Manual Integrations

12.3.1 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. See TestAmerica policy: CA-Q-S-002, Manual Integration. Manual integrations are denoted with a “M” flag on the Target quantitation report.

12.4 Use the total integrated area that results from a common baseline (include all of the area down to the baseline) between the retention time limits established, to quantify total extractable petroleum hydrocarbons in the sample.

12.5 Quantitation of Hydrocarbons

12.5.1 Total Area Response is the total response in the chosen retention time window.

12.6 Dilutions

12.6.1 If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range.

12.6.2 It may be necessary to dilute samples due to matrix.

12.7 Carryover

12.7.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.7.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

12.7.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 OA-2 evaluation:
 - 13.1.1 For hydrocarbons, the analyst should rely primarily on pattern recognition.
 - 13.1.2 Retention time windows are used for identification, but the “pattern” produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the pattern resembles the standard chromatogram.
 - 13.1.3 Weathering can make pattern determination difficult. Often the early eluting peaks of the hydrocarbon are diminished and peaks may not be in the anticipated ratios. If severe weathering is indicated, the analyst may base hydrocarbon pattern on the general shape of the chromatography (e.g. diesel crown) and later eluting peaks.
- 13.2 8015 evaluation:
 - 13.2.1 For 8015, evaluation is based on carbon ranges, not pattern. All peaks observed within the prescribed carbon range are used in quantitation.
 - 13.2.2 If the peaks observed appear as a single peak (e.g. PAHs) rather than “hydrocarbon like” (multi-peaks), the analyst may exclude them from quantitation. An NCM is written to document the reason for the exclusion.
- 13.3 Weathering, single peaks and comments regarding pattern should be documented using the NCM process.
- 13.4 The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.
- 13.5 Method Blank
 - 13.5.1 Acceptance Criteria:
 - 13.5.1.1 No target analytes may be present in the method blank above the reporting limit.
 - 13.5.1.2 The method blank must have acceptable surrogate recoveries.
 - 13.5.1.3 Corrective Action for Method Blanks not meeting acceptance criteria:
 - 13.5.1.3.1 Method Blank Contamination – Blank contamination above the RL (>1/2 RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.
 - 13.5.1.3.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.
- 13.6 Laboratory Control Sample (LCS)
 - 13.6.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.
 - 13.6.1.1 The LCS must have acceptable surrogate recoveries. .
 - 13.6.1.2 Corrective Action for LCS not meeting acceptance criteria:
 - 13.6.1.2.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. . In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM

- 13.6.1.2.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.
- 13.6.1.2.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.
- 13.6.1.2.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM
- 13.6.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
- 13.6.3 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.
- 13.6.4 Corrective Action for MS/MSD not meeting acceptance criteria:
 - 13.6.4.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.
- 13.6.5 Sample result evaluation
 - 13.6.5.1 Dilutions
 - 13.6.5.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
 - 13.6.5.1.1.1 Dilution: Sample– An NCM is written to document the reason for the dilution.
 - 13.6.5.1.1.2 Dilution: Surrogate(s) and/or spike(s) diluted out– An NCM is written to document the reason for the dilution.
 - 13.6.5.2 Carryover
 - 13.6.5.2.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
 - 13.6.5.2.2 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.
 - 13.6.5.2.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.
 - 13.6.5.3 Insufficient Sample
 - 13.6.5.3.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative. An NCM is written to document the insufficient volume.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, reporting limits, and QC acceptance limits, are maintained in the LIMs.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

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- 14.3 Training qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 Waste Streams Produced by the Method
 - 16.1.1 The following waste streams are produced when this method is carried out.
 - 16.1.1.1 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".
 - 16.1.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", Method 8000C
- 17.2 SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" Method 8015B and D.
- 17.3 IOWA Method OA-2 University Hygienic Laboratory, Iowa City, IA, rev. 07/27/93.
- 17.4 St. Louis Quality Assurance Manual (ST-QAM), current revision.
- 17.5 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.6 TestAmerica Policy CA-Q-S-002, Manual Integration
- 17.7 TestAmerica Policy CA-T-P-002, Selection of Calibration Points
- 17.8 Associated SOPs
 - 17.8.1 ST-OP-0001, Labware Preparation for Organic Analysis
 - 17.8.2 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Water and Soils, Based on SW-846 3500 Series, 3600 Series, and 600 Series
 - 17.8.3 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.8.4 ST-QA-0002, Standard and Reagent Preparation

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- 17.8.5 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."
- 17.8.6 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
- 17.8.7 ST-QA-0016, IDL/MDL Determination
- 17.8.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 MODIFICATIONS FROM REFERENCE METHOD

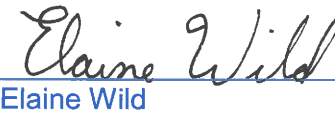
- 18.1 Chapter 1 of SW-846 states that the Method Blank should not contain any analyte of interest at or above the Method Detection Limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

19.0 CHANGES TO PREVIOUS SOP REVISION

- 19.1 No Changes, Annual Review.
- 19.2 Rev 16;
 - 19.2.1 Annual Review, No Changes
- 19.3 Rev 17:
 - 19.3.1 Updated scope in section 1.0.
 - 19.3.2 Removed Gasoline Range Organics (GRO) from the section 2.0.
 - 19.3.3 Removed the definition for gasoline for method 8015B in section 3.0.
 - 19.3.4 Updated equipments and supplies throughout section 6.0.
 - 19.3.5 Updated calibration standards in sections 7.4, 7.5 and 7.6.
 - 19.3.6 Updated the retention time standard being analyzed daily in section 10.2.
 - 19.3.7 Updated initial calibration in section 10.3.
 - 19.3.8 Updated ICV and CCV information in sections 10.4 and 10.5.
 - 19.3.9 Updated the storage temperature for extracts in section 11.0.
 - 19.3.10 Added external calibration calculations to Section 12.2
 - 19.3.11 Updated the quantitation of hydrocarbons in section 12.5.
 - 19.3.12 Removed the reference to "Quantims" and "Clouseau".
 - 19.3.13 Updated corrective action for non-conformances in section 13.0.
 - 19.3.14 Corrected references to Corporate Policies in Sections 17.6 & 17.7.
- 19.4 Rev 18:
 - 19.4.1 Added carbon range retentiontime marker to Section 7
 - 19.4.2 Changed "stock" to "primary" for clarification in Section 7
 - 19.4.3 Added six point requirement for quadratic curves to Section 10.
 - 19.4.4 Added requirement for NCM when excluding peaks to Section 13.
 - 19.4.5 Added location of Control Limits (found in LIMS) to Section 13.
- 19.5 Rev 19 (06/02/2014)
 - 19.5.1 Updated equipment and supplies in Section 6
 - 19.5.2 Added names of marker standards in section 7.5.1 and 10.2.2
 - 19.5.3 Corrected fragmented sentences in section 10.3.5
 - 19.5.4 Added retention time windows criteria in section 10.6.1.4
- 19.6 Rev 20 (06/22/2015)
 - 19.6.1 Updated ICV and CCV information in Section 10.4 and 10.5.
 - 19.6.2 Added "D" method criteria
 - 19.6.3 Annual Review, no changes (09/23/2016)

Title: PCB GC ANALYSIS
[SW-846 8000B/8000C/8082A; EPA 608]
SW-846 8000D MW
10-28-16

Approvals (Signature/Date):

 Dennis Konopka Extractables Manager	10/28/16 Date	 Michael Ridenhower Health & Safety Manager / Coordinator	10/28/16 Date
 Kristen Ely Quality Assurance Manager	10/28/16 Date	 Elaine Wild Laboratory Director	10/28/16 Date

This SOP was previously identified as SOP No. ST-GC-0015 Rev. 19

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes procedures to be used for the analysis of polychlorinated biphenyls (PCB) by GC/ECD. The PCBs are determined and quantitated as multi-component Aroclor mixes.
- 1.2 Sample preparation techniques are described in SOP ST-OP-0002.
- 1.3 This SOP is based on EPA SW-846 Methods 8000B, 8000C, 8000D and 8082A, and EPA Method 608.
- 1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.4.1 Additional compounds may be amendable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

- 2.1 Aqueous samples are prepared for analysis using the separatory funnel liquid / liquid extraction technique. Solid samples are prepared using sonication. Wipes are extracted by autoshaker.
- 2.2 After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. The internal standardization procedure is used.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers
- 3.2 Lower Limit of Quantitation – LLOQ. SOP refers to this as the reporting limit (RL) The LLOQ is verified annually (see SOP ST-QA-0016 for details).

4.0 INTERFERENCES

- 4.1 Interferences in GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.
- 4.2 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). For PCBs the most common cleanup procedure is the Sulfuric Acid cleanup (Method 3665A).
- 4.3 Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, DDD) may interfere. When suspected, a standard of DDT analogs should be injected to determine which of the aroclor peaks may be subject to said interference, [DDT may interfere with the last major Aroclor 1254 peak in soil/sediment samples.]

- 4.4 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or high biased results.
- 4.5 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and interferences to sample extracts. Strict attention to glassware cleaning and handling and demonstration of solvent purity will lead to minimization of these interferences.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
- 5.2.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.2.2 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- 5.3 PRIMARY MATERIALS USED
- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	OSHA Exposure Limit (2)	Signs and symptoms of exposure/Unusual Hazards
Hexane	Flammable Irritant	500 ppm- (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limits refer to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 GC/ECD system: The lab utilizes a Hewlett Packard GC 5890 dual ECD system and an Agilent GC 6890 dual micro ECD system with autosampler.
 - 6.1.1 Columns used Restek Rtx-CLPesticides 30 meter, 0.53 mmID, 0.5 μ m df; Restek Rtx-CLPesticides2 30 meter, 0.52 mmID, 0.42 μ m df
 - 6.1.2 GC column types, and instrument run conditions are posted in the maintenance calendar and reside in the Chemstation method.
- 6.2 Data System – Chemstation for acquisition and Chrom™ for data processing.
- 6.3 Amber and/or clear glass vials. Crimp top seals.
- 6.4 Disposal pipettes.
- 6.5 Micro syringes – 10- μ L, 250- μ L, 500- μ L, 1000- μ L. Hamilton 1700 series.
- 6.6 Volumetric flasks, Class A

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 See recipes for standards in the TALS Reagent module. Information listed in appendix to this SOP.
- 7.3 PCB primary standard solutions:
 - 7.2.1 Primary standards are prepared by dilution of neat liquid Aroclor mix 1016/1260, and from single aroclor mixes in hexane. Primary standards must be replaced after 6 months or the manufacturer's expiration date whichever is shorter. Standards must be stored in refrigerator or freezer at $\leq 6^{\circ}\text{C}$.
- 7.4 Working standards:
 - 7.7.1 The working standards are prepared in hexane from the primary standard solution for a minimum of five concentration levels of the Aroclor mix 1016/1260 and one level of the single aroclors. Working standards must be replaced after 6 months or manufacturer's expiration date whichever is shorter. All working standards expire after six months or at the expiration date of their stock standards, whichever comes sooner.
- 7.5 Gases for carrier and make-up: Hydrogen carrier, Nitrogen make-up.
- 7.6 Decachlorobiphenyl (surrogate)
- 7.7 An Internal Standard (IS) solution is prepared. Compounds in the I.S. Mix are: 1-Bromo-2-Nitrobenzene
- 7.8 Internal Standards are added to all standards and extracts to result in 50 ng injected onto the column.
- 7.9 Copper powder
 - 7.9.1 Remove oxides (if powder is dark) by treating with dilute nitric acid, rinse with organic-

free reagent water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.

- 7.10 DDT analog standard – used when possible interference from DDT analogs is present. This is a single standard containing DDT, DDE and DDD at a concentration judged sufficient by the analyst.
- 7.11 Initial Calibration Verification (ICV) spiking standard is similar to calibration standards, but are from a different source or vendor and are prepared and stored in the same way as calibration standards.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Water samples are unpreserved and stored at >0 to $\leq 6^{\circ}$ C.
- 8.3 Soil samples are refrigerated at >0 to $\leq 6^{\circ}$ C.
- 8.4 Extracts must be refrigerated at $\leq 6^{\circ}$ C.
- 8.5 Sample extracts need to be isolated from all potential contaminants and all standards.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
- 9.1.2 A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples, and/or 24 hours (12 hours for GC/MS).
- 9.1.3 An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
- 9.1.4 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.5 Each analytical batch may contain up to 20 environmental samples, a Method Blank (MB), a single Laboratory Control Sample (LCS) and a Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair. In the event that there is insufficient sample to analyze an MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.1.6 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual “sample batch” does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 Method Blank (MB)

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every batch (20 or fewer samples of the same matrix).

- 9.2.3 DI water is used as the blank matrix for water batches.
- 9.2.4 Sodium sulfate is used as the blank matrix for solid batches.
 - 9.2.4.1 South Carolina requires the use of Ottawa sand for the Method Blank
- 9.3 **Laboratory Control Sample (LCS)**
 - 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.3.2 An LCS must be prepared with every batch.
 - 9.3.3 DI water, spiked with the analytes of interest is used as the LCS for water batches.
 - 9.3.4 Sodium sulfate, spiked with the analytes of interest is used as the LCS for solid batches (South Carolina requires the use of Ottawa sand for the LCS solid matrix).
- 9.4 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)**
 - 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.4.2 Additional MS/MSDs do not count towards the 20 samples in an analytical batch.
 - 9.4.3 An MS/MSD can be prepared with every batch, although it is not a method requirement. If there is insufficient sample to perform an MS/MSD, a duplicate LCS is analyzed.
- 9.5 **Procedural Variations/Nonconformance and Corrective Action**
 - 9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
 - 9.5.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Internal standard calibration is used.
- 10.2 Initial Calibration
 - 10.2.1 Prepare an Aroclor 1016/1260 and Decachlorobiphenyl (surrogate) standard at a minimum of five concentration levels. (Six points are required if a quadratic (second order) curve is used.) The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in Table 1. [NOTE: Quadratic regression is not allowed for South Carolina compliance work.]
 - 10.2.1.1 If a specific Aroclor is of interest for a particular project, that Aroclor may be used for the five point calibration rather than the 1016 / 1260 mix. See Client Requirements Sheet or Memo.
 - 10.2.1.2 A single point calibration for Aroclor 1221, 1232, 1242, 1248, 1254, 1262, and 1268 is performed with every initial calibration for pattern recognition.
 - 10.2.1.2.1 If any of the above single point Aroclors are detected, samples may be required to be re-analyzed under a five point calibration for the aroclor found. See Client Requirement Sheet to determine if this is needed.
 - 10.2.1.3 Select 3-5 major peaks in the analyte pattern. Calculate the response using the area of these individual peaks.
 - 10.2.1.3.1 Aroclor 1221 uses only 3 peaks due to the limited amount of peaks available to choose from.
 - 10.2.2 Add the internal standard mixture to result in 50 ng on column.
 - 10.2.3 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any

significant changes in instrument operating parameters, and major instrument maintenance (e.g., ECD replacement).

- 10.2.4 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Corporate policy, "Calibration Point Selection", CA-Q-P-003.

10.3 SW 8082 criteria

- 10.3.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 20\%$.
- 10.3.2 If the %RSDs in the initial calibration is $> 20\%$, then calibration using a linear regression may be employed.
- 10.3.2.1 If a linear regression curve is used, the intercept of the curve at zero response must be less than + or – the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.
- 10.3.2.2 If a linear regression curve is used, r must be ≥ 0.99
- 10.3.2.3 For Samples requiring adherence to SW 846 Methods 8000B and 8000D (including South Carolina compliance work), forcing through zero is NOT allowed.
- 10.3.2.4 Weighting of data points
- 10.3.2.4.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it may be preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4 Acceptance criteria independent of calibration model

- 10.4.1 Either of the procedures Percent Error or Relative Standard Error (RSE) may be used to determine calibration function acceptability for linear and non-linear curves. Both evaluate the difference between the measured and the true amounts or concentrations used to create the model.
- 10.4.1.1 $\%Error = \frac{x_i - x'_i}{x_i} \times 100$
- 10.4.1.2 Where x'_i = measured amount of the analyte at calibration level i in mass or concentration units and x_i = the true amount of the analyte at calibration level i in mass or concentration units.
- 10.4.1.3 % Error should be $\leq 30\%$. For some data uses $\leq 50\%$ may be acceptable for the lowest calibration point.
- 10.4.1.4 Calculation of Relative Standard Error:
- 10.4.1.5

$$RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

Where X_i = True amount of the calibration level i , in mass or concentration units
 X'_i = measured amount of analyte in calibration level i , in mass or concentration units

p = number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3,)

n = number of calibration points

10.4.1.6 RSE acceptance criteria for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If not defined in the method, use $\pm 20\%$

10.5 608 Criteria

10.5.1 Method 608 only requires a 3 point calibration. We routinely perform a 5 point calibration; however, 2 points may be removed from a curve if necessary to meet 608 calibration criteria. The lowest level of the curve must be at or below the reporting limit. The other standards define the working range of the detector.

10.5.1.1 Refer to the TestAmerica Corporate policy, "Calibration Point Selection", CA-Q-T-002.

10.5.2 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $< 10\%$.

10.5.3 If the %RSDs in the initial calibration is $\geq 10\%$, then calibration using a linear regression may be employed.

10.5.3.1 If a linear regression curve is used, the intercept of the curve at zero response must be less than + or – the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.

10.5.3.2 If a linear regression curve is used, r^2 must be ≥ 0.990

10.5.3.3 Use of $1/\text{Concentration}^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

10.5.3.3.1 Weighting of data points

10.5.3.3.2 The points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points.

$1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.6 Initial Calibration Verification (ICV)

10.6.1 An initial calibration verification standard must be a different standard source than the one used for the initial calibration.

10.6.1.1 The ICV is not performed for the single point aroclors.

10.6.2 An ICV must be performed with every initial calibration.

10.6.2.1 A passing ICV may be used as the opening CCV for a set of samples run following the ICV.

10.6.3 The ICV performance must be within $\pm 20\%$ D for 8082A and $\pm 15\%$ for Method 608..

10.6.3.1 Only the analytes present in the ICAL are evaluated for the ICV criteria.

10.6.3.2 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).

10.6.3.3 Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.7 Continuing Calibration Verification (CCV)

10.7.1 A CCV must be run at the start of each 24 hour period.

- 10.7.2 A CCV may be a second source or the same source as the initial calibration standards and should be made to represent the midpoint of the curve.
- 10.7.3 Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV).
 - 10.7.3.1 Analyte response factors must be verified after every 20 samples (10 samples for DOD) and at the end of the analysis run through the analysis of a CCV..
- 10.7.4 It is adequate to verify calibration with a single mixture of Aroclors 1016 and 1260.
 - 10.7.4.1 For projects with specific Aroclor requirements, a specific Aroclor may be included in the daily calibration check.
- 10.7.5 The calibration verification is acceptable if the %D for both 1016/1260 and the surrogate (DCB) is $\leq 20\%$ for 8082A and $\leq 15\%$ for Method 608..
 - 10.7.5.1 The same criterion is used if other Aroclor verifications are performed.
 - 10.7.5.2 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument.
 - 10.7.5.3 If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed.
 - 10.7.5.4 If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration).
- 10.8 Retention Time (RT) Windows
 - 10.8.1 Retention Time (RT) windows must be determined for all analytes.
 - 10.8.2 Establishing RT windows:
 - 10.8.2.1 Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used).
 - 10.8.2.2 If three days of analytical data are not available, use a default RT window of ± 0.01 minutes. At the end of the batch evaluate all CCVs and LCS in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.
 - 10.8.2.3 Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 min, in which case the window is set at ± 0.01 min. For example, if the maximum RT deviation for a specific analyte is 0.008 min, then the RT window is set at ± 0.012 min.
 - 10.8.2.4 **NOTE:** For the multi-component analytes, Toxaphene and Technical Chlordane, the maximum deviation must be evaluated for each of the 3 to 6 major peaks used for sample calculations .
 - 10.8.2.5 The center of the retention time window is the retention time from the CCV performed at the beginning of the analytical run. For samples run during the same shift as the initial calibration, use the retention time of the mid-point standard from the initial calibration. Some clients may have specific requirements regarding the updating of RT windows. Review the Client Requirement Memo for instructions.
 - 10.8.2.6 Retention time windows for analytes of interest must not overlap
 - 10.8.2.7 Method 8000B requires a new retention time window study be performed annually or when the analytical column from a new vendor or different stationary phase is used. 8000C and 8000D also requires a new study when the column is clipped during maintenance.
 - 10.8.2.8 Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.
- 10.9 Ongoing evaluation of retention time windows

- 10.9.1 Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below)
 - 10.9.1.1 Matrix spike analytes may fall outside the retention time window if there is a large non-target peak coeluting with the analyte in the matrix spike
- 10.9.2 If any analytes fall outside the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.
 - 10.9.2.1.1 Retention time windows should be reliably narrower than ± 0.03 min. If RT windows wider than this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained
- 10.9.3 Retention Time Criteria
 - 10.9.3.1 The retention times of AR 1016/1260 (and other aroclors if applicable) in each continuing calibration must be within the retention time windows established.
 - 10.9.3.2 The retention times of AR 1016/1260 (and other aroclors if applicable) are updated using the 1st CCV or RT marker of the day.
- 10.10 Method Detection Limit Studies
 - 10.10.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. The study must encompass both columns. See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs

11.0 PROCEDURE

- 11.1 Samples are prepared following ST-OP-0002
- 11.2 Allow standards, samples and sample extracts to reach ambient temperature before analysis.
- 11.3 Sulfur Removal
 - 11.3.1 Sulfur Removal with Copper Powder
 - 11.3.1.1 Transfer 1.0 mL of sample extract, and associated QC, into labeled vials.
 - 11.3.1.2 Add approximately 2g cleaned copper powder to the vial.
 - 11.3.1.3 Mix for one minute on a mechanical shaker.
 - 11.3.1.4 Allow phases to separate.
 - 11.3.1.5 Separate extract from copper by drawing the extract off with a disposable pipette.
 - 11.3.1.6 An NCM is written to document that a copper clean-up was done.
 - 11.3.1.7 Transfer the supernate to a clean, labeled vial.
- 11.4 All analysis conditions and injection volumes for samples must be the same as for the calibration standards
- 11.5 Add internal standard to the extract to result in 50ng injected on column.
- 11.6 Sample Introduction
 - 11.6.1 Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

- 11.7 Perform all qualitative and quantitative measurements. When the standards and extracts are not being used, refrigerate them at $\leq 6^{\circ}\text{C}$, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 Internal Standard Calculations

12.2.1 Analyte Concentration ($\mu\text{g/L}$) in sample

Concentration ($\mu\text{g/L}$):

$$[C] = \frac{A_x * I_s * V_t * D}{A_{is} * RRF_{avg} * V_o * V_i}$$

Where:

$[C]$	=	Analyte Concentration in sample ($\mu\text{g/L}$)
A_x	=	peak area or peak height
I_s	=	Amount of each internal standard injected (ng)
V_t	=	volume of concentrated extract (mL)
D	=	Dilution factor
A_{is}	=	Peak area or peak height of each internal standard
RRF_{avg}	=	Average relative response factor
V_o	=	Volume of water extracted (L)
V_i	=	Injection volume

- 12.2.2 See Chrom software for additional calculations.

12.3 Manual Integrations

- 12.3.1 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. See TestAmerica policy: CA-Q-S-002, "Manual Integrations". Manual integrations are denoted with an "M" flag on the Chrom quantitation report.

12.4 Identification of Aroclors

- 12.4.1 Tentative identification of an Aroclor occurs when multi-component peaks are found within their respective retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required.
- 12.4.2 Definitive Aroclor identification is based primarily on pattern recognition. Retention times and retention time windows are used to tentatively identify Aroclors, but the fingerprint produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

- 12.4.3 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern. Source-specific information, such as historical data, indicating the anticipation of Aroclors must be documented. The pattern of peaks can serve as confirmation depending of the client specific project requirements.

12.5 Quantitation of Aroclors

- 12.5.1 Use three to five major peaks when calibrating Aroclors. Choose peaks distinctive of the individual Aroclor. Any manual integration made in the ICAL levels must be noted and be made in any subsequent samples to maintain consistency with the initial calibration. These same three to five peaks are then used to calculate the response/concentration of the Aroclor(s) when present in a sample.

12.5.1.1 For Aroclor 1221 only three peaks are used due to the limited number of peaks available.

12.5.1.2 In instances where less than five peaks are used those peaks that are not used are said to be “dropped” and an NCM must be written. When quantitating Aroclors 1016/1260 in an LCS/D and/or MS/MSD, all five peaks must be used. In samples, less than the standard five peaks may be used to quantitate target analytes if there are demonstrated matrix interferences and/or if multiple, overlapping Aroclors are present. If there is a predominance of one Aroclor that elutes next to and shares peaks with another Aroclor and it is apparent to the analyst that the lesser Aroclor’s concentration is elevated significantly by the more dominant Aroclor, then three peaks may be dropped and an NCM written. It is never allowable to quantitate an Aroclor using only one peak.

- 12.5.2 If well distinguishable Aroclor patterns are present, then multiple Aroclors are quantitated and reported.

12.5.2.1 Aroclor elution times may overlap and one or more Aroclor peaks may be “shared” with another Aroclor. When this occurs, only the predominant Aroclor is quantitated and reported. Aroclors sharing elution time and peaks include: 1016, 1232, 1242, 1248 – these cannot be identified together (unless quantitating an MS/D where Aroclor 1016 is known to be present). Aroclors 1260 and 1262 also share peaks; only one of these can be reported (unless quantitating and MS/D where 1260 is known to be present).

12.5.3 Dual Column Quantitation

12.5.3.1 Dual column confirmation is required for positive Aroclor identification. A secondary column using an alternate phase is employed and the sample is injected simultaneously into both a primary and secondary column. Elution times often differ, as does overall pattern/fingerprint of Aroclors between the two columns. Determination of target analytes on the secondary column is made in the same way as on the primary column. Target analytes may be reported from either column.

12.5.3.2 Method 8000C states to report the lower result of the two columns, unless the Client SOW requires that the higher result be reported. Method 8000B requires the reporting of the higher of the two columns. See Client Requirement Sheet for determination.

12.5.3.2.1 For non-detect (ND) results, report from the primary channel if all QC and CCVs are acceptable. If the QC and CCVs are only acceptable on the secondary column, report the non-detects from this column.

12.5.3.2.2 If the %D between the two columns is greater than 40%, report the higher result if there are obvious chromatographic interferences on the column with the lower result.

12.5.3.2.3 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or

operator) during peak integration. If no anomalies are noted, review the chromatographic conditions.

12.5.3.2.3.1 If there is no evidence of chromatographic problems, report the lower result for method 8000C and the higher result for method 8000B. The data user should be advised of the disparity between the results on the two columns.

12.5.3.2.3.2 Use the higher result if there is obvious chromatographic interference on the column with the lower result.

12.5.3.3 The QC should be reported from the column that reflects the column used for the majority of the samples associated with the QC.

12.5.3.4 The surrogate should be reported from the column used for the reporting of the sample results.

12.6 Dilutions

12.6.1 If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample data is “E” flagged and the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Target analytes with resulting concentrations lower than the dilution adjusted RL should be flagged with a “J” qualifier.

12.6.2 It may be necessary to dilute samples due to matrix.

12.7 Carryover

12.7.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.7.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

12.7.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank (MB)

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit. SW846 Method 8000D Revision 5 requires that the Method Blank be less than ½ the Reporting Limit (aka LLOQ).

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet. South Carolina requires blank be below the RL. If blank > RL, take corrective action and re-analyze associated samples.

13.2.1.3 The Method Blank must have acceptable surrogate recoveries.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – Blank contamination above the RL (>1/2 RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.

13.2.2.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate

recovery is high and the samples are non-detect, the data may be reported with an NCM.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). . South Carolina requires the LCS to recover within limit of 70 -130%. If recovery is outside limits take corrective action and re-analyze associated samples.

13.3.1.2 The LCS must have acceptable surrogate recoveries.

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM

13.3.2.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.

13.3.2.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.

13.3.2.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM.

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.

13.5 Surrogate

13.5.1 All Surrogates should be within established control limits for accuracy (%Recovery).

13.5.2 Corrective Action for Surrogate not meeting acceptance criteria:

13.5.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.6 Sample Result Evaluation

13.6.1 Dilutions

13.6.2 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.1.1.1 Dilution: Sample– An NCM is written to document the reason for the dilution

13.1.1.2 Dilution: Surrogate(s) and/or spikes diluted out– Dilution: Surrogate(s) and/or spike(s) diluted out– An NCM is written to document the reason for the dilution.

13.6.3 Carryover

- 13.6.3.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
- 13.6.3.2 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.
- 13.6.3.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.
- 13.6.4 Internal Standards
 - 13.6.4.1 Acceptance Criteria
 - 13.6.4.1.1 If the area for the internal standard in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, corrective action must be taken.
 - 13.6.4.1.2 If the area for the internal standard in the samples, spikes and blanks changes by a factor of two (-50% to +100%) from the areas determined in the continuing calibration analyzed that day, corrective action must be taken. The samples, spikes or blanks should be reanalyzed or the data should be qualified. (Some programs require that the midpoint of the initial calibration be used for ISTD monitoring. See the project CRM for specifics.)
 - 13.6.4.2 Corrective Action for Internal Standards not meeting acceptance criteria:
 - 13.6.4.2.1 Internal Standard excursion – high – High ISTD recovery indicates a potential low bias to the analytical result. Instrument maintenance, if required, is done and affected samples are reanalyzed. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.
 - 13.6.4.2.2 Internal Standard excursion – low – Low ISTD recovery indicates the potential for a high bias to analytical results. Samples with positive hits above the RL for the analytes associated to the poor ISTD recovery require re-analysis. Instrument maintenance, if required, is done. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.
- 13.7 Insufficient Sample
 - 13.7.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report case narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.1 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".

16.2.1.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

16.2.1.4 Expired primary and working PCB standards shall be segregated and placed into the proper satellite accumulation container specifically for PCB waste which is located within the GC lab.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000B, 8000C and 8000D..

17.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8082A, 8081B Update IV, February 2007 and EPA 608 Method.

17.3 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision

17.4 Corporate Environmental Health and Safety Manual (CW-E-M-001), current revision

17.5 TestAmerica Policy CA-Q-S-002, Manual Integrations

17.6 TestAmerica Policy CA-Q-P-003, Calibration Point selection

17.7 Associated SOPs

17.7.1 ST-OP-0001, Labware Preparation for Organic Analysis

17.7.2 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Water and Soils, Based on SW-846 3500 Series, 3600 Series, 8151A and 600 Series

- 17.7.3 ST-OP-0003, Extraction of PCB in Oil
- 17.7.4 ST-QA-0002, Standard and Reagent Preparation
- 17.7.5 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."
- 17.7.6 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
- 17.7.7 ST-QA-0016, IDL/MDL Determination
- 17.7.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process
- 17.7.9 ST-PM-0002, Sample Receipt and Chain of Custody

18.0 MODIFICATIONS FROM REFERENCE METHOD

- 18.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.
 - 18.1.1 Method Blanks for South Carolina compliance work MUST be below the RL
- 18.2 The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.
- 18.3 Method 608 only requires a 3 point calibration. We routinely perform a 5 point calibration; however, 2 points may be removed from a curve if necessary to meet 608 calibration criteria. The lowest level of the curve must be at or below the reporting limit.
- 18.4 SW846 Method 8000C requires that new retention time windows be established if a GC column has been shortened during maintenance. Given the matrices of the sample the laboratory receives, and the number of times the GC column may require clipping, TestAmerica St. Louis does not perform a RT study after clipping a column. RT studies done by the laboratory show that, historically, RT windows have not been greater than the method allowed 0.03 minutes. The lab defaults to a 0.03 minute RT window as allowed by the method.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Updated the table one regarding the levels of calibration of Aroclor 1016/1260 and the amount of the surrogate used, Decachlorobipenyl.
- 19.2 Rev 10:
 - 19.2.1 Removing holding times for PCB's in section 8.0 per method
- 19.3 Revision 11:
 - 19.3.1 Removed references to QuantIMS and Clouseau – replaced with LIMS.
 - 19.3.2 Added software information to Section 6.
 - 19.3.3 Added composition of Method Blank and LCS to Section 9.
 - 19.3.4 Added requirement for 6 points for non-linear curves to Section 10.
 - 19.3.5 Added Reporting limit calculations to Section 12
 - 19.3.6 Added specific corrective actions to Section 13
 - 19.3.7 Updated text in Section 15 to include methods beyond those approved by EPA.
- 19.4 Rev 12 (4/21/14):
 - 19.4.1 Removed continuous extractions in section 2.
 - 19.4.2 Replaced Target with Chrom data systems in section 6.
 - 19.4.3 Updated location of instrument run setting in section 6.

- 19.4.4 Added location of standard recipes to section 7.
- 19.4.5 Updated retention time window study requirements in section 10.
- 19.4.6 Added exception to SW846 requirements in section 18.
- 19.4.7 Updated Initial Calibration sequence example following Table 1.
- 19.5 Revision 13 (9/18/14):
 - 19.5.1 Section 18 updated to reflect requirements for Method Blanks associated with South Carolina compliance work.
 - 19.5.2 Returned continuous liquid/liquid extraction to Section 2.1.
- 19.6 Revision 14 (10/30/14)
 - 19.6.1 Revision done to combine this SOP with requirements for South Carolina compliance work originally found in SOP ST-GC-0015SC, Rev 1.
 - 19.6.2 Updated Section 10 to include instruction that forcing through zero is NOT allowed for South Carolina compliance work.
 - 19.6.3 Updated Section 10.7 to include requirements from both 8000B and 8000C regarding timing of running retention time window studies.
 - 19.6.4 Added calculations to Section 12.
 - 19.6.5 Updated Section 12.5.3 to include instruction from both 8000B and 8000C in regards to dual column reporting requirements.
 - 19.6.6 Updated Section 13.3 to include South Carolina requirements for LCS recoveries.
- 19.7 Revision 15 (12/01/2014)
 - 19.7.1 Section 4 updated to address possible interference from the DDT analogs
 - 19.7.2 Section 6 updated to include specific columns used.
 - 19.7.3 Section 10 was updated to include a note that quadratic calibrations are not allowed for South Carolina compliance work; MDL study requirements add.
 - 19.7.4 Section 13 was updated to include the 70 – 130% LCS recovery requirement for South Carolina compliance work.
 - 19.7.5 Added standard preparation and concentration information to the appendices.
- 19.8 Revision 16 (04/24/2015)
 - 19.8.1 Added IS standards to Section 7.
 - 19.8.2 Update Section 10.
 - 19.8.2.1 Changed calibration on internal Section 10.1
 - 19.8.2.2 Added IS to initial calibration, Section 10.2
 - 19.8.2.3 Updated CCV requirements, Section 10.6
 - 19.8.2.4 Updated retention time updates, Section 10.7
 - 19.8.3 Included IS standards in procedure, Section 11.
 - 19.8.4 Updated acceptance criteria for IS, and surrogates, Section 13.
 - 19.8.5 Updated Appendix 1,
- 19.9 Revision 17 (06/12/2015)
 - 19.9.1 Removed reference to CLLE extraction in Section 2.1
 - 19.9.2 Removed external standard calculations from Section 12; add internal standard calculations.

- 19.10 Revision 18 (10/23/15)
 - 19.10.1 Corrected the CCV frequency requirement in Section 10.6.3.1.
- 19.11 Revision 19 (5/23/16)
 - 19.11.1 Sections 10.2 and 11.4 were updated to correct the Internal Standard amount – from 20ng to 50ng.
 - 19.11.2 Section 10.4 the r^2 criteria was updated
 - 19.11.3 Section 11.3 was updated to add the requirement to write an NCM to documents when Copper clean-up is done.
- 19.12 Revision 20 (10/28/16)
 - 19.12.1 Updated section 10 for retention time establishment and evaluation
 - 19.12.2 Added references to SW 846 Method 8000D
 - 19.12.3 Added reference to the LLOQ (Reporting Limit)
 - 19.12.4 Added explanations and calculations for % Error and Relative Standard Error

Table 1								
Calibration Levels ng/ml								
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	
Aroclor 1016/1260	50	100 Reporting Limit	250	500	1000 ICV/CCV	2000	4000	
The surrogate is included with all calibration mixes at the following levels								
Decachlorobiphenyl	2.5	5	12.5	25	50	100	200	

* Level 1 is optional.

Aroclors 1232, 1221, 1242, 1248, 1254, 1262 and 1268 may be quantitated within the range 100 to 4000 ng/mL

Analytical Sequence

Example Initial Calibration

Injection #

- 1 Conditioning standard
- 2 Solvent blank
- 3 Aroclor 1221
- 4 Aroclor 1232
- 5 Aroclor 1242
- 6 Aroclor 1248
- 7 Aroclor 1262
- 8 Aroclor 1268
- 9-15 Aroclor 1254 Levels 1-7
- 16 Independent Calibration Verification (ICV) standard
- 17-23 Aroclor 1016/1260 Levels 1-7
- 24 Independent Calibration Verification (ICV) standard
- 25-34 Sample Injections (max.10)
- 35 Aroclor 1016/1260 Level 5

Appendix 1

Standards:

Surrogate Stock – 1000 ug/mL

IS Standard Stock – 100ug/mL

Aroclors Stock – 1000 ug/mL

(1221, 1232, 1242, 1248, 1254, 1016/1260, 1262, 1268

Intermediates:

Aroclor(s) Intermediate (excluding 1016/1260) – 1 mL Aroclor stock + 50 µl of Surrogate stock to 25 mL with hexane = 40 ug/mL Aroclor/0.2 ug/ml surrogate

IS Intermediate – 0.5 mL IS stock to 500 mL with hexane = 1 ug/mL

Working Standards:

1016/1260 working – 1 ml of 1016/1260 stock + 50 µ of Surrogate stock to 250 ml with hexane = 4 ug/ml
1016.1260/0.2 ug/ml surrogate

Aroclor(s) working – 5 ml of aroclor intermediate to 20 ml with hexane = 4 ug/ml aroclor/0.2 ug/ml surrogate

Calibration:
Aroclor 1016/1260 and 1254

Calibration point	Dilution of Working Std.	Concentration (ug/ml)
1	80x	0.05
2	40x	0.10
3	20x	0.20
4	8x	0.50
5	4x	1.00
6	2x	2.00
7	1x	4.00
ICV (second source)	4x	1.00

Calibration:
Aroclor 1221, 1232, 1242, 1248, 1262, 1268

Calibration point	Dilution of Working Std.	Concentration (ug/ml)
5	4x	1.00

**Reagent ID: 8082SurrDeca_00018**

Type:	ASTD	Expiration Date:	02/28/2015
Description:	Decachlorobiphenyl Solution	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Saulters, Camme N
Storage Location:	Organic Prep Standards Storag	Vendor:	Ultra Scientific
Reagent Volume:	1.000 mL	Vendor Lot #:	CC-3147Z
Creation Date:	08/26/2013	Vendor Cat #:	PPS-150
Container(s):	190362		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
DCB Decachlorobiphenyl (Surr)					1004.00000	ug/mL

**Reagent ID: 8082 Spike_00018**

Type:	ASTD	Expiration Date:	06/01/2019
Description:	8082 spike aroclor 1060/1260 (PCB)	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Saulters, Camme N
Storage Location:	Organic Prep Standards Storag	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A094177
Creation Date:	08/26/2013	Vendor Cat #:	32039
Container(s):	190372		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1016					1000.00000	ug/mL
PCB-1260					1000.00000	ug/mL

**Reagent ID: Aroclor 1254_00010**

Type:	ASTD	Expiration Date:	08/31/2016
Description:	Aroclor 1254 Solution	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Ultra Scientific
Reagent Volume:	1.000 mL	Vendor Lot #:	CE-2632
Creation Date:	01/15/2013	Vendor Cat #:	PP-352
Container(s):	74708		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1254					100.00000	ug/mL
PCB-1254 Peak 1					100.00000	ug/mL
PCB-1254 Peak 2					100.00000	ug/mL
PCB-1254 Peak 3					100.00000	ug/mL
PCB-1254 Peak 4					100.00000	ug/mL
PCB-1254 Peak 5					100.00000	ug/mL



Reagent ID: Ar 1221_00002

Type:	ASTD	Expiration Date:	12/31/2018
Description:	Aroclor 1221 Standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A090667
Creation Date:	07/08/2013	Vendor Cat #:	32007
Container(s):	158536		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1221					1000.00000	ug/mL
PCB-1221 Peak 1					1000.00000	ug/mL
PCB-1221 Peak 2					1000.00000	ug/mL
PCB-1221 Peak 3					1000.00000	ug/mL



Reagent ID: Ar 1268_00004

Type:	ASTD	Expiration Date:	12/31/2019
Description:	Aroclor 1268 Standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A097795
Creation Date:	06/09/2014	Vendor Cat #:	32410
Container(s):	381748		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1268					1000.00000	ug/mL
PCB-1268 Peak 1					1000.00000	ug/mL
PCB-1268 Peak 2					1000.00000	ug/mL
PCB-1268 Peak 3					1000.00000	ug/mL
PCB-1268 Peak 4					1000.00000	ug/mL
PCB-1268 Peak 5					1000.00000	ug/mL

**Reagent ID: Ar 1242_00002**

Type:	ASTD	Expiration Date:	11/30/2018
Description:	Aroclor 1242 Standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A090182
Creation Date:	07/08/2013	Vendor Cat #:	32009
Container(s):	158544		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1242					1000.00000	ug/mL
PCB-1242 Peak 1					1000.00000	ug/mL
PCB-1242 Peak 2					1000.00000	ug/mL
PCB-1242 Peak 3					1000.00000	ug/mL
PCB-1242 Peak 4					1000.00000	ug/mL
PCB-1242 Peak 5					1000.00000	ug/mL

**Reagent ID: Ar 1232_00002**

Type:	ASTD	Expiration Date:	11/30/2018
Description:	Aroclor 1232	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A090290
Creation Date:	07/08/2013	Vendor Cat #:	32008
Container(s):	158538		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1232					1000.00000	ug/mL
PCB-1232 Peak 1					1000.00000	ug/mL
PCB-1232 Peak 2					1000.00000	ug/mL
PCB-1232 Peak 3					1000.00000	ug/mL
PCB-1232 Peak 4					1000.00000	ug/mL
PCB-1232 Peak 5					1000.00000	ug/mL



Reagent ID: Ar 1262_00002

Type:	ASTD	Expiration Date:	06/30/2019
Description:	Aroclor 1262 Standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A094073
Creation Date:	07/08/2013	Vendor Cat #:	32409
Container(s):	158599		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1262					1000.00000	ug/mL
PCB-1262 Peak 1					1000.00000	ug/mL
PCB-1262 Peak 2					1000.00000	ug/mL
PCB-1262 Peak 3					1000.00000	ug/mL
PCB-1262 Peak 4					1000.00000	ug/mL
PCB-1262 Peak 5					1000.00000	ug/mL

**Reagent ID: Ar 1248_00002**

Type:	ASTD	Expiration Date:	04/30/2019
Description:	Aroclor 1248 Standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hunt, Joseph B
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A092864
Creation Date:	07/08/2013	Vendor Cat #:	32010
Container(s):	158550		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1248					1000.00000	ug/mL
PCB-1248 Peak 1					1000.00000	ug/mL
PCB-1248 Peak 2					1000.00000	ug/mL
PCB-1248 Peak 3					1000.00000	ug/mL
PCB-1248 Peak 4					1000.00000	ug/mL
PCB-1248 Peak 5					1000.00000	ug/mL

**Title: PESTICIDE GAS CHROMATOGRAPHIC ANALYSIS
[SW-846 8081B; EPA 608]**

Approvals (Signature/Date):

 10/28/16
Dennis Koropka Date
Extracables Manager

 10/28/16
Michael Ridenhower Date
Health & Safety Manager / Coordinator

 10/28/16
Kristen Ely Date
Quality Assurance Manager

 10/28/16
Elaine Wild Date
Laboratory Director

This SOP was previously identified as SOP No. ST-GC-0016 Rev. 19

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes procedures to be used for the analysis of Pesticides by GC/ECD.
- 1.2 Sample preparation techniques are described in SOP ST-OP-0002.
- 1.3 This SOP is based on SW-846 Methods 8000B, 8000C, 8000D, 8081B and EPA Method 608.
- 1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.4.1 Additional compounds may be amendable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

- 2.1 Aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction. Solid samples are prepared using sonication. Waste dilution and wipes are extracted by autoshaker.
- 2.2 After the initial preparation step, the sample is introduced to the GC, equipped with capillary columns and dual Electron Capture Detectors (ECD). Concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. The internal standardization procedure is used.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Lower Limit of Quantitation – LLOQ. SOP refers to this as the reporting limit (RL). The LLOQ is verified annually. (See SOP ST-QA-0016 for details,

4.0 INTERFERENCES

- 4.1 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.
- 4.2 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620).
- 4.3 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results.
- 4.4 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and interferences to sample extracts. Strict attention to glassware cleaning and handling and demonstration of solvent purity will lead minimization of these interferences.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve

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- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
- 5.2.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.2.2 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- 5.2.3 The ECD contains a source of radiation.
- 5.3 PRIMARY MATERIALS USED
- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 GC/ECD system: The lab utilizes an Agilent GC 6890 dual micro ECD system with auto-sampler.
- 6.1.1 The GC column type, and instrument run conditions are posted in the maintenance log and reside in the Chemstation method.
- 6.1.2 Columns used: Restek Rtx-CLPesticides 30 meter, 0.53 mmID, 0.5 μ m df and Restek Rtx-CLPesticides2 30 meter, 0.53 mmID, 0.42 μ m df
- 6.2 Data System – Chemstation for acquisition and Chrom for data processing
- 6.3 Disposable pipettes

- 6.5 Clear vials and silicon crimp seals
- 6.6 Micro syringes- 10- μ L, 250- μ L, 500- μ L, 1000- μ L. Hamilton 1700 series
- 6.7 Volumetric flasks, Class A
- 6.8 Analytical Balance, capable of weighing ± 0.01 g
- 6.9 Instrument run conditions can be found in the front of the maintenance calendars.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 Pesticide stock standard solutions must be replaced after 6 months, once opened, or manufacturer's expiration date whichever is shorter. Pesticide calibration solutions must be refrigerated at ≤ 6 °C and protected from light. Intermediate and working standards must be replaced at least every six months, or the stock solutions expiration date, whichever is sooner. Additionally standards are discarded if comparison with check standards indicates a problem.
- 7.3 See reagent log for specific information regarding standards and reagents. See Appendix 1 and 2 for examples.
- 7.4 ICV standards, NIST traceable:
 - 7.4.1 The Pesticide ICV standard is a second source from the calibration standard.
 - 7.4.2 ICV standard is prepared and stored in the same way as calibration standards
- 7.5 Internal Standards
 - 7.5.1 An Internal Standard solution is prepared comprised of 1-Bromo-2-Nitrobenzene. The internal standard is added to all standards and extracts to result in 50ng on column.
- 7.6 Surrogate Standards
 - 7.6.1 Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards
- 7.7 Column Degradation Evaluation Mix (PEM Standard)
 - 7.7.1 A standard containing 4,4'-DDT and Endrin must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after 6 months, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. Refer to [Table 1](#) for details of the column degradation evaluation mix.
- 7.8 Gases for carrier and make-up: Hydrogen carrier, Nitrogen make-up

8.0 SAMPLE PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Water samples are unpreserved and stored at >0 and ≤ 6 °C.
- 8.3 Soil samples are refrigerated at >0 and ≤ 6 °C.

- 8.3 Soil samples are refrigerated at >0 and ≤ 6 °C.
- 8.4 The extraction holding time for Pesticides analysis in waters is 7 days.
- 8.5 The extraction holding time for Pesticides analysis in soil/solid matrix is 14 days.
- 8.6 Extracts must be refrigerated at ≤ 6 °C and analyzed within 40 days of the end of the extraction.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD an LCS Duplicate (LCSD) is prepared and analyzed.

9.2 Method Blank (MB)

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 DI water is used as the blank matrix for water batches.
- 9.2.4 Sodium sulfate is used as the blank matrix for solid batches.
 - 9.2.4.1 South Carolina requires the use of Ottawa sand for the Method Blank

9.3 Laboratory Control Sample (LCS)

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.
- 9.3.3 DI water, spiked with the analytes of interest is used as the LCS for water batches.
- 9.3.4 Sodium sulfate, spiked with the analytes of interest is used as the LCS for solid batches
 - 9.3.4.1 South Carolina requires the use of Ottawa sand for the LCS solid matrix..

9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.4.2 For Method 608 a matrix spike must be performed at a frequency of one per ten samples.

9.5 Surrogate

- 9.5.1 A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.
- 9.5.2 Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.

9.6 Procedural Variations/ Nonconformance and Corrective Action

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

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10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Internal standard calibration is used.
- 10.2 Column Degradation Evaluation (PEM)
 - 10.2.1 The column evaluation mix must be injected before each initial calibration, the beginning of an analytical sequence and every subsequent 12 hours of continuous analysis.
 - 10.2.2 The degradation of DDT and Endrin must be calculated and each shown to be $\leq 15\%$ before calibration can proceed.
 - 10.2.3 If the breakdown of DDT and/or Endrin exceeds the limits given above, corrective action must be taken. This action may include:
 - 10.2.3.1 Replacement of the injection port liner or the glass wool.
 - 10.2.3.2 Cutting off a portion of the injection end of a capillary column.
 - 10.2.3.3 Replacing the GC column.
- 10.3 Initial Calibration
 - 10.3.1 Prepare a Pesticide standard at a minimum of five concentration levels. (Six points are required if a quadratic (second order) curve is used.) Add the internal standard mixture to result in 50ng injected on column. Both columns must pass calibration criteria. Single component analytes must be 80% resolved. The low level standard should be at or below the reporting limit (LLOQ). The other standards define the working range of the detector. Six points are required for quadratic (2nd order) curves
 - 10.3.2 A single point calibration for Technical Chlordane and Toxaphene is performed with every initial calibration for pattern recognition.
 - 10.3.2.1 Select 3-5 major peaks in the multi-component analyte pattern. The area of each peak is used to calculate the concentration for each peak. An average of the concentrations is used.
 - 10.3.2.2 If Toxaphene or Technical Chlordane is detected, sample will require re-analysis under a five point calibration for the analyte found. See Client Requirements Sheet to determine if this is needed.
 - 10.3.3 The analyst may include a full 5 point calibration for any of the multi-component analytes with the initial calibration.
 - 10.3.4 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., ECD replacement).
 - 10.3.5 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the corporate policy, "Selection of Calibration Points", CA-Q-P-003.
 - 10.3.6 Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount concentration of analyte in the calibration standard is defined as the response factor (RF) or calibration factor (CF).
- 10.4 SW 8081 criteria
 - 10.4.1 SW-846 chromatographic methods allow the use of both linear and non-linear models for the calibration data.
 - 10.4.1.1 The first way is to begin with the simplest approach, the linear model through the origin, and then progress through other options until the calibration acceptance criteria are met. The second way is to use technical knowledge of the detector response to the target compound to choose the calibration model.
 - 10.4.1.2 The option for non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance.
 - 10.4.2 Linear calibration using the average response factor

- 10.4.2.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 20\%$ for each target analyte.
- 10.4.2.2 If the %RSDs in the initial calibration is $> 20\%$, then calibration using a linear regression may be employed.
- 10.4.3 Linear calibration using a least squares regression
- 10.4.3.1 The intercept of the curve at zero response must be less than $\pm$ the reporting limit for the analyte.
- 10.4.3.2 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990 .
- 10.4.3.3 Linear calibration using a least squares regression, forcing thru zero
- 10.4.3.3.1 Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 *before* the regression is calculated, thereby setting the bias to favor the low end of the calibration range by “pivoting” the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression through zero for some calibrations.
- 10.4.3.3.2 Curve must still meet criteria in 10.4.3.1 and 10.4.3.2.
- 10.4.3.3.3 For samples requiring adherence to SW846 Method 8000B and 8000D (i.e. South Carolina compliance work), forcing through zero is NOT allowed.
- 10.4.3.4 Linear calibration using a least squares regression, weighting of data points
- 10.4.3.4.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. For this reason it may be preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/x^2$ weighting) to improve accuracy at the low end of the curve.
- 10.4.3.4.2 Curve must still meet criteria in 10.4.3.1 and 10.4.3.2.
- 10.4.4 Non-linear calibration
- 10.4.4.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches have not met the acceptance criteria, a non-linear calibration model may be employed. Non linear calibration requires 6 points.
- 10.4.4.1.1 It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance. Thus, non-linear calibrations are not to be employed for analytes shown to consistently exhibit linear calibration for the analytes of interest.
- 10.4.4.2 The intercept of the curve at zero response must be less than $+$ or $-$ the reporting limit for the analyte.
- 10.4.4.3 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990 .
- 10.4.4.4 A quadratic calibration curve requires six standards.
- 10.4.4.5 Non-linear calibrations are NOT allowed for South Carolina compliance work.
- 10.4.5 **Acceptance criteria independent of calibration model**
- 10.4.5.1 Either of the procedures Percent Error or Relative Standard Error (RSE) may be used to determine calibration function acceptability for linear and non-linear curves. Both evaluate the difference between the measured and the true amounts or concentrations used to create the model.
- 10.4.5.2 $\%Error = \frac{x_i - x_i'}{x_i} \times 100$

- 10.4.5.3 Where x'_i = measured amount of the analyte at calibration level i in mass or concentration units and x_i = the true amount of the analyte at calibration level i in mass or concentration units.
- 10.4.5.4 % Error should be $\leq 30\%$. For some data uses $\leq 50\%$ may be acceptable for the lowest calibration point.
- 10.4.5.5 Calculation of Relative Standard Error:
- 10.4.5.6

$$RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

Where X_i = True amount of the calibration level i , in mass or concentration units

X'_i = measured amount of analyte in calibration level i , in mass or concentration units

p = number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3,)

n = number of calibration points

- 10.4.5.7 RSE acceptance criteria for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If not defined in the method, use $\pm 20\%$

10.5 608 Criteria

- 10.5.1 Method 608 only requires a 3-point calibration.
- 10.5.1.1 We routinely perform a 5-point calibration; however, 2 points may be removed from the curve if necessary to meet 608 calibration criteria.
- 10.5.1.2 Refer to the corporate policy, "Selection of Calibration Points", CA-Q-P-003.
- 10.5.2 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $< 10\%$.
- 10.5.3 If the %RSDs in the initial calibration is $\geq 10\%$, then calibration using a linear regression may be employed. See section 10.4.3 for criteria.

10.6 Initial Calibration Verification (ICV)

- 10.6.1 An initial calibration verification standard must be a different standard source than the one used for the initial calibration.
- 10.6.2 An ICV must be performed with every initial calibration.
- 10.6.3 The ICV performance must be within $\pm 20\%$ D for Method 8081B and $\pm 15\%$ D for Method 608.
- 10.6.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.7 Continuing Calibration Verification (CCV)

- 10.7.1 A CCV may be the same source or second source as the calibration.
- 10.7.2 Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run through the analysis of a mid-level calibration standard.

- 10.7.3 SW 8081 criteria
 - 10.7.3.1 The calibration verification is acceptable if the %D for each single component pesticide is $\leq 20\%$.
 - 10.7.3.2 The same criteria will be used if Technical Chlordane or Toxaphene verifications are performed.
 - 10.7.3.3 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed. If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration).
- 10.7.4 608 criteria
 - 10.7.4.1 At the beginning of each 24-hour long (or less) analytical sequence, demonstrate that the instrument calibration is still within the linear range established by the initial calibration through the analysis of mid-level standards of each compound of interest.
 - 10.7.4.2 If the analyzed concentration of each analyte is within $\pm 15\%$ of the true value, the daily calibration check is successful, and samples may be analyzed until 24-hours have elapsed from the injection of the first daily calibration standard.
- 10.8 Retention Time (RT) Windows
 - 10.8.1 Retention Time (RT) windows must be determined for all analytes.
 - 10.8.2 Establishing RT windows
 - 10.8.3 Make an injection of all analytes of interest each day over a three day period. Calculate the mean and the standard deviation of the three retention times for each analyte.
 - 10.8.3.1 If three days of analytical data are not available, use a default RT window of ± 0.01 minutes. At the end of the batch evaluate all CCVs and LCS in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.
 - 10.8.3.2 Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 min, in which case the window is set at ± 0.01 min. For example, if the maximum RT deviation for a specific analyte is 0.008 min, then the RT window is set at ± 0.012 min.
 - NOTE:** For the multi-component analytes, Toxaphene and Technical Chlordane, the maximum deviation must be evaluated for each of the 3 to 6 major peaks used for sample calculations
 - 10.8.4 The center of the retention time window is the retention time from the CCV performed at the beginning of the analytical run. For samples run during the same shift as the initial calibration, use the retention time of the mid-point standard from the initial calibration. Some clients may have specific requirements regarding the updating of RT windows. Review the Client Requirement Memo for instructions.
 - 10.8.5 Retention time windows for analytes of interest must not overlap
 - 10.8.6 Method 8000B requires a new retention time window study be performed annually or when the analytical column from a new vendor or different stationary phase is used. 8000C and 8000D also requires a new study when the column is clipped during maintenance.
 - 10.8.6.1 Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.
- 10.9 Ongoing evaluation of retention time windows
 - 10.9.1 Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day. All analytes for all subsequent CCVs, LCS and

matrix spikes must fall within the retention time window (except as discussed below)

- 10.9.1.1 Matrix spike analytes may fall outside the retention time window if there is a large non-target peak coeluting with the analyte in the matrix spike
- 10.9.2 If any analytes fall outside the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.
- 10.9.3 Retention time windows should be reliably narrower than ± 0.03 min. If RT windows wider than this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained.
- 10.9.4 Retention Time Criteria
 - 10.9.4.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established.
- 10.10 Method Detection Limit Studies
 - 10.10.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. The study must encompass both columns. See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs.

11.0 PROCEDURE

- 11.1 Allow standards, samples and sample extracts to reach ambient temperature before analysis
- 11.2 Add Internal Standard mixture to the extracts to result on 50ng injected on column.
- 11.3 All analysis conditions and injection volumes for samples must be the same as for the calibration standards.
- 11.4 Sample Introduction
 - 11.4.1 Semi-volatile analytes are introduced by direct injection of the extract. Samples, standards and QC must be introduced using the same procedure.
- 11.5 Perform all qualitative and quantitative measurements. When the standards and extracts are not being used, refrigerate them at $\leq 6^{\circ}\text{C}$, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 Internal Standard Calculations

Definitions:

Response (target) = the response (either area or height) of the target compound

IS= Internal Standard or Isotope Dilution Analyte

M1= 1st Degree Coefficient

M2= 2nd Degree Coefficient

B= Constant

RelResponse= Relative Response

Internal Standard:

Relative Response=

$$\frac{\text{Response(target)} * \text{IS(Concentration)}}{\text{IS(Response)}}$$

Average:

$$\text{Concentration(target)} = \frac{\text{Relative Response}}{M1}$$

Linear

$$\text{Concentration(target)} = \frac{\text{Relative Response} - B}{M1}$$

12.3 Breakdown calculations:

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD+DDE)}}{\text{sum of all peak areas (DDT+DDE+DDD)}} \times 100$$

$$\% \text{ breakdown of endrin} = \frac{\text{sum of degradation peak areas (aldehyde+ketone)}}{\text{sum of all peak areas (endrin+aldehyde+ketone)}} \times 100$$

12.4 See TALS software for additional calculations.

12.5 Manual Integrations

12.5.1 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. See TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices. Manual integrations are denoted with a "M" flag on the Target quantitation report.

12.6 Dilutions

12.6.1 If the concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed.

12.6.2 A dilution should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the client specific project requirements.

12.7 Carryover

12.7.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.

12.7.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

12.7.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, carryover may have occurred.

12.7.1.2.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

12.7.1.3 If the chromatographic profile resembles the previous sample, the results are questionable.

12.7.1.3.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

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- 12.8.1 For method 8000C, report the lower result of the two columns, unless the Client SOW requires that the higher result be reported. See Client Requirements Sheet for determination. Method 8000B requires reporting the higher of the two columns.
- 12.8.1.1 For non-detect (ND) results, report from the A channel, unless there is evidence of chromatographic interference in the A channel's performance.
- 12.8.1.2 If one result is significantly higher (e.g., > 40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the lower result for 8000C and the higher result for 8000B. The data user should be advised of the disparity between the results on the two columns.
- 12.8.1.3 Use the higher result if there is obvious chromatographic interference on the column with the lower result.
- 12.8.1.3.1 If the CCV performance on one of the two channels is outside acceptance criteria due to confirmed matrix interference, report sample data from the column with acceptable performance, irrespective of it being the higher or lower result.
- 12.8.2 The QC should be reported from the column that reflects the column used for the majority of the samples associated with the QC.
- 12.8.3 The surrogate should be reported from the column that reflects the column used for the majority of the analytes associated with a sample.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.
- 13.2 Method Blank
- 13.2.1 Acceptance Criteria:
- 13.2.1.1 No target analytes may be present in the method blank above the reporting limit. SW846 Method 8000D Revision 5 requires that the Method Blank be less than ½ the Reporting Limit (aka LLOQ).
- 13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements sheet. If not, perform necessary corrective action and re-analyse the associated samples.
- 13.2.1.3 The method blank must have acceptable surrogate recoveries.
- 13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:
- 13.2.2.1 Method Blank Contamination – Blank contamination above the RL (>½ RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL or greater than 10 times the amount detected in the method blank.
- 13.2.2.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.
- 13.3 Laboratory Control Sample (LCS)
- 13.3.1 Acceptance Criteria:
- 13.3.1.1 All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

- 13.3.1.1.1 For long analyte spike list, marginal exceedances (ME) are allowed as follows:
- 13.3.1.1.2 Marginal exceedance is not allowed for South Carolina compliance work, LCS recovery must fall within 70 -130% of the true value or corrective action taken and associated samples re-analyzed.)
- 13.3.1.1.3 < 11 analytes in LCS, no analytes allowed in ME of the LCS control limit.
- 13.3.1.1.4 11 – 30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit.
- 13.3.1.1.5 31 – 50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit.
- 13.3.1.1.6 51 – 70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit.
- 13.3.1.1.7 71 – 90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit.
- 13.3.1.1.8 > 90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit.
- 13.3.1.1.9 No LCS recoveries may be outside the Marginal Exceedance limit.
- 13.3.1.1.10 Marginal exceedances must be random. If the same LCS analyte exceeds the control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.
- 13.3.1.1.11 The LCS should have acceptable surrogate recoveries.
- 13.3.2 Corrective Action for LCS not meeting acceptance criteria:
 - 13.3.2.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM
 - 13.3.2.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.
 - 13.3.2.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.
 - 13.3.2.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM
- 13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
 - 13.4.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD)
 - 13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:
 - 13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.
- 13.5 Surrogate
 - 13.5.1 All Surrogates should be within established control limits for accuracy (%Recovery).
 - 13.5.2 Corrective Action for Surrogate not meeting acceptance criteria:
 - 13.5.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.
- 13.6 Internal Standards

- 13.6.1 Acceptance criteria: the ISTD must elute within 30 seconds and have a response of 50% to +100% as compared to the most recent (preceding) calibration standard or corrective action must be taken.
 - 13.6.1.1 High internal standard recovery indicates a low bias to the analytical results. Instrument maintenance, if required is done and the affected samples are re-analyzed. If internal standards are outside criteria on the re-analysis, a matrix interference is suspected and the data is reported with an NCM.
 - 13.6.1.2 Low internal standard recovery indicates the potential for high bias to analytical results. Samples with positive hits above the reporting limit for analytes associated with the poor ISTD require re-analysis. Instrument maintenance is done, if required. If ISTD recovery are outside criteria in the reanalysis, a matrix interference is suspected and data is reported with an NCM.
- 13.7 Sample result evaluation
 - 13.7.1 Dilutions
 - 13.7.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
 - 13.7.1.2 Dilution: Sample – An NCM is written to document the reason for the dilution.
 - 13.7.1.3 Dilution: Surrogate(s) and/or Spike(s) diluted out – Dilution: Surrogate(s) and/or spike(s) diluted out – An NCM is written to document the reason for the dilution
 - 13.7.2 Carryover
 - 13.7.2.1 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be demonstrated to not have occurred.
 - 13.7.2.1.1 The carryover affected analyses are not reported, unless specifically requested by the client or there was insufficient extract/sample remaining to reanalyze.
 - 13.7.3 Insufficient Sample
 - 13.7.3.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative. An NCM is written to document the insufficient volume..

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
- 16.2.1 The following waste streams are produced when this method is carried out.
- 16.2.1.1 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".
- 16.2.1.2 Vials containing sample extract will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "C".

17.0 REFERENCES

- 17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Methods 8000B, 8000C, and 8000D.
- 17.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8081B.
- 17.3 USEPA Wastewater Method 608.
- 17.4 TestAmerica Quality Assurance Manual (ST-QAM), current revision
- 17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.6 TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices
- 17.7 TestAmerica Policy CA-Q-P-003, Selection of Calibration Points
- 17.8 Associated SOPs, current revisions;
- 17.8.1 ST-OP-0001, Labware Preparation for Organic Analysis
- 17.8.2 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Water and Soils, Based on SW-846 3500 Series, 3600 Series, and 600 Series
- 17.8.3 ST-PM-0002, Sample Receipt and Chain of Custody
- 17.8.4 ST-QA-0002, Standard and Reagent Preparation
- 17.8.5 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."
- 17.8.6 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
- 17.8.7 ST-QA-0016, IDL/MDL Determination
- 17.8.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.
 - 18.1.1 The Method Blank MUST be below the RL for South Carolina compliance work.
- 18.2 The surrogate calibration curve is calculated from last curved that is run before samples are injected.
- 18.3 SW.8846 requires that new retention time windows be established if a GC column has been shortened during maintenance. Given the matrices of the sample the laboratory receives, and the number of times the GC column may require clipping, TestAmerica St. Louis does not perform a RT study after clipping a column. RT studies done by the laboratory show that, historically, RT windows have not been greater than the method allowed 0.03 minutes. The lab defaults to a 0.03 minute RT window as allowed by the method.
- 18.4 Method 608 only requires a 3 point calibration. The laboratory routinely performs a 5 point calibration.

19.0 CHANGES TO PREVIOUS REVISION

- 19.1 Updated section 4.2 by removing Gel Permeation Chromatography (Method 3640) and Sulfur cleanup (Method 3660).
- 19.2 Rev 11:
 - 19.2.1 Annual Review, No Changes.
- 19.3 Rev 12:
 - 19.3.1 Annual Review, No Changes.
- 19.4 Rev 13:
 - 19.4.1 Added new equipment and supplies to section 6.0.
 - 19.4.2 Updated information regarding reagent log in section 7.0.
 - 19.4.3 Updated instructions for initial calibrations in section 10.3.
 - 19.4.4 Updated non-linear calibration point requirements in section 10.4.
 - 19.4.5 Updated requirements for performing RT window studies.
- 19.5 Rev 14:
 - 19.5.1 Added new safety concerns in section 5.2
 - 19.5.2 Updated location of where GC column type information can be found in section 6.0
 - 19.5.3 Added software information to Section 6
 - 19.5.4 Added method blank and LCS composition to Section 9..
 - 19.5.5 Updated initial calibration peak calculations in section 10.3.
 - 19.5.6 Updated quadratic calibration curve requirements in section 10.4.4.
 - 19.5.7 Removed references to QuantIMS and Clouseau – replaced with LIMS.
 - 19.5.8 Added reporting limit calculations to Section 12.
 - 19.5.9 Added specific corrective actions to Section 13.

- 19.6 Rev 15 (06/02/14)
 - 19.6.1 Added location of instrument run conditions in section 6.9
 - 19.6.2 Updated processing software in section 6.2
 - 19.6.3 Updated information on PEM standard in section 7.6.1
 - 19.6.4 Corrected requirements for breakdown in section 10.2.2
 - 19.6.5 Corrected storage requirements for standard in section 11.4
 - 19.6.6 Updated software for calculations in section 12.3
 - 19.6.7 Updated information for surrogate calibration in section 18.2
 - 19.6.8 Updated Table 1
 - 19.6.9 Updated Initial Calibration Sequence
- 19.7 Revision 16 (9/16/14)
 - 19.7.1 Section 10.2 removed the incorrect reference to the location of the breakdown calculation
 - 19.7.2 Section 10.4 updated to note that non-linear calibrations are not allowed for South Carolina compliance samples
 - 19.7.3 Section 18 updated to list requirements for South Carolina regarding Method Blank acceptability
- 19.8 Revision 17 (10/31/14)
 - 19.8.1 Calculations for breakdown products (DDT and endrin) added to Section 12.
 - 19.8.2 Combined this SOP with requirements for South Carolina from SOP ST-GC-0016SC Rev 1 and retired that SOP.
- 19.9 Revision 18 (12/1/14) additional information for South Carolina added:
 - 19.9.1 Columns used added to Section 6
 - 19.9.2 MDL information added to Section 10CS specific limits added to Section 13
 - 19.9.3 Standard and reagent information updated/added to Appendix 1. Appendix 2 added for preparation of working standards and calibration levels.
- 19.10 Revision 19 (12/3/15)
 - 19.10.1 Changed SOP to reflect the use of internal standard calibration. Removed references to external standard calibration.
 - 19.10.2 Updated calibration criteria for EPA method 608.
 - 19.10.3 Added resolution requirement to Initial Calibration Section.
- 19.11 Revision 20 (10/28/16)
 - 19.11.1 Added references to SW 846 Method 8000D
 - 19.11.2 Add references to the LLOQ (Reporting limit)
 - 19.11.3 Added explanations and calculations for % Error and Relative Standard Error.
 - 19.11.4 Updated Section on establishing and evaluating Retention Time Windows.

Table 1	
Column Degradation Evaluation Mix	
Component	Concentration (ug/L)
4,4'-DDT	100.000
Endrin	50.000

Analytical Sequence

Initial Calibration

Conditioning Standard	
Hexane Blank	
Instrument Blank	
Breakdown Mix (PEM)	
Individual mix	All levels
Individual mix ICV	
Technical Chlordane	All levels
Technical Chlordane ICV	
Toxaphene	All levels
Toxaphene ICV	
2,4'-DD*/HCB Mix (if needed)	All levels
2,4'-DD*/HCB Mix ICV (if needed)	
Sample injections (maximum 10)	
Continuing Calibration Verification (CCV)	Mid level (for needed target analytes)
Sample injections (maximum 10)	
Continuing Calibration Verification (CCV)	Mid level (for needed target analytes)

Breakdown check (beginning of an analytical run and subsequently every 12 hours of continuous analysis)

Appendix 1



Reagent ID: 8081tox_00016

Type:	ASTD	Expiration Date:	01/02/2015
Description:	Toxaphene	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hardin, Charles E
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0101579
Creation Date:	06/11/2014	Vendor Cat #:	32071
Container(s):	395834		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Toxaphene					5000.00000	ug/mL
Toxaphene Peak 1					5000.00000	ug/mL
Toxaphene Peak 2					5000.00000	ug/mL
Toxaphene Peak 3					5000.00000	ug/mL
Toxaphene Peak 4					5000.00000	ug/mL
Toxaphene Peak 5					5000.00000	ug/mL

**Reagent ID: 8081mixab3SEC_00002**

Type:	ASTD	Expiration Date:	01/02/2015
Description:	individual mix	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hardin, Charles E
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A097850
Creation Date:	06/11/2014	Vendor Cat #:	32415
Container(s):	400232		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
4,4'-DDD					2000.00000	ug/mL
4,4'-DDE					2000.00000	ug/mL
4,4'-DDT					2000.00000	ug/mL
Aldrin					2000.00000	ug/mL
alpha-BHC					2000.00000	ug/mL
alpha-Chlordane					2000.00000	ug/mL
beta-BHC					2000.00000	ug/mL
delta-BHC					2000.00000	ug/mL
Dieldrin					2000.00000	ug/mL
Endosulfan I					2000.00000	ug/mL
Endosulfan II					2000.00000	ug/mL
Endosulfan sulfate					2000.00000	ug/mL
Endrin					2000.00000	ug/mL
Endrin aldehyde					2000.00000	ug/mL
Endrin ketone					2000.00000	ug/mL
gamma-BHC (Lindane)					2000.00000	ug/mL
gamma-Chlordane					2000.00000	ug/mL
Heptachlor					2000.00000	ug/mL
Heptachlor epoxide					2000.00000	ug/mL
Methoxychlor					2000.00000	ug/mL



Reagent ID: 8081mixab3_00002

Type:	ASTD	Expiration Date:	01/02/2015
Description:	individual mix	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hardin, Charles E
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0101061
Creation Date:	06/11/2014	Vendor Cat #:	32415
Container(s):	400231		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
4,4'-DDD					2000.00000	ug/mL
4,4'-DDE					2000.00000	ug/mL
4,4'-DDT					2000.00000	ug/mL
Aldrin					2000.00000	ug/mL
alpha-BHC					2000.00000	ug/mL
alpha-Chlordane					2000.00000	ug/mL
beta-BHC					2000.00000	ug/mL
delta-BHC					2000.00000	ug/mL
Dieldrin					2000.00000	ug/mL
Endosulfan I					2000.00000	ug/mL
Endosulfan II					2000.00000	ug/mL
Endosulfan sulfate					2000.00000	ug/mL
Endrin					2000.00000	ug/mL
Endrin aldehyde					2000.00000	ug/mL
Endrin ketone					2000.00000	ug/mL
gamma-BHC (Lindane)					2000.00000	ug/mL
gamma-Chlordane					2000.00000	ug/mL
Heptachlor					2000.00000	ug/mL
Heptachlor epoxide					2000.00000	ug/mL
Methoxychlor					2000.00000	ug/mL

**Reagent ID: 8081surr_00017**

Type:	ASTD	Expiration Date:	11/30/2020
Description:	Pest Surr	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hardin, Charles E
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0105135
Creation Date:	10/30/2014	Vendor Cat #:	32000
Container(s):	490048		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
DCB Decachlorobiphenyl (Surr)					200.00000	ug/mL
Tetrachloro-m-xylene					200.00000	ug/mL

**Reagent ID: 8081techchlor_00014**

Type:	ASTD	Expiration Date:	03/31/2017
Description:	Technical Chlordane sec	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hardin, Charles E
Storage Location:	GC and HPLC Standards Stora	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A094164
Creation Date:	04/30/2013	Vendor Cat #:	32072.sec
Container(s):	146584		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Technical Chlordane					5000.00000	ug/mL
Technical Chlordane Peak 1					5000.00000	ug/mL
Technical Chlordane Peak 2					5000.00000	ug/mL
Technical Chlordane Peak 3					5000.00000	ug/mL
Technical Chlordane Peak 4					5000.00000	ug/mL
Technical Chlordane Peak 5					5000.00000	ug/mL

APPENDIX 2

Standards:

Surrogate Stock – 200 ug/ml

Individuals Stock – 2000 ug/ml

Technical Chlordane Stock – 5000 ug/ml

Toxaphene Stock – 5000 ug/ml

Intermediates:

Surrogate Intermediate – 1 ml stock Q.S. to 10 ml with hexane = 20 ug/ml

Individuals Intermediate – 100 ul of stock Q.S. to 10 ml with hexane = 20 ug/ml

Technical Chlordane Intermediate - 1 ml of stock Q.S. to 5 ml with hexane = 1000 ug/ml

Toxaphene Intermediate – 1 ml of stock Q.S. to 5 ml with hexane = 1000 ug/ml

Working standards:

Individuals working – 250 ul of Individuals Intermediate + 250 ul of Surrogate Intermediate Q.S. with 50 ml hexane = 0.1 ug/ml Individuals / 0.1 ug/ml Surrogate

Technical Chlordane working – 50 ul of Technical Chlordane Intermediate Q.S. with 50 ml hexane = 1 ug/ml

Toxaphene working – 200 ul of Toxaphene Intermediate Q.S. with 50 ml hexane = 4 ug/ml

Calibration:

Toxaphene

Calibration point	Dilution of Working Std.	Concentration (ug/ml)
1	20x	0.200
2	10x	0.400
3	4x	1.000
4	2x	2.000
5	1x	4.000
ICV (second source)	4x	1.000

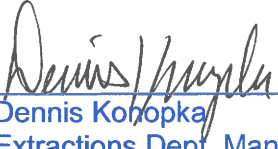
Technical Chlordane

Calibration point	Dilution of Working Std.	Concentration (ug/ml)
1	20x	0.050
2	10x	0.100
3	4x	0.250
4	2x	0.500
5	1x	1.000
ICV (second source)	4x	0.250

Individuals and surrogate

Calibration point	Dilution of Working Std.	Concentration (ug/ml)
1	20x	0.005
2	10x	0.010
3	4x	0.025
4	2x	0.050
5	1x	0.100
ICV (second source)	4x	0.025

**Title: HERBICIDE GAS CHROMATOGRAPHIC ANALYSIS
[SW-846 8151A]**

Approvals (Signature/Date):	
 Dennis Kozopka Extractions Dept. Manager	<u>10/31/16</u> Date
 Michael Ridenhower Health & Safety Manager / Coordinator	<u>10/31/16</u> Date
 Kristen Ely Quality Assurance Manager	<u>10/31/16</u> Date
 Elaine Wild Laboratory Director	<u>10/31/16</u> Date

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes procedures to be used for the analysis of Herbicides by GC/ECD.
- 1.2 Sample preparation techniques are described in SOP ST-OP-0007.
- 1.3 This SOP is based on EPA SW-846 Method 8000B, 8000C, 8000D and 8151A.
- 1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.4.1. Additional compounds may be amendable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

- 2.1 Aqueous samples are separatory funnel extracted in diethylether, hydrolyzed and esterified with TMS diazomethane. Solid samples are sonicator extracted in diethylether, hydrolyzed and esterified with TMS diazomethane. Waste samples are extracted using the waste dilution procedure followed by esterification.
- 2.2 After the initial preparation step, the sample is introduced to the instrument equipped with capillary columns and dual Electron Capture Detectors (ECD). Concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. The external standardization procedure is used.
- 2.3 Herbicides are spiked and prepared as methyl acid and analyzed as their methyl esters.

3.0 DEFINITIONS

- 3.1 See the TestAmerica ST. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers,
- 3.2 Lower Limit of Quantitation – LLOQ. SOP refers to this as the reporting limit (RL) The LLOQ is verified at least annually (see SOP ST-QA-0016 for details).

4.0 INTERFERENCE

- 4.1 Organic acids, especially chlorinated acids, interfere with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure. Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the ECD. The hydrolysis may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids.
- 4.2 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660).
- 4.3 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
- 5.2.1. The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.2.2. There are areas of high voltage in both the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- 5.3 PRIMARY MATERIALS USED
- 5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 GC/ECD system: The lab utilizes a Hewlett Packard GC 5890 dual ECD system and an Agilent GC 6890 dual micro ECD system
- 6.1.1. The GC column type and instrument run conditions are posted in the maintenance calendar and reside in the Chemstation method.
- 6.1.2. Columns used: Restek RTX Pest 1 and Pest 2 with guard.
- 6.2 Data System – Chemstation for acquisition and Chrom™ for data processing.
- 6.3 Amber vials. Crimp top seals.

- 6.4 Disposal pipettes
- 6.5 Micro syringes – 10- μ L, 250- μ L, 500- μ L, 1000- μ L Hamilton 1700 series
- 6.6 Volumetric flasks, Class A
- 6.7 Analytical Balance, capable of weighing ± 0.01 grams.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 See recipes for standards in the TALS Reagent module and in SOP ST-OP-0007.
- 7.3 Herbicide stock standard solutions must be replaced after 6 months or manufacturer's expiration date whichever is shorter. Calibration solutions must be refrigerated at $\leq 6^{\circ}\text{C}$ and protected from light. Intermediate and working standards must be replaced at least every six months, or the stock solutions expiration date, whichever is sooner. Additionally standards are discarded if comparison with check standards indicates a problem. Stock standard solutions of free acids degrade more quickly and should be replaced after two months, or sooner if comparison with check standards indicates a problem.
- 7.4 ICV standards, NIST traceable:
 - 7.4.1 The Herbicide ICV standard is a second source from the calibration standard.
 - 7.4.2 ICV standard is prepared and stored in the same way as calibration standards.
- 7.5 Standards used in calibration are the methyl ester forms, as opposed to the methyl acids used for spiking in the extraction process
 - 7.5.1 Method 8151 requires that the calibration standards be derivitized from the free acid form to the methyl ester form in the laboratory.
- 7.6 Derivatization of Standards
 - 7.6.1 Add 1mL of room temperature standard to a 10mL certified, calibrated or class A concentrator tip.
 - 7.6.2 Add 0.5mL of Ether.
 - 7.6.3 Bring up to 4mL with hexane and mix with a pipet.
 - 7.6.4 Add 0.5mL of Diazomethane and stir until mixture is yellow.
 - 7.6.4.1 Diazomethane should be kept in a refrigerated location.
 - 7.6.5 Let sit 1 hour.
 - 7.6.6 Bring up to 10mL with hexane.
 - 7.6.6 Add solid salicylic acid until it reaches the 3 mL mark of the tube.
 - 7.6.7 Let standard sit until bubbling stops and acid settles.
 - 7.6.8 Vial and label standard.
- 7.7 Gases for carrier and make-up: Hydrogen carrier, Nitrogen make-up

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Water samples are unpreserved and stored >0 to $\leq 6^{\circ}\text{C}$.
- 8.3 Soil samples are refrigerated >0 to $\leq 6^{\circ}\text{C}$.

- 8.4 Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid samples.
- 8.5 For TCLP leachates, the extraction is initiated within seven days of the completion of TCLP leach tumbling, excluding the filtration step.
- 8.6 Extracts must be refrigerated at $<6^{\circ}$ and analyzed within 40 days of the start of extraction.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1. A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
- 9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3. For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2 Method Blank (MB)

- 9.2.1. A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2. A method blank must be prepared with every sample batch.
- 9.2.3. DI water is used as the blank matrix for water batches.
- 9.2.4. Sodium sulfate is used as the blank matrix for solid batches. (Note: South Carolina requires the use of Ottawa sand for the Method Blank)

9.3 Laboratory Control Sample (LCS)

- 9.3.1. An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2. An LCS must be prepared with every sample batch.
- 9.3.3. DI water, spiked with the analytes of interest is used as the LCS for water batches.
- 9.3.4. Sodium sulfate, spiked with the analytes of interest is used as the LCS for solid batches (Note: South Carolina requires the use of spiked Ottawa sand for the LCS)

9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1. A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5 Surrogate

- 9.5.1. A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.
- 9.5.2. Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.

9.6 Procedural Variations/ Nonconformance and Corrective Action

- 9.6.1. Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

- 9.6.2. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 External standard calibration is used.
- 10.2 Calibrations standards requiring compliance with SW846 methods are to be derivatized from the free acid form to the methyl ester form in the laboratory.
- 10.2.1. If calibration is performed using herbicide acid calibration standards, the standards have to have been analyzed in the same manner as the samples. TestAmerica St. Louis uses derivative acids for the initial calibration. (Section 7.6)
- 10.3 Initial Calibration
- 10.3.1. Prepare a Herbicide standard at a minimum of five concentration levels (six points are required if a quadratic (second order) curve is used.). The low level standard should be at or below the reporting limit. The other standards define the working range of the detector.
- 10.3.2. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., ECD replacement).
- 10.3.3. Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
- 10.3.4. Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount concentration of analyte in the calibration standard is defined as the response factor (RF) or calibration factor (CF).
- 10.3.5. SW-846 chromatographic methods allow the use of both linear and non-linear models for the calibration data.
- 10.3.5.1 The first way is to begin with the simplest approach, the linear model through the origin, and then progress through other options until the calibration acceptance criteria are met. The second way is to use technical knowledge of the detector response to the target compound to choose the calibration model.
- 10.3.5.2 The option for non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance.
- 10.3.5.3 For SW846 Methods 8000B and 8000D, the curve must not be forced through zero.
- 10.3.6. **Linear calibration using the average response factor**
- 10.3.6.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 20\%$ for each target analyte.
- 10.3.6.2 If the %RSDs in the initial calibration is $> 20\%$, then calibration using a linear regression may be employed.
- 10.3.7. **Linear calibration using a least squares regression**
- 10.3.7.1 The intercept of the curve at zero response must be less than + or – the reporting limit for the analyte.
- 10.3.7.2 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990 .
- 10.3.7.3 **Linear calibration using a least squares regression, forcing thru zero(not allowed for SW846 8000B, 8000D)**
- 10.3.7.3.1 Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 *before* the regression is calculated, thereby setting the bias to favor the low end of the

calibration range by “pivoting” the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression through zero for some calibrations.

10.3.7.3.2 Curve must still meet criteria in 10.4.3.1 and 10.4.3.2.

10.3.7.4 **Linear calibration using a least squares regression, weighting of data points**

10.3.7.4.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. For this reason it may be preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) to improve accuracy at the low end of the curve.

10.3.7.4.2 Curve must still meet criteria in 10.4.3.1 and 10.4.3.2.

10.3.8. **Non-linear calibration**

10.3.8.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches have not met the acceptance criteria, a non-linear calibration model may be employed. Non linear calibration requires 6 points.

10.3.8.1.1 It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance. Thus, non-linear calibrations are not to be employed for analytes shown to consistently exhibit linear calibration for the analytes of interest.

10.3.8.2 The intercept of the curve at zero response must be less than + or – the reporting limit for the analyte.

10.3.8.3 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990 .

10.3.8.4 A quadratic calibration curve requires six standards.

10.3.8.5 South Carolina does not allow the use of non-linear regression

10.4 Acceptance criteria independent of calibration model

10.4.1. Either of the procedures Percent Error or Relative Standard Error (RSE) may be used to determine calibration function acceptability for linear and non-linear curves. Both evaluate the difference between the measured and the true amounts or concentrations used to create the model.

$$10.4.1.1 \text{ \%Error} = \frac{x_i - x'_i}{x_i} \times 100$$

10.4.1.2 Where x'_i = measured amount of the analyte at calibration level i in mass or concentration units and x_i = the true amount of the analyte at calibration level i in mass or concentration units.

10.4.1.3 % Error should be $\leq 30\%$. For some data uses $\leq 50\%$ may be acceptable for the lowest calibration point.

10.4.1.4 Calculation of Relative Standard Error:

10.4.1.5

$$10.4.1.6 \quad RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

10.4.1.7 Where X_i = True amount of the calibration level i , in mass or concentration units10.4.1.8 X'_i = measured amount of analyte in calibration level i , in mass or concentration units10.4.1.9 p = number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3,)10.4.1.10 n = number of calibration points10.4.1.11 RSE acceptance criteria for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If not defined in the method, use $\pm 20\%$

10.5 Initial Calibration Verification (ICV)

10.5.1. An initial calibration verification standard must be a different standard source than the one used for the initial calibration.

10.5.2. An ICV must be performed with every initial calibration.

10.5.3. The ICV performance must be within $\pm 30\%$ D criteria.

10.5.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.6 Continuing Calibration Verification (CCV)

10.6.1. A CCV may be the same source or second source as the calibration.

10.6.2. Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run through the analysis of a mid-level calibration standard.

10.6.3. **SW846 8000C**: The CCV performance must be with $\pm 20\%$ D criteria.

10.6.3.1 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed. If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration)

10.6.3.2 **SW846 8000B**: The CCV performance must be with $\pm 15\%$ D criteria

10.7 Retention Time (RT) Windows

10.7.1. Retention Time (RT) windows must be determined for all analytes.

10.7.1.1 Establishing RT windows:

10.7.1.1.1 Make an injection of all analytes of interest each day over a three day period. Calculate the mean and standard deviation of the three retention times for each analyte.

10.7.1.1.2 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period or 0.03 minutes, whichever is greater. Historically, calibrations RT windows have not been greater than 0.03 minutes. Windows larger than 0.03 minutes are indicative of equipment issues.

10.7.1.1.3 The center of the retention time window is the retention time from the CCV performed at the beginning of the analytical run. For samples run during the same shift as the initial calibration, use the retention time of the mid-point standard from the initial calibration. Some clients may have specific requirements regarding the updating of RT windows. Review the Client Requirement Memo for instructions.

10.7.1.1.4 A new retention time window study is performed annually or when a new column is used.

10.7.1.1.4.1 Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

10.7.2. Retention Time Criteria

10.7.2.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established.

10.8 Method Detection Limit Studies

10.7.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. The study must encompass both columns. See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs

11.0 PROCEDURE

11.1 Allow standards, samples and sample extracts to reach ambient temperature before analysis.

11.2 All analysis conditions and injection volumes for samples must be the same as for the calibration standards.

11.3 Sample Introduction

11.3.1. Herbicide analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.4 Perform all qualitative and quantitative measurements. When the standards and extracts are not being used, refrigerate them $> 0^{\circ}\text{C}$ & $\leq 6^{\circ}\text{C}$, protected from light in screw cap vials equipped with un-pierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 External Standard Calculations

12.2.1 *Analyte Concentration ($\mu\text{g/L}$) in sample*

Concentration ($\mu\text{g/L}$):

$$[C] = \frac{A_x * V_t * D}{\overline{CF} * V_i * V_s}$$

Where:

$[C]$	=	Analyte Concentration in sample ($\mu\text{g/L}$)
A_x	=	Area of peak (response)
V_t	=	Total volume of extract (μL)
D	=	Dilution factor
$\overline{CF}$	=	Calibration factor ($\overline{RF}$ in target) – Response factor
V_i	=	Volume of extract injected (μL)
V_s	=	Volume of sample extracted

12.2.2 On column concentration

On Column Concentration ($\mu\text{g/mL}$):

$$[OC] = \frac{A_x}{\overline{CF}}$$

Where:

$[OC]$ = On Column Concentration [typically expressed in $\mu\text{g/mL}$ (ppm)]

Then substitute/derive

$$[C] = [OC] \left(\frac{V_t * D}{V_i * V_s} \right)$$

When on column concentration $[OC]$ is equal to the CAL-AMT (calibration amount) of the low level standard needed to support the reporting limit ($\mu\text{g/L}$) and we solve the equation for concentration ($\mu\text{g/L}$)

Then

$$[C] \equiv RL \equiv [OC] \left(\frac{V_t * D}{V_i * V_s} \right)$$

Where:

RL = Reporting Limit

12.2.3 See Target software for additional calculations.

12.3 Manual Integrations

12.3.1 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. See TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices. Manual integrations are denoted with a “M” flag on the Target quantitation report.

12.4 Dilutions

- 12.4.1 If the concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed.
- 12.4.2 A dilution should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the client specific project requirements.
- 12.5 Carryover
 - 12.5.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
 - 12.5.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.
 - 12.5.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, carryover may have occurred.
 - 12.5.1.2.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.
 - 12.5.1.3 If the chromatographic profile resembles the previous sample, the results are questionable.
 - 12.5.1.3.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.
- 12.6 Dual column quantitation
 - 12.6.1 8000B requires the reporting of the higher result of the two columns. For 8000C and 8000D, report the lower result of the two columns, unless the Client SOW requires that the higher result be reported. See Client Requirements Sheet for determination.
 - 12.6.1.1 For non-detect (ND) results, report from the A channel, unless there is evidence of chromatographic interference in the A channel's performance.
 - 12.6.1.2 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result for 8000B and the lower result for 8000C and 8000D. The data user should be advised of the disparity between the results on the two columns.
 - 12.6.1.3 Use the higher result if there is obvious chromatographic interference on the column with the lower result.
 - 12.6.1.4 If the CCV performance on one of the two channels is outside acceptance criteria due to confirmed matrix interference, report sample data from the column with acceptable performance, irrespective of it being the higher or lower result.
 - 12.6.2 The QC should be reported from the column that reflects the column used for the majority of the samples associated with the QC.
 - 12.6.3 The surrogate should be reported from the column that reflects the column used for the majority of the analytes associated with a sample.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: STL-QA-0036.
- 13.1 Method Blank
 - 13.1.1 Acceptance Criteria:
 - 13.1.1.1 No target analytes may be present in the method blank above the reporting limit. SW846 Method 8000D requires that the Method Blank be less than ½ the Reporting Limit (aka LLOQ).

- 13.1.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above $\frac{1}{2}$ RL), will be noted on the client requirements sheet.
- 13.1.1.3 The Method Blank must have acceptable surrogate recoveries.
- 13.1.2 Corrective Action for Method Blanks not meeting acceptance criteria:
 - 13.1.2.1 Method Blank Contamination – Blank contamination above the RL ($> \frac{1}{2}$ RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are $<$ RL or greater than 10 times the amount detected in the method blank. South Carolina requires that the method blank be below the RL.
 - 13.1.2.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.
- 13.2 Laboratory Control Sample (LCS)
 - 13.2.1 Acceptance Criteria:
 - 13.2.1.1 All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).
 - 13.2.1.1.1 For long analyte spike list, marginal exceedances (ME) are allowed as follows:
 - 13.2.1.1.2 < 11 analytes in LCS, no analytes allowed in ME of the LCS control limit.
 - 13.2.1.1.3 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit.
 - 13.2.1.1.4 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit.
 - 13.2.1.1.5 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit.
 - 13.2.1.1.6 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit.
 - 13.2.1.1.7 > 90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit.
 - 13.2.1.1.8 No LCS recoveries may be outside the Marginal Exceedance limit.
 - 13.2.1.1.9 Marginal exceedances must be random. If the same LCS analyte exceeds the control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.
 - 13.2.2 Marginal Exceedance is not recognized by the state of South Carolina. LCS recovery must meet the criteria of 70 – 130% for all compounds.
 - 13.2.3 The LCS should have acceptable surrogate recoveries.
 - 13.2.4 Corrective Action for LCS not meeting acceptance criteria:
 - 13.2.4.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM
 - 13.2.4.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.
 - 13.2.4.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.
 - 13.2.4.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM
- 13.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

- 13.3.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).
- 13.3.2 Corrective Action for MS/MSD not meeting acceptance criteria:
 - 13.3.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.
- 13.4 Surrogate
 - 13.4.1 All Surrogates should be within established control limits for accuracy (%Recovery).
 - 13.4.2 Corrective Action for Surrogate not meeting acceptance criteria:
 - 13.4.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.
- 13.5 Sample result evaluation
 - 13.5.1 Dilutions
 - 13.5.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
 - 13.5.1.2 Dilution: Sample– An NCM is written to document the reason for the dilution.
 - 13.5.1.3 Dilution: Surrogate(s) and/or Spike(s) diluted out– Dilution: Surrogate(s) and/or spike(s) diluted out– An NCM is written to document the reason for the dilution
 - 13.5.2 Carryover
 - 13.5.2.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
 - 13.5.2.2 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.
 - 13.5.2.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.
- 13.6 Insufficient Sample
 - 13.6.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.
- 14.5 An annual MDL study is required for the State of South Carolina. See SOP ST-QA-0016 for determination of MDLs.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department...

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
 - 16.2.1 The following waste streams are produced when this method is carried out.
 - 16.2.1.1 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".
 - 16.2.1.2 Vials containing sample extract will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "C".

17.0 REFERENCES

- 17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8000B,, 8000C and 8000D..
- 17.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8151A
- 17.3 TestAmerica Quality Assurance Manual (ST-QAM), current revision
- 17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.5 TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices
- 17.6 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
- 17.7 Associated SOPs
 - 17.7.1 ST-OP-0001, Labware Preparation for Organic Analysis
 - 17.7.2 ST-OP-0007, Extraction and Cleanup of Herbicides from Water and Soils, Based on SW-846 8151A
 - 17.7.3 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.7.4 ST-QA-0002, Standard and Reagent Preparation
 - 17.7.5 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."
 - 17.7.6 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.7.7 ST-QA-0016, IDL/MDL Determination

17.7.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 MODIFICATIONS, CLARIFICATIONS TO THE REFERENCE METHOD

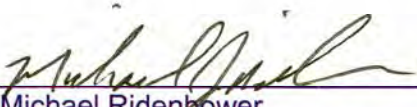
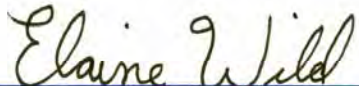
- 18.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.
- 18.1.1 For South Carolina compliance samples the method blank MUST be below the reporting limits for ALL analytes.
- 18.2 SW846 requires that new retention time windows be established if a GC column has been shortened during maintenance. Given the matrices of the sample the laboratory receives, and the number of times the GC column may require clipping, TestAmerica St. Louis does not perform a RT study after clipping a column. RT studies done by the laboratory show that, historically, RT windows have not been greater than the method allowed 0.03 minutes. The lab defaults to a 0.03 minute RT window as allowed by the method.

19.0 CHANGES TO PREVIOUS REVISION

- 19.1 Columns used added to Section 6.
- 19.2 Section 7.5 updated to include derivatization of free acids for SW846 compliance
- 19.3 Updated Section 10.3.8 to include South Carolina requirement that non-linear calibration is not to be used.
- 19.4 Updated Section 13.2 to disallow marginal exceedance for South Carolina LCS and added South Carolina LCS recovery limits.
- 19.5 Added South Carolina annual MDL requirement to Section 14.
- 19.6 Rev 10:
- 19.6.1 Annual Review, No Changes.
- 19.7 Rev 11:
- 19.7.1 Added section 7.6, instructions for the derivitization of standards.
- 19.7.2 Added statement regarding the use of derivitized acids for initial calibrations to section 10.2.1.
- 19.8 Rev 12:
- 19.8.1 Updated requirements for performing RT window studies.
- 19.9 Rev 13:
- 19.9.1 Removed references to Quantims and Clouseau – replaced with LIMS.
- 19.9.2 Added software information to Section 6.
- 19.9.3 Added composition of Method Blank and LCS to Section 9.
- 19.9.4 Added requirement for 6 points for non-linear curves to Section 10.
- 19.9.5 Added Reporting limit calculations to Section 12.
- 19.9.6 Added specific corrective actions to Section 13.
- 19.10 Rev 14: (4/21/14)
- 19.10.1 Updated extraction method of solid samples in Section 2.
- 19.10.2 Changed data system to Chrom in Section 6.
- 19.10.3 Updated location of instrument run conditions in Section 6.
- 19.10.4 Updated location of standard recipes in Section 7.
- 19.10.5 Updated retention time study requirements in Section 10.
- 19.11 Revision 15 (9/16/14)

- 19.11.1 Section 18 updated to include South Carolina requirements for the Method Blank.
- 19.12 Revision 16 (12/1/14)
 - 19.12.1 Updated Section 7 clarifying requirements for derivitization of standards.
 - 19.12.2 Noted in Section 10.7.3 that non-linear curves are not allowed for South Carolina compliance work.
 - 19.12.3 Added Section 10.7 documenting MDL study requirements.
 - 19.12.4 Added 8000B requirements for dual column result reporting to Section 12.6
 - 19.12.5 Added note to Section 13.1.2 that Method Blanks must be below the RL for South Carolina compliance work.
 - 19.12.6 Annual Review, No Changes (06/17/16)
- 19.13 Revision 17 (10/31/16)
 - 13.13.1 Added references to SW 846 Method 8000D
 - 13.13.2 Added definition and calculations for % Error and Relative Percent Difference.
 - 13.13.3 Added definition and verification requirement for the LLOQ (Reporting Limit)
 - 13.13.4 Updated sample and extract temperature requirements.

Title: HAZARDOUS WASTE MANAGEMENT PLAN

Approvals (Signature/Date):	
 Michael Ridenhower Health & Safety Manager / Coordinator	<u>9/10/13</u> Date
 Marti Ward Quality Assurance Manager	<u>9-10-13</u> Date
 Elaine Wild Laboratory Director	<u>9/10/13</u> Date

This SOP was previously identified as SOP No. ST-HS-0004 Rev. 15

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1.0 SCOPE AND APPLICATION

- 1.1 This procedure describes the Hazardous Waste Management Program used at the TestAmerica St. Louis Laboratory. The hazardous waste is managed in an occupationally safe, environmentally responsible, legally compliant, and economically efficient manner.
- 1.2 This procedure outlines all aspects of the Hazardous Waste Management Program, including, but not limited to, Collection and Accumulation of Hazardous Waste, Disposal of Samples after Analysis, Guidelines for Discharge to Sewer, Sampling and Neutralization of Hazardous Waste Accumulation Containers, Safety Inspection of 90-Day Storage Room (Main Waste Storage Area) and Hazardous Waste Spill Response., Hazardous Waste Manifesting, Use of Land Ban and Land Disposal Restriction (LDR) Forms, Reporting to Regulatory Agencies.

2.0 SUMMARY

- 2.1 The accumulation and disposal of all wastes will comply with all applicable federal, state and local regulations regarding hazardous waste. The Environmental Health and Safety Coordinator is responsible for defining waste streams, designating accumulation containers, and for processing waste in the main waste storage area.
- 2.2 The disposal of all wastes shall comply with all federal, state, and local regulations concerning hazardous waste disposal. Samples will be retained until disposal according to the Terms and Conditions established with each client. The project manager is responsible for identifying the samples that may be removed from the storage areas and collected for disposal. The project manager is also responsible for identifying the samples that must be returned to the client rather than accumulated and disposed of by the laboratory. The sample control department is responsible for removing the samples from the storage areas and segregating and disposing of the samples into the designated waste streams. The Sample Control Department will document the disposal of each sample via the Sample Tracking Utility (STU) in order to track each sample "Cradle-to-Grave".
- 2.3 The TestAmerica St. Louis laboratory, has defined 18 waste streams. These streams are generated in the individual laboratories and accumulated into satellite accumulation containers. Once filled, these containers are transported to the 90-day storage area where they are then sampled for analysis for their hazardous constituents before being disposed. The disposal of wastes at TestAmerica St. Louis is intended to satisfy all federal, state and local regulations regarding hazardous wastes.
- 2.4 Sampling and Neutralization of Hazardous Waste Accumulation
 - 2.4.1 Containers Wastes are generated in the individual laboratories and accumulated into satellite containers. The satellite containers are ultimately collected into drums, which are then analyzed for hazardous constituents. Those drums, which are shown to be non-hazardous after review of the analytical data, are then discharged to the sewer (after neutralization if needed) by the Environmental Health & Safety Coordinator or designee.
 - 2.4.2 No Chemical or waste used/generated in the laboratory is directly discharged to the sanitary sewer.
- 2.5 Safety Inspection of 90-Day Storage Room (Main Waste Storage Area)
- 2.6 Hazardous Waste Spill Response
- 2.7 Hazardous Waste Manifesting, Use of Land Ban and Land Disposal Restriction (LDR) Forms
 - 2.7.1 All shipments of hazardous wastes from TestAmerica St. Louis to EPA permitted TSDF's must adhere to DOT regulations.

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- 2.7.2 All shipments of hazardous waste must be manifested.
- 2.7.3 The Environmental Health & Safety Coordinator is responsible for filing the quarterly report with the Missouri Department of Natural Resources for Hazardous Waste Activity, the Biennial Report of Hazardous Waste Activity to the EPA, and any other reports that may be necessary to any other regulatory agency concerning the generation and disposal of hazardous waste.
 - 2.7.3.1 A quarterly Hazardous Waste Report Summary is required to be filed with the Missouri Department of Natural Resources.
 - 2.7.3.2 A Biennial Report is required to be filed with the U.S. EPA every even numbered year.
 - 2.7.3.3 An Exception report is required to be filed with the Missouri Department of Natural Resources when a completed manifest is not returned from the treatment, storage and disposal facility within forty-five (45) days after delivery of the waste. This is to be tracked by the EHSC.
 - 2.7.3.4 Other reports, such as the EPA PCB Activity Report, may be necessary depending on the type of waste generated.

3.0 DEFINITIONS

- 3.1 See the TestAmerica Quality Assurance Manual (QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 **Accumulation Time** - the period which hazardous wastes may be legally held on site prior to a transfer to a treatment, storage, or disposal facility (TSDF), (45 CFR 262.4).
- 3.3 **Disposal** - the shipment of and final disposition of hazardous waste at an EPA permitted disposal facility.
- 3.4 **Disposal Facility** - a facility or part of a facility at which hazardous waste is intentionally placed into, or on, any land or water, and at which wastes will remain after closure.
- 3.5 **Hazardous Waste** - hazardous waste as it is defined in 40 CFR 261.3.
- 3.6 **Hazardous Waste Management** - the orderly process of minimization, source separation, collection, accumulation, storage, transportation, treatment and disposal of hazardous waste.
- 3.7 **Satellite Accumulation** - the collection of hazardous waste at or near a site of generation that is under the direct control of the generator.
- 3.8 **Sample** - solid waste, water, soil or air which is collected for the sole purpose of testing to determine its characteristics or composition.
- 3.9 **Solid Waste** - solid, liquid, semi-solid or contained gaseous material which is discarded after it has served its intended purpose.
- 3.10 **Source Separation** - segregation of wastes into discrete waste categories for the purpose of accumulation, treatment, storage, or disposal.
- 3.11 **Transportation** - the movement of hazardous materials by air, rail, highway, or water.
- 3.12 **Treatment, Storage and Disposal Facility (TSDF)** - an EPA permitted facility that treats, stores or disposes of hazardous waste.

- 3.13 **Treatment** - any method that renders hazardous waste non-hazardous or less hazardous.
- 3.14 **Waste Accumulation** - the gathering and/or collection of hazardous wastes at a location other than the satellite accumulation location (40 CFR 262.34).
- 3.15 **Waste Collection** - the satellite accumulation of hazardous wastes.
- 3.16 **Waste Minimization** - any change in operational procedures that results in the reduction of hazardous waste volume or toxicity.
- 3.17 **Waste Packaging** - the proper containment and labeling of wastes in DOT approved containers for shipment to a TSDF (40 CFR 171 - 177).
- 3.18 **Waste Transportation** - moving and manifesting of waste to an off-site TSDF.
- 3.19 **Uniform Hazardous Waste Manifest** - EPA Form 8700-22 or equivalent state specific form that is originated, signed and maintained by the generator for the purpose of tracking hazardous waste shipments (40 CFR 262.23).
- 3.20 **Chlorinated Waste** - Any organic solution containing more than 25 ppm chlorine or other halogens, excluding PCBs.
- 3.21 **Non-chlorinated Waste**- Any solution containing non-halogenated organic compounds greater than 25 ppm.
- 3.22 **Acidic Waste** - Aqueous solutions with a pH less than 2.5
- 3.23 **Basic Waste** - Aqueous solutions with a pH greater than 10.5.
- 3.24 **Non-hazardous - Wastes** with a pH greater than 5.5 and less than 10.5, organic contamination less than 25 ppm, metals less than 1 ppm (mercury less than 0.02 ppm), and no licensable amount of radioactivity.
- 3.25 **Radioactive** - Wastes known to contain radioactive material.
- 3.26 **Laboratory Waste** - Any by-product from extraction, analysis or clean-up process including, but not limited to spent samples, initial wash and first rinse waste, contaminated: gloves, bench top paper, pipettes, etc.
- 3.27 **Publicly Owned Treatment Works (POTW)** - A publicly owned sanitary sewer system.
- 3.28 **Treatment, Storage, and Disposal Facility (TSDF)** - An EPA permitted facility that treats, stores, and disposes of hazardous waste

4.0 INTERFERENCES

- 4.1 Cross contamination of samples may occur if the sampling device is used for sampling different containers.
- 4.2 The sampling device must be thoroughly cleaned between each sample using a compatible solvent or water. If the device cannot be thoroughly cleaned, then obtain a new sampling device.

5.0 SAFETY

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- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 Protective Equipment
- 5.2.1 Personnel that remove hazardous waste from the satellite containers in each laboratory must wear latex or nitrile safety glasses (Silver Shield gloves for solvents), a lab coat, and appropriate gloves. When bulking liquid waste in the Main Waste Accumulation Area a face shield is required.
- 5.2.2 Personnel that use the waste compactor located in the 90-day Waste Storage Area (waste room) must use hearing protection while operating the equipment. The equipment may not be used for extended periods, not to exceed 1-hour on any given day.
- 5.2.3 All handling of full waste drums will be done with the use of a drum dolly. Movement of waste drums to a secondary spill container in the Main Waste Accumulation Area will be done so with the use of the supplied ramp.
- 5.2.4 Grounding equipment must be used for accumulation containers that hold flammable liquid waste in the Main Waste Accumulation Storage Area. Ground the accumulation container using a grounding cable attached to a grounding rod.
- 5.2.5 Spill kits are located in each main laboratory group's area. Thus, there is one located in the Main lab, the Rad lab hallway, and the Main Waste Accumulation Area. They are equipped with at least the following:
- 5.2.5.1 Universal Gel Absorbent
 - 5.2.5.2 Spill Booms
 - 5.2.5.3 Chemical resistant booties
 - 5.2.5.4 Solvent gloves
 - 5.2.5.5 Latex gloves
 - 5.2.5.6 Chemical absorbent pads
 - 5.2.5.7 Face shield
 - 5.2.5.8 Chemical resistant trash bag
 - 5.2.5.9 Tyvek body suits
 - 5.2.5.10 Directions for spill clean-up

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Designated satellite accumulation containers.
- 6.2 DOT approved drum or container
- 6.3 Grounding equipment, where applicable (for Flammables)
- 6.4 Funnel
- 6.5 Face Shield
- 6.6 Plastic Apron
- 6.7 Sample Bottles
- 6.8 Bottle Labels

6.9 Chain of Custody

7.0 REAGENTS AND STANDARDS

- 7.1 The reagents in this section are not used for chemical analysis and thus are not required to follow documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 Universal Absorbent spill kit
- 7.3 Absorbent paper/booms
- 7.4 Mercury spill kit

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection, except of its' waste drums. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures.
- 8.2 Waste drum samples are collected in accordance with the sample volumes and preservative information is given in ST-PM-0002.
- 8.3 Samples handling, storage and hold times are discussed in the analysis SOPs:
 - 8.3.1 Gross Alpha/Beta: ST-RC-0020
 - 8.3.2 Total Metals: ST-IP-0002 (solids ICP), ST-MT-0007 (solids mercury)- and ST-IP-0013 (liquids ICP), ST-MT-0005 (liquids mercury)
 - 8.3.3 TOC, ST-WC-0016
 - 8.3.4 PCB, ST-OP-0002
 - 8.3.5 Additionally analyses may be needed, please reference individual method SOPs.

9.0 QUALITY CONTROL

- 9.1 Nonconformance and Corrective Action
 - 9.1.1 Failure to comply with the guidelines within this document may result in legal liabilities, unsafe management operations, and/or wastes that cannot be accepted by TSDF's and unlawful transportation of hazardous waste.
 - 9.1.2 Failure to submit reports listed within this document could result in legal action taken by the State of Missouri and/or the U.S. EPA with fines and increased hazardous waste taxes.
- 9.2 Quality controls for the analytical methods used in testing waste drum samples is defined in section 9 of each analytical SOP.

10.0 CALIBRATION

- 10.1 For pH meter calibration, please see SOP: ST-WC-0011
- 10.2 Individual analysis calibrations are specified in each analytical SOP.

11.0 PROCEDURE

- 11.1 Hazardous Waste Collection

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11.1.1 Specific definitions of each waste stream are provided in *Disposal of Samples, Table 1*

WASTE CATEGORY	STORAGE DRUM TYPE - SAA	STORAGE DRUM TYPE – Waste Room
Aqueous Acidic Waste = pH <5.5	30/55 Gallon Closed Head Poly Drum – UN Rated, marked “A” or “B”	30/55 Gallon Closed Head Poly Drum – UN Rated, marked “A” or “B”
Aqueous Base Waste = pH >10.5	30/55 Gallon Closed Head Poly Drum – UN Rated, marked “A” or “B”	30/55 Gallon Closed Head Poly Drum – UN Rated, marked “A” or “B”
Non-Hazardous Liquid	30/55 Gallon Closed Head Poly Drum – UN Rated, marked “A” or “B”	30/55 Gallon Closed Head Poly Drum, marked “A” or “B”
Mixed Solvent	5 Gallon Carboy	55 Gallon Closed Head Steel Drum – UN Rated, marked “D”
Pyridine	5 Gallon Carboy	5 Gallon Carboy
Non-Regulated Sodium Sulfate	5 Gallon Steel Pail	55 Gallon Open Head Steel Drum, marked “C”
Non-Regulated Soil/Debris	5 Gallon Steel Pail	55 Gallon Open Head Steel Drum, marked “C”
Vials - Flammable Solvent	5 Gallon Lever-Actuated Flip Top Steel Waste Container	55 Gallon Open Head Steel Drum – UN Rated, marked “C”
Vials – Acidic	55 Gallon Closed Head Poly Drum, marked “E”	55 Gallon Closed Head Poly Drum, marked “E”
Fluorescent Bulbs	Original containers	Secondary Fiber Board Box Designed for Fluorescent Bulb Shipment
Lab Pack Material	Original containers	*Suitable Secondary Containment
Radioactive Liquid – Acidic	2 liter poly containers	30/55 Gallon Closed Head Poly Drum, marked “A” or “B”
Radioactive Liquid Scintillation Cocktail Solutions	Original containers (vial boxes)	55 Gallon Open Head Steel Drum – UN Rated, marked “C”
Radioactive Solids/Debris	5 Gallon Steel Pails	55 Gallon Open Head Steel Drums – UN Rated, marked “C”
Radioactive Dry Active Waste	5 Gallon Steel Pails or Specially Designed Rectangular Fiberboard Boxes	55 Gallon Open Head Steel Drums – UN Rated, marked “C”
PCB Contaminated Waste Solids	5 Gallon Steel Pails	30/55 Gallon Open Head Steel Drums – UN Rated, marked “C”
Mercury Waste (Broken Thermometers, etc.)	2 Liter Poly Container with Lid	2 Liter Poly Container with Lid
* - “Suitable” = containers made of compatible material that will contain entire quantity should the original containers leak.		

11.1.2 Satellite Accumulation Containers

11.1.2.1 Lab coat, safety glasses and appropriate chemical resistant gloves must be worn when transferring wastes into the satellite accumulation containers.

11.1.2.2 The satellite accumulation containers, smaller than 30-gallons, must use secondary spill containment. Note: It is not practical or safe to place 30-gallon or 55-gallon satellite accumulation containers into secondary spill containers. Also, certain “Dump Daily” containers may not need secondary containment. See the EHSC for guidance.

11.1.2.3 All Satellite Accumulation Containers will be labeled at a minimum with the contents of the container, the date the accumulation started (unless it is a “Dump

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- Daily” SAA, see 3.4.1.4), and the name of the area that the container resides in. These containers are usually smaller than 30 gallons in size.
- 11.1.2.4 In some instances, small (5-gallon or less) waste containers may be used that are not required to have an accumulation start date listed on the label. These containers are known as “Dump Daily” waste containers and shall only contain waste during an active shift. At the end of the employee’s shift the container must be emptied into a permanent waste drum.
 - 11.1.2.5 The satellite containers will be inspected daily by the individuals working in their specific laboratory departments.
 - 11.1.2.6 The satellite containers must be kept closed when not in active use.
 - 11.1.2.7 Waste that is generated at the bench is segregated and accumulated into accumulation containers designated for that particular area. Only wastes that are compatible with the waste stream designated for each container may be disposed into a satellite container.
 - 11.1.2.8 Radioactive samples and wastes generated from these samples must be segregated from other wastes generated in the laboratory.
 - 11.1.2.9 When transferring the wastes into the satellite containers, proceed slowly and carefully in order to avoid a spill. Know the locations of the spill kits in the area.
 - 11.1.2.10 If a spill occurs, immediately alert all personnel in the vicinity and contact the lab supervisor and the Environmental Health & Safety Coordinator.
 - 11.1.2.11 Contain the spill in as small of an area as possible.
 - 11.1.2.12 The EHSC will then oversee the clean up of the spill and dispose of the cleaning material in the appropriate waste container.
 - 11.1.2.13 When a satellite container is 95% full it will be transferred to the 90-day storage area.
 - 11.1.2.14 In specific cases, client waste may be segregated to avoid large quantities of radioactive or TSCA waste from being generated. Client samples will be consolidated based upon analyzed results, prior knowledge, preservation type, etc. according to the waste stream.
- 11.1.3 Accumulation Containers in the Main Waste Accumulation Storage Area
- 11.1.3.1 Satellite containers need to be emptied on a periodic basis to minimize exposure to hazardous constituents.
 - 11.1.3.2 Transfer the satellite accumulation containers to the Main Waste Accumulation Storage Area when it becomes equal to or less than 95% full.
 - 11.1.3.3 Obtain the assistance of another associate to help in the transfer process of the satellite accumulation container.
 - 11.1.3.4 Ensure that the waste is placed in the designated 55-gallon accumulation container.
 - 11.1.3.5 The 55-gallon accumulation containers must be labeled.
 - 11.1.3.6 Lab coat, safety glasses, face shield, funnel, grounding equipment, and appropriate chemical resistant gloves must be used when transferring the wastes from the satellite container to the 55-gallon accumulation container.
 - 11.1.3.7 When transferring the contents from the satellite containers, proceed slowly and carefully in order to avoid a spill. Know the location of the spill kits and emergency equipment in the area.
 - 11.1.3.8 Grounding equipment must be used for accumulation containers that hold flammable liquid waste. Ground the accumulation container using grounding cable attached to a grounding rod.
 - 11.1.3.9 If a spill occurs, immediately contact the supervisor and the Environmental Health & Safety Coordinator.
 - 11.1.3.10 Make sure that the 55-gallon drum is closed when finished transferring the waste.

- 11.1.3.11 Record the drum entry into the Waste Disposal Logbook, entering the date of entry, the next assigned drum number as listed in the Waste Disposal Logbook, the satellite accumulation area, and any additional comments that may be appropriate.
- 11.1.3.12 Assign the drum number to the top of the drum. This is a unique identification number used to track each specific drum.
- 11.1.3.13 Locate the waste label on the drum and write in today's date and the words "waste room", showing that the drum is now in final storage and has 90 days to leave the facility. All 30 and 55-Gallon accumulation containers must be DOT approved shipping containers (UN Rated) labeled with the DOT shipping name which shows the contents of the drum, the start date, and the name of the area that the container resides in.
- 11.1.3.14 Record the name of the dept. that is disposing of the waste, the date, and the purpose of entering the Main Waste Accumulation Storage Area on the clipboard posted on the outside of the Main Waste Accumulation Storage Area.
- 11.1.3.15 Locate the Drum Labeling Information Board located in the waste room. Use this to identify the correct labels needed to place on your next drum that will be used in place of the one just entered into the waste room.
- 11.1.3.16 Find the same type of drum to use to replace the last one. Consult the EH&S Coordinator for assistance if needed.
- 11.1.4 Unknown Wastes
 - 11.1.4.1 Contact the Environmental Health & Safety Coordinator for any non-standard wastes or neat chemicals (expired standards) needing to be managed for disposal.
 - 11.1.4.2 The Environmental Health & Safety Coordinator will arrange for analytical analysis to determine the appropriate waste stream.
 - 11.1.4.3 When a 55-gallon accumulation drum is 95% full, it will be capped and labeled by the Environmental Health & Safety Coordinator. The drum will then be sampled to determine its constituents as described in this SOP.
 - 11.1.4.4 Hazardous Waste that has been entered into the Main Waste Accumulation Area may be stored there for no more than 90-days. By the 90th day the Hazardous Waste must be shipped to the approved TSDF.

Laboratory	Type of Waste	Description of Waste
Organic Extraction	Aqueous Base Waste	Water mixed w/ sodium hydroxide
	Aqueous Acidic Waste	Water mixed with nitric, hydrochloric, or sulfuric acid
	Mixed Solvent Waste	Acetone, Methylene Chloride, Hexane, Acetonitrile, Ether
	Sodium Sulfate	Sodium sulfate mixed with soil
	Radioactive Solids	Dry Active Waste (towels, PPE, bench paper, etc.)
GC & GC/MS	Radioactive Soil	Soil emitting radioactive activity
	Vials of Mixed Solvent	Sample extracts
VOA	Radioactive Solids	Dry Active Waste (towels, PPE, bench paper, etc.)
	Non-Reg. H ₂ O	Non-hazardous waste water
	Radioactive Soil	Soil emitting radioactive activity
Wet Chemistry	Radioactive Solids	Dry Active Waste (towels, PPE, bench paper, etc.)
	Aqueous Base Waste	Ammonium Hydroxide, Sodium Hydroxide
	Aqueous Acidic Waste	Nitric, hydrochloric, or sulfuric acid
Metals	Pyridine	Pyridine waste
	Radioactive Solids	Dry Active Waste (towels, PPE, bench paper, etc.)
	Aqueous Acidic Waste	Nitric, hydrochloric, or sulfuric acid
Radiochemistry	Radioactive Solids	Dry Active Waste (towels, PPE, bench paper, etc.)
	Radioactive Soil	Soil emitting radioactive activity
	Aqueous Acidic Waste	Nitric, hydrochloric, or sulfuric acid

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	Aqueous Base Waste Radioactive Solids Radioactive Soil Scintillation Cocktail	Ammonium Hydroxide, Sodium Hydroxide Dry Active Waste (towels, PPE, bench paper, etc.) Soil emitting radioactive activity Scintillation vials
TCLP	Aqueous Acidic Waste Radioactive Solids Radioactive Soil	Nitric, hydrochloric, or sulfuric acid Dry Active Waste (towels, PPE, bench paper, etc.) Soil emitting radioactive activity
Sample Control	Aqueous Acidic Waste Aqueous Base Waste Radioactive Soil Non-Reg. Soil/Debris Radioactive Solids VOA Vials	Nitric, hydrochloric, or sulfuric acid Ammonium Hydroxide, Sodium Hydroxide Soil emitting radioactive activity Non-Hazardous soil & debris Dry Active Waste (towels, PPE, bench paper, etc.) Acidified (hydrochloric or sulfuric) aqueous sample
Waste Room (Lab Pack)	Universal Waste Expired Materials Non-Reg. Soil/Debris PCB's Radioactive Solids VOA Vials	Fluorescent Bulbs, Batteries Misc. material not disposed of in general waste streams Non-Hazardous soil & debris Materials contaminated with >50 PPM PCB's Dry Active Waste (towels, PPE, bench paper, etc.) Acidified (hydrochloric or sulfuric) aqueous sample

11.2 Sample Disposal

- 11.2.1 The project manager identifies samples that are ready for disposal and then notifies the Sample Control staff.
- 11.2.2 The Sample Control Department removes the samples from the storage areas and segregates them by waste streams, (Table 1). The Sample Control Department then disposes of the waste in the appropriate container as designated by the Environmental Health & Safety Coordinator.
- 11.2.3 It is the responsibility of the project managers to designate samples for disposal according to the specific requirements of their clients.
- 11.2.4 Some projects have unique sample disposal procedures. The Environmental Health & Safety Department and the Sample Control Department must be informed of these unique procedures by the project manager. The Environmental Health & Safety Coordinator will assist in the correct disposal of these samples.
- 11.2.5 The project manager notifies samples control that the analysis for a series of samples is complete and no longer must be stored as discrete samples.
- 11.2.6 The Sample Control Department removes the samples from the storage areas. Lab coat, safety glasses, and appropriate chemical resistant gloves must be worn when pulling samples from the storage coolers.
- 11.2.7 The Sample Control Department segregates the samples according to the specified waste streams. See Table 1 for a list and definitions of the waste streams identified in the laboratory.
 - 11.2.7.1 Hazardous waste streams are segregated for disposal by a permitted treatment, storage and disposal facility (TSDF).
 - 11.2.7.2 Radioactive samples, Categories 2, 3 and 4 are segregated for storage and disposal by a licensed disposal facility, or returned to the client.
 - 11.2.7.3 All samples exhibiting the characteristics listed in Table 1 are added to the correct accumulation drum by the sample control department.
- 11.2.8 All marking labels must be destroyed such that information is not legible on the containers after their contents have been emptied. The only exception is for containers that are being shipped for incineration, as this will destroy the markings.
- 11.2.9 All samples determined to be non-hazardous may be discharged to the sanitary sewer by the Environmental Health & Safety Coordinator in accordance with *Guidelines for Discharge to the Sewer* found in this SOP.
- 11.2.10 All samples determined to be hazardous are disposed using an EPA permitted TSDF.

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- 11.2.11 Soil samples are collected in drums such that every 5th container is randomly selected for compositing. At completion of filling the drum, the selected samples are composited and analysis is performed according to the direction of the Environmental Health & Safety Coordinator. After review of the analysis, the appropriate disposal action is determined. All non-hazardous soils are processed by incineration in compliance with our USDA permit.
- 11.2.12 All lab waste and expired samples not returned to the client are segregated and ultimately accumulated into appropriately sized accumulation containers. Samples are collected from these containers and analyzed for hazardous and/or radioactive constituents to properly characterize the waste prior to disposal. Once they are sampled, acid and base accumulation drums are neutralized to a pH acceptable for sewerage, a pH between 5.5 and 10.5.

Table 1
Definitions for Hazardous Waste Streams

Waste Stream Drum	Description	
1. Aqueous Acidic Liquids	pH<5.5 readily dispersible in water.	Acid
2. Aqueous Basic Liquids	pH>10.5 readily dispersible in water.	Base
3. Waste Flammable Solvents	Halogenated Solvents <25% Non-Halogenated Solvents 50-100% Waste Oils (non-PCB containing) <50%	Flam
4. Non-Rad, non RCRA Solids	non-hazardous soil, debris, lab trash	Non-Reg
5. Solids, soil containing PCB's	wet or dry solids known to contain PCB's >50ppm, may contain glassware	PCB
6. Rad acidic liquid	acidic liquids known to contain licensable quantity of radioactive material.	Rad-acid
7. Rad Scintillation Cocktail	organic liquid phase known to contain licensable amount of radioactive material	Rad- Vial
8. Radioactive Solids	wet or dry solids, including resins, known to contain licensable amount of radioactive material (Cat 2,3,4)	Rad- Soil
9. Radioactive Dry Active Waste	combustible, dry solids including plastic bags, absorbent paper, lab trash, planchettes, and protective equipment used to prevent the spread of radioactive contamination.	DAW
10. Non-hazardous liquids	pH>5.5, pH<10.5, organic contamination known to <25ppm, metals less than 1ppm (<.02 ppm for Mercury). Includes ground or surface water samples.	Non- Reg
11. Vials from GC, GCMS, & Standards	vials that contain sample and/or standards residue.	Flam- Vial
12. Aqueous acidic solutions containing metals	liquids that contain concentrated amounts of metals such as standards and digestates with concentrations > 1.0ppm (>0.02ppm for Mercury).	Acid
13. Pyridine Waste >2% Pyridine	acidic waste containing 2% pyridine or greater	Acidw/
14. Mercury Waste	liquids that contain concentrated amounts of mercury such as standards and digestates with concentrations > 0.02ppm.	N/A

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15. Universal Waste	Batteries, light bulbs from emergency lights and other sources.	N/A
16. Standards containing Silvex, Tri-, Tetra-, or Penta-Chlorophenol	Expired standards containing the four listed hazardous components	Acid
17. Non-Hazardous Soil/Solids	All soil or solid waste not having a radioactive or RCRA hazardous constituent.	Non- Reg

11.3 Sampling of Waste Drums

11.3.1 The samples are logged into the LIMS for specific analyses after they are collected and then stored in the 90-day storage area until analyzed.

11.3.2 Sampling Liquids

11.3.2.1 A sample is collected from a 30 or 55-gallon accumulation barrel using a pump.

11.3.2.2 A portion of the contents is collected from the accumulation drum at several depths and locations in order to obtain a representative sample.

11.3.2.3 Safety glasses, lab coat and appropriate chemical resistant gloves must be worn while collecting samples.

11.3.2.4 The contents of the drum are collected into the sample container(s).

11.3.2.5 The sample containers are labeled with the following information:

11.3.2.5.1 Drum

11.3.2.5.2 Number

11.3.2.5.3 Sample Date

11.3.2.6 The following analyses are required for complete characterization or analyses of unknown liquid wastes:

11.3.2.6.1 Gross Alpha/Beta

11.3.2.6.2 Total Metals

11.3.2.6.3 TOC

11.3.2.6.4 PCB

11.3.2.6.5 Gamma Scan may be needed later if the Gross Alpha/Beta analysis shows radioactive activity.

11.3.2.7 Additional analyses may be required due to varying acceptance criteria from treatment, storage, and disposal facilities.

11.3.2.8 The samples are logged into the LIMS for specific analyses after they are collected and then stored in the 90-day storage area until analyzed.

11.3.2.9 After initial characterization, generator knowledge may be used to determine the waste stream of the accumulation container. The Environmental Health & Safety Coordinator may require one or more of the above analyses to confirm waste streams.

11.3.2.10 Once the sampling process has been completed, neutralization of the drum needs to be performed if the contents of the drum's pH is less than 5.5 or greater than 10.5. Remember that when adjusting the pH either way in a drum, excessive heat may build up. Follow these directions precisely as to not damage the drum.

11.3.2.11 For drums showing pH less than 5.5 add approximately 5000 grams of sodium hydroxide pellets *slowly* to the drum, taking about 3-4 minutes to add full amount. Do not add quickly as it may result in a violent reaction. Use a long stirring device, such as a broom handle, to mix and dissolve the pellets. Check the pH. If still below 5.5, now add sodium hydroxide pellets in smaller increments of 500 grams. Due to the buffering action of the contents of the drum it usually takes a while to adjust the pH. After a certain point that buffering changes rapidly, adjusting the pH in a large swing.

11.3.2.12 For drums showing pH higher than 10.5 add approximately 2.5 liters of concentrated nitric acid or hydrochloric acid. Using expired bottles of acid is suggested as to not waste these since they cannot be used for analytical

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- purposes. If the pH has not dropped below 10.5, add smaller amounts of acid, in 250 ml increments, stirring and checking the pH each time.
- 11.3.3 Sampling Solids
- 11.3.3.1 Any and all radiochemistry analytical data will be reviewed for samples that are collected into waste drums that could potentially meet the description of a radioactive solid waste as defined by DOT, the NRC, or other local, State, or Federal regulations.
- 11.3.3.2 A sample is collected from a 55-gallon accumulation barrel using a random composite of 20% of the samples placed in the drum. This sampling is to provide data for correct waste characterization and drum profiling with the proper TSDF.
- 11.3.3.3 Safety glasses, lab coats, and appropriate chemical resistant gloves must be used while collecting composite samples.
- 11.3.3.4 The sample containers are labeled with the following information:
- 11.3.3.4.1 Drum Number
- 11.3.3.4.2 Sample Date
- 11.3.3.5 The following analyses are required for complete characterization or analyses of unknown solid wastes:
- 11.3.3.5.1 Total Metals
- 11.3.3.5.2 Gross Alpha/Beta
- 11.3.3.5.3 Gamma Scan
- 11.3.3.5.4 PCB
- 11.3.3.6 Additional analyses (especially further radiochemistry parameters) may be required due to varying acceptance criteria from treatment, storage, and disposal facilities.
- 11.3.3.7 The samples are logged into the LIMS for specific analyses after they are collected and then stored in the 90-day storage area until analyzed.
- 11.3.3.8 After initial characterization, generator knowledge may be used to determine the waste stream of the accumulation container. The Environmental Health & Safety Coordinator may require one or more of the above analyses to confirm waste streams.
- 11.3.3.9 Wastes found to be hazardous will be disposed using an EPA permitted treatment, storage and disposal facility.
- 11.3.3.10 After the drum has been analyzed, the data is reviewed by the Environmental Health & Safety Coordinator to determine if the waste is hazardous or non-hazardous.
- 11.3.3.11 Hazardous waste is disposed using an EPA permitted treatment and disposal facility.
- 11.3.3.12 Non-hazardous aqueous waste may be discharged to the sewer after the criteria from Table 2 have been met.
- 11.3.3.13 Drums shown to be non-hazardous are placed near the floor drain located in the shipping and receiving area.
- 11.3.3.13.1 The siphon hose is filled with water from the tap. One end is then placed in the drum, and the other is placed in the drain located under the sink.
- 11.3.3.13.2 The drum is then allowed to empty into the drain.
- 11.3.3.13.3 The empty drum is rinsed with water and stored until re-use.

Table 2
Criteria for Disposal VIA Sewer

Parameter	Daily Average Limit* (mg/L) – Large River
Antimony	0.5
Arsenic	0.4

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Barium	10.0
Beryllium	0.4
Cadmium	0.7
Chromium	5.0
Copper	2.7
Cyanide, Amenable	0.4
Iron	150.0
Lead	0.4
Mercury	0.01
Nickel	2.3
Oil & Grease	200.0
Phenolic Compounds	7.0
Selenium	0.2
Silver	0.5
Zinc	3.0
Total Toxic Organics	Not to exceed 5.52 mg/L at any time
Temperature	Not to exceed 140 degrees F (60 degrees C) at any time
pH	Shall be in the range of 5.5 to 10.5 s.u. at all times
* - Excursions may be allowed pursuant to the continuous monitoring provisions of Article X, Section 2, Subsection C.	

11.4 Shipping

- 11.4.1 Upon completion of characterization, the waste barrels will be classified according to the proper shipping name found in 49 CFR 172.101.
- 11.4.2 The proper hazard class or packing group and labeling requirements will be determined by using 49 CFR 172.101, Hazardous Materials Table.
- 11.4.3 The proper labels will be filled out and applied to the container as specified in 49 CFR Subpart E and as requested by the transporter.
- 11.4.4 When setting guidelines as to specific radioactive waste limits, TestAmerica St. Louis has determined that waste classified as being above the limits set forth by the Department of Transportation (DOT) for a radioactive material shall be classified as a radioactive waste. Material deemed lower than the DOT established limits shall be considered non-radioactive.

11.5 Manifesting

- 11.5.1 Manifests are obtained from the state that is the waste's final destination. If that state does not have a manifest, then a Missouri State Manifest must be used. Missouri manifests may be obtained from the waste broker being used to transport the waste. They may also be obtained from the Missouri Department of Natural Resources. Department of Environmental Quality, Waste Management Program, P.O. Box 176, Jefferson City, MO 65102.
- 11.5.2 An example of the Uniform Hazardous Waste Manifest with instructions can be found in the Appendix to 40 CFR 262, or state instructions accompany individual state manifests.
- 11.5.3 Generator Numbers for STL St. Louis:
EPA ID: MOD981709272
MO ID: 000551
- 11.5.4 The EPA and Missouri ID numbers must be used for all shipments.
- 11.5.5 Use of Land Ban and Land Disposal Restriction Forms
- 11.5.6 Land Ban Forms and Land Disposal Restriction Forms are required for shipments of waste that are defined as Hazardous Waste under RCRA regulations. Discuss the shipment with the waste broker prior to shipment to negotiate which wastes being shipped needs to be accompanied by these forms.
- 11.5.7 Generally the waste vendor will prepare these. If so, the EHSC or RCRA trained designee must review the forms for accuracy prior to shipping the waste from the facility.

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- 11.5.8 All solid waste drums being shipped from the facility must be frisked using a handheld frisker calibrated to read in counts-per-minute (CPM). A record shall be kept with the manifest which includes the following:
 - 11.5.8.1 Shipment date,
 - 11.5.8.2 Manifest #,
 - 11.5.8.3 Drum #,
 - 11.5.8.4 Waste stream,
 - 11.5.8.5 Area background,
 - 11.5.8.6 Highest reading noted and area of drum where highest reading was captured,
 - 11.5.8.7 Frisker serial #, model #, and most recent calibration date
- 11.5.9 All drum numbers must be documented on each manifest so that each manifest directly shows which drums were shipped under that specific manifest.
- 11.5.10 Drums being sent out as non-radioactive must be lower than free-release criteria, which is less than 100 CPM above area background.
- 11.6 An inspection of the 90-day storage area will be conducted once per week by the Environmental Health & Safety Coordinator or designee and documented in the Waste Inspection Log Book.
 - 11.6.1 This inspection is designed to identify potential hazards and recommend remedial and follow-up actions necessary to correct those hazards that may exist and to comply with state and federal regulations.
 - 11.6.2 Safety inspections are an effective means for minimizing potential hazards when routinely conducted and follow-up action is maintained.
 - 11.6.3 During this inspection the dates of the storage drums will be checked as well to assure compliance with regulations stating that hazardous waste will not be stored for a period greater than 90 days. All hazardous waste shall be shipped to the approved TSDF prior to the 90th day in storage.
 - 11.6.4 The Environmental Health & Safety Coordinator or designee will inspect the 90-day storage area at least once per week.
 - 11.6.5 The following guidelines are to be used when making the inspection and noted in the Log Book.
 - 11.6.5.1 Required signs must be posted: No Smoking; Authorized Personnel Only; and any sign denoting required safety equipment.
 - 11.6.5.2 General Housekeeping: No paper, boxes, trash, etc. found on the floor or in the aisles.
 - 11.6.5.3 All containers must be inspected for: leaks, cleanliness and grounding (steel drums only).
 - 11.6.5.4 All containers must be capped when not in active use.
 - 11.6.5.5 Any odors must be investigated and resolved.
 - 11.6.5.6 All necessary DOT and EPA labels must be in place on those drums known to be hazardous.
 - 11.6.5.7 Accumulation start dates must be noted on all accumulation containers.
 - 11.6.5.8 Minimum safety equipment available in the area must include: Eyewash Station, Fire Extinguisher, and Spill Kits.
 - 11.6.5.9 Incompatible materials must be segregated by a secondary containment.
 - 11.6.5.10 The date and person conducting the inspection must be recorded on the checklist.
 - 11.6.5.11 Any deficiencies, corrective actions, and dates of completion must be included on the inspection checklist.
- 11.7 Records Management
 - 11.7.1 All waste management records must be kept in the Hazardous Waste Management files until such time that they are archived for permanent document storage.
 - 11.7.2 A quarterly report of all wastes generated and/or shipped must be filed with the Missouri Department of Natural Resources within 45 days after the end of the quarter.

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- 11.7.3 All records relating to waste storage, handling, and shipping that are generated will be kept on file with the Environmental Health & Safety Coordinator until such time that they are archived for permanent document storage. These records shall include but not be limited to:
 - 11.7.3.1 Analysis from waste samples and all data used to classify waste for proper disposal
 - 11.7.3.2 Waste manifests
 - 11.7.3.3 LDR notifications & Land Ban Forms
 - 11.7.4 The Missouri Department of Natural Resources' Generator's Hazardous Waste Report Summary Sheet must be filed within forty-five days after the end of a calendar quarter. The end of the quarters are as follows:
 - 11.7.4.1 First Quarter, September 30.
 - 11.7.4.2 Second Quarter December 31.
 - 11.7.4.3 Third quarter, March 31.
 - 11.7.4.4 Fourth Quarter, June 30.
 - 11.7.5 Instructions for completing this report can be found with the report sent by the Missouri Department of Natural Resources.
 - 11.7.6 Copies of manifests must accompany this report.
 - 11.7.7 U.S. Environmental Protection agency Biennial Reports must be filled out every even numbered year by March 15. Instructions for completing this form will be mailed when it is time to complete the report from EPA Region VII office.
 - 11.7.8 Exception Reports must be filed with the Missouri Department of Natural Resources when a completed manifest is not returned from the TSDF within forty-five (45) days after the waste is received at the facility.
 - 11.7.9 This report shall consist of a letter addressed to the department explaining the methods used to try and obtain the completed manifest from the TSDF. A copy of the incomplete manifest must accompany this letter.
 - 11.7.10 Information and assistance may be obtained for any of these requirements by calling the Missouri Department of Natural Resources, Hazardous Waste Program.
- 11.8 Spills
- 11.8.1 Solvent, acid, and base material spills are cleaned up using the Universal Gel Absorbent. However, Universal Gel Absorbent is not to be used for Hydrofluoric acid. Instead, HF Acid Spill Absorbent and Neutralizer must be used. The spill is contained and cleaned up with the absorbent material being disposed in the appropriate waste container.
 - 11.8.2 Soil/Solid spills are cleaned up by gathering the spilled material and placing it in an appropriate container, and then washing the area thoroughly with water and collecting the water and waste.
 - 11.8.3 Metallic mercury is collected using a mercury pump or aspirator and the waste being collected in a polypropylene container. Mercury that is unable to be collected by a pump is sprinkled with Hg Absorb where an amalgam is formed which is disposed according to all applicable regulations.
 - 11.8.4 The material safety data sheet (MSDS) for the spilled material should be consulted before attempting to clean a spill.
 - 11.8.5 Acid, Base, Solvent Spills
 - 11.8.5.1 Equipment Checklist
 - 11.8.5.1.1 Universal Gel Absorbent spill kit
 - 11.8.5.1.2 Waste Container
 - 11.8.5.1.3 Broom/dust pan
 - 11.8.5.2 Never respond to a spill alone. Call for help and notify the supervisor and Environmental Health & Safety Department. Make sure that at least one other person responds to the spill.
 - 11.8.5.3 Be familiar with the safety equipment in the area as it may be needed.
 - 11.8.5.4 Never enter the area of the spill before knowing the exact nature of the spill as

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- toxic vapors may be present.
- 11.8.5.5 Never step into spilled material.
 - 11.8.5.6 Don the appropriate personal protective equipment.
 - 11.8.5.7 In some instances it may be advisable to dilute the acid with water (added very slowly) before attempting to neutralize and clean up the spill. Check the MSDS before diluting with water.
 - 11.8.5.8 Create a dike around the spill with absorbent or booms.
 - 11.8.5.9 Spread universal gel absorbent over the spill applying sufficient agent to obtain a consistent gelatin-like change throughout. Add in small amounts as a little absorbent goes a long way.
 - 11.8.5.10 Please note, the Universal Gel Absorbent quickly turns the liquid into a thick gel making it easier to clean up but it DOES NOT NEUTRALIZE the acid or base. Pick up and dispose of the gelatin waste in plastic containers with sealable lids for acids and bases. Do not use metal as the remaining corrosive material will erode it. Do not use glass as there is a risk of breakage. Metal containers shall be used to dispose of solvent material.
 - 11.8.5.11 The container must be labeled as to contents.
 - 11.8.5.12 Dispose of the waste as per the instructions of the Environmental Health & Safety Department.
 - 11.8.5.13 Rinse the area with water and collect the content into an appropriate container for disposal as directed by the Environmental Health & Safety Department.
- 11.8.6 Spills of Soils or other Solids
- 11.8.6.1 Never respond to a spill alone. Call for help and contact the supervisor and the Environmental Health and Safety Department. Make sure that at least one other person responds to the spill.
 - 11.8.6.2 Wear the appropriate protective equipment and clean up the spill by gathering the material with a broom or absorbent paper. Be careful not to stir up any dust or particles or step in the material.
 - 11.8.6.3 With dusty spills, it may be necessary to mist the materials with water before beginning to gather the material.
 - 11.8.6.4 Place the collected material into a container and dispose in the appropriate waste drum. Decontaminate all reusable cleaning materials and dispose of that waste in the appropriate waste container.
 - 11.8.6.5 If the material is known not to contain any hazards, it may be disposed in the sanitary trash.
 - 11.8.6.6 Clean the area of the spill with water after the material is collected and dispose in the appropriate waste container.
- 11.8.7 Metallic Mercury Spill
- 11.8.7.1 Equipment Checklist
 - 11.8.7.1.1 Mercury Spill Kit (includes aspirator, vacuum pump, and amalgam)
 - 11.8.7.1.2 Plastic bottles with lids for waste.
 - 11.8.7.2 Never step into the spilled material.
 - 11.8.7.3 Remove any exposed gold, copper, or silver jewelry before beginning cleanup.
 - 11.8.7.4 Clear the general spill area before cleanup is begun. Notify the supervisor and the Environmental Health & Safety Department immediately.
 - 11.8.7.5 Do not allow mercury to come into contact with exposed skin or eyes.
 - 11.8.7.6 Never respond to a spill alone. Always make sure that at least one other person responds to the spill.
 - 11.8.7.7 Using a wooden or plastic spatula, gather as much mercury together as possible.
 - 11.8.7.8 Collect this material using a mercury aspirator or a mercury vacuum pump.
 - 11.8.7.8.1 Squeeze the bulb to exhaust air from the aspirator
 - 11.8.7.8.2 Place the tip next to the mercury drops and sharply release the bulb.

- 11.8.7.8.3 Repeat until the bulb feels about half full. Empty the aspirator into a polypropylene bottle labeled "Impure Mercury."
- 11.8.7.9 Open the mercury absorbent container.
- 11.8.7.10 Apply a narrow strip of activated absorbent to the spill surface.
- 11.8.7.11 Push the absorbent across the contaminated area using a wooden or plastic spatula.
- 11.8.7.12 Repeat this process until the entire area has been covered at least two times.
- 11.8.7.13 Transfer the absorbent into a wide mouth poly jar and allow it to dry before closing.
- 11.8.7.14 Wipe the entire area with a sponge wetted with warm soapy water.
- 11.8.7.15 Collect and dispose of the waste material and all materials used to clean up the spill in a polypropylene container.
 - 11.8.7.15.1 Label the container "Mercury Waste Materials"
- 11.8.7.16 Mercury Vapor Absorbent should be used to absorb mercury vapors given off by small amounts of mercury remaining in areas inaccessible to physical clean up.
- 11.8.7.17 Sprinkle the absorbent into any cracks or areas where the presence of mercury is suspected.
- 11.8.7.18 Check the mercury vapor levels periodically using a Sensidyne Pump and direct read tube. Renew the absorbent as necessary.
- 11.8.7.19 After the mercury vapor levels are no longer detected above 0.1 ppm, collect the amalgam and dispose of contaminated materials according to the instructions given by the Environmental Health & Safety Department.

12.0 DEMONSTRATION OF CAPABILITY

- 12.1 Laboratory personnel responsible for handling hazardous waste shall be trained in hazardous waste management procedures to the degree appropriate for their position.
- 12.2 At a minimum, training will include
 - 12.2.1 *Collection and Accumulation of Hazardous Wastes*
 - 12.2.2 *Disposal of Samples After Analysis*
 - 12.2.3 *Hazardous Waste Spill Response*
 - 12.2.4 *Laboratory Safety/Chemical Hygiene Plan Training*
- 12.3 Training will be conducted by the Environmental Health and Safety Coordinator or designee.
- 12.4 Training records will be maintained in the individual's personal training files.

13.0 REFERENCES

- 13.1 Title 29, code of Federal Regulations, Parts 1900 - 1910, U.S. Department of Labor (OSHA).
- 13.2 Title 40, Code of Federal Regulations, Parts 240 - 281, U.S. Environmental Protection Agency, Protection of the Environment.
- 13.3 Title 10, Code of Federal Regulations, Parts 19, 20, 33, Nuclear Regulatory Commission Energy.
- 13.4 Title 10, Code of State Regulations, Part 25, Missouri Department of Natural Resources, Hazardous Waste Management Program.
- 13.5 Title 49, Code of Federal Regulations, Chapter 1. Subchapter C (171-177), Hazardous Materials Regulations, Department of Transportation.
- 13.6 St. Louis Metropolitan Sewer District Ordinance No. 8472.

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- 13.7 Test Methods for Evaluation of Solid Wastes, Vol. II, Field Manual; Physical/Chemical Methods, SW846, current revision.
- 13.8 TestAmerica Quality Assurance Manual (QAM), current revision
- 13.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 13.10 Associated SOPs
 - 13.10.1 ST-HS-0001, "Waste Minimization Plan"
 - 13.10.2 ST-HS-0003, "St. Louis Facility Contingency Plan"
 - 13.10.3 ST-IP-0002, "Acid Digestion of Soils"
 - 13.10.4 ST-IP-0013, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS"
 - 13.10.5 ST-MT-0005, "Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption"
 - 13.10.6 ST-MT-0007, "Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic absorption Spectroscopy"
 - 13.10.7 ST-OP-0002, "Extraction and Clean-up of Organic Compounds from Waters and Soils"
 - 13.10.8 ST-PM-0002, "Sample Receipt and Chain of Custody"
 - 13.10.9 ST-QA-0002, "Standards and Reagent Preparation"
 - 13.10.10 ST-RC-0020, "Determination of Gross Alpha/Beta Activity"
 - 13.10.11 ST-WC-0011, "Analysis of pH in Water"
 - 13.10.12 ST-WC-0016, "Total Organic Carbon"

14.0 CHANGES FROM PREVIOUS REVISION

- 14.1 Annual Review - No Changes
- 14.2 Rev 14;
 - 14.2.1 Annual Review, No Changes
- 14.3 Rev 15:
 - 14.3.1 Annual Review, No Changes
- 14.4 Rev 16: (9/11/13)
 - 14.4.1 Removed references to appendices no longer part of this SOP from Section 11.
- 14.5 Annual Review – No Changes (4/23/15)
- 14.6 Annual Review, No Changes (05/19/2016)

**Title: ACID DIGESTION OF SOILS
[SW-846 3050B FOR ICP, ICP/MS]**

Approvals (Signature/Date):


Kristen Ely
Inorganic Manager

7/25/16
Date


Michael Ridenhower
Health & Safety Manager / Coordinator

Date


Marti Ward
Quality Assurance Manager

7/25/16
Date


Elaine Wild
Laboratory Director

Date

This SOP was previously identified as SOP No. ST-IP-0002 Rev. 18

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1.0 SCOPE AND APPLICATION

- 1.1 This procedure describes the preparation of soil samples for the analysis of metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma Atomic Emission/Mass Spectrometry (ICP/MS).
- 1.2 This procedure is based on SW-846 Method 3050B.
- 1.3 Additional metals may be processed by this method, assuming that performance criteria of the determinative method are met.
- 1.4 This method is not a total digestion, but will dissolve almost all metals that could become “environmentally available”. By design, metals bound in silicate structures are not dissolved by this procedure as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludge, waste and sediment.
- 1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory’s Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1 A representative 0.5 g (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. Next the digestate is refluxed with hydrochloric acid for ICP, ICP/MS analysis. The digestates are then diluted to 50 mL.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Total Metals: The concentration determined on an unfiltered sample following digestion.

4.0 INTERFERENCES

- 4.1 Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc). Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.
- 4.2 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.
- 4.3 Specific analytical interferences are discussed in the respective analytical SOPs: ST-MT-0001 (ICP/MS) and ST-MT-0003 (ICP).

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and

reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added. Hydrogen peroxide (H₂O₂) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H₂O₂ to avoid a reaction and possible violent effervescence, or boiling over of the digestion. A splash/splatter hazard is possible and a face shield should be worn.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrofluoric Acid	Poison Corrosive Dehydrator	3 ppm-TWA	Severely corrosive to the respiratory tract. Corrosive to the skin and eyes. Permanent eye damage may occur. Skin contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.
Nitric Acid	Corrosive Oxidizer Poison	2ppm (TWA) 4ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1ppm (TWA)	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

Ceiling – At no time should this exposure limit be exceeded.

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Hot block, capable of maintaining a temperature of 90 – 95 °C.
- 6.2 Thermometer, temperature range of 0 – 250 °C.
- 6.3 Laboratory fume hood.
- 6.4 Hot block digestion vessels
- 6.5 Watch glasses, ribbed or equivalent
- 6.6 Vacuum filters – 0.45 µm
- 6.7 Analytical balance capable weighing to the nearest 0.001g.
- 6.8 Pipettes or reagent dispensers, micro pipettes 0.05 - 10 mL
- 6.9 Ultra-Pure PTEE Boiling Stones
- 6.10 Centrifuge tubes
- 6.11 Vacuum pump
- 6.12 Tongue Depressors (for taking aliquots of sample)

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 DI water: Obtained by the use of a Milli-Q System (ASTM Type I)
- 7.3 Matrix Spike Standard, NIST traceable
- 7.4 ERA soil laboratory control samples (LCS) or LCS aqueous standard, NIST traceable
- 7.5 Nitric acid (HNO₃), concentrated, trace metal grade
- 7.6 Hydrochloric acid (HCl), concentrated, trace metal grade
- 7.7 30% Hydrogen peroxide (H₂O₂), AR Select; Ultrex Grade (for Nb, TA and Sn.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Samples are to be collected in plastic or glass containers.
- 8.3 All soils must be refrigerated to $4 \pm 2^{\circ}\text{C}$.
- 8.4 The analytical holding time for metals is 6 months.

9.0 QUALITY CONTROL

- 9.1 Batch
 - 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
 - 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
 - 9.1.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), and a Matrix Spike/ Matrix Spike Duplicate (MS/MSD). In the event that there is insufficient sample to analyze a sample duplicate, a LCS Duplicate (LCSD) is prepared and analyzed.
 - 9.1.3.1. Matrix Spike (MS) and Sample Duplicate (DU) may be performed upon client request, and are noted in LIMS.
- 9.2 Method Blank
 - 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
 - 9.2.2 A method blank must be prepared with every sample batch.
 - 9.2.3 For Soil analyses, the method blank is comprised of boiling chips.
- 9.3 Laboratory Control Sample
 - 9.3.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.3.2 A LCS must be prepared with every sample batch.
 - 9.3.3 For Soil analyses, the LCS is a purchased Standard Reference Material (SRM)
 - 9.3.3.1. If requested, or if the target analytes are not in the SRM, the laboratory may perform a LCS consisting of boiling chips fortified with target analytes
- 9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)
 - 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.
 - 9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.
 - 9.4.4 If there is insufficient sample to perform a MS/MSD, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate

precision.

9.5 Sample Duplicate

- 9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.
- 9.5.2 A Sample Duplicate is only performed upon client request.
- 9.5.3 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.

9.6 Procedural Variations/ Nonconformance and Corrective Action

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Hot block/Hot plate must be checked daily when in use.

- 10.1.1 Temperature is documented in LIMS.
- 10.1.2 A calibrated thermometer is suspended in sand in a digestion vessel of water and brought to the proper temperature.
 - 10.1.2.1. See SOP ST-QA-0005 for information regarding calibration of thermometers.

10.2 Instrument calibration is discussed in the respective analytical SOPs: ST-MT-0001 (ICP/MS) and ST-MT-0003 (ICP).

11.0 PROCEDURE

NOTE: For Tc99 soil prep, see SOP ST-RC-0125 (Determination of Technetium-99 Using Eichrom TEVA Resin). For the alternate antimony soil prep, see section 11.26.

- 11.1 Label digestion vessel with sample ID or QC identifier.
- 11.2 Homogenize the sample by mixing thoroughly. See SOP ST-QA-0038 for details.
- 11.3 Weigh a 0.5 – 0.6 g portion of sample and the batch QC and record the weight in LIMS.
 - 11.3.1 Larger sample sizes may be used upon client request. Reagent volumes are adjusted to reflect the sample volume used.
- 11.4 Measure 0.5 - 0.6 g of boiling chips into a digestion vessel for the MB (and LCS, if applicable. See section 9.3)
- 11.5 Spike LCS (is applicable, see section 9.3) and MS/MSD with spiking mix applicable to the requested analysis.
 - 11.5.1 Document spiking volumes and standard numbers in LIMS.
- 11.6 Add 2.5 mL 1:1 HNO₃

- 11.7 Place watch glasses on digestion vessels
- 11.8 Place digestion vessels in hot block at 90 - 95°C and reflux for 10-15 minutes.
- 11.8.1 **Do not allow the sample to boil or go dry during the digestion.** Allowing so may result in the loss of volatile metals. If this occurs the sample must be re-prepared. Antimony is easily lost by volatilization from hydrochloric media.
- 11.9 Take samples out and cool.
- 11.10 Add 1.25 mL HNO₃.
- 11.11 Return covered vessels to hot block.
- 11.12 Reflux at 90 - 95°C for 30 minutes.
- 11.13 Add DI water as needed to ensure that the volume of solution is not reduced to less than 5 mL.
- 11.13.1 If brown fumes are observed, add additional 1.25 mL aliquots of concentrated nitric acid until no more fumes are given off.
- 11.13.2 If additional reagents are added to a sample, they must also be added to the MB and LCS.
- 11.14 Remove vessels from block and allow the samples to cool.
- 11.15 Add 1 mL of DI water and 2 mL of 30 % H₂O₂.
- 11.15.1 Use Ultrex grade for Nb, Ta and Sn
- 11.15.2 Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. If this occurs, add a minimal amount of DI water until sample settles, to ensure no loss of sample. If any amount of sample is lost, a re-digestion must be done.
- 11.15.3 If additional reagents are added to a sample, they must also be added to the MB and LCS.
- 11.16 Return covered vessel to hot block and heat sample until effervescence subsides.
- 11.17 While in block, continue adding 30% H₂O₂ in 1 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.
- 11.4.17.1 NOTE: Do not add more than a total of 5 mL of 30 % H₂O₂. If more than 5 mL of 30 % H₂O₂ is needed then the sample will be re-prepped using a smaller aliquot of sample.
- 11.17.1 If additional reagents are added to a sample, they must also be added to the MB and LCS.
- 11.18 Continue heating for 1 hour.
- 11.19 Remove vessel from hot block and allow to cool.
- 11.20 Add 2.5 mL HCL and reflux, on hot block, for an additional 15 minutes without boiling.
- 11.21 Remove vessel from hot block and allow to cool.
- 11.22 Wash down the vessel wall with DI water to dissolve any precipitation to a final volume 50 mL.
- 11.23 The digestate is filtered using a vacuum pump and a 0.45 µm filter. See Filtration SOP, ST-IP-0015.

- 11.23.1 Alternatively, particles in the digestate may be minimized by allowing the sample to settle before analysis.
- 11.24 Sample preparation is complete. Store digestates in designated cabinet. Print out the load sheet, which is then given to the analyst.
- 11.25 Hot Acid Prep for Antimony
- 11.25.1 Label digestion vessel with sample ID or QC identifier.
- 11.25.2 Homogenize the sample by mixing thoroughly. See SOP ST-QA-0038 for details.
- 11.25.3 Weigh a 0.5 g – 0.6g portion of sample and record the weight on in LIMS.
- 11.25.4 Weigh out 0.5g - 0.6g boiling chips for MB and LCS and record the weight in LIMS
- 11.25.5 Spike the LCS/MS/MSD with spiking mix applicable to the requested analysis.
- 11.25.5.1. Document spiking volumes and standard numbers in LIMS
- 11.25.6 Add 2.5 mL of HNO_3 and 2.5 mL of HCL.
- 11.25.7 Place digestion vessels in the hot block at 90-95°C and heat for 15 minutes.
- 11.25.8 Filter through vacuum filter while samples are still hot.
- 11.25.9 Rinse filter with 1.2 mL of hot HCL.
- 11.25.10 Rinse filter with 5 mL of hot DI water 4 times for a total of 20 mL.
- 11.25.11 Rinse the sample debris off the filter paper and place back into original digestion vessel.
- 11.25.12 Add 2.5 mL of HCL.
- 11.25.13 Return vessels to hot block.
- 11.25.14 Place watch glasses on the digestion vessels.
- 11.25.15 Reflux at 90-95°C for 20 minutes.
- 11.25.16 Filter while still hot through vacuum filter, adding original filtrate.
- 11.25.17 Rinse 3 times with DI water.
- 11.25.18 Bring samples to a final volume of 50 mL with DI water.
- 11.25.19 Sample preparation is complete. Store digestates in designated cabinet. Print out the load sheet, which is then given to the analyst.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 Specific calculations are included in the respective analytical SOPs: ST-MT-0001 (ICP/MS) and ST-MT-0003 (ICP).

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 Data assessment, acceptance criteria and corrective actions are included in the respective analytical

SOPs: ST-MT-0001 (ICP/MS) and ST-MT-0003 (ICP).

- 13.2 Data assessment does not pertain to this sample preparation procedure.
- 13.3 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036.

14.0 METHOD PERFORMANCE AND DEMONSTRATIONS OF CAPABILITY

- 14.1 Method performance data, reporting limits, and QC acceptance limits, are maintained in LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
 - 16.2.1 The following waste streams are produced when this method is carried out.
 - 16.2.2 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
 - 16.2.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 3050B.
- 17.2 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision

- 17.3 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions
- 17.4 Associated SOPs, current revisions
 - 17.4.1 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.4.2 ST-QA-0002, Standard and Reagent Preparation
 - 17.4.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
 - 17.4.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
 - 17.4.5 ST-QA-0038, Procedure for Compositing and Subsampling
 - 17.4.6 ST-RC-0125 Determination of Technetium-99 Using Eichrom Teva Resin
 - 17.4.7 ST-MT-0001, Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry
 - 17.4.8 ST-MT-0003, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method for Trace Element Analysis
 - 17.4.9 ST-IP-0015, Filtration Procedure

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCED METHOD

- 18.1 TestAmerica St. Louis uses a 0.5g sample aliquot size instead of the 1g aliquot in the method. TestAmerica St. Louis can achieve the necessary detections without using such a large sample volume.
- 18.2 Additionally, sample reagent volumes needed for digestion have been reduced to reflect the reduction in sample volume.
- 18.3 HCL is used for both ICP and ICPMS analysis. Our ICPMS has collision cell technology, which reduces the interference caused by chloride.
- 18.4 The final acid concentration in the digestate is 5% HCL and 5%HNO3 (v/v %).

19.0 CHANGES TO PREVIOUS REVISION

- 19.1 Glass beads added to equipment (Section 6)
- 19.2 Balance capability and pipette volumes changed in section 6.
- 19.3 Additional information added to LCS composition in section 9.3.
- 19.4 Plunger removed from section 11.21 due to zinc contamination.
- 19.5 Blank & LCS composition updated in section 11.23.
- 19.6 Grammatical corrections.
- 19.7 Rev. 13; updated the Hydrogen peroxide from reagent grade to ultrex grade in section 7.0.
- 19.8 Rev. 13; copied 9.3.3.2 and added it to the end of section 9.2.
- 19.9 Rev. 13; Updated the use of glass beads in section 9.2 and cause of contamination issues in section 9.3.3.2.
- 19.10 Rev. 13; Adjusted reagent volumes used to reflect sample volume used in section 11.3.3.2.
- 19.11 Rev 14:
 - 19.11.1 Removed filter step from section 2.1
 - 19.11.2 Added HF acid to safety table in Section 5
 - 19.11.3 Added TC-99 soil prep equipment to Section 6
 - 19.11.4 Added TC-99 soil prep reagents and standards to Section 7.
 - 19.11.5 Added soil prep for TC-99, section 11.24
 - 19.11.6 Added Eichrom method references to Section 17.
- 19.12 Rev 15:
 - 19.12.1 Annual Review, No Changes.
- 19.13 Rev 16:
 - 19.13.1 Removed Technetium 99 tracer prep procedure from section 7.0 replaced Tc-99m with

- rhenum as tracer.
- 19.13.2 Updated solid sample preparation for Technetium 99 by ICPMS in 11.24.
- 19.13.3 Removed references to Q'tims – old laboratory LIMS system.
- 19.14 Rev. 17:
 - 19.14.1 Grammatical corrections throughout
 - 19.14.2 Removed Tc99 soil prep (and all related references) . Referenced the Rad SOP.
 - 19.14.3 Added procedure for insufficient sample for batch QC, section 9.4.4
 - 19.14.4 Added sample duplicate only done by client request in section 9.5.2
 - 19.14.5 Clarified through out the SOP that references to a digestion log was referring to LIMS, not an actual log book
 - 19.14.6 Changed sample volume in section 11.3
 - 19.14.7 Updated section 11.25 (Hot Antimony Prep) to make it resemble the prep steps in our normal prep.
- 19.15 Rev. 18: (2/27/2015)
 - 19.15.1 Replace references to glass beads with boiling chips
 - 19.15.2 Updated 11.3.1 (added at client request, and that reagents would be adjusted accordingly)
 - 19.15.3 Added modifications to section 18
 - 19.15.4 Updated References
- 19.16 Rev. 19: (7/25/2016)
 - 19.16.1 Minor clarifications made throughout
 - 19.16.2 Filtration is now the normal procedure, not the alternative.
 - 19.16.3 Added notation that if additional reagents are added to samples, they must also be added to the MB and LCS
 - 19.16.4 Specified Ultrex grade for Nb, TA and Sn.

**Title: ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS
FOR TOTAL METALS FOR ANALYSIS BY ICP OR ICP/MS
[SW-846 3010A; EPA 200.7, 200.8]**

Approvals (Signature/Date):

Kristen Ely 8/11/16
Kristen Ely Date
Inorganics Manager

Michael J. Ridenhower 8/12/16
Michael Ridenhower Date
Health & Safety Manager / Coordinator

Marti Ward 8/12/16
Marti Ward Date
Quality Assurance Manager

Elaine Wild 8/11/16
Elaine Wild Date
Laboratory Director

This SOP was previously identified as SOP No. ST-IP-0013 Rev. 21

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1.0 SCOPE AND APPLICATION

- 1.1 This Standard Operating Procedure is applicable to the preparation of digestates from aqueous samples, including SPLP, CWET and TCLP extracts, and wastes that contain suspended solids, for subsequent analysis by ICP (Inductively Coupled Argon Plasma Spectroscopy) or ICP/MS (Inductively Coupled Argon Plasma Mass Spectroscopy) for total metals.
- 1.2 This SOP is based on SW846 Method 3010A, EPA Method 200.7 and EPA Method 200.8.
- 1.3 Additional metals may be processed by this method, assuming that performance criteria of the determinative method are met.
- 1.4 It is mandatory that Clean Water Act (CWA) samples be digested per this procedure prior to analysis.
- 1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1 Water samples are refluxed with nitric acid in a hot block vessel until the digestate is light in color. The digestate is reduced to a low volume and further refluxed with hydrochloric acid. The cooled digestate is brought to a known volume for analysis by ICP or ICP/MS.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Laboratory Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Total Metals: The concentration of target metals determined on an unfiltered sample after vigorous acid digestion. (samples may be filtered AFTER digestion)
- 3.3 Dissolved Metals: Those elements which pass through a 0.45 µm membrane filter (Sample is filtered BEFORE acidification and digestion))

4.0 INTERFERENCES

- 4.1 Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc). Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.
- 4.2 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm- (TWA) 4 ppm- (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm- (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

6.1 Hot block or other heating source capable of maintaining a temperature of 90 to 95 °C.

6.2 Laboratory fume hood.

6.3 Thermometer, temperature range of 0 – 200 °C

6.4 Adjustable pipettes (0.1-1mL)

6.5 Adjustable Reagent Dispensers

6.6 50-mL hot block digestion vessels (Class A)

6.7 Watch glasses

- 6.8 Vacuum pump
- 6.9 Vacuum manifold
- 6.10 0.45 µm vacuum filter units (with plugs)

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 DI water Type 1: Obtained by the Milli-Q System
- 7.3 Nitric acid, concentrated, trace metal grade or better.
- 7.4 Hydrochloric acid, concentrated, trace metal grade or better
- 7.5 LCS standard, NIST traceable
- 7.6 Matrix Spike standard, NIST traceable

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002. Aqueous samples must be acidified at the time of collection to a pH of 2 or less with nitric acid.
- 8.2 If samples are preserved by the lab, the samples must sit 24 hours before digestion.
- 8.3 Sample containers may be either plastic or glass, and must be pre-cleaned by vendor.
- 8.4 Sample digestion and analysis must be completed within six months (180 days) of sample collection.
- 8.5 Leachates must be digested and analyzed within six months (180 days) of the beginning of the leaching.

9.0 QUALITY CONTROL

- 9.1 **Batch**
 - 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
 - 9.1.2 Instrument/equipment conditions must be the same for all standards, samples and QC samples.
 - 9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and a Matrix Spike/ Matrix Spike Duplicate (MS/MSD).). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Sample Duplicate (DU) may be performed upon client request, and are noted in LIMS.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.2.1 For Water analyses, the method blank is comprised of a 2% HNO₃ solution.

9.3 **Laboratory Control Sample**

9.3.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.2.1 For Water analyses, the LCS is comprised of a 2% HNO₃ solution, fortified with target analytes.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 For **6010B, 6010C, 6010D, 6020, 6020A and 6020B**:

9.4.3.1 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.4.4 For **200.7 and 200.8**:

9.4.4.1 MS/MSD are performed every 10 samples or per batch whichever is shorter.

9.4.5 If there is insufficient sample to perform a MS/MSD, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume.

9.5 **Sample Duplicate (DU)**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.

9.5.3 A Sample Duplicate is only performed when client requested.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Hot block must be checked daily when in use.

10.1.1 Temperature is documented in the LIMS.

- 10.1.2 A calibrated thermometer is suspended in sand in a digestion vessel of water and brought to the proper temperature.
 - 10.1.2.1 See SOP ST-QA-0005 for information regarding calibration of thermometers.
- 10.2 Instrument calibration is discussed in the respective analytical SOPs: ST-MT-0001 (ICP/MS), and ST-MT-0003 (ICP).

11.0 PROCEDURE

- 11.1 For Dissolved metals, the sample must be filtered BEFORE preservation
 - 11.1.1 See SOP ST-IP-0015 (Filtration Procedure)
- 11.2 For Total metals, the sample must be preserved before digestion. See section 8.0
- 11.3 Label digestion vessel with sample ID or QC identifier.
- 11.4 Transfer 50 mL of well-mixed sample to a digestion vessel.
 - 11.4.1 If a smaller aliquots of sample is used the applicable reagents are reduced accordingly. Alternatively the sample may require dilutions to reduce interferences. If the sample is diluted during the digestion bring total volume to 50 mL with Type 1 DI water prior to adding the reagents.
 - 11.4.2 For the MB and LCS, use 50 mL of 2% HNO₃ solution.
 - 11.4.3 Spike LCS and MS/MSD with spiking mix applicable to the requested analysis.
 - 11.4.4 Document spiking volumes and standard numbers in LIMS
- 11.5 When preparing TCLP leachate samples, aliquot 20 mL of leachate and dilute to 50 mL with DI, unless client project requirements specify no dilution.
 - 11.5.1 Note: this dilution applies to all QC samples.
- 11.6 When preparing CWET samples, aliquot 10 mL of leachate and dilute to 50 mL with DI, unless client project requirements specify no dilution.
 - 11.6.1 Note: this dilution applies to all QC samples.
- 11.7 When preparing SPLPE & SPLPW transfer 50 mL of well mixed sample to a digestion vessel
- 11.8 Digestion
 - 11.8.1 Add 2.5 mL of concentrated nitric acid..
 - 11.8.2 Place watch glasses on top of all digestion vessels.
 - 11.8.3 Place the digestion vessel in a hot block at 90 °C – 95 °C and evaporate, without boiling , for approximately 10 to 12 hrs.
 - 11.8.3.1 Note: do not allow sample to go to dryness, as loss of target metals may result.
 - 11.8.3.2 If the sample changes color with the addition of nitric acid, repeat the addition of acid and refluxing until digestion is complete, which is indicated by the appearance of a light colored liquid which does not change with continue refluxing.
 - 11.8.4 Cool vessel.
 - 11.8.5 Add 5.0 mL of 1:1 HCl to the digestate, and place back in hot block for an additional 15 minutes.
 - 11.8.6 Cool the samples; wash down the vessel wall with DI water to dissolve any precipitation to a final volume 50 mL.

- 11.8.7 Cap and mix sample thoroughly and allow too settle.
- 11.8.8 For some samples (such as those with large amounts of sediment) filtration may be needed before analysis
 - 11.8.8.1 NOTE: The MB and LCS must also be filtered
 - 11.8.8.2 Label clean digestion tubes
 - 11.8.8.3 Attach filter to the sample digestion tube. Attach the clean, labeled digestion tube to the other side of the filter.
 - 11.8.8.4 Attach vacuum manifold to the vacuum pump, turn on the pump.
 - 11.8.8.5 Either use finger or place plug over hole in filter
 - 11.8.8.6 Invert, and attach to the vacuum manifold
 - 11.8.8.7 Once all liquid has gone through, turn off the vacuum and remove the sample.
 - 11.8.8.8 Cap the sample
- 11.8.9 Sample preparation is complete. Store digestates in designated cabinet, and transfer the load sheet to the analyst.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the ST-QAM.
- 12.2 Specific calculations are included in the respective analytical SOPs: ST-MT-0001 (ICP/MS), and ST-MT-0003 (ICP).

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 Data assessment, acceptance criteria and corrective actions are included in the respective analytical SOPs: ST-MT-0001 (ICP/MS) and ST-MT-0003 (ICP).
- 13.2 Data assessment does not pertain to this sample preparation procedure.
- 13.3 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036.

14.0 METHOD PERFORMANCE AND DEMONSTRATIONS OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in ST-QAM .
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in ST-QAM.

- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
- 16.2.1 The following waste streams are produced when this method is carried out.
- 16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A"
- 16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, 3rd Edition Revision 1, July, 1992, Method 3010A & 3005A.
- 17.2 EPA Method 200.7, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry," Revision 4.4.
- 17.3 EPA Method 200.8, "Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry", Revision 5.4.
- 17.4 TestAmerica St. Louis Laboratory Quality Assurance Manual (ST-QAM), current revision
- 17.5 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.6 Associated SOPs, current revisions:
- 17.6.1 ST-PM-0002, Sample Receipt and Chain of Custody
- 17.6.2 ST-QA-0002, Standard and Reagent Preparation
- 17.6.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
- 17.6.4 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
- 17.6.5 ST-QA-0016, IDL/MDL Determination
- 17.6.6 ST-QA-0036, Non-conformance Memorandum (NCM) Process

- 17.6.7 ST-IP-0004, Labware Preparation for Inorganic and Trace Metal Analysis
- 17.6.8 ST-IP-0015, Filtration Procedure
- 17.6.9 ST-MT-0001, Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry
- 17.6.10 ST-MT-0003, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method for Trace Element Analysis

18.0 MODIFICATIONS TO THE REFERENCED METHODMETHOD

- 18.1 3010A
 - 18.1.1 TestAmerica St. Louis uses a 50 mL sample aliquot size instead of the 100 mL aliquot in the method. TestAmerica St. Louis can achieve the necessary detections without using such a large sample volume.
 - 18.1.2 Additionally, sample reagent volumes needed for digestion have been reduced to reflect the reduction in sample volume.
 - 18.1.3 Acid concentrations have been modified to optimize recoveries and to combine prep methods. The final acid concentration in the digestate is 5% HCL and 5% HNO₃ (v/v%):
- 18.2 200.7, 200.8
 - 18.2.1 TestAmerica St. Louis uses a 50 mL sample aliquot size instead of the 100 mL aliquot in the method. TestAmerica St. Louis can achieve the necessary detections without using such a large sample volume.
 - 18.2.2 The method allows direct analysis, if the sample is a drinking water, and the turbidity of the sample is < 1NTU. TestAmerica St. Louis digests all samples, unless requested otherwise by the client
 - 18.2.3 Acid concentrations have been modified to optimize recoveries and to combine prep methods. The final acid concentration in the digestate is 5% HCL and 5% HNO₃ (v/v%):
 - 18.2.4 The method specifies the use of a hot plate, and that an uncovered Griffin beaker containing 50mL of water should be maintained at a temperature approximately to, but no higher than 85°C. But once the beaker is covered with a watch glass the temperature of the water will rise to approximately 95°C. TestAmerica St. Louis measures the temperature in a closed container, and so uses a range of 90-95°C.

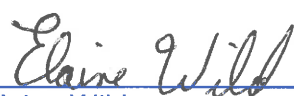
19.0 CHANGES TO PREVIOUS REVISION

- 19.1 Removed Plunger Filter from section 6.0.
- 19.2 Up dated section 11.10 regarding the use of plunger filters.
- 19.3 Rev 17:
 - 19.3.1 Updated digestion time for sample to evaporate in section 11.7.
 - 19.3.2 Updated where spiking volumes records to be stored in section 11.2.4.
 - 19.3.3 Updated pipette and reagent dispenser range used in section 6.0.
- 19.4 Revision 18:
 - 19.4.1 Updated matrix for method blank and LCS for Au, Ta and Pd in section 9 and 11
- 19.5 Revision 19:
 - 19.5.1 Corrected digestate cleaning method in section 8.2
 - 19.5.2 Removed reference to spiking concentrations in section 11.5.
 - 19.5.3 Updated section 15.1
 - 19.5.4 Updated QAM reference in Section 14.
- 19.6 Revision 20
 - 19.6.1 Changed starting matrix for MB and LCS, from DI to 2% HNO₃
 - 19.6.2 Changed final acid concentration, from 3% HNO₃/2%HCL, to 5%HNO₃/5%HCL
 - 19.6.3 Added modifications to section 18, clarified which method was being modified.

- 19.6.4 Removed Table 1 (DOE low level spike)- not reference in SOP, levels can be found in LIMS.
- 19.6.5 Added reference to dissolved metals
- 19.6.6 Added reference to filtration procedure
- 19.7 Revision 21 (08/28/2015)
 - 19.7.1 Grammatical corrections throughout
 - 19.7.2 Section 11: removed reference to low level TCLP digestion.
- 19.8 Rev. 22 (08/12/2016)
 - 19.8.1 Made clarifications throughout
 - 19.8.2 added vacuum manifold to supplies
 - 19.8.3 added use of watch glasses to Section 11
 - 19.8.4 added filtration procedure for samples that have already been digested
 - 19.8.5 added reference to methods 6010D and 6020B

**Title: ANALYSIS OF NITROAROMATIC AND NITROAMINE
EXPLOSIVES BY HPLC
[SW846 8000C and 8330B]**

Approvals (Signature/Date):

 Aaron Dickson Operations Manager	<u>8/3/16</u> Date	 Michael Ridenhower Health & Safety Manager / Coordinator	<u>8/3/16</u> Date
 Marti Ward Quality Assurance Manager	<u>8-3-16</u> Date	 Elaine Wild Laboratory Director	<u>8/3/16</u> Date

This SOP was previously identified as SOP No. ST-LC-0002 Rev. 20

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1.0 SCOPE AND APPLICATION

- 1.1 This procedure provides instructions for the analysis of nitroaromatic and nitramine explosives by high performance liquid chromatography (HPLC) with UV detection in water, soil and sediment samples.
- 1.2 This SOP uses Low-level solid phase extraction method 3535A for low concentration water samples and the sonication extraction method 3550C or a shaker table (8330B) for soil, sediment, and wipe samples.
- 1.3 This SOP is based on EPA SW-846 Method 8000C and 8330B.
- 1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.4.1 Additional compounds may be amenable to this procedure. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument. When an MDL study can not be conducted, the MDL is set equal to the reporting limit.

2.0 SUMMARY OF METHOD

- 2.1 After the initial preparation step, (SOP ST-OP-0008) the sample is introduced to the HPLC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Analysis on a second column provides confirmation of the identification of the target analyte, as does spectra identification, if client requested.

3.0 DEFINITIONS

- 3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

- 4.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks.
- 4.2 2,4-DNT and 2,6-DNT elute at similar retention times (retention time difference of ~0.2 minutes). A large concentration of one isomer may mask the response of the other isomer. If these isomers cannot be resolved, an isomeric mixture should be reported.
- 4.3 Meta and para-nitrotoluene co-elute on the confirmation column.
- 4.4 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain Tetryl should be diluted with acetonitrile prior to filtration. SAMPLES EXPECTED TO CONTAIN TETRYL SHOULD NOT BE EXPOSED TO TEMPERATURES ABOVE ROOM TEMPERATURE
- 4.5 Degradation products of Tetryl appear as a shoulder on the 2,4,6-TNT peak. Tetryl has also been observed to degrade into 4-amino-2,6-dinitrotoluene.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Primary Materials Used

5.2.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (Methyl Alcohol)	Poison Flammable	200 ppm (TWA)	Inhalation symptoms may include headache, drowsiness, nausea, vomiting, blurred vision and blindness. Symptoms of skin contact may parallel inhalation exposure and can cause skin to become dry and cracked. Eye contact can cause irritation.
Acetonitrile	Flammable Poison	40 ppm (TWA)	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 HPLC system: The lab utilizes two HPLC systems, one Agilent 1100 and one Agilent 1200. Both HPLC equipped with Quaternary pump and DAD (Diode array detector). Chemstation software collects data which is transferred to Chrom server. The data then is automatically processed and is uploaded to the LIMS through Chrom after data is reviewed.

6.2 Restek, Ultra C-18 5um (250 x 4.6mm) [primary/quantitative column].

6.3

6.4 Restek, Ultra BiPh 5um (150 x 4.6 mm) [confirmation column]

6.5 High pressure injection syringes, 10-μL, 25-μL, 50-μL, 100-μL, 500-μL, 1000-μL, Hamilton liquid syringe or equivalent

6.6 Volumetric flasks, 10-mL, 100-mL, 1000-mL with screw tops

6.7 Wipes- 4-ply rayon/polyester 4"x4" Avant Gauze PRM25444

6.8 Sand – reagent grade

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7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 HPLC grade water
- 7.3 Acetonitrile, HPLC grade
- 7.4 Methanol, HPLC grade
- 7.5 HPLC Mobile Phases. The following solutions:
 - 7.5.1 HPLC Primary Mobile Phase: 55 ($\pm$ 2%) /45 ($\pm$ 2%) methanol/water at 1.0 mL/min [isocratic], UV detection at 254 nm. Note: The 215 nm wavelength is also monitored for nitroglycerin and PETN, if applicable. Special analytes may be detected at other wavelengths.
 - 7.5.2 HPLC Confirmation Mobile Phase: 40/60 methanol/water at 1.0 ($\pm$ 0.4) mL/min., UV detection at 254 nm. Note: The 215 nm wavelength is also monitored for nitroglycerin and PETN, if applicable.
- 7.6 Stock Standards
 - 7.6.1 It is recommended that diluted standards be purchased as stock solutions with a concentration no greater than 1000 mg/L (ppm). Standard solutions may also be prepared using Standard Analytical Reference Materials (SARMs). **Before neat materials are to be used, contact the EH&S Coordinator immediately for revision of this procedure.** IF USING NEAT STANDARDS PLEASE NOTE: HMX, RDX, Tetryl, and 2,4,6-TNT are explosives and the neat material must be handled carefully. See section 5.0 Safety, for guidance. Drying at ambient temperature requires several days. DO NOT DRY AT HEATED TEMPERATURES!
 - 7.6.1.1 Place about 10 mg (weighed to the nearest 0.1 mg) of a single analyte into a 10-mL volumetric flask and dilute to volume with acetonitrile. Invert the flask several times until dissolved. Calculate the concentration of the stock solution from the actual weight used (nominal concentration = 1,000 mg/L).
 - 7.6.1.2 Store at $< 6^{\circ}\text{C}$ and protect from light.
 - 7.6.1.3 Stock standards must be replaced after **six months** or sooner if comparison with quality control samples indicates a problem.
 - 7.6.1.4 Standard solutions may also be purchased as prepared certified solutions. The vendor specified expiration dates will apply until ampoules are opened and mix is used.
 - 7.6.2 Intermediate Stock Standard Solutions
 - 7.6.2.1 Prepare an intermediate stock standard solution at 2000 ug/L for low level and 20,000 for high level per analyte in acetonitrile.
NOTE: If the analytical column cannot achieve separation of 2,4-DNT and 2,6-DNT, it will be necessary to prepare a separate intermediate stock solution of 2,6-DNT.
 - 7.6.2.2 Intermediate stock standards must be replaced after **30 days** or sooner if comparison with quality control samples indicates a problem.
 - 7.6.3 Calibration Standards
 - 7.6.3.1 Calibration standards, at a minimum of five concentration levels, are prepared through dilution of the intermediate stock standard solutions by 50% (v/v) with 500 microliters HPLC Grade Water. One of the concentration levels should be at or below the reporting limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the

working range of the HPLC. See Table 2 for suggested calibration standard concentrations.

- 7.6.3.2 Calibration standards must be stored at a temperature less than zero degrees C at ($< 0^{\circ}\text{C}$) and stored in the dark, and prepared fresh on the day of calibration.
- 7.6.4 Surrogate Standards
 - 7.6.4.1 1,2-Dinitrobenzene is the surrogate typically used for this method. The surrogate standards are prepared and stored in the same way as the calibration standards.
 - 7.6.4.2 Table 2 lists suggested concentrations for surrogate standards for initial calibration.
 - 7.6.4.3 The working surrogate mix concentration is $40\text{ }\mu\text{g/mL}$.
- 7.6.5 Quality Control (QC) Standards
 - 7.6.5.1 Calibration verification standards (second source verification standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independently prepared from that used for the calibration standards.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Aqueous samples should be collected in 1-L glass bottles and soil samples should be collected in 4-oz glass jars. Samples are protected from light and stored at $4 \pm 2^{\circ}\text{C}$. Sample extracts are stored at a temperature $< 0^{\circ}\text{C}$, protected from light and isolated from all potential contaminants and all standards.
- 8.3 Holding Times
 - 8.3.1 Extraction is initiated within 7 days of the sampling date for aqueous samples, and 14 days of the collection date for solid and waste samples.
 - 8.3.2 In the case, where drying/homogenization techniques and/or incremental sampling techniques are employed, the hold time may be assessed from when the composited sample is created, in the field or in the laboratory. Homogenization and incremental sampling techniques are diverse and vary depending on the nature of the material, methodologies and project objectives. Please reference the project's Client Requirements Memorandum regarding compositing and hold times.

9.0 QUALITY CONTROL

- 9.1 **Batch**
 - 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
 - 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
 - 9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.
 - 9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.
- 9.2 **Method Blank (MB)**
 - 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure. DI water is used for water samples, reagent sand for soils and solids, and Avant Gauze wipe for wipes.
 - 9.2.2 A method blank must be prepared with every sample batch.

9.3 **Laboratory Control Sample (LCS)**

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. DI water is used for water samples, reagent sand for soils and solids, and Avant Gauze wipe for wipes.
- 9.3.2 An LCS must be prepared with every sample batch.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5 **Surrogate**

- 9.5.1 A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.
- 9.5.2 Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.

9.6 **Instrument Blanks**

- 9.6.1 An instrument blank (consisting of 1:1 acetonitrile to 5 g/L calcium chloride solution) is periodically analyzed to assess instrument performance.

9.7 **Procedural Variations/ Nonconformance and Corrective Action**

- 9.7.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.7.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 **Initial Calibration**

- 10.1.1 External standard calibration is used. Prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in Table 2.
- 10.1.1.1 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., UV lamp replacement).
- 10.1.1.2 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the corporate policy, "Selection of Calibration Points."
- 10.1.2 SW-846 chromatographic methods allow the use of both linear and non-linear models for the calibration data.
- 10.1.2.1 The first way is to begin with the simplest approach, the linear model through the origin, and then progress through other options until the calibration acceptance criteria are met. The second way is to use technical knowledge of the detector response to the target compound to choose the calibration model.
- 10.1.2.2 The option for non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance.

- 10.1.3 **Linear calibration using the average response factor**
 - 10.1.3.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 20\%$ for each target analyte.
 - 10.1.3.2 If the %RSDs in the initial calibration is $> 20\%$, then calibration using a linear regression may be employed.
- 10.1.4 **Linear calibration using a least squares regression**
 - 10.1.4.1 The intercept of the curve at zero response must be less than $\pm$ the reporting limit for the analyte.
 - 10.1.4.2 r (correlation coefficient) must be ≥ 0.995 **OR** r^2 (coefficient of difference) must be ≥ 0.990 .
 - 10.1.4.3 **Linear calibration using a least squares regression, forcing thru zero**
 - 10.1.4.3.1 Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 *before* the regression is calculated, thereby setting the bias to favor the low end of the calibration range by “pivoting” the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression through zero for some calibrations.
 - 10.1.4.3.2 Curve must still meet criteria in 10.4.3.1 and 10.4.3.2.
 - 10.1.4.4 **Linear calibration using a least squares regression, weighting of data points**
 - 10.1.4.4.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. For this reason it may preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/x^2$ weighting) to improve accuracy at the low end of the curve.
 - 10.1.4.4.2 Curve must still meet criteria in 10.4.3.1 and 10.4.3.2.
- 10.1.5 **Non-linear calibration**
 - 10.1.5.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches have not met the acceptance criteria, a non-linear calibration model may be employed.
 - 10.1.5.1.1 It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance. Thus, non-linear calibrations are not be employed for analytes shown to consistently exhibit linear calibration for the analytes of interest.
 - 10.1.5.2 The intercept of the curve at zero response must be less than $\pm$ the reporting limit for the analyte.
 - 10.1.5.3 r (correlation coefficient) must be ≥ 0.995 **OR** r^2 (coefficient of difference) must be ≥ 0.990 .
 - 10.1.5.4 a quadratic calibration curve requires six standards.
- 10.2 Initial Calibration Verification (ICV)
 - 10.2.1 An initial calibration verification standard must be analyzed before any samples are analyzed for the initial calibration curve. The value calculated is compared to the expected value and should meet the 30% difference criteria or client requirements before the samples are analyzed. The ICV standard is a separate/second source standard.
 - 10.2.2 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

- 10.3 Continuing Calibration Verification.
- 10.3.1 Analyte response factors must be verified at the beginning of each, after every 10 samples, and at the end of the analysis sequence through the analysis of a mid-level calibration standard.
- Any individual compounds with %D $\leq$ 20% meet the calibration criteria.
 - It is not necessary to run a calibration standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.
 - If highly contaminated samples are expected, it is acceptable to analyze blanks or rinses at any point in the run.
- 10.3.2 Corrective Actions for Continuing Calibration: If the %D for any analyte is $>$ 20% corrective action must be taken. This may include minor instrument adjustments, reconditioning or stabilizing the HPLC system, followed by reanalyzing the standard. If the %D is still unacceptable, a new calibration curve must be prepared.
- 10.3.3 Corrective Action for Samples: Any samples which are not bracketed by compliant continuing calibration standards must be re-injected.
- 10.4 Retention Time Windows
- 10.4.1 Retention Time Windows:
- 10.4.1.1 Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three day period. Calculate the standard deviation of the three retention times for each analyte. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.
- 10.4.1.2 The centers of the windows are updated with the midpoint standard of the initial calibration or the opening daily CCV.
- 10.4.1.3 If the retention time window as calculated above is less than $\pm$ 0.2 minutes, use $\pm$ 0.2 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.
- 10.4.1.4 The laboratory must calculate new retention time windows each time a new column is installed. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.
- 10.4.2 Corrective Action for Retention Times
- 10.4.2.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed.

11.0 PROCEDURE

- 11.1 Sample Introduction
- 11.1.1 Add 500 μ L Water HPLC Grade to 500 μ L sample extract and QC extract.
- 11.1.2 Allow extracts to warm to ambient temperature before injection.
- 11.1.3 Samples are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.
- 11.2 Perform all qualitative and quantitative measurements.
- 11.3 Return extract to freezer when not in use.
- 11.3.1 Once HPLC Grade water addition is made to the extract, and the extract is frozen, the extract is no longer suitable for analysis. If reanalysis is needed, a fresh extract aliquot must be taken through 11.1.1 – 11.1.3.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Qualitative Identification

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- 12.1.1 Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the Reporting Limit (RL).
- 12.1.2 The primary column is used normally used for reporting.
 - 12.1.2.1 When sample results are confirmed using two dissimilar columns or with two dissimilar detectors, the agreement between the quantitative results should be evaluated after the identification has been confirmed. Large differences in the numerical results from the two analyses may be indicative of positive interferences with the higher of the results which could result from poor separation of target analytes, or the presence of a non-target compound. However, they may also result from other causes.
 - 12.1.2.2 Calculate the RPD between the two concentrations using the formula below.

$$RPD = \frac{(\text{The difference between C1 and C2})}{(\text{the average of C1 and C2})} \times 100$$

where C1 and C2 are concentrations on the two columns

- 12.1.2.3 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. A rising baseline may cause the incorrect integration of the peak for the lower result
 - 12.1.2.4 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, it may be appropriate to report the lower result. Regardless of the presence or absence of chromatographic problems, the data user must be advised of the disparity between the two results. Data will be flagged and an NCM written..
 - 12.1.3 If requested, the laboratory will report confirmed analyte levels which are calculated to be below the RL but above the laboratory MDL. The data for these hits will be flagged to indicate that the value is estimated.
 - 12.1.4 The confirmation column, when used, meets all the same QC requirements as the Primary column.

12.2 Calibration Range

- 12.2.1 If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50%) of the calibration range.

12.3 Dilutions

- 12.3.1 Samples may be screened to determine the appropriate dilution for the initial run.
- 12.3.2 Reporting Dilutions
The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12.4 Interferences

- 12.4.1 If peak detection is prevented by interferences, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5 Calculations are performed using Chrom.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.
- 13.2 Method Blank
 - 13.2.1 No target analytes may be present in the method blank above the reporting limit.
 - 13.2.2 The method blank must have acceptable surrogate recoveries.
 - 13.2.3 Corrective Action for Method Blanks not meeting acceptance criteria:
 - 13.2.3.1 Method Blank Contamination – Blank contamination above the RL ($>1/2$ RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are $< RL$ or greater than 10 times the amount detected in the method blank.
 - 13.2.3.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM
- 13.3 Laboratory Control Sample (LCS)
 - 13.3.1 Acceptance Criteria:
 - 13.3.1.1 All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).
 - 13.3.1.1.1 For long analyte spike list, marginal exceedances (ME) are allowed as follows:
 - 13.3.1.1.2 < 11 analytes in LCS, no analytes allowed in ME of the LCS control limit.
 - 13.3.1.1.3 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit.
 - 13.3.1.1.4 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit.
 - 13.3.1.1.5 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit.
 - 13.3.1.1.6 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit.
 - 13.3.1.1.7 > 90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit.
 - 13.3.1.1.8 No LCS recoveries may be outside the Marginal Exceedance limit.
 - 13.3.1.1.9 Marginal exceedances must be random. If the same LCS analyte exceeds the control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.
 - 13.3.1.2 The LCS should have acceptable surrogate recoveries.
 - 13.3.1.3 Corrective Action for LCS not meeting acceptance criteria:
 - 13.3.1.3.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM
 - 13.3.1.3.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.
 - 13.3.1.3.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.
 - 13.3.1.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM.

- 13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
 - 13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).
 - 13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:
 - 13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.
- 13.5 Surrogate
 - 13.6.1 All Surrogates should be within established control limits for accuracy (%Recovery).
 - 13.6.2 Corrective Action for Surrogate not meeting acceptance criteria:
 - 13.6.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration. In cases where the surrogate recovery is high and the associated analytes in the sample are non-detect, the data may be reported with an NCM. Samples with low surrogate recovery require re-prep and re-analysis.
- 13.6 Sample result evaluation
 - 13.6.1 Dilutions
 - 13.6.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
 - 13.6.1.1.1 Dilution: Sample– An NCM is written to document the reason for the dilution.
 - 13.6.1.1.2 Dilution: Internal Standard diluted out– An NCM is written to document the reason for the dilution
 - 13.6.1.1.3 Dilution: Internal Standard and/or Spike(s) diluted out An NCM is written to document the reason for the dilution.
 - 13.6.2 Carryover
 - 13.6.2.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
 - 13.6.2.2 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.
 - 13.6.2.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.
- 13.7 Insufficient Sample
 - 13.7.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, maintained in LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
 - 16.2.1 The following waste streams are produced when this method is carried out.
 - 16.2.1.1 Solvent and water from the HPLC. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".
 - 16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, January 1998, Method 8330A.
- 17.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, October 2006, Method 8330B.
- 17.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, December 1996, Section 8000C.
- 17.4 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, November 1998, Method 3535A.
- 17.5 TestAmerica Quality Assurance Manual (ST-QAM), current revision
- 17.6 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.7 TestAmerica Policy CA-Q-S-001, Acceptable Manual Integration Practices
- 17.8 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
- 17.9 Associated SOPs, current revisions
 - 17.9.1 ST-QA-0002, Standard and Reagent Preparation
 - 17.9.2 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.9.3 ST-QA-0016, IDL/MDL Determination
 - 17.9.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process

- 17.9.5 ST-QA-0038 Procedure for Compositing and Subsampling
- 17.9.6 ST-PM-0002 Sample Receipt and Chain of Custody
- 17.9.7 ST-OP-0008 Extraction of Nitroaromatic and Nitroamine Explosives for Waters and Soils, Based on SW-846 3500 Series Methods

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 None

19.0 CHANGES TO PREVIOUS SOP REVISION

- 19.1 Rev. 12 No Changes, Annual Review.
- 19.2 Revision 13:
 - 19.2.1 Section 6.1, changed the number of Agilent pieces of equipment used.
 - 19.2.2 Section 12.1.3.1, changed the qualifier assigned to "S".
- 19.3 Revision 14:
 - 19.3.1 Updated the number of HPLC systems used in section 6.1.
 - 19.3.2 Updated the use of Phenomenex Synergi Polar columns in section 6.4.
- 19.4 Revision 15:
 - 19.4.1 Updated the steps for preparing low level intermediate stock standard in section 7.7.2.
 - 19.4.2 Updated the amount of calcium chloride solution needed to make QC extract in section 11.0.
 - 19.4.3 Updated grammatical errors.
- 19.5 Revision 16:
 - 19.5.1 Inserted requirement for quadratic curve for non-linear calibrations in section 10.1.
 - 19.5.2 Updated information regarding retention times in section 10.4.
- 19.6 Revision 17:
 - 19.6.1 Grammatical and formatting corrections through out
 - 19.6.2 Removal of QuantIMS and Clouseau references and replaced with LIMS
 - 19.6.3 Section 6.0 updated to add equipment
 - 19.6.4 Section 8.0 updated
 - 19.6.5 Section 9.0 updated to indicate the composition of the MB and LCS
- 19.7 Revision 18: (8/8/2014)
 - 19.7.1 Updated section 6 equipment list
 - 19.7.2 Section 7, removal of QC Standard 16 mg/mL and 40 ug/mL verbiage
 - 19.7.3 Section 12, removed further confirmation of hits identified by C18 from Qualitative Identification section and updated calculation software.
 - 19.7.4 Updated section 13 with corrective actions
 - 19.7.5 Updated section 15
- 19.8 Revision 19: (1/16/2015)
 - 19.8.1 Updated section 6.1: equipment list – added details about pump and detector.
 - 19.8.2 Updated location of **method performance data, Reporting Limits, and QC acceptance limits in Section 14.1**
- 19.9 Revision 20 (5/23/16)
 - 19.9.1 Updated columns used in Section 6
 - 19.9.2 Changed Calcium Chloride to HPLC Grade Water in Section 11
- 19.10 Revision 21 (8/3/16)
 - 19.10.1 Updated Section 12.1 regarding column reporting.

- 19.10.2 Replaced calcium chloride solution in Section 7 with HPLC grade water
- 19.10.3 Added co-elution note for nitrotoluene isomers to Section 4.

Calibration Standards
Water (Final Concentration) µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
HMX	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
RDX	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
1,3,5-Trinitrobenzene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
1,3-Dinitrobenzene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
Tetryl	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
Nitrobenzene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
2,4,6-Trinitrotoluene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
4-Amino-2,6-dinitrotoluene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
2-Amino-2,4-dinitrotoluene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
2,6-Dinitrotoluene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
2,4-Dinitrotoluene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
2-Nitrotoluene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
3-Nitrotoluene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
4-Nitrotoluene	0.125	0.25	1.25	2.50	5.00	12.5	25.0	50.0
Nitroglycerin	0.312	0.624	3.12	6.24	12.5			
PETN	0.312	0.624	3.12	6.24	12.5			
MNX	0.025	0.05	0.125	0.25	0.625	1.25	2.50	5.00
DNX	0.025	0.05	0.125	0.25	0.625	1.25	2.50	5.00
TNX	0.025	0.05	0.125	0.25	0.625	1.25	2.50	5.00

Soil (Final Concentration) µg/kg

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
HMX	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
RDX	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
1,3,5-Trinitrobenzene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
1,3-Dinitrobenzene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
Tetryl	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
Nitrobenzene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
2,4,6-Trinitrotoluene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
4-Amino-2,6-dinitrotoluene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
2-Amino-2,4-dinitrotoluene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
2,6-Dinitrotoluene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
2,4-Dinitrotoluene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
2-Nitrotoluene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
3-Nitrotoluene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
4-Nitrotoluene	125	250	1250	2500.00	5000.00	12500	25000.0	50000.0
Nitroglycerin	312	624	3120	6240.00	12480.0			
PETN	312	624	3120	6240.00	12480.0			
MNX	25	50	125	250.00	625	1250	2500.00	5000.00
DNX	25	50	125	250.00	625	1250	2500.00	5000.00
TNX	25	50	125	250.00	625	1250	2500.00	5000.00

*NOTE: Level concentration may be slightly varied.

Title: GC/MS SEMIVOLATILES ANALYSIS
[SW-846 8270D; EPA 625]

Approvals (Signature/Date):

 10/28/16
Dennis Konopka
Extractable Manager

 10/28/16
Mike Ridenhower
Health & Safety Manager / Coordinator

 10/28/16
Kristen Ely
Quality Assurance Manager

 10/28/16
Elaine Wild
Laboratory Director

This SOP was previously identified as SOP No. ST-MS-0001 Rev. 21

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.
- 1.2 This SOP is based on SW-846 Method 8000B, 8000C;800D and 8270D and EPA method 625.
- 1.3 The following compounds are documented in the method as problematic:
 - 1.3.1 Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
 - 1.3.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
 - 1.3.3 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
 - 1.3.4 Hexachlorophene may not be amenable to analysis by this method.
- 1.4 N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from Diphenylamine.
- 1.5 3-Methylphenol cannot be separated from 4-Methylphenol by the conditions specified in this method.
- 1.6 Phthalic acid decomposes in the gas chromatographic inlet and cannot be distinguished from Phthalic anhydride.
- 1.7 Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be reported as Azobenzene.
- 1.8 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.8.1 Additional compounds may be amendable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

- 2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel. Continuous liquid-liquid extraction may also be used. Solid samples are extracted with methylene chloride / acetone using sonication. Waste dilution is used for organic or unusual matrix samples. The sample extract is concentrated to a volume of 1 mL, 5 mL or 10 mL, and analyzed by GC/MS. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.
- 2.2 The use of selected ion monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electro impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification, since less mass spectral information is available. Instead of scanning everything in a retention time range, SIM looks for specific ions (qualitative and quantitative) that are placed in retention time groups. The ions used for qualitative and quantitative purposes are the same for scan and SIM analysis. SIM is not allowed for South Carolina work, as the laboratory does not hold certification there for the SIM method.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Lower Limit of Quantitation – LLOQ. SOP refers to this as the reporting limit (RL) The LLOQ is verified annually (see SOP ST-QA-0016 for details.)
- 3.3 SIM –Selected Ion Monitoring

4.0 INTERFERENCES

- 4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.
- 4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.
- 4.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.
- 4.4 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

- 5.2.1 Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile, Silver Shield, or similar gloves must be used.
- 5.2.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.2.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- 5.2.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 PRIMARY MATERIALS USED

- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Gas Chromatograph/Mass Spectrometer System: HP 6890/5973 - An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source. Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in [Table 1](#) when 50 ng of the GC/MS tuning standard is injected through the GC.
- 6.1.1 Column: Restek RXI-5Sil MS, 30 meters, 0.25mm ID, 0.25 μ m df
- 6.2 Data System:
- 6.2.1 ChemStation software system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the length of the chromatographic program.
- 6.2.2 Target software system allows the searching of any GC/MS data file for ions of a specified mass and plots such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software allows integrating the abundances in any EICP for a specified time or scan-number limit. Also, for the non-target compounds with a mass spectrum that meets the required criteria, software must be available that allows for the comparison of sample spectra against the reference library spectra.
- 6.2.3 Data Library: NIST05
- 6.3 Carrier gas: Ultra high purity helium
- 6.4 Instrument columns and run conditions are posted in the instrument maintenance calendar.
- 6.5 Amber vials. Crimp top seals
- 6.6 Disposal pipettes
- 6.7 Micro syringes- 10 μ L, 250 μ L, 500 μ L, 1000 μ L. Hamilton 1700 series, Agilent Gold Standard
- 6.8 Volumetric flasks, Class A
- 6.9 Analytical Balance, capable of weighing $\pm$ 0.01 grams.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

- 7.2 See recipes for standards and QC samples in the LIMS Reagent Log program. See Appendix 1 of this SOP
- 7.3 At a minimum, a five point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to [Table 3](#) for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.
- 7.4 An Internal Standard (IS) solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.
- 7.5 Internal Standards are added to all standards and extracts to result in 40 ng injected onto the column. SIM Analysis Internal Standards are added to all standards and extracts to result in 4 ng injected onto the column.
- 7.6 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared.
- 7.7 ICV standards, NIST traceable:
- 7.7.1 The Semivolatile ICV standard is a second source from the calibration standard, where a second viable source is available.
- 7.7.2 ICV standard is prepared and stored in the same way as calibration standards.
- 7.8 Standards are to be refrigerated at $\leq 6^{\circ}\text{C}$ when not in use. Refrigeration at less than -10°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least 6 months after opening.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Water samples are collected in amber glass, unpreserved and stored at $4 \pm 2^{\circ}\text{C}$.
- 8.3 Soil samples are refrigerated at $4 \pm 2^{\circ}\text{C}$.
- 8.4 The extraction holding time for Semivolatiles analysis in waters is 7 days.
- 8.5 The extraction holding time for Semivolatiles in soil/solid matrix is 14 days.
- 8.6 Extracts must be refrigerated at $\leq 6^{\circ}\text{C}$ and analyzed within 40 days of the beginning of the extraction.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2 -Method Blank (MB)

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 DI water is used for the Method Blank.
- 9.2.4 Sodium sulfate is used as the method blank for solid matrices.
 - 9.2.4.1 South Carolina requires the use of Ottawa sand for the Method Blank.

9.3 Laboratory Control Sample (LCS)

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.
- 9.3.3 The LCS is comprised of sodium sulfate fortified with the target analyte(s).
 - 9.3.3.1 South Carolina requires the use of Ottawa sand for the LCS solid matrix.

9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5 Surrogate

- 9.5.1 A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.
- 9.5.2 Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.

9.6 Procedural Variations/ Nonconformance and Corrective Action

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION**10.1 Internal standard calibration is used.**

- 10.1.1 Internal Standard Calibration Procedure: Internal standards are listed in [Table 5](#). Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.
 - 10.1.1.1 Compounds are assigned to the IS, generally with the closest retention time. See [Table 5](#).

10.2 Instrument Tuning

- 10.2.1 The GC/MS system must be checked to see if acceptable performance criteria are achieved for DFTPP (decafluorotriphenylphosphine). See [Table 1](#) in this SOP.
 - 10.2.1.1 The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.
 - 10.2.1.2 **8270** - At the beginning of every twelve hour shift.
 - 10.2.1.3 **625** - At the beginning of every 24 hour shift.
 - 10.2.1.3.1 The time period begins at the moment of injection of DFTPP.
- 10.2.2 Inject 50 ng of the GC/MS tuning standard into the GC/MS system. Obtain a background-corrected mass spectrum of DFTPP and confirm that all the key m/z criteria in [Table 1](#) are

achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

- 10.2.3 Degradation of DDT to DDE and DDD should not exceed 20%.

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD \% DDE)}}{\text{sum of all peak areas (DDT \% DDE \% DDD)}} \times 100$$

- 10.2.4 Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

$$\text{Tailing Factor} = BC/AB$$

Where the peak is defined as follows:

AC is the width at 10% height; DE is the height of peak and B is the height at 10% of DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height.

10.3 **Initial Calibration**

- 10.3.1 Prepare calibration standards at a minimum of five concentration levels, six points for a quadratic fit, (see [Table 3](#) for suggested concentrations) for each parameter of interest. It may be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. The low level standard should be at or below the reporting limit. The other standards define the working range of the detector.
- 10.3.2 Add the internal standard mixture to result in 40 ng on column. The concentrations of all analytes are listed in [Table 3](#). Add the internal standard mixture to result in 4ng on column for SIM analysis.
- 10.3.3 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. The low level standard must be at or below the reporting limit.
- 10.3.4 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica corporate policy, "Calibration Curves."
- 10.3.5 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.
- 10.3.6 A new calibration curve must be generated after major changes to the system and may be required when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance (e.g., cleaning the ion source).
- 10.3.7 Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount concentration of analyte in the calibration standard is defined as the response factor (RF) or calibration factor (CF).
- 10.3.8 Structural isomers that produce very similar mass spectra (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene) should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution is visibly verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification (CCV) level.
- 10.3.9 **Initial Calibration Criteria (8270D)**
- 10.3.9.1 Minimum Response Factors
- 10.3.9.2 See [Table 4](#) in this SOP for the minimum response factors. These minimum response factors are prescribed by SW method 8270D. For analytes not given a minimum response factor by the method, St. Louis has established a default minimum response

factor of 0.01 for compound, except for Famphur, Hexachlorophene, Kepone and Phthalic Anhydride which have a minimum response factor of 0.001.

10.3.9.2.1 SW-846 chromatographic methods allow the use of both linear and non-linear models for the calibration data.

10.3.9.3 The first way is to begin with the simplest approach, the linear model through the origin, and then progress through other options until the calibration acceptance criteria are met. The second way is to use technical knowledge of the detector response to the target compound to choose the calibration model.

10.3.9.4 The option for non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance.

10.3.9.5 **Linear calibration using the average response factor**

10.3.9.5.1 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 20\%$ for each target analyte.

10.3.9.5.2 If the %RSD in the initial calibration is $> 20\%$, then calibration using a linear regression may be employed.

10.3.9.6 **Linear calibration using a least squares regression**

The intercept of a linear calibration at zero response (i.e. the y-intercept) must have an absolute value less than the reporting limit for each analyte. Client requirements may be tighter, please check Client Requirement Memorandum (CRM) if identified in comments.

Note, for analyses utilizing an internal standard the Target variable “b” does NOT equal the y-intercept. For analyses utilizing an internal standard, the Target variable “b” must be multiplied by the associated internal standard concentration to derive the concentration at the y-intercept.

10.3.9.6.1 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990 .

10.3.9.6.2 When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve.

10.3.9.6.3 It is not necessary to re-analyze a low concentration standard; rather the data system can recalculate the concentrations.

10.3.9.6.4 The recalculated concentration of the low calibration point should be within $\pm 30\%$ of the standard's true concentration.

10.3.9.6.4.1 Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered “out of control” and corrective action should be taken.

10.3.9.7 **Linear calibration using a least squares regression, forcing thru zero**

10.3.9.7.1 Do not include an X-value of zero as a calibration point. Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 *before* the regression is calculated, thereby setting the bias to favor the low end of the calibration range by “pivoting” the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression through zero for some calibrations.

10.3.9.7.2 Curve must still meet criteria in 10.3.8.6.1 and 10.3.8.6.2

10.3.9.7.3 For samples requiring adherence to method 8000B and 8000D,, forcing through zero is **NOT** allowed. This includes South Carolina compliance work.

10.3.9.8 **Linear calibration using a least squares regression, weighting of data points**

10.3.9.8.1 In linear, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration; for this reason it may be preferable to increase the weighting of the lower concentration points, $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting), to improve accuracy at the low end of the curve.

10.3.9.8.2 Curve must still meet criteria in 10.3.8.6.1 and 10.3.8.6.2

10.3.9.9 Non-linear calibration

10.3.9.9.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches have not met the acceptance criteria, a non-linear calibration model may be employed.

10.3.9.9.2 The use of non-linear calibrations or second order regression calibrations are not allowed for South Carolina compliance samples.

10.3.9.9.3 It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance. Thus, non-linear calibrations are not to be employed for analytes shown to consistently exhibit linear calibration for the analytes of interest.

10.3.9.9.3.1 These compounds are not to use non-linear calibrations:

1,4-Dioxane; Pyridine; n-Nitrosodimethylamine;
2-Fluorophenol; Aniline; Bis(2-chloroethyl)ether; Phenol-d5;
Phenol; 2-Chlorophenol; 1,3-Dichlorobenzene; 1,4-Dichlorobenzene; 1,2-Dichlorobenzene; Benzyl Alcohol;
2-Methylphenol; N-nitrosodipropylamine; Hexachloroethane; 3 and 4-Methylphenol; Nitrobenzene-d5; Nitrobenzene; Isophorone;
2-Nitrophenol;
2,4-Dimethylphenol; Bis(2-chloroethoxy) methane;
2,4-Dichlorophenol; 1,2,4-Trichlorobenzene; Naphthalene;
Hexachlorobutadiene; 4-Chloro-3-Methylphenol;
2-Methylnaphthalene; 2,4,6-Trichlorophenol;
2-Fluorobiphenyl; 2,4,5-Trichlorophenol; 2-Chloronaphthalene;
Dimethylphthalate; Acenaphthylene; Acenaphthene; Dibenzofuran;
Diethylphthalate; Fluorene; 4-Chlorophenyl-phenylether; N-Nitrosodiphenylamine; Azobenzene; 4-Bromophenyl-phenylether;
Hexachlorobenzene; Phenanthrene; Anthracene; Carbazole; Di-n-Butylphthalate; Fluoranthene; Pyrene; Terphenyl-d14;
Butylbenzylphthalate; Benzo(a)Anthracene; Chrysene;
bis(2-ethylhexyl)Phthalate; 2-Picoline;
n-Nitrosomethylethylamine; Methyl methanesulfonate;
n-Nitrosodiethylamine; Ethyl Methanesulfonate; Pentachloroethane;
Acetophenone; n-Nitrosopyrrolidine;
n-Nitrosomorpholine; O-Toluidine; n-Nitrosopiperidine; o,o,o-Triethyl-Phosphorothioate; 2,6-Dichlorophenol;
Hexachloropropene; Benzothiazole;
n-Nitrosodi-n-butylamine; Safrole;
1,2,4,5-Tetrachlorobenzene; cis-Isosafrole; trans-Isosafrole; 1,4-Dinitrobenzene; 1,3-Dinitrobenzene; Pentachlorobenzene; 1-Naphthylamine; 2-Naphthylamine; Thionazin; 5-Nitro-o-toluidine;
Tri-n-butylphosphate; Sulfotep; Diallyl; Phorate; Phenacetin; Tris (2-chloroethyl) phosphate; 4-Aminobiphenyl; Pronamide;
Pentachloronitrobenzene; Disulfoton; Parathion; Isodrin; Aramite;
p- (Dimethylamino) azobenzene; Chlorobenzilate; 2-Acetylaminofluorene; 4,4'-Methylenebis (2)-Chloroaniline; 7,12-Dimethylbenz (a) anthracene;

3-Methylcholanthrene; Isosafrole; Octachlorostyrene;

Methyl methacrylate;

Ethyl methacrylate; Benzaldehyde; Caprolactam;

1-Methylnaphthalene; Biphenyl; Atrazine.

10.3.8.9.2.2 EPA Method 8000C suggests a 20% RSD limit be used when evaluating a calibration. The above compound list was constructed based on the 20% RSD criteria. TestAmerica St. Louis reserves the right to employ different calibration models when client mandated criteria are less than the 20% criteria found in method 8000C.

10.3.9.9.4 The intercept of the curve at zero response must be less than + or – the reporting limit for the analyte.

10.3.9.9.5 r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990 .

10.3.9.9.6 Due to the nature of SIM analysis, non-linear calibrations may be used.

10.3.10 Acceptance criteria independent of calibration model

10.3.10.1 Either of the procedures Percent Error or Relative Standard Error (RSE) may be used to determine calibration function acceptability for linear and non-linear curves. Both evaluate the difference between the measured and the true amounts or concentrations used to create the model.

$$10.3.10.2 \quad \%Error = \frac{x_i - x'_i}{x_i} \times 100$$

10.3.10.3 Where x'_i = measured amount of the analyte at calibration level i in mass or concentration units and x_i = the true amount of the analyte at calibration level i in mass or concentration units.

10.3.10.4 % Error should be $\leq 30\%$. For some data uses $\leq 50\%$ may be acceptable for the lowest calibration point.

10.3.10.5 Calculation of Relative Standard Error:

10.3.10.6

$$RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

Where X_i = True amount of the calibration level i , in mass or concentration units

X'_i = measured amount of analyte in calibration level i , in mass or concentration units

p = number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3,)

n = number of calibration points

10.3.10.7 RSE acceptance criteria for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If not defined in the method, use $\pm 20\%$

10.3.11 625 criteria

10.3.11.1 Method 625 only requires a 3 point calibration. We routinely perform a 6 point calibration; however, 3 points may be removed from the curve if necessary to meet 625 calibration criteria.

10.3.11.1.1 Refer to the TestAmerica corporate policy, "Calibration Curves."

10.3.11.2 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $< 35\%$.

10.3.11.3 If the %RSD in the initial calibration is $> 35\%$, then calibration using a linear regression may be employed.

10.3.11.4 If a linear regression curve is used, the intercept of the curve at zero response must be less than $\pm$ the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.

- 10.3.11.5 Use of $1/\text{Concentration}^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.
- 10.3.11.6 Weighting of data points
 - 10.3.11.6.1 The points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4 **Initial Calibration Verification (ICV)**

- 10.4.1 An initial calibration verification standard is a different standard source than the one used for the initial calibration.
- 10.4.2 An ICV must be performed with every initial calibration.
- 10.4.3 The ICV performance must be within $\pm 30\%$ Difference for average CF and average RF curves and %Drift is used for evaluating linear and quadratic curves.
 - 10.4.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.5 **Continuing Calibration Verification (CCV)**

- 10.5.1 At the start of each 12 hour period (8270) or 24 hour period (EPA 625) the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria. See [Table 1](#) in this SOP.
- 10.5.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.
- 10.5.3 A CCV standard is analyzed every analysis tune clock immediately following the DFTPP tune.
 - 10.5.3.1 **EPA 8270** – for each 12 hour tune time period
 - 10.5.3.2 **EPA 625** – for each 24 hour tune time period
- 10.5.4 The CCV can be the same source or a second source from the calibration.
- 10.5.5 The internal standard response must be within 50-200 area counts (-50% to 100%) of the response in the mid level of the initial calibration. The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.
- 10.5.6 **EPA 8270 Criteria**
 - 10.5.6.1 Minimum Response Factors
 - 10.5.6.2 See [Table 4](#) in this SOP for the minimum response factors. These minimum response factors are prescribed by SW-846 method 8270D. For analytes not given a minimum response factor by the method, St. Louis has established a default minimum response factor of 0.01 per compound, except for Famphur, Hexachlorophene, Kepone and Phthalic Anhydride which have a minimum response factor of 0.001.
 - 10.5.6.3 The CCV performance must be with $\pm 20\%$ Difference for average CF and average RF curves and %Drift is used for evaluating linear and quadratic curves.
 - 10.5.6.4 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then sample analysis may continue; however the preceding samples must be reanalyzed. If this second CCV does

not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration).

10.5.7 **EPA 625 Criteria**

10.5.7.1 For each target analyte %D must be < 20%.

10.5.8 Calibration excursions are to be documented via a NCM.

10.6 Retention Time (RT) windows

10.6.1 Relative Retention Time (RRT)

10.6.1.1 In addition to normalizing the response (peak area) of the target compound to the response of the internal standard in that sample or extract for that injection, the retention times of the target compound and the internal standard may be used to calculate the relative retention time (RRT) of the target compound.

10.6.1.2 The RRT is expressed as a unit-less quantity:

$$\text{RRT} = \frac{\text{Retention time of the analyte}}{\text{Retention time of the internal standard}}$$

10.6.1.3 The RRT of each target analyte in each calibration standard should agree within ± 0.06 RRT units.

10.6.1.4 It is recognized here that with increasing retention times of the internal standard, target analytes will be able to more easily meet this criterion. Thus, care should be exercised when selecting the appropriate internal standards by retention times. The process of selecting internal standards to quantify target analytes should also include consideration of retention times as they should be similar.

10.6.1.5 If this criterion is not met and unless there are no other indicators of a component's identification such as a very unique but a high probability mass spectral match then that component may not be considered as identified by relative retention time.

10.6.1.6 The RRT evaluation allows the analyst to compensate for modest shifts in the chromatographic conditions that can occur due to interferences and simple day-to-day instrument variability. Many methods that employ internal standard calibration use more than one internal standard and the target compounds are related to the internal standards on the basis of the similarity of their respective chromatographic retention times (see [Table 5](#)).

10.6.2 Internal standard retention time

10.6.2.1 The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.6.3 Retention Time Criteria

10.6.3.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established.

10.7 Method Detection Limit Studies

10.7.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. . See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs

11.0 PROCEDURE

11.1 Samples are prepared following ST-OP-0002.

- 11.2 South Carolina requires a separate certification for SIM analysis. At this time TestAmerica St. Louis does not hold that certification. SIM analysis can not be used for South Carolina compliance samples.
- 11.3 South Carolina requires a separate certification for LVI (Limited Volume Extraction). At this time TestAmerica St. Louis does not hold that certification. LVI analysis can not be used for South Carolina compliance work.
- 11.4 All standards and extracts are allowed to warm to room temperature before injecting.
- 11.5 All samples must be analyzed using the same instrument conditions as the initial calibration.
- 11.6 Add internal standard to the extract to result in 40ng injected on column. Mix thoroughly before injection into the instrument.
- 11.6.1 Add internal standard to the extract to result in 4ng injected on column for SIM analysis.
- 11.7 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.
- 11.8 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.
- 11.9 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate at -10°C to -20°C (if it can be demonstrated that analytes do not fall out of solution at this temperature), protected from light in screw cap vials equipped with un-pierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 External Standard Calculations
- 12.1.1 See instrument software (Target/Chrom) for calculations.
- 12.2 Manual Integrations
- 12.2.1 Identified compounds are reviewed for proper integration. Integrations are performed automatically by the data system. If necessary, manual integrations are performed and are documented by the analyst. Manual integrations are denoted with "M" flag on the Target quantitation report. See TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices.
- 12.3 Qualitative identification
- 12.3.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.
- 12.3.1.1 Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated. The following analytes should be carefully reviewed:
- | | | |
|------------------------|-----------------------|---------------------------|
| 1,4-Dichlorobenzene-d4 | Aniline | Bis (2-Chloroethyl) ether |
| 1,3-Dichlorobenzene | 1,4-Dichlorobenzene | 1,2-Dichlorobenzene |
| Benzyl alcohol | 2-Methylphenol | 3,4-Methylphenol |
| 2,4-Dichlorophenol | 2,4,6-Trichlorophenol | 2,4,5-Trichlorophenol |
| Phenanthrene | Anthracene | Benz (a) anthracene |

Bis (2-ethylhexyl) phthalate	Chrysene	Di-n-octyl phthalate
Benzo (b) fluoranthene	Benzo (k) fluoranthene	Indeno (1,2,3-cd) pyrene
Benzo (g,h,i) perylene	p-Phenylenediamine	Safrole
Cis-Isosafrole	Trans-Isosafrole	1,4-Dinitrobenzene
1,3-Dinitrobenzene	1-Naphthylamine	2-Naphthylamine
2,3,4,6-Tetrachlorophenol	Dinoseb	Sulfotepp
Diallate 1 & 2	Methapyrilene	Aramite 1 & 2

- 12.3.2 The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- 12.3.3 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- 12.3.4 For Method 8270D the relative intensities of ions should agree to within $\pm 30\%$ between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance should be between 20 and 80 percent.) For Method 625, the relative peak heights of the ions must fall within $\pm 20\%$ of the reference spectra.
- 12.3.4.1 See [Table 2](#) for primary, secondary and tertiary ion assignments.
- 12.3.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.
- 12.3.6 Retention time criteria for samples
- 12.3.6.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.
- 12.3.6.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.
- 12.4 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client.
- 12.4.1 TICs are done as follows:
- 12.4.1.1 The computer will give quality matches in order from most likely to least likely. In order for us to call a TIC a certain compound, the quality match must be at least 90%. However, if the next two quality matches are within (around) 10% quality match of the first choice, the compound will be identified as an unknown because it is too close to call. Unknowns are put into a group if possible (such as Unknown alkanes) but if a group is not available it will be called Unknown. A compound will be also called unknown if the top three matches are all different groups of compounds and the quality match is $< 90\%$ (ex. If the top choice is an alkane, the second choice is an alcohol, the third choice is an acid).
- 12.4.1.2 The first 30 TICs, based on abundance, will be identified in a sample, unless a different number is specified by the client. See client requirement sheet.
- 12.5 Dilutions
- 12.3.7 If the concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed.
- 12.5.1 A dilution should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the client specific project requirements.

- 12.5.2 Samples may be diluted initially if the project reporting limits are above the laboratory's routine calibration lower limit, if there is physical evidence of matrix, or historical knowledge of the site.

12.6 Carryover

- 12.6.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
- 12.6.1.1 If a sample analyzed after a sample with high concentrations has negative results or is non-detect, carryover did not occur.
- 12.6.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, carryover may have occurred.
- 12.6.1.2.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 This SOP lists requirements for the standard Quality Assurance criteria followed at TestAmerica St. Louis. If a client or program requires stricter quality controls (i.e. DoD, DOE, SC DHEC) the analyst is directed to the Client Requirement Memo for that client/project for limits.

- 13.2 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036. Steps taken for out-of-control situations include demonstrating that the cause of the out-of-control situation was addressed and demonstration that a return to control was obtained.

13.3 Method Blank

13.3.1 Acceptance Criteria:

- 13.3.1.1 No target analytes may be present in the method blank above the reporting limit. SW846 Method 8270D Revision 5 requires that the Method Blank be less than ½ the Reporting Limit (aka LLOQ)

- 13.3.1.2 The method blank must have acceptable surrogate recoveries.

- 13.3.1.3 Corrective Action for Method Blanks not meeting acceptance criteria:

- 13.3.1.3.1 Method Blank Contamination – Blank contamination above the RL (>1/2 RL for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are < RL (LLOQ) or greater than 10 times the amount detected in the method blank.

- 13.3.1.3.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.

- 13.3.1.3.3 For South Carolina compliance work, the Method Blank concentration must meet the 8270D Update V method requirement.

13.4 Laboratory Control Sample (LCS)

- 13.4.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD).

- 13.4.1.1 For long analyte spike lists, marginal exceedances (ME) are allowed as follows:

- 13.4.1.2 less than 11 analytes in LCS, no analytes allowed in ME of the LCS control limit.

- 13.4.1.3 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit.

- 13.4.1.4 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit.

- 13.4.1.5 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit.

- 13.4.1.6 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit.

- 13.4.1.7 More than 90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit.

- 13.4.1.8 No LCS recoveries may be outside the Marginal Exceedance limit.

- 13.4.1.9 Marginal exceedances must be random. If the same LCS analyte exceeds the control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.
- 13.4.1.10 Marginal exceedance is not allowed by all programs. See Project/Program CRM for details. The use of marginal exceedances is not allowed for South Carolina Compliance samples.
- 13.4.2 The LCS should have acceptable surrogate recoveries.
- 13.4.3 Corrective Action for LCS not meeting acceptance criteria:
 - 13.4.3.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.
 - 13.4.3.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.
 - 13.4.3.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.
 - 13.4.3.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM.
- 13.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
 - 13.5.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).
 - 13.5.2 Corrective Action for MS/MSD not meeting acceptance criteria:
 - 13.5.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.
- 13.6 Sample result evaluation
 - 13.6.1 Dilutions
 - 13.6.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
 - 13.6.1.2 Dilution: Sample– An NCM is created when dilutions are required.
 - 13.6.1.3 Dilution: Surrogate(s)/spikes diluted out– An NCM is generated to document the surrogates/spikes being diluted out.
 - 13.6.2 Carryover
 - 13.6.2.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
 - 13.6.2.2 If a sample analyzed after a sample with high concentrations is non-detect for the high concentration analyte, carryover did not occur.
 - 13.6.2.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.
 - 13.6.3 Internal Standards
 - 13.6.3.1 Acceptance Criteria:
 - 13.6.3.1.1 If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, corrective action must be taken.

- 13.6.3.1.2 If the EICP area for any of the internal standards in samples, spikes and blanks changes by a factor of two (-50% to +100%) from the areas determined in the continuing calibration analyzed that day, corrective action must be taken. The samples, spikes or blanks should be reanalyzed or the data should be qualified. (Some programs may require that the midpoint of the initial calibration be used for ISTD monitoring. See the project CRM for specifics.)
- 13.6.3.2 Corrective Action for Internal Standards not meeting acceptance criteria:
 - 13.6.3.2.1 Internal Standard excursion – high – High ISTD recovery indicates a potential low bias to analytical results. Instrument maintenance, if required, is done and affected samples are reanalyzed. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.
 - 13.6.3.2.2 Internal Standard excursion – low – Low ISTD recovery indicates the potential for a high bias to analytical results. Samples that are non-detect for affected analytes may be reported with an NCM. Samples with positive hits above the RL for analytes associated with the poor ISTD recovery require re-analysis. Instrument maintenance, if required, is done. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.
- 13.6.4 Surrogate
 - 13.6.4.1 All Surrogates should be within established control limits for accuracy (%Recovery).
 - 13.6.4.2 Corrective Action for Surrogate not meeting acceptance criteria:
 - 13.6.4.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.
- 13.7 Insufficient Sample
 - 13.7.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, an NCM is created and a narrative comment stating such is included in the report's Case Narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the QAM.
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
 - 16.2.1 The following waste streams are produced when this method is carried out.
 - 16.2.1.1 Auto-sample vials containing Methylene Chloride are to be disposed of in the appropriate solvent vial waste accumulation container located within the GC/MS lab, for temporary storage. Once this temporary container is full or once it reaches a one-year collection time, this container must be dumped into the permanent solvent vial waste container located in the 90-day storage area, which is marked as a Type "C" waste accumulation container.
 - 16.2.1.2 Waste Methylene Chloride rinses are to be collected and disposed of within the solvent waste accumulation container located in the Organic Prep. Lab. This temporary storage container shall be dumped on a daily basis into the permanent waste accumulation container located in the 90-day storage area which is marked as a Type "D" waste drum.

17.0 REFERENCES

- 17.1 SW846, Test Methods for Evaluating Solid Waste, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Method 8000B, 8000C, 8000D and 8270D.
- 17.2 40CFR Part 136: "Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A, "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", Code of Federal Regulations, Revised July1, 1995, Method 625.
- 17.3 TestAmerica St. Louis Quality Assurance Manual (QAM), current revision.
- 17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-002) and St. Louis Facility Addendum (ST-HS-0002), current revision.
- 17.5 TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices
- 17.6 TestAmerica Policy CA-Q-P-003, Selection of Calibration Points
- 17.7 Associated SOPs, current revisions
 - 17.7.1 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW-846 3500 Series, 3600 Series, and 600 Series Methods
 - 17.7.2 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.7.3 ST-QA-0002, Standard and Reagent Preparation
 - 17.7.4 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.
 - 17.7.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.7.6 ST-QA-0016, IDL/MDL, LOD/LOQ Determination
 - 17.7.7 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

19.0 CHANGES TO PREVISION SOP REVISION

- 19.1 Table reference in Section 6.1 was corrected.
- 19.2 Y-intercept requirements added to Section 10.
- 19.3 Added requirement for 6 levels for a quadratic curve to Section 10
- 19.4 Added CLP allowance for reporting data within 10% of upper standard without dilution to Section 12
- 19.5 Clarification of criteria for TIC reporting added to Section 12.4.
- 19.6 [Table 1](#): clarified Tune criteria and added allowance of other published DFTPP Tune criteria (i.e. EPA CLP)
- 19.7 Added [Table 5](#), a listing of internal standards and associated analytes
- 19.8 Revision 13:
- 19.8.1 Grammatical /spelling corrections
- 19.8.2 Added SIM analysis to section 11
- 19.9 Revision 14:
- 19.9.1 Removed QuantIMS and Clouseau references – replaced with LIMs
- 19.9.2 Created hyperlinks to tables
- 19.9.3 Appended LVI Calibration Levels to [Table 3](#)
- 19.9.4 Combined fragmented [Table 5](#) into one table
- 19.9.5 Added table of potentially mis-identifiable analytes to Section 12.3.
- 19.9.6 Removed CLP allowance for reporting data within 10% of upper standard without dilution from Section 12.
- 19.9.7 Revised Section 13 to remove Clouseau corrective action references and to provide specific corrective actions for non-conformances.
- 19.10 Revision 15:
- 19.10.1 Section 3, updated SIM definition
- 19.10.2 Section 7.5 Added SIM requirement
- 19.10.3 Section 7.7 ICV standard 2nd source where available to acquire
- 19.10.4 Section 10.1 corrected table references
- 19.10.5 Section 10.2 Added % breakdown calculation and added Benzidine and pentachlorophenol requirements
- 19.10.6 Section 10.3.8.9.2.1 removed compounds that are not to use non-linear calibration model
- 19.10.7 Removed 12.6.1.3
- 19.10.8 Section 13.6.2.3 removed chromatographic profile reference
- 19.11 Revision 16:
- 19.11.1 Section 1.1 Added a missing period
- 19.11.2 Section 10.2.1.2 Added a missing period
- 19.11.3 Section 10.2.2 Added a missing period
- 19.11.4 Section 10.5.2 Added a missing period
- 19.11.5 Section 12.2.1 Added a missing period
- 19.11.6 Section 13.4.3.1 Added a missing period
- 19.11.7 Section 13.4.3.4 Added a missing period
- 19.11.8 Section 10.3.8.2 Took comma out after Kepone and added the word “and”
- 19.11.9 Section 10.5.6.2 Took comma out after Kepone
- 19.11.10 Section 17.5 Changed policy number from 001 to 002
- 19.11.11 Section 2.1 Added possible concentration of 5 mL
- 19.11.12 Added Surrogates to Section 13

- 19.11.13 Table 3 – Changed calibration concentration levels
- 19.12 Revision 17:
- 19.12.1 Added note to Section 11 that South Carolina requires certification for SIM analysis.
- 19.12.2 Updated Section 13.4 to disallow the use of marginal exceedance for South Carolina compliance work.
- 19.12.3 Updated Section 10.3.8 to disallow the use of non-linear or second order calibrations for South Carolina compliance work.
- 19.13 Revision 18 (11/3/14):
- 19.13.1 Combined SOP with SOP ST-MS-0001SC Rev 1 to include the South Carolina DHEC requirements.
- 19.13.2 Table 7 added to SOP - containing information on standards and reagents.
- 19.14 Revision 19 (12/1/14)
- 19.14.1 Added liquid-liquid continuous extraction to Section 2.1.
- 19.14.2 Added column used to Section 6
- 19.14.3 Updated Section 10.3 to clarify South Carolina requirements regarding forcing through zero
- 19.14.4 Added Section 10.7 to list MDL information
- 19.14.5 Added Section 11.3 to disallow LVI procedure for South Carolina compliance work
- 19.14.6 Added standard preparation and concentration information to the SOP appendix
- 19.15 Revision 20 (11/16/15)
- 19.15.1 Added section 10.3.8 to provide a reference to 8270D isomer resolution being <50%
- 19.15.2 Edited section 10.7.1 to remove reference to using dual column
- 19.16 Revision 21 (9/20/2016)
- 19.16.1 South Carolina requirement to use Ottawa sand for Method Blank and LCS in soil batches was added to Section 9.
- 19.16.2 Defined the use of % Difference and % Drift in specific curve models in Section 10.
- 19.16.3 Corrected ion intensities for Method 625 from 30% to 20%.
- 19.17 Revision 22 (10/28/16)
- 19.17.1 Incorporated requirements from 8270 Rev 5
- 19.17.1.1 Updated Method blank requirements
- 19.17.1.2 Added reference to LLOQ (RL)
- 19.17.1.3 Added % Error and Relative Percent Error information to Section 10.

Table 1
DFTPP Key Ions and Ion Abundance Criteria*

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

* Tune criteria in use is a combination of 8270C and 8270D which is more stringent than either method. Alternatively, other documented tuning criteria (e.g. EPA CLP) may be used provided method performance is not adversely affected.

Table 2
Analytes in Approximate Retention Time Order and Characteristic Ions

Primary Standard			
Analyte	Primary	Secondary	Tertiary
1,4 Dioxane	88	58	43
n-Nitrosodimethylamine	74*	42	44
Pyridine	79	52	—
Dimethylformamide	44	73	42
Cyclohexanol	57	82	67
2-Fluorophenol (Surrogate Standard)	112	64	63**
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	65
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	111
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	111
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	111
2-Methylphenol	108*	107	79
2,2'-oxybis(1-chloropropane) ¹	45	77	121
3&4-Methylphenol	107	108	79
n-Nitroso-di-n-propylamine	70	42	101
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107*	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123

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Table 2
Analytes in Approximate Retention Time Order and Characteristic Ions

Primary Standard			
Analyte	Primary	Secondary	Tertiary
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54**
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	—
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	—
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153*	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	—
4-Nitrophenol	109*	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	105	51

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Table 2
Analytes in Approximate Retention Time Order and Characteristic Ions

Primary Standard			
Analyte	Primary	Secondary	Tertiary
n-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332**	141
Azobenzene	77	51**	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	203
Benzidine	184	92	185
Pyrene	202	200	203
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

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* primary/secondary and/or tertiary ions are switched from order in Method

** not listed in the method

Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Methyl methacrylate	69	41	39
Ethyl methacrylate	69	41	39
2-Picoline	93	66	92
n-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
2-Fluorophenol (Surrogate Standard)	112	64	63**
n-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Benzaldehyde	77	106	51
Phenol-d5 (Surrogate Standard)	99	42	71
Pentachloroethane	117	119	167
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
Acetophenone	105	77	120
n-Nitrosopyrrolidine	100	41	42
n-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	—
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
n-Nitrosopiperidine	114	42	55
O,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	—
Naphthalene-d8 (Internal Standard)	136	68	54**
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
Benzothiazole	135	108	69
Caprolactam	55	113	42
p-Phenylenediamine	108	80	—
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
Phthalic anhydride	104	76	50
1-methylnaphthalene	142	141	115

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Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole, cis	162	104	131
2-Fluorobiphenyl (Surrogate Standard)	172	171	—
Isosafrole, trans	162	104	131
Biphenyl	154	153	152
1,4-Dinitrobenzene	168	75	50
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Acenaphthene-d10 (Internal Standard)	164	162	160
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	—
2-Naphthylamine	143	115	—
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	107	96	143
1,3,5-Trinitrobenzene	213*	75	120
2,4,6-Tribromophenol (Surrogate Standard)	330	332**	141**
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate 1	86	234	43
Diallate 2	86	234	43
Dimethoate	87	93	125
4-Aminobiphenyl	169	168	170
Pentachloronitrobenzene	237	142	214
Phenanthrene-d10 (Internal Standard)	188	94	80
Pronamide	173	175	145
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	75
Parathion	109	97	291
Isodrin	193	66	195

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Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Kepone	272	274	237
Methapyrilene	97	58**	—
Octachlorostyrene	308	343	154
Terphenyl-d14 (Surrogate Standard)	244	122	212
Aramite 1	185	319	—
Aramite 2	185	319	—
p-(Dimethylamino)azobenzene	120*	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	—
2-Acetylaminofluorene	181	180	223
Famphur	218	125	93
Chrysene-d12 (Internal Standard)	240	120	236
Hexachlorophene	196	198	209
7,12-Dimethylbenz(a)anthracene	256	241	120
Perylene-d12 (Internal Standard)	264	260	265
3-Methylcholanthrene	268	252	126

* primary/secondary and/or tertiary ions are switched from order in Method

** not listed in the method

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Table 3
Calibration Levels, Primary Standard, µg/mL³

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
1,4 Dioxane	1	2	5	10	20	30	40	50	60
Pyridine	1	2	5	10	20	30	40	50	60
n-Nitrosodimethylamine	1	2	5	10	20	30	40	50	60
Dimethylformamide	1	2	5	10	20	30	40	50	60
Cyclohexanol	1	2	5	10	20	30	40	50	60
Aniline	1	2	5	10	20	30	40	50	60
Phenol	1	2	5	10	20	30	40	50	60
Bis(2-chloroethyl)ether	1	2	5	10	20	30	40	50	60
2-Chlorophenol	1	2	5	10	20	30	40	50	60
1,3-Dichlorobenzene	1	2	5	10	20	30	40	50	60
1,4-Dichlorobenzene	1	2	5	10	20	30	40	50	60
Benzyl alcohol	1	2	5	10	20	30	40	50	60
1,2-Dichlorobenzene	1	2	5	10	20	30	40	50	60
2-Methylphenol	1	2	5	10	20	30	40	50	60
2,2'-oxybis(1-chloropropane) ¹	1	2	5	10	20	30	40	50	60
3&4-Methylphenol	1	2	5	10	20	30	40	50	60
n-Nitroso-di-n-propylamine	1	2	5	10	20	30	40	50	60
Hexachloroethane	1	2	5	10	20	30	40	50	60
Nitrobenzene	1	2	5	10	20	30	40	50	60
Isophorone	1	2	5	10	20	30	40	50	60
2-Nitrophenol	1	2	5	10	20	30	40	50	60
2,4-Dimethylphenol	1	2	5	10	20	30	40	50	60
Benzoic acid	1	2	5	10	20	30	40	50	60
bis(2-Chloroethoxy)methane	1	2	5	10	20	30	40	50	60
2,4-Dichlorophenol	1	2	5	10	20	30	40	50	60
1,2,4-Trichlorobenzene	1	2	5	10	20	30	40	50	60
Naphthalene	1	2	5	10	20	30	40	50	60
4-Chloroaniline	1	2	5	10	20	30	40	50	60
Hexachlorobutadiene	1	2	5	10	20	30	40	50	60

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Table 3
Calibration Levels, Primary Standard, µg/mL³

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
4-Chloro-3-methylphenol	1	2	5	10	20	30	40	50	60
2-Methylnaphthalene	1	2	5	10	20	30	40	50	60
Hexachlorocyclopentadiene	1	2	5	10	20	30	40	50	60
2,4,6-Trichlorophenol	1	2	5	10	20	30	40	50	60
2,4,5-Trichlorophenol	1	2	5	10	20	30	40	50	60
2-Chloronaphthalene	1	2	5	10	20	30	40	50	60
2-Nitroaniline	1	2	5	10	20	30	40	50	60
Dimethyl phthalate	1	2	5	10	20	30	40	50	60
Acenaphthylene	1	2	5	10	20	30	40	50	60
3-Nitroaniline	1	2	5	10	20	30	40	50	60
Acenaphthene	1	2	5	10	20	30	40	50	60
2,4-Dinitrophenol	2	4	10	20	40	60	80	100	120
4-Nitrophenol	2	4	10	20	40	60	80	100	120
Dibenzofuran	1	2	5	10	20	30	40	50	60
2,4-Dinitrotoluene	1	2	5	10	20	30	40	50	60
2,6-Dinitrotoluene	1	2	5	10	20	30	40	50	60
Diethylphthalate	1	2	5	10	20	30	40	50	60
4-Chlorophenyl phenyl ether	1	2	5	10	20	30	40	50	60
Fluorene	1	2	5	10	20	30	40	50	60
4-Nitroaniline	1	2	5	10	20	30	40	50	60
4,6-Dinitro-2-methylphenol	2	4	10	20	40	60	80	100	120
N-Nitrosodiphenylamine	1	2	5	10	20	30	40	50	60
Azobenzene ²	1	2	5	10	20	30	40	50	60
4-Bromophenyl phenyl ether	1	2	5	10	20	30	40	50	60
Hexachlorobenzene	1	2	5	10	20	30	40	50	60
Pentachlorophenol	1	2	5	10	20	30	40	50	60
Phenanthrene	1	2	5	10	20	30	40	50	60
Anthracene	1	2	5	10	20	30	40	50	60
Carbazole	1	2	5	10	20	30	40	50	60
Di-n-butyl phthalate	1	2	5	10	20	30	40	50	60
Fluoranthene	1	2	5	10	20	30	40	50	60

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Table 3
Calibration Levels, Primary Standard, µg/mL³

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Benzidine	1	2	5	10	20	30	40	50	60
Pyrene	1	2	5	10	20	30	40	50	60
Butyl benzyl phthalate	1	2	5	10	20	30	40	50	60
3,3'-Dichlorobenzidine	1	2	5	10	20	30	40	50	60
Benzo(a)anthracene	1	2	5	10	20	30	40	50	60
Bis(2-ethylhexyl)phthalate	1	2	5	10	20	30	40	50	60
Chrysene	1	2	5	10	20	30	40	50	60
Di-n-octylphthalate	1	2	5	10	20	30	40	50	60
Benzo(b)fluoranthene	1	2	5	10	20	30	40	50	60
Benzo(k)fluoranthene	1	2	5	10	20	30	40	50	60
Benzo(a)pyrene	1	2	5	10	20	30	40	50	60
Indeno(1,2,3-cd)pyrene	1	2	5	10	20	30	40	50	60
Dibenz(a,h)anthracene	1	2	5	10	20	30	40	50	60
Benzo(g,h,i)perylene	1	2	5	10	20	30	40	50	60

¹ 2,2'-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

² Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

³ Lower concentration standards may be analyzed on a project specific basis.

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Methyl methacrylate	1	2	5	10	20	30	40	50	60
Ethyl methacrylate	1	2	5	10	20	30	40	50	60
2-Picoline	1	2	5	10	20	30	40	50	60
n-Nitrosomethylethylamine	1	2	5	10	20	30	40	50	60
Methyl methanesulfonate	1	2	5	10	20	30	40	50	60
n-Nitrosodiethylamine	1	2	5	10	20	30	40	50	60
Ethyl methanesulfonate	1	2	5	10	20	30	40	50	60
Benzaldehyde	1	2	5	10	20	30	40	50	60

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Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Pentachloroethane	1	2	5	10	20	30	40	50	60
Acetophenone	1	2	5	10	20	30	40	50	60
n-Nitrosopyrrolidine	1	2	5	10	20	30	40	50	60
n-Nitrosomorpholine	1	2	5	10	20	30	40	50	60
o-Toluidine	1	2	5	10	20	30	40	50	60
n-Nitrosopiperidine	1	2	5	10	20	30	40	50	60
O,o,o-Triethyl-Phosphorothioate	1	2	5	10	20	30	40	50	60
A,a-Dimethyl-phenethylamine	1	2	5	10	20	30	40	50	60
2,6-Dichlorophenol	1	2	5	10	20	30	40	50	60
Hexachloropropene	1	2	5	10	20	30	40	50	60
Benzothiazole	1	2	5	10	20	30	40	50	60
Caprolactam	1	2	5	10	20	30	40	50	60
p-Phenylenediamine	1	2	5	10	20	30	40	50	60
n-Nitrosodi-n-butylamine	1	2	5	10	20	30	40	50	60
Safrole	1	2	5	10	20	30	40	50	60
Phthalic anhydride	1	2	5	10	20	30	40	50	60
1-Methylnaphthalene	1	2	5	10	20	30	40	50	60
1,2,4,5-Tetrachlorobenzene	1	2	5	10	20	30	40	50	60
Isosafrole, cis	.5	1	2.5	5	10	15	20	25	30
Isosafrole, trans	.5	1	2.5	5	10	15	20	25	30
Biphenyl	1	2	5	10	20	30	40	50	60
1,4-Dinitrobenzene	1	2	5	10	20	30	40	50	60
1,4-Naphthoquinone	1	2	5	10	20	30	40	50	60
1,3-Dinitrobenzene	1	2	5	10	20	30	40	50	60
Pentachlorobenzene	1	2	5	10	20	30	40	50	60
1-Naphthylamine	1	2	5	10	20	30	40	50	60
2-Naphthylamine	1	2	5	10	20	30	40	50	60
2,3,4,6-Tetrachlorophenol	1	2	5	10	20	30	40	50	60
5-Nitro-o-toluidine	1	2	5	10	20	30	40	50	60
Thionazin	1	2	5	10	20	30	40	50	60
1,3,5-Trinitrobenzene	1	2	5	10	20	30	40	50	60
Sulfotepp	1	2	5	10	20	30	40	50	60
Phorate	1	2	5	10	20	30	40	50	60

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Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Phenacetin	1	2	5	10	20	30	40	50	60
Diallate 1	.5	1	2.5	5	10	15	20	25	30
Diallate 2	.5	1	2.5	5	10	15	20	25	30
Dimethoate	1	2	5	10	20	30	40	50	60
4-Aminobiphenyl	1	2	5	10	20	30	40	50	60
Pentachloronitrobenzene	1	2	5	10	20	30	40	50	60
Pronamide	1	2	5	10	20	30	40	50	60
Disulfoton	1	2	5	10	20	30	40	50	60
2-sec butyl-4,6-dinitrophenol (Dinoseb)	1	2	5	10	20	30	40	50	60
Methyl parathion	1	2	5	10	20	30	40	50	60
4-Nitroquinoline-1-oxide	1	2	5	10	20	30	40	50	60
Parathion	1	2	5	10	20	30	40	50	60
Isodrin	1	2	5	10	20	30	40	50	60
Kepone	1	2	5	10	20	30	40	50	60
Famphur	1	2	5	10	20	30	40	50	60
Methapyrilene	1	2	5	10	20	30	40	50	60
Octachlorostyrene	1	2	5	10	20	30	40	50	60
Aramite 1	.5	1	2.5	5	10	15	20	25	30
Aramite 2	.5	1	2.5	5	10	15	20	25	30
p-(Dimethylamino)azobenzene	1	2	5	10	20	30	40	50	60
p-Chlorobenzilate	1	2	5	10	20	30	40	50	60
3,3'-Dimethylbenzidine	1	2	5	10	20	30	40	50	60
Hexachlorophene	10	20	50	100	200	300	400	500	600
2-Acetylaminofluorene	1	2	5	10	20	30	40	50	60
Dibenz (a,j)acridine	1	2	5	10	20	30	40	50	60
7,12-Dimethylbenz(a)anthracene	1	2	5	10	20	30	40	50	60
3-Methylcholanthrene	1	2	5	10	20	30	40	50	60
2-Fluorophenol (Surrogate Standard)	1	2	5	10	20	30	40	50	60
Phenol-d5 (Surrogate Standard)	1	2	5	10	20	30	40	50	60
Nitrobenzene-d5 (Surrogate Standard)	1	2	5	10	20	30	40	50	60
2-Fluorobiphenyl (Surrogate Standard)	1	2	5	10	20	30	40	50	60
2,4,6-Tribromophenol (Surrogate Standard)	1	2	5	10	20	30	40	50	60
Terphenyl-d14 (Surrogate Standard)	1	2	5	10	20	30	40	50	60

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Calibration Levels SIM Standard, ug/mL

Naphthalene	0.2	0.5	1.0	2.0	5.0	10.0
Acenaphthylene	0.2	0.5	1.0	2.0	5.0	10.0
Acenaphthene	0.2	0.5	1.0	2.0	5.0	10.0
Fluorene	0.2	0.5	1.0	2.0	5.0	10.0
Phenanthrene	0.2	0.5	1.0	2.0	5.0	10.0
Pyrene	0.2	0.5	1.0	2.0	5.0	10.0
Benzo(a)anthracene	0.2	0.5	1.0	2.0	5.0	10.0
Chrysene	0.2	0.5	1.0	2.0	5.0	10.0
Benzo(b)fluoranthene	0.2	0.5	1.0	2.0	5.0	10.0
Benzo(k)fluoranthene	0.2	0.5	1.0	2.0	5.0	10.0
Benzo(a)pyrene	0.2	0.5	1.0	2.0	5.0	10.0
Indeno(1,2,3-cd)pyrene	0.2	0.5	1.0	2.0	5.0	10.0
Dibenz(a,h)anthracene	0.2	0.5	1.0	2.0	5.0	10.0
Anthracene	0.2	0.5	1.0	2.0	5.0	10.0
Fluoranthene	0.2	0.5	1.0	2.0	5.0	10.0
Benzo(g,h,i)perylene	0.2	0.5	1.0	2.0	5.0	10.0
2-Methylnaphthalene	0.2	0.5	1.0	2.0	5.0	10.0

Table 3

LVI Calibration Levels, Primary Standard, µg/mL³

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
1,4 Dioxane	1	2	5	10	20	30	40	50	60
Pyridine	1	2	5	10	20	30	40	50	60

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Table 3
LVI Calibration Levels, Primary Standard, µg/mL³

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
n-Nitrosodimethylamine	1	2	5	10	20	30	40	50	60
Dimethylformamide	1	2	5	10	20	30	40	50	60
Cyclohexanol	1	2	5	10	20	30	40	50	60
Aniline	1	2	5	10	20	30	40	50	60
Phenol	1	2	5	10	20	30	40	50	60
Bis(2-chloroethyl)ether	1	2	5	10	20	30	40	50	60
2-Chlorophenol	1	2	5	10	20	30	40	50	60
1,3-Dichlorobenzene	1	2	5	10	20	30	40	50	60
1,4-Dichlorobenzene	1	2	5	10	20	30	40	50	60
Benzyl alcohol	1	2	5	10	20	30	40	50	60
1,2-Dichlorobenzene	1	2	5	10	20	30	40	50	60
2-Methylphenol	1	2	5	10	20	30	40	50	60
2,2'-oxybis(1-chloropropane) ¹	1	2	5	10	20	30	40	50	60
3&4-Methylphenol	1	2	5	10	20	30	40	50	60
n-Nitroso-di-n-propylamine	1	2	5	10	20	30	40	50	60
Hexachloroethane	1	2	5	10	20	30	40	50	60
Nitrobenzene	1	2	5	10	20	30	40	50	60
Isophorone	1	2	5	10	20	30	40	50	60
2-Nitrophenol	1	2	5	10	20	30	40	50	60
2,4-Dimethylphenol	1	2	5	10	20	30	40	50	60
Benzoic acid	1	2	5	10	20	30	40	50	60
bis(2-Chloroethoxy)methane	1	2	5	10	20	30	40	50	60
2,4-Dichlorophenol	1	2	5	10	20	30	40	50	60
1,2,4-Trichlorobenzene	1	2	5	10	20	30	40	50	60
Naphthalene	1	2	5	10	20	30	40	50	60
4-Chloroaniline	1	2	5	10	20	30	40	50	60
Hexachlorobutadiene	1	2	5	10	20	30	40	50	60
4-Chloro-3-methylphenol	1	2	5	10	20	30	40	50	60
2-Methylnaphthalene	1	2	5	10	20	30	40	50	60
Hexachlorocyclopentadiene	1	2	5	10	20	30	40	50	60
2,4,6-Trichlorophenol	1	2	5	10	20	30	40	50	60
2,4,5-Trichlorophenol	1	2	5	10	20	30	40	50	60

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Table 3
LVI Calibration Levels, Primary Standard, µg/mL³

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
2-Chloronaphthalene	1	2	5	10	20	30	40	50	60
2-Nitroaniline	1	2	5	10	20	30	40	50	60
Dimethyl phthalate	1	2	5	10	20	30	40	50	60
Acenaphthylene	1	2	5	10	20	30	40	50	60
3-Nitroaniline	1	2	5	10	20	30	40	50	60
Acenaphthene	1	2	5	10	20	30	40	50	60
2,4-Dinitrophenol	2	4	10	20	40	60	80	100	120
4-Nitrophenol	2	4	10	20	40	60	80	100	120
Dibenzofuran	1	2	5	10	20	30	40	50	60
2,4-Dinitrotoluene	1	2	5	10	20	30	40	50	60
2,6-Dinitrotoluene	1	2	5	10	20	30	40	50	60
Diethylphthalate	1	2	5	10	20	30	40	50	60
4-Chlorophenyl phenyl ether	1	2	5	10	20	30	40	50	60
Fluorene	1	2	5	10	20	30	40	50	60
4-Nitroaniline	1	2	5	10	20	30	40	50	60
4,6-Dinitro-2-methylphenol	2	4	10	20	40	60	80	100	120
N-Nitrosodiphenylamine	1	2	5	10	20	30	40	50	60
Azobenzene ²	1	2	5	10	20	30	40	50	60
4-Bromophenyl phenyl ether	1	2	5	10	20	30	40	50	60
Hexachlorobenzene	1	2	5	10	20	30	40	50	60
Pentachlorophenol	1	2	5	10	20	30	40	50	60
Phenanthrene	1	2	5	10	20	30	40	50	60
Anthracene	1	2	5	10	20	30	40	50	60
Carbazole	1	2	5	10	20	30	40	50	60
Di-n-butyl phthalate	1	2	5	10	20	30	40	50	60
Fluoranthene	1	2	5	10	20	30	40	50	60
Benzidine	1	2	5	10	20	30	40	50	60
Pyrene	1	2	5	10	20	30	40	50	60
Butyl benzyl phthalate	1	2	5	10	20	30	40	50	60
3,3'-Dichlorobenzidine	1	2	5	10	20	30	40	50	60
Benzo(a)anthracene	1	2	5	10	20	30	40	50	60
Bis(2-ethylhexyl)phthalate	1	2	5	10	20	30	40	50	60

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Table 3
LVI Calibration Levels, Primary Standard, µg/mL³

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Chrysene	1	2	5	10	20	30	40	50	60
Di-n-octylphthalate	1	2	5	10	20	30	40	50	60
Benzo(b)fluoranthene	1	2	5	10	20	30	40	50	60
Benzo(k)fluoranthene	1	2	5	10	20	30	40	50	60
Benzo(a)pyrene	1	2	5	10	20	30	40	50	60
Indeno(1,2,3-cd)pyrene	1	2	5	10	20	30	40	50	60
Dibenz(a,h)anthracene	1	2	5	10	20	30	40	50	60
Benzo(g,h,i)perylene	1	2	5	10	20	30	40	50	60

¹2,2'-oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

²Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

³Lower concentration standards may be analyzed on a project specific basis.

LVI Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
Methyl methacrylate	1	2	5	10	20	30	40	50	60
Ethyl methacrylate	1	2	5	10	20	30	40	50	60
2-Picoline	1	2	5	10	20	30	40	50	60
n-Nitrosomethylethylamine	1	2	5	10	20	30	40	50	60
Methyl methanesulfonate	1	2	5	10	20	30	40	50	60
n-Nitrosodiethylamine	1	2	5	10	20	30	40	50	60
Ethyl methanesulfonate	1	2	5	10	20	30	40	50	60
Benzaldehyde	1	2	5	10	20	30	40	50	60
Pentachloroethane	1	2	5	10	20	30	40	50	60
Acetophenone	1	2	5	10	20	30	40	50	60
n-Nitrosopyrrolidine	1	2	5	10	20	30	40	50	60
n-Nitrosomorpholine	1	2	5	10	20	30	40	50	60
o-Toluidine	1	2	5	10	20	30	40	50	60
n-Nitrosopiperidine	1	2	5	10	20	30	40	50	60
O,o,o-Triethyl-Phosphorothioate	1	2	5	10	20	30	40	50	60
A,a-Dimethyl-phenethylamine	1	2	5	10	20	30	40	50	60

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LVI Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
2,6-Dichlorophenol	1	2	5	10	20	30	40	50	60
Hexachloropropene	1	2	5	10	20	30	40	50	60
Benzothiazole	1	2	5	10	20	30	40	50	60
Caprolactam	1	2	5	10	20	30	40	50	60
p-Phenylenediamine	1	2	5	10	20	30	40	50	60
n-Nitrosodi-n-butylamine	1	2	5	10	20	30	40	50	60
Safrole	1	2	5	10	20	30	40	50	60
Phthalic anhydride	1	2	5	10	20	30	40	50	60
1-Methylnaphthalene	1	2	5	10	20	30	40	50	60
1,2,4,5-Tetrachlorobenzene	1	2	5	10	20	30	40	50	60
Isosafrole, cis	.5	1	2.5	5	10	15	20	25	30
Isosafrole, trans	.5	1	2.5	5	10	15	20	25	30
Biphenyl	1	2	5	10	20	30	40	50	60
1,4-Dinitrobenzene	1	2	5	10	20	30	40	50	60
1,4-Naphthoquinone	1	2	5	10	20	30	40	50	60
1,3-Dinitrobenzene	1	2	5	10	20	30	40	50	60
Pentachlorobenzene	1	2	5	10	20	30	40	50	60
1-Naphthylamine	1	2	5	10	20	30	40	50	60
2-Naphthylamine	1	2	5	10	20	30	40	50	60
2,3,4,6-Tetrachlorophenol	1	2	5	10	20	30	40	50	60
5-Nitro-o-toluidine	1	2	5	10	20	30	40	50	60
Thionazin	1	2	5	10	20	30	40	50	60
1,3,5-Trinitrobenzene	1	2	5	10	20	30	40	50	60
Sulfotepp	1	2	5	10	20	30	40	50	60
Phorate	1	2	5	10	20	30	40	50	60
Phenacetin	1	2	5	10	20	30	40	50	60
Diallate 1	.5	1	2.5	5	10	15	20	25	30
Diallate 2	.5	1	2.5	5	10	15	20	25	30
Dimethoate	1	2	5	10	20	30	40	50	60
4-Aminobiphenyl	1	2	5	10	20	30	40	50	60
Pentachloronitrobenzene	1	2	5	10	20	30	40	50	60
Pronamide	1	2	5	10	20	30	40	50	60
Disulfoton	1	2	5	10	20	30	40	50	60

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LVI Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8	Level 9
2-sec butyl-4,6-dinitrophenol (Dinoseb)	1	2	5	10	20	30	40	50	60
Methyl parathion	1	2	5	10	20	30	40	50	60
4-Nitroquinoline-1-oxide	1	2	5	10	20	30	40	50	60
Parathion	1	2	5	10	20	30	40	50	60
Isodrin	1	2	5	10	20	30	40	50	60
Kepone	1	2	5	10	20	30	40	50	60
Famphur	1	2	5	10	20	30	40	50	60
Methapyrilene	1	2	5	10	20	30	40	50	60
Octachlorostyrene	1	2	5	10	20	30	40	50	60
Aramite 1	.5	1	2.5	5	10	15	20	25	30
Aramite 2	.5	1	2.5	5	10	15	20	25	30
p-(Dimethylamino)azobenzene	1	2	5	10	20	30	40	50	60
p-Chlorobenzilate	1	2	5	10	20	30	40	50	60
3,3'-Dimethylbenzidine	1	2	5	10	20	30	40	50	60
2-Acetylaminofluorene	1	2	5	10	20	30	40	50	60
Dibenz (a,j)acridine	1	2	5	10	20	30	40	50	60
Hexachlorophene	10	20	50	100	200	300	400	500	600
7,12-Dimethylbenz(a)anthracene	1	2	5	10	20	30	40	50	60
3-Methylcholanthrene	1	2	5	10	20	30	40	50	60
2-Fluorophenol (Surrogate Standard)	1	2	5	10	20	30	40	50	60
Phenol-d5 (Surrogate Standard)	1	2	5	10	20	30	40	50	60
Nitrobenzene-d5 (Surrogate Standard)	1	2	5	10	20	30	40	50	60
2-Fluorobiphenyl (Surrogate Standard)	1	2	5	10	20	30	40	50	60
2,4,6-Tribromophenol (Surrogate Standard)	1	2	5	10	20	30	40	50	60
Terphenyl-d14 (Surrogate Standard)	1	2	5	10	20	30	40	50	60

Table 4
Minimum Response Factor Criteria

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010

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Table 4
Minimum Response Factor Criteria

Semivolatile Compounds	Minimum Response Factor (RF)
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.600
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachlorethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010

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Table 4
Minimum Response Factor Criteria

Semivolatile Compounds	Minimum Response Factor (RF)
2,6-Dinitrotulene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010

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Table 4
Minimum Response Factor Criteria

Semivolatile Compounds	Minimum Response Factor (RF)
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

TestAmerica St. Louis has established a default minimum response factor of 0.01 for compound not identified in this table, except for Famphur, Hexachlorophene, Kepone, Phthalic Anhydride which have a minimum response factor of 0.001.

Table 5
Semi-Volatile Internal Standards with Corresponding Analytes*

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
1,4-Dioxane	Acetophenone	cis-Isosafrole	5-Nitro-o-toluidine	Benzidine	Benzo(b)fluoranthene
Methyl methacrylate	N-Nitrosopyrrolidine	1,2,4,5-Tetrachlorobenzene	4,6-Dinitro-2-methylphenol	Pyrene	Benzo(k)fluoranthene
Pyridine	N-Nitrosomorpholine	Hexachlorocyclopentadiene	N-Nitrosodiphenylamine	Terphenyl-d14	7,12-Dimethyl benz(a)anthracene
N-Nitrosodimethylamine	O-Toluidine	2,4,6-Trichlorophenol	Tri-n-butyl phosphate	Aramite 1	Hexachlorophene
N,N-Dimethylformamide	Nitrobenzene-d5	2,4,5-Trichlorophenol	Azobenzene	Kepone	Benzo(a)pyrene
Ethyl methacrylate	Nitrobenzene	2-Fluorobiphenyl	Sulfotep	Aramite 2	3-methylcholanthrene
2-Picoline	N-Nitrosopiperidine	trans-Isosafrole	Diallate 1	p-(dimethylamino) azobenzene	Indeno (1,2,3-cd) pyrene
N-Nitrosomethylethylamine	Isophorone	Biphenyl	1,3,5-Trinitrobenzene	Chlorobenzilate	Dibenz(a,h)anthracene
Methyl methanesulfonate	2-Nitrophenol	2-Chloronaphthalene	Phorate	3,3'-Dimethylbenzidine	Benzo(g,h,i)perylene
2-Fluorophenol	2,4-Dimethylphenol	2-Nitroaniline	4-Bromophenyl phenyl ether	Butyl benzyl phthalate	
Cyclohexanol	Bis (2-chloroethoxy) methane	1,4-Naphthoquinone	Phenacetin	2-Acetylaminofluorene	
N-Nitrosodiethylamine	o,o,o-Triethylphosphorothioate	1,4-Dinitrobenzene	Diallate 2	Famphur	
Ethyl methanesulfonate	Benzoic acid	Dimethylphthalate	Hexachlorobenzene	Benzo (a) anthracene	
Benzaldehyde	2,4-Dichlorophenol	1,3-Dinitrobenzene	Dimethoate	4,4'-methylenebis (2-Chloroaniline)	
Phenol-d5	a,a-Dimethylphenethylamine	Acenaphthylene	Atrazine	3,3'-Dichlorobenzidine	
Phenol	1,2,4-Trichlorobenzene	2,6-Dinitrotoluene	Tris(2-chloroethyl) phosphate	Chrysene	
Aniline	Naphthalene	3-Nitroaniline	4-Aminobiphenyl	Bis (2-ethylhexyl) phthalate	
Pentachloroethane	4-Chloroaniline	Acenaphthene	Pentachlorophenol	Di-n-octyl phthalate	
Bis (2-chloroethyl) ether	2,6-Dichlorophenol	2,4-Dinitrophenol	Pronamide		
2-Chlorophenol	Hexachloropropene	4-Nitrophenol	Pentachloronitrobenzene		
1,3-Dichlorobenzene	Hexachlorobutadiene	Dibenzofuran	Phenanthrene		

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Table 5
Semi-Volatile Internal Standards with Corresponding Analytes*

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
1,4-Dichlorobenzene	Benzothiazole	Pentachlorobenzene	Disulfoton		
1,2-Dichlorobenzene	Caprolactam	2,4-Dinitrotoluene	Anthracene		
Benzyl alcohol	N-Nitroso-di-n-butylamine	1-Naphthylamine	Dinoseb		
2-Methylphenol	p-Phenylenediamine	2-Naphthylamine	Carbazole		
Bis (2-chloroisopropyl) ether	4-Chloro-3-methylphenol	2,3,4,6-Tetrachlorophenol	Methyl parathion		
3,4-Methylphenol	Safrole	Diethylphthalate	Di-n-butyl phthalate		
N-Nitroso-di-n-propylamine	2-Methylnaphthalene	Fluorene	Parathion		
Hexachloroethane		4-Chlorophenyl phenyl ether	4-Nitroquinoline-1-oxide		
		Thionazin	Methapyrilene		
		4-Nitroaniline	Isodrin		
		2,4,6-Tribromophenol	Fluoranthene		

* ISTD assignment is based on instrument operating conditions and column type and may vary slightly from this listing.

Table 6a Acid Surrogates with Corresponding Analytes

2-Fluorophenol	Phenol-d5	2,4,6-Tribromophenol
none	Phenol	2,4,6-Trichlorophenol
	2-Chlorophenol	2,4,5-Trichlorophenol
	2-Methylphenol	2,4-Dinitrophenol
	3,4-Methylphenol	4-Nitrophenol
	2-Nitrophenol	2,3,4,6-
	2,4-Dimethylphenol	Tetrachlorophenol
	Benzoic acid	4,6-Dinitro-2-methyl
	2,4-Dichlorophenol	phenol
	2,6-Dichlorophenol	Pentachlorophenol
	4-Chloro-3-methyl	
	-phenol	

Table 6b Base/Neutral Surrogates with Corresponding Analytes

Nitrobenzene-d5	2-Fluorobiphenyl	Terphenyl-d14
1,4-Dioxane	Benzothiazole	Phenanthrene
Methyl methacrylate	Caprolactam	Disulfoton
Pyridine	N-Nitrosodi-n-butylamine	Anthracene
N-Nitrosodimethylamine	p-Phenylenediamine	Dinoseb
Dimethylformamide	Safrole	Carbazole
Ethyl methacrylate	2-Methylnaphthalene	Methyl parathion
2-Picoline	1-Methylnaphthalene	Di-n-butyl phthalate
N-Nitrosomethylethylamine	cis-Isosafrole	Parathion
Methyl methanesulfonate	1,2,4,5-Tetrachlorobenzene	4-Nitroquinoline-1-oxide
Cyclohexanol	Hexachlorocyclopentadiene	Methapyrilene
N-Nitrosodiethylamine	trans-Isosafrole	Isodrin
Ethyl methanesulfonate	Biphenyl	Fluoranthene
Benzaldehyde	2-Chloronaphthalene	Benzidine
Aniline	2-Nitroaniline	Pyrene
Pentachloroethane	1,4-Naphthoquinone	Aramite 1 & 2
Bis (2-Chloroethyl) ether	1,4-Dinitrobenzene	Kepone
1,2-Dichlorobenzene	Dimethyl phthalate	p-(dimethylamino)
1,3-Dichlorobenzene	1,3-Dinitrobenzene	azobenzene

Table 6b Base/Neutral Surrogates with Corresponding Analytes

Nitrobenzene-d5	2-Fluorobiphenyl	Terphenyl-d14
1,4-Dichlorobenzene	Acenaphthylene	Chlorobenzilate
Benzyl alcohol	2,6-Dinitrotoluene	3,3-Dimethylbenzidine
2,2'-oxybis (1-Chloro propane)	3-Nitroaniline	Butyl benzyl phthalate
Acetophenone	Acenaphthene	2-Acetylaminofluorene
N-Nitrosopyrrolidine	Dibenzofuran	Famphur
N-Nitrosodinpropylamine	Pentachlorobenzene	Benz (a) anthracene
N-Nitrosomorpholine	2,4-Dinitrotoluene	4,4'-Methylenebis (2-Chloro
o-Toluidine	1-Naphthylamine	-aniline)
Hexachloroethane	2-Naphthylamine	3,3'-Dichlorobenzidine
	Diethyl phthalate	Chrysene

Nitrobenzene	Fluorene	Bis (2-ethylhexyl) phthalate
N-Nitrosopiperidine	4-Chlorophenyl phenyl ether	Di-n-octyl phthalate
Isophorone	Thionazin	Benzo (b) fluoranthene
Bis (2-Chloroethoxy)	5-Nitro-o-toluidine	Benzo (k) fluoranthene
Methane	4-Nitroaniline	7,12-Dimethylbenz (a) anthracene
O,o,o-Triethylphosphoro	N-Nitrosodiphenylamine	Hexachlorophene
Thioate	Tri-n-butyl phthalate	Benzo (a) pyrene
A,a-Dimethylphenethyl	Azobenzene	3-methylcholanthrene
Amine	Sulfotepp	Indeno (1,2,3-cd) pyrene
1,2,4-Trichlorobenzene	Diallate 1 & 2	Dibenz (a,h) anthracene
Naphthalene	1,3,5-Trinitrobenzene	Benzo (g,h,i) perylene
4-Chloroaniline	Phorate	
Hexachloropropene	4-Bromophenyl phenyl ether	Phenacetin
Hexachlorobutadiene		
Hexachlorobenzene	Dimethoate	
	Atrazine	
	Tris(2-chloroethyl)phosphate	
	4-Aminobiphenyl	
	Pronamide	Pentachloronitrobenzene

APPENDIX 1

Standard preparations:

SCAN Intermediates

Internal Standard at 2000 ppm (I.S.): 1 mL to 2.5 = 800 ppm

8270:

8270 Surrogate at 5000 ppm: 0.4 mL to 10 mL CH₂CL₂ = 200 ppm

Benzoic Acid at 2000 ppm: 1 mL to 10 mL CH₂CL₂ = 200 ppm

Cyclohexanol at 2000 ppm: 1 mL to 10 mL CH₂CL₂ = 200 ppm

List 1 STD 1 at 1000 ppm: 2 mL to 10 mL CH₂CL₂ = 200 ppm

List 1 STD 2 at 2000 ppm: 1 mL to 10 mL CH₂CL₂ = 200 ppm

List 1 STD 7 at 2000 ppm: 1 mL to 10 mL CH₂CL₂ = 200 ppm

N,N-Dimethylformamide at 5000ppm: 0.4 mL to 10 mL CH₂CL₂ = 200 ppm

SIM Intermediate

Internal Standard at 2000 (I.S.): 0.1 mL to 2.5 mL = 80 ppm

CAL mix 5 at 2000 ppm: 1 mL to 10 mL CH₂CL₂ = 200 ppm

200 ppm PAH intermediate: 1 mL to 10 mL CH₂CL₂ = 20 ppm

8270 Surrogate at 5000 ppm: 0.04 mL to 10 mL CH₂CL₂ = 20 ppm

Working Standards Levels from 200 ppm Intermediates 8270:

FV = 1mL for all levels, 800 ppm I.S. addition to each level of 0.05 mL = 40 ppm

Level 1: 1 ppm = 0.005 mL of 200 ppm intermediate

Level 2: 2 ppm = 0.01 mL of 200 ppm intermediate

Level 3: 5 ppm = 0.025 mL of 200 ppm intermediate

Level 4: 10 ppm = 0.05 mL of 200 ppm intermediate

Level 5: 20 ppm = 0.1 mL of 200 ppm intermediate

Level 6 (CCV): 30 ppm = 0.15 mL of 200 ppm intermediate

Level 7: 40 ppm = 0.2 mL of 200 ppm intermediate

Level 8: 50 ppm = 0.25 mL of 200 ppm intermediate

Level 9: 60 ppm = 0.3 mL of 200 ppm intermediate

ICV: 30 ppm = 0.05 of 2nd source ICV 200 ppm intermediate

Working SIM Standards Levels from 20 ppm Intermediates:

FV = 1mL for all levels, 80 ppm I.S. addition to each level of 0.05 mL = 4 ppm

Level 1: 0.1 ppm = 0.005 mL of 20 ppm intermediate

Level 2: 0.2 ppm = 0.01 mL of 20 ppm intermediate

Level 3: 0.5 ppm = 0.025 mL of 20 ppm intermediate

Level 4 (CCV): 1 ppm = 0.05 mL of 20 ppm intermediate

Level 5: 2 ppm = 0.1 mL of 20 ppm intermediate

Level 6: 5 ppm = 0.25 mL of 20 ppm intermediate

Level 7: 10 ppm = 0.5 mL of 20 ppm intermediate

ICV: 1 ppm = 0.05 mL of 2nd source ICV 20 ppm intermediate

**Reagent ID: SV IS Mix_00103**

Type:	ASTD	Expiration Date:	06/30/2020
Description:	SV Internal Standard Mix	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Kuessner, Melissa A
Storage Location:	Semi-volatiles Standards Storage	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0104707
Creation Date:	08/18/2014	Vendor Cat #:	31206
Container(s):	429301		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,4-Dichlorobenzene-d4					2000.00000	ppm
Acenaphthene-d10					2000.00000	ppm
Chrysene-d12					2000.00000	ppm
Naphthalene-d8					2000.00000	ppm
Perylene-d12					2000.00000	ppm
Phenanthrene-d10					2000.00000	ppm

**Reagent ID: List 1 Std 1_00005**

Type:	ASTD	Expiration Date:	02/13/2015
Description:	8270 List 1 Std #1 Megamix parent std	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Kuessner, Melissa A
Storage Location:	Semi-volatiles Standards Storage	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0101615
Creation Date:	08/11/2014	Vendor Cat #:	567672
Container(s):	423987		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,1'-Biphenyl					1000.00000	ppm
1,2,4,5-Tetrachlorobenzene					1000.00000	ppm
1,2,4-Trichlorobenzene					1000.00000	ppm
1,2-Dichlorobenzene					1000.00000	ppm
1,3-Dichlorobenzene					1000.00000	ppm
1,3-Dinitrobenzene					1000.00000	ppm
1,4-Dichlorobenzene					1000.00000	ppm
1,4-Dioxane					1000.00000	ppm
1-Methylnaphthalene					1000.00000	ppm
2,2'-oxybis[1-chloropropane]					1000.00000	ppm
2,3,4,6-Tetrachlorophenol					1000.00000	ppm
2,4,5-Trichlorophenol					1000.00000	ppm
2,4,6-Trichlorophenol					1000.00000	ppm
2,4-Dichlorophenol					1000.00000	ppm
2,4-Dimethylphenol					1000.00000	ppm
2,4-Dinitrophenol					2000.00000	ppm
2,4-Dinitrotoluene					1000.00000	ppm
2,6-Dinitrotoluene					1000.00000	ppm
2-Chloronaphthalene					1000.00000	ppm
2-Chlorophenol					1000.00000	ppm
2-Methylnaphthalene					1000.00000	ppm
2-Methylphenol					1000.00000	ppm
2-Nitroaniline					1000.00000	ppm
2-Nitrophenol					1000.00000	ppm
3 & 4 Methylphenol					1000.00000	ppm
3-Nitroaniline					1000.00000	ppm
4,6-Dinitro-2-methylphenol					2000.00000	ppm

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**Reagent ID: List 1 Std 1_00005**

Type:	ASTD	Expiration Date:	02/13/2015
Description:	8270 List 1 Std #1 Megamix parent std	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Kuessner, Melissa A
Storage Location:	Semi-volatiles Standards Storage	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0101615
Creation Date:	08/11/2014	Vendor Cat #:	567672
Container(s):	423987		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
4-Bromophenyl phenyl ether					1000.00000	ppm
4-Chloro-3-methylphenol					1000.00000	ppm
4-Chloroaniline					1000.00000	ppm
4-Chlorophenyl phenyl ether					1000.00000	ppm
4-Nitroaniline					1000.00000	ppm
4-Nitrophenol					2000.00000	ppm
Acenaphthene					1000.00000	ppm
Acenaphthylene					1000.00000	ppm
Acetophenone					1000.00000	ppm
Aniline					1000.00000	ppm
Anthracene					1000.00000	ppm
Azobenzene					1000.00000	ppm
Benzo[a]anthracene					1000.00000	ppm
Benzo[a]pyrene					1000.00000	ppm
Benzo[b]fluoranthene					1000.00000	ppm
Benzo[g,h,i]perylene					1000.00000	ppm
Benzo[k]fluoranthene					1000.00000	ppm
Benzyl alcohol					1000.00000	ppm
Bis(2-chloroethoxy)methane					1000.00000	ppm
Bis(2-chloroethyl)ether					1000.00000	ppm
Bis(2-ethylhexyl) phthalate					1000.00000	ppm
Butyl benzyl phthalate					1000.00000	ppm
Carbazole					1000.00000	ppm
Chrysene					1000.00000	ppm
Dibenz(a,h)anthracene					1000.00000	ppm
Dibenzofuran					1000.00000	ppm
Diethyl phthalate					1000.00000	ppm

**Reagent ID: List 1 Std 1_00005**

Type:	ASTD	Expiration Date:	02/13/2015
Description:	8270 List 1 Std #1 Megamix parent std	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Kuessner, Melissa A
Storage Location:	Semi-volatiles Standards Storage	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0101615
Creation Date:	08/11/2014	Vendor Cat #:	567672
Container(s):	423987		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Dimethyl phthalate					1000.00000	ppm
Di-n-butyl phthalate					1000.00000	ppm
Di-n-octyl phthalate					1000.00000	ppm
Fluoranthene					1000.00000	ppm
Fluorene					1000.00000	ppm
Hexachlorobenzene					1000.00000	ppm
Hexachlorobutadiene					1000.00000	ppm
Hexachlorocyclopentadiene					1000.00000	ppm
Hexachloroethane					1000.00000	ppm
Hexadecane					1000.00000	ppm
Indeno[1,2,3-cd]pyrene					1000.00000	ppm
Isophorone					1000.00000	ppm
Naphthalene					1000.00000	ppm
n-Decane					1000.00000	ppm
Nitrobenzene					1000.00000	ppm
N-Nitrosodimethylamine					1000.00000	ppm
N-Nitrosodi-n-propylamine					1000.00000	ppm
n-Octadecane					1000.00000	ppm
Pentachlorophenol					2000.00000	ppm
Phenanthrene					1000.00000	ppm
Phenol					1000.00000	ppm
Pyrene					1000.00000	ppm
Pyridine					1000.00000	ppm

**Reagent ID: 8270 Surr Std_00019**

Type:	ASTD	Expiration Date:	02/28/2018
Description:	8270 Surrogate parent standard	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Winkler, Jeff S
Storage Location:	Semi-volatiles Standards Storage	Vendor:	Restek
Reagent Volume:	5.000 mL	Vendor Lot #:	A093638
Creation Date:	08/28/2013	Vendor Cat #:	567685
Container(s):	198745		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
2,4,6-Tribromophenol					5000.00000	ppm
2-Fluorobiphenyl (Surr)					5000.00000	ppm
2-Fluorophenol					5000.00000	ppm
Nitrobenzene-d5					5000.00000	ppm
Phenol-d5					5000.00000	ppm
Terphenyl-d14					5000.00000	ppm

**Title: DETERMINATION OF VOLATILE ORGANICS BY GC/MS
[SW-846 8260; EPA 624; DW 524.2]**

Approvals (Signature/Date):	
 Andrew Buettner Volatiles Organics Manager	3-6-17 Date
 Michael Ridenhower Health & Safety Manager / Coordinator	3/6/17 Date
 Kristen Ely Quality Assurance Manager	3/6/17 Date
for  Elyne Wild Laboratory Director	3/6/17 Date

This SOP was previously identified as SOP No. ST-MS-0002 Rev. 25

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges and other solid matrices.
- 1.2 This SOP is applicable to SW-846 method 8000B, 8000C, 8000D, 8260B, 8260C, Drinking water method 524.2, and EPA 624.
- 1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.
- 1.4 The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 1 to 200 µg/L for 5 mL waters, 0.5 to 40 µg/L for 25 mL purge waters, 5 to 200 µg/kg for low-level soils, and 250 to 25,000 µg/kg for high-level soils.
- 1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.5.1 Additional compounds may be amenable to this method. The minimum requirement for non-standard analytes is that the reporting limit be set at the lowest required concentration that can actually be detected by the instrument, and when an MDL study can not be conducted, the MDL be set equal to the reporting limit.

2.0 SUMMARY OF METHOD

- 2.1 Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.
- 2.2 In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and flushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components that are detected with a mass spectrometer.
- 2.3 Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.
- 2.4 The use of selected ion monitoring (SIM) is acceptable for applications requiring quantitation limits below the normal range of electro impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification, since less mass spectral information is available. Instead of scanning everything in a retention time range, SIM looks for specific ions (qualitative and quantitative) that are placed in retention time groups. The ions used for qualitative and quantitative purposes are the same for scan and SIM analysis. In addition to using the Scan mode, the laboratory also runs a SIM method for 1,4 dioxane. Details are available in Appendix 1 of this SOP.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 SIM – selected ion monitoring
- 3.3 LLOQ – lower limit of quantitation (Reporting limit)

4.0 INTERFERENCES

- 4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water or purchased HPLC water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants.
- 4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. Trip Blanks, prepared from reagent water, can serve as a check on conditions during transportation from the field to the laboratory.
- 4.3 Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.
- 4.4 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.
- 4.5 Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent can be used. A blank spiked with this agent must be analyzed with the sample because of the non-target interferences associated with the agent.
- 4.6 Methylene Chloride, Acetone, and 2-Butanone are potential laboratory contaminants. Concentrations up to five times the level observed in the method blank, in associated laboratory samples, may be attributed to the presence of these compounds in the laboratory.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 **SPECIFIC SAFETY CONCERNS OR REQUIREMENTS**

- 5.2.1 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.2.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.
- 5.2.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 PRIMARY MATERIALS USED

- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Micro syringes- 1.0 µL, 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL, 1000 µL. Hamilton 1700 series
- 6.2 Volumetric flasks, Class A
 - 6.2.1 Thoroughly rinse glassware with purge and trap grade methanol 3 times
 - 6.2.2 Place glassware in warm to hot oven to bake off the methanol
 - 6.2.3 Allow glassware to cool to room temperature before use.
- 6.3 Analytical Balance, capable of weighing ± 0.001 g
- 6.4 Syringe: 5 or 25 mL glass with Luerlok tip, if applicable to the purging device.
- 6.5 Vials: 20 and 40 mL with Teflon lined screw caps
- 6.6 Spatula: Disposable wooden tongue depressors
- 6.7 Disposable pipettes
- 6.8 pH paper: Wide range and 0.3 to 2.3

- 6.9 Glass beads: Store in a drying oven.
- 6.10 Gas Chromatograph/Mass Spectrometer System:
 - 6.10.1 Gas Chromatograph: Hewlett Packard GC 5890 and Agilent 6890 system with temperature programming.
 - 6.10.2 Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorb heater.
 - 6.10.3 Gas Chromatographic Capillary Columns:
 - 6.10.3.1 Mass Spectrometer: Hewlett Packard 5970, 5972 and 5973 mass spectrometers capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode.
 - 6.10.3.2 Columns used: Restek RTX-VMS, 30 meters, 0.25mm inside diameter, 1.4 µm film thickness; Restek RTX-VMS, 40 meter, 0.18mm inside diameter, 1 µm film thickness
 - 6.10.4 GC/MS interface: In general split/splitless injector are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.
 - 6.10.5 Instrument setup and operating conditions are maintained in each instruments daily maintenance calendar.
 - 6.10.6 Data System:
 - 6.10.6.1 ChemStation software system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.
 - 6.10.6.2 Target software system allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software allows integrating the abundances in any EICP between for a specified time or scan-number limit. Also, for the non-target compounds, software a mass spectrum that meets the required criteria when 50ng of 4-Bromofluorobenzene (BFB) are must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library.
- 6.10.5.2.1 Data Library: NIST 98
- 6.11 Carrier gas: Ultra high purity helium
- 6.12 Make up gas: Ultra high purity helium
- 6.13 Purge gas: Gasified liquid-Nitrogen or Ultra high purity helium.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 Methanol: Purge and Trap Grade
- 7.3 Water: HPLC grade or equivalent.
- 7.4 See recipes for standards and QC samples in the Reagents section of TALS.[Also examples included in Appendix 2 and 3.

- 7.5 Calibration Standards and Surrogates
 - 7.5.1 Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at 0 to -20 °C.
 - 7.5.2 Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer.
 - 7.5.2.1 See SOP: ST-QA-0002 for expiration date criteria.
- 7.6 Initial calibration verification (ICV) standards are similar to calibration standards, but are from a completely different source.
- 7.7 Internal standards: Internal standards are added to all samples, standards, and blank analyses.
- 7.8 Tuning standard: A 25 ng/μL 4-Bromofluorobenzene standard is made up that will deliver 50 ng (or 25 ng) on column upon injection.
- 7.9 Sodium bisulfate, crystal
- 7.10 Ottawa sand
- 7.11 Ascorbic acid, ACS Reagent Grade, granular
- 7.12 Sodium Thiosulfate, ACS Reagent Grade, granular

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should review the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Aromatic volatiles water samples are preserved with 1:1 HCl and stored at 4 ± 2 °C. Analysis hold time is 14 days from collection.
- 8.3 Aqueous samples are stored in glass containers with Teflon lined septa at 4 ± 2 °C, with minimum headspace.
- 8.4 Soil samples are refrigerated at 4 ± 2 °C. Analysis hold time is 14 days from collection.
 - 8.4.1 Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4 ± 2 °C. The extracts are stored with minimum headspace.
- 8.5 For 5035 analysis
 - 8.5.1 Solid samples, for low level analysis, may be field preserved with sodium bisulfate solution, or collected unpreserved using the Encore™ (or equivalent) sampler and shipped to the laboratory within 48 hours of sampling.
 - 8.5.1.1 Following shipment back to the lab the soil is preserved with sodium bisulfate or the sample is extruded into an empty, clean sealed vial and frozen within 48 hours of sampling.
 - 8.5.1.2 It is recommended that two Encore (or equivalent) samplers be used for each field sample position, to allow for any reruns than may be necessary.

- 8.5.2 Solid samples, for medium level analysis, may be field extracted with methanol, or collected unpreserved using the Encore (or equivalent)TM sampler and shipped to the laboratory within 48 hours of sampling.
- 8.5.2.1 It is recommended that two Encore (or equivalent) samplers be used for each field sample position, to allow for any reruns than may be necessary.
 - 8.5.2.2 Solid samples – field extracted with methanol
 - 8.5.2.2.1. Prepare a 2 oz sample container by adding 25 mL purge and trap grade methanol. (If a 5 g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).
 - 8.5.2.2.2. Seal the bottle and attach a label.
 - 8.5.2.2.3. Weigh the bottle to the nearest 0.01 g and note the weight on the label.
 - 8.5.2.2.4. Ship with appropriate sampling instructions.
 - 8.5.2.2.4.1. At client request, the methanol addition and weighing may also be performed in the field.
 - 8.5.2.2.4.2. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.
 - 8.5.2.3 Solid samples – field extracted with methanol
 - 8.5.2.3.1. When the samples are returned to the lab, extrude the (nominal) 5 g (or 25 g) sample into a pre-weighed VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.
- 8.6 An additional sample is collected for percent moisture determination.
- 8.7 EPA 524.2 And 624 Sample Dechlorination and Preservation
- 8.7.1 If specified by the client that actual drinking water samples(Method 524.2) and/or wastewater samples (Method 624) containing residual chlorine are to be analyzed by the laboratory, the following procedure shall be used:
 - 8.7.1.1 Finished drinking water samples suspected of containing residual chlorine shall have 25 mg of ascorbic acid added to each 40 mL VOA vial prior to filling. If the requested target analytes are not gases at room temperature or are not listed in Table 7 of EPA Method 524.2, sodium thiosulfate is recommended to reduce residual chlorine (3 mg sodium thiosulfate for each 40 mL VOA vial). Wastewater samples for Method 624 will be collected in vials pre-filled with sodium thiosulfate.
 - 8.7.1.2 If the sample containers are prepared by the laboratory for filling by the sample collector, the ascorbic acid or sodium thiosulfate will be placed into unpreserved VOA vials (do not mix the ascorbic acid or sodium thiosulfate with the HCl used for preservation).
 - 8.7.1.3 It will be the sample collector's responsibility to add additional ascorbic acid or sodium thiosulfate if a diethyl-p-phenylenediamine (DPD) test kit indicates residual chlorine in excess of 5 mg/L (25 mg ascorbic acid or 3 mg sodium thiosulfate per each 5 mg/L of residual chlorine).
 - 8.7.1.4 After filling the VOA vial, the sample collector should adjust the sample to pH < 2 by carefully adding two drops of 1:1 HCl for each 40 mL of sample. Samples should be sealed and mixed for one minute. (Note: If the sample is to be analyzed for trihalomethanes (THMs) only, the HCl preservation step may be omitted if sodium thiosulfate was used for dechlorination.)
 - 8.7.1.5 If a sample foams vigorously when HCl is added, the sample should be discarded. Fill fresh VOA vials containing ascorbic acid or sodium thiosulfate, but do not acidify. A note should be placed on the chain-of-

custody that is submitted with the samples that they were “not acidified”. These samples have a 24 hold time if target analytes other than THMs are to be analyzed.

8.7.1.6 Samples should be maintained at 4 ± 2 °C until analysis.

8.7.1.7 If the proper preservative was not added to the empty vial prior to sampling, the sample will need to be checked post analysis for residual chlorine. If present, the analytical results will be flagged and a non-conformance memo will be written to document the issue.

8.8 At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and sub-sampled in the lab.

8.8.1 This is an older procedure based on method 5030.

9.0 QUALITY CONTROL

9.1 Batch

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), Matrix Spike (MS) and Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual “sample batch” does not exceed 20 environmental samples. MS/MSD must be performed for each separate QC code.

9.1.5 Unless medium level (i.e. an extraction batch) the blank, LCS and MS/MSD MUST run in the same 12 hour clock. The definition of a non-extraction batch is a maximum of 20 samples run in a 12 hr clock.

9.2 Method Blank

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 For Water analyses, the method blank is comprised of HPLC water.

9.2.3 For Soil analyses, the method blank is comprised of glass beads, or Ottawa sand (required by South Carolina)

9.2.4 For water and low soil method 8260 and 524.2 analyses

9.2.4.1 A method blank must be analyzed with every 12 hour analytical clock and/or with each batch of 20 samples, whichever occurs first.

9.2.5 For water method 624 analyses

9.2.5.1 A method blank must be analyzed with every 24 hour analytical clock and with each batch of 20 samples, whichever occurs first..

9.2.6 For medium level soil method 8260 analyses

9.2.6.1 A method blank must be prepared with every medium level soil batch (20 or fewer samples of the same matrix). The medium level method blank is tied to

an extraction and does not require repeated analysis with each sample analysis batch.

9.2.7 Method blank population studies shall be performed periodically. The data from such studies shall be used to identify systemic contamination issues.

9.3 Laboratory Control Sample

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 For Water analyses, the LCS is comprised of HPLC water fortified with Volatiles.

9.3.2.1 Under current methodology, the CCV may also serve as the LCS; however, due to limitations in the LIMS, the CCV and LCS should **never** be generated from the same injection.

9.3.3 For Soil analyses, the LCS is comprised of glass beads or Ottawa sand fortified with Volatiles. South Carolina requires the use of Ottawa sand.

9.3.4 For method 8260 and 524.2 analyses

9.3.4.1 A LCS must be analyzed with every 12 hour analytical clock and with each batch of 20 samples, whichever occurs first..

9.3.5 For method 624 analyses

9.3.5.1 A LCS must be analyzed with every 24 hour analytical clock and with each batch of 20 samples.

9.3.5.2 A LCS must be prepared with every high level soil batch (20 or fewer samples of the same matrix).

9.3.6 For medium level soil method 8260 analyses

9.3.7 An LCS must be prepared with every medium level soil batch (20 or fewer samples of the same matrix).

9.3.7.1 The medium level LCS is tied to an extraction and does not require repeated analysis with each sample analysis batch.

9.4 Matrix Spike/Matrix Spike Duplicate

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 **8260** – A MS/MSD must be analyzed with every 12 hour analytical clock and with each batch of 20 samples, whichever occurs first..

9.4.3 **524.2** – Method 524.2 does not require a MS/MSD be performed though it may be requested by the client. In the absence of client QC criteria for this MS/MSD, the laboratory will use 70 – 130% recovery limits, and RPD of 20%, as advisory criteria.

9.4.4 **624** - A MS/MSD must be analyzed with every 24 hour analytical clock and with each batch of 20 samples, whichever occurs first.

9.5 Surrogate

9.5.1 A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.

9.5.2 Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.

9.6 Procedural Variations/ Nonconformance and Corrective Action

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See

SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Internal standard calibration is used.

10.1.1 Internal standard calibration is used. The internal standards are listed in this SOP. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard.

10.2 Instrument Tuning

10.2.1 Each GC/MS system must be hardware-tuned to meet the abundance criteria, listed in this SOP, for a maximum of a 50 ng (8260 and 624) and 25 ng (524.2) injection or purging of BFB. These criteria must be met

10.2.1.1 **8260** – for each 12 hour time period

10.2.1.2 **524.2** – for each 12 hour time period

10.2.1.3 **624** – for each 24 hour time period

10.2.1.3.1. The time period begins at the moment of injection of BFB.

10.2.2 Inject the method appropriate amount of GC/MS tuning standard into the GC/MS system. Obtain a background-corrected mass spectra of BFB and confirm that all the key m/z criteria in [Table 2](#) are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3 Initial Calibration

10.3.1 Prepare a multi-point calibration curve by fortifying 5 mL, or 25 mL depending on the required analysis, of HPLC water with incrementing concentrations of Volatiles standard.

10.3.1.1 Samples preserved with sodium bisulfate require a sodium bisulfate curve.

10.3.2 The initial calibration contains a minimum of 5 points, for each target analyte (6 points are required for a quadratic fit). The low level standard must be at or below the reporting limit (LLOQ). The other standards define the working range of the detector, with the highest level standard establishing the linear range of the instrument.

10.3.2.1 Note 624 requires a minimum 3 point calibration. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector, with the highest level standard establishing the linear range of the instrument.

10.3.3 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, any significant changes in instrument operating parameters, and major instrument maintenance.

10.3.4 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

10.3.5 Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount concentration of analyte in the calibration standard is defined as the response factor (RF) or calibration factor (CF).

10.3.6 **Initial Calibration Criteria (SW 8000B/8260B):**

10.3.6.1 The % RSD of the calibration check compounds (CCC) must be less than 30%. CCC are as follows:

- Vinyl Chloride
- 1,1-Dichloroethene
- Chloroform

- 1,2-Dichloropropane
- Toluene
- Ethylbenzene

10.3.6.2 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.3.6.3 The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor.

10.3.6.3.1. A **minimum response factor of 0.01** must be achieved for all other volatile target analytes.

Compound	Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	>0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

10.3.6.4 If all %RSDs in the initial calibration are $\leq 15\%$, then all analytes may use average response factor for calibration.

10.3.6.5 The analyst should evaluate analytes with %RSD > 15%.

10.3.6.5.1. Evaluate whether the problem is related to the analytical range. The low standard or the high standard having a response that is out of line with the others typically expresses this. For criteria regarding the removing calibration points from the curve, refer to STL policy P-T-001, Selection of Calibration Points.

10.3.6.6 For SW846 Method 8000B

10.3.6.6.1. If the average of all %RSDs in the calibration is > 15%, the analyst should consider instrument maintenance to improve the linearity of response.

10.3.6.6.2. When a linear regression curve is used, the intercept of the curve at zero response must be less than $\pm$ the reporting limit for the analyte. Client requirements may be tighter.(see Section 10.3.7.6.1).

10.3.6.6.3. If a linear regression curve is used, r must be ≥ 0.995 .

10.3.6.6.4. When a linear model is employed use of $1/\text{Concentration}^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve and to better control the intercept at zero response.

10.3.6.6.5. The regression may not be forced through zero when using a linear model. South Carolina compliance work does NOT allow forcing through zero. See specific client requirements to determine if this technique is allowed for specific sample groups.

10.3.7 Initial Calibration criteria (8000C/8000D/8260C and 524.2):

10.3.7.1 Minimum Response Factors (SW 8260C only)

- 10.3.7.1.1. See [Table 4](#) in this SOP for recommended minimum response factors prescribed by method 8260. For analytes not given a minimum response factor by the method, TestAmerica St. Louis has established a default of 0.01 and 0.001 depending on the nature of the compound.
- 10.3.7.2 SW-846 8260C and 524.2 chromatographic methods allow the use of both linear and non-linear models for the calibration data.
- 10.3.7.3 The first way is to begin with the simplest approach, the linear model through the origin, and then progress through other options until the calibration acceptance criteria are met. The second way is to use technical knowledge of the detector response to the target compound to choose the calibration model.
- 10.3.7.4 The option for non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance.
- 10.3.7.5 **Linear calibration using the average response factor**
- 10.3.7.5.1. The Relative Standard Deviation (RSD) of the calibration points from the curve used must be $\leq 20\%$ for each target analyte.
- 10.3.7.5.2. If the %RSD in the initial calibration is $> 20\%$, then calibration using a linear regression may be employed.
- 10.3.7.6 **Linear calibration using a least squares regression**
- 10.3.7.6.1. The intercept of a linear calibration at zero response (i.e. the y-intercept) must have an absolute value less than the reporting limit for each analyte. Client requirements may be tighter, please check Client Requirement Memorandum (CRM) if identified in comments.
- 10.3.7.6.2. r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990 .
- 10.3.7.6.3. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve.
- 10.3.7.6.4. It is not necessary to re-analyze a low concentration standard, rather the data system can recalculate the concentrations.
- 10.3.7.6.5. The recalculated concentration of the low calibration point should be within $\pm 30\%$ of the standard's true concentration.
- 10.3.7.6.5.1. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control" and corrective action should be taken.
- 10.3.7.7 **Linear calibration using a least squares regression, forcing thru zero**
- 10.3.7.7.1. Forcing the curve through zero is not the same as including the origin as a fictitious point in the calibration. In essence, if the curve is forced through zero, the intercept is set to 0 *before* the regression is calculated, thereby setting the bias to favor the low end of the calibration range by "pivoting" the function around the origin to find the best fit and resulting in one less degree of freedom. It may be appropriate to force the regression through zero for some calibrations.
- 10.3.7.7.2. Curve must still meet criteria in 10.3.7.6.1 and 10.3.7.6.2
- 10.3.7.8 **Linear calibration using a least squares regression, weighting of data points**
- 10.3.7.8.1. In a linear model, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. For this reason it may be preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$

weighting (often called $1/X^2$ weighting) to improve accuracy at the low end of the curve.

10.3.7.8.2. Curve must still meet criteria in 10.3.7.6.1 and 10.3.7.6.2.

10.3.7.9 Non-linear calibration

10.3.7.9.1. In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches have not met the acceptance criteria, a quadratic model may be employed. All quadratic curves require a minimum of 6 pts.

10.3.7.9.2. It is not EPA's intent to allow non-linear calibration to be used to compensate for detector saturation or to avoid proper instrument maintenance. Thus, non-linear calibrations are not to be employed for analytes shown to consistently exhibit linear calibration for the analytes of interest.

10.3.7.9.3. The intercept of the curve at zero response must be less than $\pm$ the reporting limit for the analyte. (Some clients may have tighter criteria; check Client Requirement Memos)

10.3.7.9.4. r (correlation coefficient) must be ≥ 0.995 OR r^2 (coefficient of difference) must be ≥ 0.990 .

10.3.7.9.5. The regression may be forced through zero when using a non-linear model. See specific client requirements to determine if this technique is allowed for specific sample groups.

10.3.7.9.6. The use of quadratic curves is not allowed for South Carolina compliance samples.

10.3.8 Acceptance criteria independent of calibration model

10.3.8.1 Either of the procedures Percent Error or Relative Standard Error (RSE) may be used to determine calibration function acceptability for linear and non-linear curves. Both evaluate the difference between the measured and the true amounts or concentrations used to create the model.

$$10.3.8.2 \quad \%Error = \frac{x_i - x'_i}{x_i} \times 100$$

10.3.8.3 Where x'_i = measured amount of the analyte at calibration level i in mass or concentration units and x_i = the true amount of the analyte at calibration level i in mass or concentration units.

10.3.8.4 % Error should be $\leq 30\%$. For some data uses $\leq 50\%$ may be acceptable for the lowest calibration point.

10.3.8.5 Calculation of Relative Standard Error:

10.3.8.6

$$RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

Where X_i = True amount of the calibration level i , in mass or concentration units
 X'_i = measured amount of analyte in calibration level i , in mass or concentration units

p = number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3,)

n = number of calibration points

10.3.8.7 RSE acceptance criteria for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If not defined in the method, use $\pm 20\%$

10.3.9 624 Criteria

- 10.3.9.1 Method 624 only requires a 3 point calibration. We routinely perform a 5 point calibration; however, 2 points may be removed from the curve if necessary to meet 624 calibration criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
- 10.3.9.2 The Relative Standard Deviation (RSD) of the calibration points from the curve used must be < 35%.
- 10.3.9.3 If the %RSD in the initial calibration is $\geq 35\%$, then calibration using a linear regression may be employed.
 - 10.3.9.3.1. If a linear regression curve is used, the intercept of the curve at small zero response must be less than $\pm$ the reporting limit for the analyte. It is recommended that for linear regression curves the line be set through the origin.
- 10.3.9.4 Use of $1/\text{Concentration}^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.
 - 10.3.9.4.1. Weighting of data points
 - 10.3.9.4.2. The points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/\text{Concentration}^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4 Initial Calibration Verification (ICV)

- 10.4.1 The initial calibration verification standard is a different standard source than the one used for the initial calibration
- 10.4.2 An ICV must be performed with each initial calibration.
- 10.4.3 The ICV performance must be within $\pm 30\%$ Difference for average CF and average RF curves and %Drift is used for evaluating linear and quadratic curves.
 - 10.4.3.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard.
- 10.4.4 Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).
 - 10.4.4.1 Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.
 - 10.4.4.2 Variance among vendor supplied standards for a few compounds is not atypical for long analyte lists. All ICV failures that cannot be associated to laboratory error will require the immediate analysis of a third standard in an attempt to characterize the bias. If a third standard is not in the laboratory it must be ordered immediately.

10.5 Continuing Calibration Verification.(CCV)

- 10.5.1 At the start of each 12 hour period (8260) or 24 hour period (624) the GC/MS tuning standard must be analyzed. A 50ng injection of BFB must result in a mass spectrum for BFB which meets the criteria. See [Table 2](#) in this SOP.

- 10.5.2 Following a successful BFB analysis, the continuing calibration standard(s) are analyzed. The standards must contain all volatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration
- 10.5.3 A CCV standard is analyzed every analysis tune clock immediately following the BFB tune.
- 10.5.3.1 **8260 and 524.2** – for each 12-hour tune time period
 - 10.5.3.2 **624** – for each 24-hour tune time period
- 10.5.4 The CCV can be the same source or a second source from the calibration.
- 10.5.5 The internal standard response must be within -50 – 100% of the response in the mid level of the initial calibration. The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.
- 10.5.6 8000B/8260B criteria:
- 10.5.6.1 The SPCC compounds must have a minimum response factor (see Initial Calibration SPCC criteria)
 - 10.5.6.2 The percent difference of the CCC compounds compared to the the initial calibration must be $\leq 20\%$.
 - 10.5.6.3 In addition, if CCC compounds are not part of the target list, the percent difference or drift of all analytes must be $\leq 20\%$, with allowance being made for up to six target compounds to have percent drift greater than 20%. Due to poor responses, the following compounds are allowed to have a %D > 60%, but less than 100%: Cyclohexane, 2-Chloroethyl vinyl ether, 2-Nitropropane, 1,4-Dioxane, Tetrahydrofuran, n-butanol and Isobutanol.
 - 10.5.6.3.1 South Carolina does not allow this exception for poor responders. If the CCC analytes are not in the target compound list all analytes must be with 30% difference or drift for calibration verification.
 - 10.5.6.4 In addition, if any target analyte's %D is > 20%, the entire target analyte list must be averaged. The average %D must be $\leq 20\%$. [NOTE: Averaging the entire target analyte list (or using the "Gand Mean") is not allowed for South Carolina compliance work.]
 - 10.5.6.5 If none of the identified CCCs are in the special calibration, the project specific target analytes %D must be < 20% or a maximum %D as agreed with the client. If there is a special project/client %D criteria it is noted on the client requirement sheet; otherwise a maximum 20% D is applied.
 - 10.5.6.6 There are instances where a small subset of the routinely calibrated analytes are needed for analysis (e.g. dilutions or project with abbreviated target analyte lists). In cases where the target analytes for analysis constitutes less than half of the total number of analytes in the calibration standard, apply the following:
 - 10.5.6.6.1. The SPCC compounds must have a response factor (see initial calibration SPCC criteria) if the SPCC is a target of interest.
 - 10.5.6.6.2. The percent difference of the CCC compounds from the initial calibration must be $\leq 20\%$, if the CCC is a target of interest. In addition, the percent difference or drift of all analytes of concern must be $\leq 60\%$, with allowance being made for, Cyclohexane, 2-Chloroethyl vinyl ether, 2-Nitropropane, 1,4-Dioxan, Tetrahydrofuran, n-butanol and Isobutanol. These compounds are allowed to have a %D > 60% but less than 100%.
 - 10.5.6.6.2.1. South Carolina does not allow this exception for poor responders. If the CCC analytes are not in the target compound list all analytes must be with 30% difference or drift for calibration verification.

10.5.6.6.3. For target analyte lists with more than 20 compounds: if any target analyte's %D is > 15%, the entire target analyte list must be averaged. The average %D must be ≤ 15%. [NOTE: This averaging is not allowed for South Carolina compliance work.]

10.5.6.6.4. For target analyte lists with less than 20 compounds: each target analyte %D must be < 15% or a maximum %D as agreed with the client.

10.5.7 **8260C criteria:**

10.5.7.1 The CCV performance must be within ± 20% Difference for average CF and average RF curves and %Drift is used for evaluating linear and quadratic curves

10.5.7.2 If a CCV has failed and the analyst can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then sample analysis may continue. If this second CCV does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e. initial calibration)

10.5.8 **624 criteria**

10.5.8.1 Continuing calibration %D criteria is given in [Table 5](#) of the Method. The column "Range for Q" is used to determine if CCV target analytes meet acceptance.

10.5.8.2 All target analytes must be within the limits prescribed.

10.5.9 **524.2 criteria**

10.5.9.1 For each target analyte %D must be less than or equal to 30%.

10.5.10 Calibration excursions are to be documented via a NCM.

10.6 Retention Time (RT) windows

10.6.1 Relative Retention Time (RRT)

10.6.1.1 In addition to normalizing the response (peak area) of the target compound to the response of the internal standard in that sample or extract for that injection, the retention times of the target compound and the internal standard may be used to calculate the relative retention time (RRT) of the target compound.

10.6.1.2 The RRT is expressed as a unit-less quantity:

$$\text{RRT} = \frac{\text{Retention time of the analyte}}{\text{Retention time of the internal standard}}$$

10.6.1.3 The RRT of each target analyte in each calibration standard should agree within ± 0.06 RRT units.

10.6.1.4 It is recognized here that with increasing retention times of the internal standard, target analytes will be able to more easily meet this criterion. Thus, care should be exercised when selecting the appropriate internal standards by retention times. The process of selecting internal standards to quantify target analytes should also include consideration of retention times as they should be similar.

10.6.1.5 If this criterion is not met and unless there are no other indicators of a component's identification such as a very unique but a high probability mass spectral match then that component may not be considered as identified by relative retention time.

10.6.1.6 The RRT evaluation allows the analyst to compensate for modest shifts in the chromatographic conditions that can occur due to interferences and simple day-to-day instrument variability. Many methods that employ internal standard calibration use more than one internal standard, and the target

compounds are related to the internal standards on the basis of the similarity of their respective chromatographic retention times.

10.6.2 Retention Time Windows

10.6.2.1 The maximum retention time window is ± 0.45 minutes from the established retention time of the target analyte in the initial calibration.

10.6.2.1.1. Establishing this maximum retention time window, ensures that the RRT criteria of $RRT \pm 0.06$ is achieved.

10.6.3 Internal standard retention time

10.6.3.1 The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.6.4 Retention Time Criteria

10.6.4.1 The retention times of all compounds in each continuing calibration must be within the retention time windows established.

10.7 Method Detection Limit Studies

10.7.1 Where required by regulatory agencies, full MDL studies are performed for the relevant analyses on an annual basis. South Carolina requires an annual MDL study. The study must encompass both columns. See SOP ST-QA-0016 for the requirements and procedures to determine and evaluate MDLs

11.0 PROCEDURE

11.1 Screening

11.1.1 Screening samples is a semi-quantitative determination and is not intended to be use as a reportable analytical result.

11.1.2 Screening may be performed on either a GC-FID or GC/MS instrument. The instrument should be capable of detecting the compounds of concern but does not adhere to any method calibration criteria or analysis tune clock times, nor are there any batch QC (eg method blanks, LCS) requirements for screening samples.

11.1.3 Water samples are screened using a minimum 0.1 mL of samples and diluting with target analyte free water to a final volume of 5 mL. Greater dilutions may be taken if the sample is suspected to have significantly high concentration of target analytes or interferents.

11.1.4 For soil samples, 1 g of sample is place in a 40 mL VOA vial with 5 mL of target analyte free water. Greater dilutions may be taken if the sample is suspected to have significantly high concentration of target analytes or interferents.

11.1.5 Sample screening may be performed utilizing a heated or non-heated purging vessel.

11.2 Allow standards, samples and sample extracts to reach ambient temperature before analysis.

11.3 All analysis conditions and injection volumes for samples must be the same for the calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.1 Water, soil and medium level extract analyses are routinely performed by heated purge.

11.3.1.1 If desired, water, TCLP and methanol extracts may be performed using a non-heated purge.

11.3.1.2 If non-heated purge is desired, the calibration and all QC samples must also be performed utilizing a non-heated purge.

11.4 Water Sample Preparation (5030B/C method)

- 11.4.1 Transfer 5 mL or 25 mL sample to a VOA vial.
- 11.4.2 Transfer the 5 mL or 25 mL sample into an empty labeled 40 mL VOA vial.
- 11.4.3 Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution)
 - 11.4.3.1 For routine TCLP samples use 0.5 mL of TCLP sample leachate with 4.5 mL reagent water and spike with surrogate and internal standards.
 - 11.4.3.1.1 Note that TCLP reporting limits will be 10 times higher than the corresponding aqueous limits)
 - 11.4.3.2 For low level TCLP samples, use 5 mL of TCLP sample leachate and spike with surrogate and internal standard.
- 11.4.4 Prepare a method blank with 5 mL or 25 mL of HPLC water. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution)
- 11.4.5 Prepare a LCS with 5 mL or 25 mL of HPLC water. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution). Add 10 µL of 25 µg/mL spiking solution
 - 11.4.5.1 For samples designated for MS/MSD analysis, add 10 µL of 25 µg/mL of spiking solution.
- 11.4.6 Check and document the pH the remaining sample.
 - 11.4.6.1 Do not check pH prior to taking aliquot for analysis.

11.5 Low-Level Soil Preparation (5035A method)

- 11.5.1 If samples arrive unpreserved, the laboratory must, within 48 hours of collection, preserve samples with sodium bisulfate or extrude sample into a clean, empty, sealed VOA vial and freeze.
 - 11.5.1.1 Check the preparation method code to determine preservation.
- 11.5.2 If samples arrive in the Encore (or equivalent) sampler and require Sodium bisulfate preservation:
 - 11.5.2.1 Pre-weigh a labeled 40 mL VOA vial.
 - 11.5.2.1.1 Label with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction.
 - 11.5.2.2 Extrude the soil sample from the Encore (or equivalent) sampler into the VOA vial.
 - 11.5.2.3 Weigh the vial to the nearest 0.01 g.
 - 11.5.2.4 Record weight in appropriate TALS prep-batch.
 - 11.5.2.5 Add a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of HPLC water.
 - 11.5.2.5.1 Soils containing carbonates may effervesce when adding the sodium bisulfate solution. If this is the case, retrieve a second Encore sample plug, add 5 mL of water instead, and freeze at < 10 °C until analysis.
 - 11.5.2.6 Seal the vial.
 - 11.5.2.7 Add 10 µL each of surrogate and internal standard through the septum to each sample and QC.
 - 11.5.2.7.1 For samples designated for MS/MSD analysis, add 10 µL of spiking solution to the vials.
 - 11.5.2.7.2 Prepare a Method Blank using 5.0 g of glass beads and 5 mL of HPLC grade water. Add 10 µL of surrogate to the vial.
 - 11.5.2.7.3 Prepare a LCS using 5.0 g glass beads, and 5 mL HPLC water. Add 10 µL of surrogate and 10 µL of spiking solution.
- 11.5.3 If samples arrive 40 mL VOA vial already preserved with Sodium bisulfate:
 - 11.5.3.1 Add 10 µL each of surrogate and internal standard through the septum to each sample and QC.
 - 11.5.3.1.1 For samples designated for MS/MSD analysis, add 10 µL of spiking solution to the vials.

11.5.3.1.2 Prepare a Method Blank using 5.0 g of glass beads and 5 mL of HPLC grade water. Add 10 µL each of surrogate and internal standard to the vial.

11.5.3.1.3 Prepare a LCS using 5.0 g glass beads, and 5 mL HPLC water. Add 10 µL each of surrogate and internal standard and 10 µL of spiking solution.

11.5.4 If samples arrive in the Encore (or equivalent) sampler and require freezing as preservation:

11.5.4.1 Pre-weigh a labeled 40 mL VOA vial.

11.5.4.1.1 Label with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction.

11.5.4.2 Extrude the soil sample from the Encore (or equivalent) sampler into the VOA vial.

11.5.4.3 Weigh the vial to the nearest 0.01 g

11.5.4.4 Record weight in appropriate TALS prep-batch.

11.5.4.5 Seal the vial.

11.5.4.6 Freeze sample until time of analysis.

11.5.4.7 Add 5 mL of HPLC water.

11.5.4.8 Seal the vial.

11.5.4.9 Add 10 µL each of surrogate and internal standard through the septum to each sample and QC.

11.5.4.9.1 For samples designated for MS/MSD analysis, add 10 µL of spiking solution to the vials.

11.5.4.9.2 Prepare a Method Blank using 5.0 g of glass beads and 5 mL of HPLC grade water. Add 10 µL each of surrogate and internal standard to the vial.

11.5.4.9.3 Prepare a LCS using 5.0 g glass beads, and 5 mL HPLC water. Add 10 µL each of surrogate and internal standard and 10 µL of spiking solution.

11.5.5 If samples arrive in 40 mL VOA vial and require freezing as preservation:

11.5.5.1 Freeze sample until time of analysis.

11.5.5.2 Add 5 mL of HPLC water

11.5.5.3 Seal the vial.

11.5.5.4 Add 10 µL each of surrogate and internal standard through the septum to each sample and QC.

11.5.5.4.1 For samples designated for MS/MSD analysis, add 10 µL of spiking solution to the vials.

11.5.5.4.2 Prepare a Method Blank using 5.0 g of glass beads and 5 mL of HPLC grade water. Add 10 µL each of surrogate and internal standard to the vial.

11.5.5.4.3 Prepare a LCS using 5.0 g glass beads, and 5 mL HPLC water. Add 10 µL each of surrogate and internal standard and 10 µL of spiking solution.

11.6 Low-Level Soil Preparation (superceded 5030 method)

11.6.1 See SOP ST-QA-0038 for the procedure for sub sampling.

11.6.2 Weigh 5 ± 0.05 g of the sample into a pre-weighed 40 mL glass labeled vial.

11.6.2.1 If the sample is suspected or known to have high concentrations of analytes, reduce the sample aliquot to 1.0 g

11.6.3 Record the weight.

11.6.4 Add 5 mL HPLC water

11.6.5 Seal the vial.

11.6.6 The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

- 11.6.7 Add 10 µL of surrogate standard through the septum.
 - 11.6.7.1 Prepare a Method Blank using 5.0 g of glass beads and 5 mL of HPLC grade water. Add 10 µL of surrogate to the vial.
 - 11.6.7.2 Prepare a LCS using 5.0g glass beads, 10 µL of surrogate and 10 µL of spiking solution.
 - 11.6.7.3 For samples designated as for MS/MSD, add 10 µL of spiking solution.
- 11.7 **Methanol Extraction of Soils (5035A method)**
 - 11.7.1 Extrude the (nominal) 5 g sample into a pre-weighed VOA vial containing 5mL methanol (25mL methanol for the 25 g sampler).
 - 11.7.2 Obtain the weight of the soil added to the vial and note in prep log (Attachment 1).
 - 11.7.3 Add 5 µL medium level surrogate to each sample and QC samples.
 - 11.7.3.1 Prepare a Method Blank using 5.0 g of glass beads and 5 mL methanol. Add 5 µL of parent surrogate to the vial.
 - 11.7.3.2 Prepare a LCS using 5.0 g glass beads, 5 mL of methanol , 5 µL of parent surrogate and 10 µL of spiking solution.
 - 11.7.3.3 For the LCS and MS/MSD, add 100 µL medium level spike mix.
 - 11.7.4 Using a vortex mixer, agitate sample for at least half a minute.
- 11.8 **Methanol Extraction of Soils (superseded 5030 method)**
 - 11.8.1 See SOP ST-QA-0038 for the procedure for sub sampling.
 - 11.8.2 Weigh 5 ± 0.05 g of the sample into a pre-weighed 40 mL glass labeled vial.
 - 11.8.2.1 If the sample is suspected or known to have high concentrations of analytes, reduce the sample aliquot to 1.0 g
 - 11.8.3 Record the weight in prep log (Attachment 1).
 - 11.8.4 Seal the vial.
 - 11.8.5 Add 5 mL of purge and trap methanol.
 - 11.8.6 Add 5 µL medium level surrogate to each sample and QC samples.
 - 11.8.6.1 Prepare a Method Blank using 5.0 g of glass beads and 5 mL methanol. Add 5µL of surrogate to the vial.
 - 11.8.6.2 Prepare a LCS using 5.0 g glass beads, 5 mL methanol, 5 µL of surrogate and 20 µL of spiking solution.
 - 11.8.6.3 For the MS/MSD, add 100 µL medium level spike mix.
 - 11.8.7 Using a vortex mixer, agitate sample for at least half a minute.
- 11.9 **Volatile Analysis:**
 - 11.9.1 Load each 40 mL VOA sample vial (and QC) in the purge and trap autosampler.
 - 11.9.1.1 Medium Level Analysis
 - 11.9.1.1.1 Rinse a glass-tight syringe with organic free water and fill the syringe with water. Bring volume to 5 mL add 5 µL of internal standards.
 - 11.9.1.1.2 Add sample methanol extract to the syringe (no more than 100 µL for a 5 mL purge).
 - 11.9.1.1.3 If less than 1 µL of methanol extract is to be added to the water, dilute the methanol extract using a serial dilution.
 - 11.9.1.1.4 Transfer methanol extract/HPLC water in syringe to a labeled 40 mL VOA vial.
 - 11.9.2 Record auto-sampler sample analysis sequence in logbook.
 - 11.9.3 Start analysis.
 - 11.9.4 After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition.
 - 11.9.4.1 For method 524.2 the desorb time should be a minimum of 2 minutes.
 - 11.9.5 After desorption, bake the trap for 5 – 10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

- 11.9.6 When the standards and extracts are not being used, refrigerate them at 4 ± 2 °C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica ST-QAM.
- 12.2 Internal Standards
- 12.2.1 Samples that exhibit low IS recoveries < 50% or high IS recoveries > 100% are reanalyzed once to confirm matrix effect.
- 12.3 Manual Integrations
- 12.3.1 Identified compounds are reviewed for proper integration. Integrations are performed automatically by the data system. If necessary, manual integrations are performed and are documented by the analyst. Manual integrations are denoted with an "M" flag on the Target quantitation report. See TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices
- 12.4 Qualitative identification
- 12.4.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification:
- 12.4.1.1 Elution of sample component at the same GC retention time as the standard component; and
- 12.4.1.2 Correspondence of the sample component and the standard component characteristic ions.
- 12.4.1.2.1 Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.
- 12.4.2 The sample component retention time must compare to within ± 0.2 minutes. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- 12.4.3 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- 12.4.4 For 8260 the relative intensities of ions should agree to within $\pm 30\%$ between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance should be between 20 and 80%.) For Method 624 the relative peak heights of the ions must fall within $\pm 20\%$ of the reference spectra. For Method 524.2 all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample and should agree within absolute 20%.
- 12.4.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.
- 12.5 Retention time criteria for samples
- 12.5.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

- 12.5.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.
- 12.6 Tentatively Identified Compounds (TICs)
- 12.6.1 If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:
- 12.6.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.
- 12.6.1.2 The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30% and 70%).
- 12.6.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.
- 12.6.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- 12.6.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or co-eluting peaks. (Data system reduction programs can sometimes create these discrepancies.)
- 12.6.1.6 Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification. Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client.
- 12.6.1.7 The first 20 TICs will be identified in a sample, unless a different number is specified by the client. See client requirement sheet.
- 12.7 Dilutions
- 12.7.1 If the concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed.
- 12.7.2 A dilution should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the client specific project requirements.
- 12.7.2.1 Aqueous samples requiring less than a 1:1000 dilution can be diluted directly using a 5 mL syringe
- 12.7.2.2 Aqueous samples requiring less than a 1:5000 dilution can be diluted directly using a 25 mL syringe
- 12.7.2.3 Low level soil samples may re-analyzed using a 1 g sample aliquot or utilizing the methanol extraction technique.
- 12.8 Carryover
- 12.8.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
- 12.8.1.1 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.
- 12.8.1.2 If a sample analyzed after a sample with high concentrations has positive results for the same analyses, carryover may have occurred.
- 12.8.1.2.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred
- 12.8.1.3 If the chromatographic profile resembles the previous sample, the results are questionable.

12.8.1.3.1 This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036. Steps taken when an out of control situation occurs include demonstrating that all issues creating the out-of-control situation have been addressed, document the problem and the action taken to correct it, document that an in-control situation has been achieved.
- 13.2 Method Blank
- 13.2.1 Acceptance Criteria:
- 13.2.1.1 No target analytes may be present in the method blank above the reporting limit.
- 13.2.1.2 The method blank must have acceptable surrogate recoveries.
- 13.2.1.3 Corrective Action for Method Blanks not meeting acceptance criteria:
- 13.2.1.3.1 Method Blank Contamination – Blank contamination above the RL (LLOQ) ($>1/2$ RL (LLOQ) for some programs – see specific Client Requirement Memos for details) requires re-prep of batch unless all associated samples are $< RL$ (LLOQ) or greater than 10 times the amount detected in the method blank.
- 13.2.1.3.2 Method Blank Surrogate excursion – If excursion is limited to the blank, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM.
- 13.2.1.3.3 For South Carolina compliance work the Method Blank concentration must be below the RL (LLOQ).
- 13.3 Laboratory Control Sample (LCS)
- 13.3.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD).
- 13.3.1.1 For long analyte spike list, marginal exceedances (ME) are allowed as follows:
- 13.3.1.2 less than 11 analytes in LCS, no analytes allowed in ME of the LCS control limit.
- 13.3.1.3 11-30 analytes in LCS, 1 analytes allowed in ME of the LCS control limit.
- 13.3.1.4 31-50 analytes in LCS, 2 analytes allowed in ME of the LCS control limit.
- 13.3.1.5 51-70 analytes in LCS, 3 analytes allowed in ME of the LCS control limit.
- 13.3.1.6 71-90 analytes in LCS, 4 analytes allowed in ME of the LCS control limit.
- 13.3.1.7 More than 90 analytes in LCS, 5 analytes allowed in ME of the LCS control limit.
- 13.3.1.8 No LCS recoveries may be outside the Marginal Exceedance limit.
- 13.3.1.9 Marginal exceedances must be random. If the same LCS analyte exceeds the control limit repeatedly, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.
- 13.3.1.10 Marginal exceedance is not allowed by all programs. See specific Project/Client CRM for details. The use of marginal exceedances is not allowed for South Carolina compliance samples. LCS recoveries must be within 70 -130% for South Carolina compliance work.
- 13.3.2 The LCS should have acceptable surrogate recoveries.
- 13.3.3 Corrective Action for LCS not meeting acceptance criteria:
- 13.3.3.1 LCS Spike Recovery excursion (high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed. In cases where the surrogate recovery is high and the samples are non-detect, the data may be reported with an NCM

- 13.3.3.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.
- 13.3.3.3 LCS Surrogate Recovery excursion – If excursion is limited to the LCS, data may be reported with an NCM. If target analytes are in control in the LCS, data may be reported with an NCM. If surrogates are also outside criteria in samples, re-prep and re-analysis is required.
- 13.3.3.4 RPD excursion for LCS/LCSD – If target analytes recoveries are in control, data may be reported with an NCM
- 13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
 - 13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).
 - 13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:
 - 13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. If affected analyte concentration in the original sample is greater than four times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to a physically evident matrix interference, the data is reported with an NCM (the physical interference must be described in the NCM). If there is no evidence of interference and the RPD as well as spike recoveries out outside limits out, sample re-prep and re-analysis are required.
- 13.5 Sample result evaluation
 - 13.5.1 Dilutions
 - 13.5.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
 - 13.5.1.2 Dilution: Sample– An NCM is created when dilutions are required.
 - 13.5.1.3 Dilution: Surrogate(s)/spikes diluted out– An NCM is generated to document the surrogates/spikes being diluted out.
 - 13.5.2 Carryover
 - 13.5.2.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
 - 13.5.2.2 If a sample analyzed after a sample with high concentrations is non-detect for the high concentration analyte, carryover did not occur.
 - 13.5.2.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, or if the chromatographic profile resembles the previous sample, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.
 - 13.5.3 Internal Standards
 - 13.5.3.1 Acceptance Criteria:
 - 13.5.3.1.1 If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence.
 - 13.5.3.1.2 If the EICP area for any of the internal standards in samples, spikes and blanks changes by a factor of two (-50% to +100%) from the areas determined in the continuing calibration analyzed that day, corrective action must be taken. The samples, spikes or blanks should be reanalyzed or the data should be qualified. (Some programs may require that the midpoint of the initial calibration be used for ISTD monitoring. See the project CRM for specifics.)
 - 13.5.3.2 Corrective Action for Internal Standards not meeting acceptance criteria:

13.5.3.2.1 Internal Standard excursion – high – High ISTD recovery indicates a potential low bias to analytical results. Instrument maintenance, if required, is done and affected samples are reanalyzed. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.

13.5.3.3 Internal Standard excursion – low – Low ISTD recovery indicates the potential for a high bias to analytical results. Samples that are non-detect for affected analytes may be reported with an NCM. Samples with positive hits above the RL (LLOQ) for analytes associated with the poor ISTD recovery require re-analysis. Instrument maintenance, if required, is done. If ISTDs are outside criteria on the re-analysis, a matrix interference is suspected and data reported with an NCM.

13.6 Surrogate

13.6.1 All Surrogates should be within established control limits for accuracy (%Recovery).

13.6.2 Corrective Action for Surrogate not meeting acceptance criteria:

13.6.2.1 Surrogate Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.7 Insufficient Sample

13.7.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, an NCM is created and a narrative comment stating such is included in the report's Case Narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

- 16.2.1 The following waste streams are produced when this method is carried out.
 - 16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
 - 16.2.1.2 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".
 - 16.2.1.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8000B, 8000C, 8260B and 8260C
- 17.2 40CFR Part 136: "Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A, "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater", Code of Federal Regulations, Revised July1, 1995, Method 624
- 17.3 USEPA Drinking Water method 524.2
- 17.4 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision.
- 17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (ST-HS-0002), current revisions
- 17.6 TestAmerica Policy CA-Q-S-002, Acceptable Manual Integration Practices
- 13.8 TestAmerica Policy CA-T-P-002, Selection of Calibration Points
- 17.7 Associated SOPs
 - 17.7.1 ST-OP-0001, Organic Labware Cleaning Procedure
 - 17.7.2 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.7.3 ST-QA-0002, Standard and Reagent Preparation
 - 17.7.4 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.
 - 17.7.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.7.6 ST-QA-0016, IDL/MDL Determination
 - 17.7.7 ST-QA-0031, VOA Holding Blank Analysis
 - 17.7.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS OF PREVIOUS REFERENCE METHOD

- 18.1 Modification to Reference Method 624
 - 18.1.1 Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.
 - 18.1.2 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.
 - 18.1.3 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

- 18.1.4 Method 624 Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely employs the Supelco K trap.
- 18.2 Modifications required for drinking water analysis by method 524.2
 - 18.2.1 The lab analyzes groundwater by method 524.2 for specific client(s); a narrative is to be included in the applicable data packages that details deviations from the method.
 - 18.2.2 Only one internal standard — Fluorobenzene — is required for this method. The lab uses 3 ISTDs.
 - 18.2.3 The same analysis run may be used to satisfy the requirements for an LCS (also known as a laboratory fortified blank, LFB) and a continuing calibration verification sample. The LCS/CCV does not need to be a second source standard.
 - 18.2.4 524.2 Section 7.1 of the source method requires that the trap packing materials be Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely uses Supelco K trap
 - 18.2.5 524.2 Section 7.8.2 of the source method requires that each calibration standard be prepared by diluting the appropriate volume of the working standard with organic-free water adjusted to pH < 2 in a volumetric flask. TestAmerica prepares calibration standards by diluting the appropriate volume of the working standard with organic-free water at neutral pH.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Section 9.1.5: Blank, LCS & MS/MSD must be run in same clock for non-medium level samples
- 19.2 Section 10.3.2: The number of calibration points required for curves is clarified
- 19.3 Section 10.3.6.6: notes added to address client requirements and y-intercept
- 19.4 Section 10.3.7.9.1: added six point requirement for all quadratic curves
- 19.5 Section 10.4.4.2: Added corrective action for ICV failure (use of third standard)
- 19.6 Section 10.3.7.9.3: added note addressing client specific requirements
- 19.7 Section 12.7.1.1: added CLP allowance for reporting data within 10% of upper standard without dilution
- 19.8 [Table 2](#): added allowance of other published BFB Tune criteria (i.e. EPA CLP)
- 19.9 Added [Attachment 1](#) – example page from VOA Medium Level Extraction log.
- 19.10 Revision 17:
 - 19.10.1 Fixed grammatical errors throughout SOP
 - 19.10.2 Updated section 9.1 regarding MS/MSD duplicates being performed for separate QC codes.
 - 19.10.3 Updated section 10.5.7 regarding CCV criteria testing.
 - 19.10.4 Removed section 10.5.8.3 regarding passing CCV percentage calibration points in the initial calibration curve.
 - 19.10.5 Updated section 11.7 and 11.8 regarding volumes of medium level surrogate and medium level spike mix used.
- 19.11 Rev 18:
 - 19.11.1 Added information for Select Ion Monitoring Procedure to Sections 2, 3 and [Appendix 1](#)
- 19.12 Rev 19:
 - 19.12.1 Section 3, updated SIM definition
 - 19.12.2 Clarified Section 9.3 stating that the LCS and CCV can be from the same source
 - 19.12.3 References to QuantiMS and Clouseau were removed and replaced with “LIMS”
 - 19.12.4 Section 12, updated Corp. SOP reference
 - 19.12.5 Section 12.7.1.1: removed the CLP allowance for reporting data within 10% of upper standard without dilution
 - 19.12.6 Section 13 was re-written to include specific corrective action scenarios.
 - 19.12.7 Section 17, updated Corp. SOP reference
 - 19.12.8 Formatting and grammatical corrections
- 19.13 Rev 20:

- 19.13.1 Section 6.10.5: indicated where instrument run conditions can be found
- 19.13.2 Section 7.4: Updated Standards Log to Reagents in TALS nomenclature
- 19.13.3 Section 10.3: updated analysis volumes used
- 19.13.4 Section 10.3: Added that non-linear regressions can be forced through origin
- 19.13.5 Section 10.3.8: Corrected RSD criteria for EPA 624 method
- 19.13.6 Section 11.1: Updated screening procedures.
- 19.13.7 Section 11.4: Added 25mL method
- 19.13.8 Section 11.5: Corrected various omissions from prep instructions
- 19.13.9 Section 18.2: referenced procedure for notification of client of deviation from EPA method.
- 19.14 Revision 21 (9/18/14):
 - 19.14.1 Section 10.3.7 updated to note that quadratic regression curves are not allowed for South Carolina compliance samples
 - 19.14.2 Section 13.3 updated to note that marginal exceedances are not allowed for South Carolina compliance work
- 19.15 Revision 22 (11/3/14)
 - 19.15.1 SOP combined with ST-MS-0002SC to document all requirements for South Carolina DHEC work.
 - 19.15.2 Added Appendix 2 to contain standard and reagent information.
- 19.16 Revision 23 (12/1/14)
 - 19.16.1 re-worded Section 2.4 to include Scan mode for 1,4-dioxane
 - 19.16.2 added column specifics to Section 6
 - 19.16.3 added Ottawa sand to Sections 7 and 9
 - 19.16.4 Update Section 10 to disallow the use of "Grand Mean" for South Carolina Compliance work
 - 19.16.5 Update %D info in Section 10.5
 - 19.16.6 Updated Section 13 to include the 70 – 130% LCS recovery required for South Carolina compliance work.
 - 19.16.7 Added Appendices with standard prep and concentration information
- 19.17 Revision 24 (05/23/2016)
 - 19.17.1 Add Nitrogen as purge gas to section 6.13
 - 19.17.2 Remove direction to multiply intercept by 10 for non-average regressions in section 10.3.7.6.2.
- 19.18 Revision 25 (9/28/16)
 - 19.18.1 Added Method 624 to Section 8.7 regarding treatment of samples containing (or expected to contain) residual chlorine.
 - 19.18.2 Added note that samples preserved with sodium bisulfate require a sodium bisulfate curve.
 - 19.18.3 Clarified the use of % difference versus % drift in Section 10
- 19.19 Revision 26 (3/6/17)
 - 19.19.1 Added LLOQ definition to Section 3.
 - 19.19.2 Added References to SW846 Method 8000D where appropriate
 - 19.19.3 Added information and calculations for RSE and % Error to Section 10.3
 - 19.19.4 Added LLOQ criteria to Section 13.2

Table 1
Internal Standards

	Standard Concentration µg/mL	Quantitation ion (5 mL purge)	Quantitation ion (25 mL purge)
Fluorobenzene	25	96	96
Chlorobenzene-d5	25	117	117
1,4-Dichlorobenzene-d4	25	152	152

Notes:

- 1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.
- 2) Surrogate and internal standards may be combined in one solution.

Surrogate and Spike concentrations are listed on the Structure and Analysis Codes, which are in the attachment to this SOP.

Table 2
BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

- BFB tuning criteria for mass 75 are 30 – 80% of mass 95 for method 524.2
- Alternatively, other documented tuning criteria (e.g. EPA CLP) may be used provided method performance is not adversely affected

Table 3
Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d ₄ (Surrogate)	67*	102*	65
Dichlorodifluoromethane	85	87	50, 101, 103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101*	103*	66
1,1-Dichloroethene	96	61	63, 98
Acrolein	56	55	58

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Iodomethane	142	127	141
Carbon disulfide	76	78	—
Trichlorotrifluoroethane	151	101	153
Acetone	58	43	—
Methylene chloride	84	86	49
tert-Butyl alcohol	59	74	—
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl tert butyl ether	73	57	—
Hexane	57	43	—
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43*	72*	—
Tetrahydrofuran	71	42	72
Chloroform	83	85	47
1,2-Dichloroethane	62	98	64
Dibromomethane	93	95	172, 174, 176
1,4-Dioxane	88	58	43, 57
Vinyl acetate	43	86	—
1,1,1-Trichloroethane	97	99	117*
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130*	95*	132
Methylcyclohexane	55	83	98
1,2-Dichloropropane	63	41*	—
Bromodichloromethane	83	85	129*
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97*	83*	85
Chlorodibromomethane	129	127*	131*
Bromoform	173	171*	175*, 254
1,2,3-Trichloropropane	110*	77	75
Toluene-d ₈ (Surrogate)	98	100	—
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91*	92*	65
4-Methyl-2-pentanone	43*	58*	85, 100
Tetrachloroethene	164	129	131, 166
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Ethylbenzene	91	106	—
Xylenes	106	91	—
Styrene	104	78	103
Dibromofluoromethane	113	111	192
Dichlorobenzene (all isomers)	146	111	148
trans 1,4-Dichloro-2-butene	53	88	75
1,1,2,2-Tetrachloroethane	83	85	131,133
Allyl Chloride	76	41	39, 78
Acetonitrile	41	40	39
Dichlorofluoromethane	67	69	—
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42, 43
Propionitrile	54	52	55
Methacrylonitrile	41	52	39
Isobutanol	43	42	74
Methyl methacrylate	41	69	39
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	—
Ethyl Acetate	43*	61*	88*
2-Nitropropane	46	43	—
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	77
1,2-Dichlorobenzene	111	146	148
1,3-Dichlorobenzene	111	146	148
1,4-Dichlorobenzene	111	146	148
Nonanol	57	98	41
t-Butyl alcohol	59	41	—
1-Chlorohexane	91	55	43
Ethanol	45	46	—
TAME	73	87	43
ETBE	59	87	41
DIPE (Diisopropyl ether)	45	87	59
2,2-Dimethylpentane	45	46	—
2,4-Dimethylpentane	57	85	43
2,2,3-Trimethylbutane	57	43	85
3,3-Dimethylpentane	43	71	85
2-Methylhexane	43	57	85
2,3-Dimethylpentane	56	71	43
3-Methylhexane	43	57	71

Characteristic ions

Compound	Primary*	Secondary	Tertiary
3-Ethylpentane	43	71	55
Heptane	43	57	71
Dimethylsulfide	94	79	45
1,3,5-Trichlorobenzene	180	182	145

The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

*Primary/secondary and/or tertiary ions are switched from order in Method based on signal intensity and co-elutions.

TABLE 4

**RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL
AND CONTINUING CALIBRATION VERIFICATION**

<u>Volatile Compounds</u>	<u>Minimum Response Factor</u>
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100
Carbon disulfide	0.100
Methyl Acetate	0.100
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

TestAmerica St. Louis has established a default minimum response factor of 0.01 for compounds not identified in this table, except for Acrolein, Acetonitrile, Isobutanol, Tetrahydrofuran, Propionitrile, n-butanol, 1,4-Dioxane, 2-chloroethyl vinyl ether, Cyclohexanone, nonanol (25 mL purge) and Acrolein, Acetonitrile, Tetrahydrofuran,

Propionitrile, Isobutanol, n-butanol, 1,4-Dioxane, Cyclohexanone (5 mL purge), which have a minimum response factor of 0.001.

Table 5
Assigned Surrogates/Internal Standards for Instruments using
Instrument MSX

Compound	Assigned Surrogate	Assigned Internal Standard
Dichlorodifluoromethane	Dibromofluoromethane	Fluorobenzene
Freon-114	Dibromofluoromethane	Fluorobenzene
Chloromethane	Dibromofluoromethane	Fluorobenzene
Vinyl Chloride	Dibromofluoromethane	Fluorobenzene
Bromomethane	Dibromofluoromethane	Fluorobenzene
Chloroethane	Dibromofluoromethane	Fluorobenzene
Trichlorofluoromethane	Dibromofluoromethane	Fluorobenzene
Diethyl Ether	Dibromofluoromethane	Fluorobenzene
1,1,2-Trichlorofluoroethane	Dibromofluoromethane	Fluorobenzene
Acrolein	Dibromofluoromethane	Fluorobenzene
Acetone	Dibromofluoromethane	Fluorobenzene
1,1-Dichloroethene	Dibromofluoromethane	Fluorobenzene
Acetonitrile	Dibromofluoromethane	Fluorobenzene
Iodomethane	Dibromofluoromethane	Fluorobenzene
Methyl Acetate	Dibromofluoromethane	Fluorobenzene
Allyl chloride	Dibromofluoromethane	Fluorobenzene
Carbon Disulfide	Dibromofluoromethane	Fluorobenzene
Methylene Chloride	Dibromofluoromethane	Fluorobenzene
Acrylonitrile	Dibromofluoromethane	Fluorobenzene
MTBE	Dibromofluoromethane	Fluorobenzene
trans-1,2-Dichloroethene	Dibromofluoromethane	Fluorobenzene
n-Hexane	Dibromofluoromethane	Fluorobenzene
1,1-Dichloroethane	Dibromofluoromethane	Fluorobenzene
1,2-Dichloroethene (total)	Dibromofluoromethane	Fluorobenzene
Vinyl acetate	Dibromofluoromethane	Fluorobenzene
2-Chloro-1,3-butadiene	Dibromofluoromethane	Fluorobenzene
2-Butoxyethanol	Dibromofluoromethane	Fluorobenzene
2-Butanone	Dibromofluoromethane	Fluorobenzene
Propionitrile	Dibromofluoromethane	Fluorobenzene
2,2-Dichloropropane	Dibromofluoromethane	Fluorobenzene
cis-1,2-Dichloroethene	Dibromofluoromethane	Fluorobenzene
Isobutanol	Dibromofluoromethane	Fluorobenzene
Ethyl Acetate	Dibromofluoromethane	Fluorobenzene
Methacrylonitrile	Dibromofluoromethane	Fluorobenzene

Compound	Assigned Surrogate	Assigned Internal Standard
Chloroform	Dibromofluoromethane	Fluorobenzene
Bromochloromethane	Dibromofluoromethane	Fluorobenzene
Tetrahydrofuran	Dibromofluoromethane	Fluorobenzene
n-butanol	1,2-Dichloroethane-d4	Fluorobenzene
1,1,1-Trichloroethane	1,2-Dichloroethane-d4	Fluorobenzene
Cyclohexane	1,2-Dichloroethane-d4	Fluorobenzene
1,1-Dichloropropene	1,2-Dichloroethane-d4	Fluorobenzene
Carbon Tetrachloride	1,2-Dichloroethane-d4	Fluorobenzene
Heptane	1,2-Dichloroethane-d4	Fluorobenzene
Benzene	1,2-Dichloroethane-d4	Fluorobenzene
1,2-Dichloroethane	1,2-Dichloroethane-d4	Fluorobenzene
Trichloroethene	1,2-Dichloroethane-d4	Fluorobenzene
Methyl cyclohexane	1,2-Dichloroethane-d4	Fluorobenzene
1,2-Dichloropropane	1,2-Dichloroethane-d4	Fluorobenzene
Methyl methacrylate	1,2-Dichloroethane-d4	Fluorobenzene
Bromodichloromethane	1,2-Dichloroethane-d4	Fluorobenzene
Dibromomethane	1,2-Dichloroethane-d4	Fluorobenzene
1,4-Dioxane	1,2-Dichloroethane-d4	Fluorobenzene
4-Methyl-2-pentanone (MIBK)	1,2-Dichloroethane-d4	Fluorobenzene
2-Chloroethyl vinyl ether	1,2-Dichloroethane-d4	Fluorobenzene
Cis-1,3-Dichloropropene	1,2-Dichloroethane-d4	Fluorobenzene
Dimethyl disulfide	1,2-Dichloroethane-d4	Fluorobenzene
2-Nitropropane	1,2-Dichloroethane-d4	Fluorobenzene
Toluene	Toluene-d8	Chlorobenzene-d5
trans-1,3-Dichloropropene	Toluene-d8	Chlorobenzene-d5
Ethyl methacrylate	Toluene-d8	Chlorobenzene-d5
1,1,2-Trichloroethane	Toluene-d8	Chlorobenzene-d5
2-Hexanone	Toluene-d8	Chlorobenzene-d5
1,3-Dichloropropane	Toluene-d8	Chlorobenzene-d5
Tetrachloroethene	Toluene-d8	Chlorobenzene-d5
Chlorodibromomethane	Toluene-d8	Chlorobenzene-d5
1,2-Dibromoethane	Toluene-d8	Chlorobenzene-d5
Chlorobenzene	Toluene-d8	Chlorobenzene-d5
1,1,1,2-Tetrachloroethane	Toluene-d8	Chlorobenzene-d5
Ethylbenzene	Toluene-d8	Chlorobenzene-d5
m,p-Xylenes	Toluene-d8	Chlorobenzene-d5
o-Xylenes	Toluene-d8	Chlorobenzene-d5
Styrene	Toluene-d8	Chlorobenzene-d5
Bromoform	Toluene-d8	Chlorobenzene-d5
Isopropylbenzene	Toluene-d8	Chlorobenzene-d5

Compound	Assigned Surrogate	Assigned Internal Standard
Cyclohexanone	Toluene-d8	Chlorobenzene-d5
1,1,2,2-Tetrachloroethane	Toluene-d8	Chlorobenzene-d5
1,2,3-Trichloropropane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Bromobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
n-Propylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
trans-1,4-dichlorobenzene-2-butene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
2-Chlorotoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3,5-Trimethylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Chlorotoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
t-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Pentachloroethane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,4-Trimethylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
sec-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Isopropyltoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,4-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
n-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Nonanal	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2-Dibromo-3-chloropropane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3,5-trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,4-Trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Hexachlorobutadiene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Naphthalene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,3-Trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Chlorophenyl methyl sulfide	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4

**Assigned Surrogates/Internal Standards for Instruments using
Instruments MSF, MSL, MSM, MSN, MSZ**

Compound	Assigned Surrogate	Assigned Internal Standard
Dichlorodifluoromethane	Dibromofluoromethane	Fluorobenzene
Freon-114	Dibromofluoromethane	Fluorobenzene
Chloromethane	Dibromofluoromethane	Fluorobenzene
Vinyl Chloride	Dibromofluoromethane	Fluorobenzene
Bromomethane	Dibromofluoromethane	Fluorobenzene

Compound	Assigned Surrogate	Assigned Internal Standard
Chloroethane	Dibromofluoromethane	Fluorobenzene
Trichlorofluoromethane	Dibromofluoromethane	Fluorobenzene
Diethyl Ether	Dibromofluoromethane	Fluorobenzene
1,1-Dichloroethene	Dibromofluoromethane	Fluorobenzene
1,1,2-Trichlorofluoroethane	Dibromofluoromethane	Fluorobenzene
Carbon Disulfide	Dibromofluoromethane	Fluorobenzene
Iodomethane	Dibromofluoromethane	Fluorobenzene
Acrolein	Dibromofluoromethane	Fluorobenzene
Allyl chloride	Dibromofluoromethane	Fluorobenzene
Methylene Chloride	Dibromofluoromethane	Fluorobenzene
Acetone	Dibromofluoromethane	Fluorobenzene
Methyl Acetate	Dibromofluoromethane	Fluorobenzene
trans-1,2-Dichloroethene	Dibromofluoromethane	Fluorobenzene
n-Hexane	Dibromofluoromethane	Fluorobenzene
Acetonitrile	Dibromofluoromethane	Fluorobenzene
MTBE	Dibromofluoromethane	Fluorobenzene
2-Chloro-1,3-butadiene	Dibromofluoromethane	Fluorobenzene
1,1-Dichloroethane	Dibromofluoromethane	Fluorobenzene
1,2-Dichloroethene (total)	Dibromofluoromethane	Fluorobenzene
Acrylonitrile	Dibromofluoromethane	Fluorobenzene
Vinyl acetate	Dibromofluoromethane	Fluorobenzene
cis-1,2-Dichloroethene	Dibromofluoromethane	Fluorobenzene
2,2-Dichloropropane	Dibromofluoromethane	Fluorobenzene
Bromochloromethane	Dibromofluoromethane	Fluorobenzene
2-Butoxyethanol	Dibromofluoromethane	Fluorobenzene
Cyclohexane	Dibromofluoromethane	Fluorobenzene
Chloroform	Dibromofluoromethane	Fluorobenzene
t-Butyl Alcohol	Dibromofluoromethane	Fluorobenzene
Diisopropyl Ether	Dibromofluoromethane	Fluorobenzene
ETBE	Dibromofluoromethane	Fluorobenzene
Ethanol	Dibromofluoromethane	Fluorobenzene
2,2-Dimethylpentane	Dibromofluoromethane	Fluorobenzene
2,4-Dimethylpentane	Dibromofluoromethane	Fluorobenzene
2,2,3-Trimethylbutane	Dibromofluoromethane	Fluorobenzene
3,3-Dimethylpentane	Dibromofluoromethane	Fluorobenzene
Ethyl Acetate	Dibromofluoromethane	Fluorobenzene
Carbon Tetrachloride	Dibromofluoromethane	Fluorobenzene
Tetrahydrofuran	Dibromofluoromethane	Fluorobenzene
1,1,1-Trichloroethane	1,2-Dichloroethane-d4	Fluorobenzene

Compound	Assigned Surrogate	Assigned Internal Standard
2-Butanone	1,2-Dichloroethane-d4	Fluorobenzene
1,1-Dichloropropene	1,2-Dichloroethane-d4	Fluorobenzene
Heptane	1,2-Dichloroethane-d4	Fluorobenzene
Benzene	1,2-Dichloroethane-d4	Fluorobenzene
Propionitrile	1,2-Dichloroethane-d4	Fluorobenzene
Methacrylonitrile	1,2-Dichloroethane-d4	Fluorobenzene
Isobutanol	1,2-Dichloroethane-d4	Fluorobenzene
1,2-Dichloroethane	1,2-Dichloroethane-d4	Fluorobenzene
Trichloroethene	1,2-Dichloroethane-d4	Fluorobenzene
Methyl cyclohexane	1,2-Dichloroethane-d4	Fluorobenzene
n-butanol	1,2-Dichloroethane-d4	Fluorobenzene
Dibromomethane	1,2-Dichloroethane-d4	Fluorobenzene
1,2-Dichloropropane	1,2-Dichloroethane-d4	Fluorobenzene
Bromodichloromethane	1,2-Dichloroethane-d4	Fluorobenzene
Methyl methacrylate	1,2-Dichloroethane-d4	Fluorobenzene
1,4-Dioxane	1,2-Dichloroethane-d4	Fluorobenzene
Cis-1,3-Dichloropropene	1,2-Dichloroethane-d4	Fluorobenzene
2-Chloroethylvinyl ether	1,2-Dichloroethane-d4	Fluorobenzene
TAME	1,2-Dichloroethane-d4	Fluorobenzene
2-Methylhexane	1,2-Dichloroethane-d4	Fluorobenzene
2,3-Dimethylpentane	1,2-Dichloroethane-d4	Fluorobenzene
3-Methylhexane	1,2-Dichloroethane-d4	Fluorobenzene
3-Ethypentane	1,2-Dichloroethane-d4	Fluorobenzene
Heptane	1,2-Dichloroethane-d4	Fluorobenzene
Toluene	Toluene-d8	Chlorobenzene-d5
Dimethyl Disulfide	Toluene-d8	Chlorobenzene-d5
2-Nitropropane	Toluene-d8	Chlorobenzene-d5
4-Methyl-2-pentanone (MEK)	Toluene-d8	Chlorobenzene-d5
trans-1,3-Dichloropropene	Toluene-d8	Chlorobenzene-d5
Tetrachloroethene	Toluene-d8	Chlorobenzene-d5
Ethyl methacrylate	Toluene-d8	Chlorobenzene-d5
1,1,2-Trichloroethane	Toluene-d8	Chlorobenzene-d5
Chlorodibromomethane	Toluene-d8	Chlorobenzene-d5
1,3-Dichloropropane	Toluene-d8	Chlorobenzene-d5
1,2-Dibromoethane	Toluene-d8	Chlorobenzene-d5
2-Hexanone	Toluene-d8	Chlorobenzene-d5
Ethylbenzene	Toluene-d8	Chlorobenzene-d5
Chlorobenzene	Toluene-d8	Chlorobenzene-d5
1,1,1,2-Tetrachloroethane	Toluene-d8	Chlorobenzene-d5

Compound	Assigned Surrogate	Assigned Internal Standard
m,p-Xylenes	Toluene-d8	Chlorobenzene-d5
o-Xylenes	Toluene-d8	Chlorobenzene-d5
Styrene	Toluene-d8	Chlorobenzene-d5
1-Chlorohexane	Toluene-d8	Chlorobenzene-d5
Bromoform	Toluene-d8	1,4-Dichlorobenzene-d4
Isopropylbenzene	Toluene-d8	1,4-Dichlorobenzene-d4
n-Propylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,1,2,2-Tetrachloroethane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Bromobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3,5-Trimethylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
2-Chlorotoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
trans-1,4-dichlorobenzene-2-butene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,3-Trichloropropane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Chlorotoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Cyclohexanone	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
t-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,4-Trimethylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Pentachloroethane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
sec-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Isopropyltoluene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,4-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
n-Butylbenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2-Dichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,3,5-trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2-Dibromo-3-chloropropane	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Hexachlorobutadiene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,4-Trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
Naphthalene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
1,2,3-Trichlorobenzene	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4
4-Chlorophenyl methyl sulfide	4-Bromofluorobenzene	1,4-Dichlorobenzene-d4

Attachment 1
Medium Level Soil Extraction Log

Analyst: _____

Extraction Date: _____

Methanol Lot ID: _____

Batch # _____

Balance ID: _____

The final volume of all extracts is 5 mL of methanol.
All weights are expressed in grams.

Sample ID	Sample Weight	Methanol volume	Sample spiked	Spike verification

QC Standards: LCS/MS/MSD_____

Surrogate_____

Appendix 1

1,4-Dioxane 8260B and 8260C SIM Method

Standards

Internal Standard Working Mix	1,4-Dioxane-d8	50 ppm
Calibration Standard Working Mix	1,4-Dioxane	50 ppm
8260 IS Working Mix	8260 IS	1 ppm

Calibration Table

Level	1,4-Diox. Mix in 5ml	1,4-Diox. Mix in 20ml	d8-1,4-Diox in 20ml	8260 IS Mix in 20ml
5 ppb	0.5 µL	2µL	40 µL	10 µL
10 ppb	1.0 µL	4µL	40 µL	10 µL
20 ppb	2.0 µL	8µL	40 µL	10 µL
50 ppb	5.0 µL	20µL	40 µL	10 µL
100 ppb	10 µL	40 µL	40 µL	10 µL
200 ppb	20 µL	80 µL	40 µL	10 µL

d8-1,4-Dioxane IS concentration in sample 100 ppb

1,4-Dioxane ICV/LCS/MS/MSD concentration 20 ppb

8260 IS concentration in samples 0.5 ppb

Need MDL verification after BFB of each clock 2 ppb (based on current MDL 0.74 ppb)

Spike d8-1,4-Dioxane and 8260 IS in all analyses

GC/MS Method – 8260SIM Concentrator Method – 8260SIM

APPENDIX 2

Standards Prep information:

WM=Working Mix (single mix of all calibrated analytes at concentration levels as indicated in Appendix 3)

Surr=Surrogate mix (single mix of all applicable surrogates at concentration levels as indicated in Appendix 3)

8260 (B & C) - all matrices)

All calibration levels are calculated from 5mL of the appropriate matrix. All calibration levels are spiked with 50ug/L Internal Standard mix.

Level 1: 0.2uL WM and 0.2uL Surr

Level 2: 0.5uL WM and 0.5uL Surr

Level 3: 1uL WM and 1uL Surr

Level 4: 2uL WM and 2uL Surr

Level 5: 4uL WM and 4uL Surr

Level 6: 10uL WM and 10uL Surr

Level 7: 20uL WM and 20uL Surr

Level 8: 40uL WM and 40uL Surr

8260 (B & C) Low Level

All calibration levels are calculated from 25mL of water. All calibration levels are spiked with 10ug/L Internal Standard mix.

Level 1: 0.5uL WM and 0.5uL Surr

Level 2: 1uL WM and 1uL Surr

Level 3: 2uL WM and 2uL Surr

Level 4: 4uL WM and 4uL Surr

Level 5: 10uL WM and 10uL Surr

Level 6: 20uL WM and 20uL Surr

Level 7: 40uL WM and 40uL Surr

APPENDIX 3

**Reagent ID: 8260MegaMixSS_00020**

Type:	ASTD	Expiration Date:	02/29/2016
Description:	8260 List 1/Std #1 MegaMix Second Source	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Working Standards Storage	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A093733
Creation Date:	10/29/2014	Vendor Cat #:	567641.SEC
Container(s):	489974		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,1,1,2-Tetrachloroethane					2000.00000	ug/mL
1,1,1-Trichloroethane					2000.00000	ug/mL
1,1,2,2-Tetrachloroethane					2000.00000	ug/mL
1,1,2-Trichloro-1,2,2-trifluoroethane					2000.00000	ug/mL
1,1,2-Trichloroethane					2000.00000	ug/mL
1,1-Dichloroethane					2000.00000	ug/mL
1,1-Dichloroethene					2000.00000	ug/mL
1,1-Dichloropropene					2000.00000	ug/mL
1,2,3-Trichlorobenzene					2000.00000	ug/mL
1,2,3-Trichloropropane					2000.00000	ug/mL
1,2,4-Trichlorobenzene					2000.00000	ug/mL
1,2,4-Trimethylbenzene					2000.00000	ug/mL
1,2-Dibromo-3-Chloropropane					2000.00000	ug/mL
1,2-Dichlorobenzene					2000.00000	ug/mL
1,2-Dichloroethane					2000.00000	ug/mL
1,2-Dichloropropane					2000.00000	ug/mL
1,3,5-Trimethylbenzene					2000.00000	ug/mL
1,3-Dichlorobenzene					2000.00000	ug/mL
1,3-Dichloropropane					2000.00000	ug/mL
1,4-Dichlorobenzene					2000.00000	ug/mL
1,4-Dioxane					40000.00000	ug/mL
2,2-Dichloropropane					2000.00000	ug/mL
2-Chlorotoluene					2000.00000	ug/mL
2-Methyl-2-propanol					20000.00000	ug/mL
3-Chloro-1-propene					2000.00000	ug/mL
4-Chlorotoluene					2000.00000	ug/mL
4-Isopropyltoluene					2000.00000	ug/mL

**Reagent ID: 8260MegaMixSS_00020**

Type:	ASTD	Expiration Date:	02/29/2016
Description:	8260 List 1/Std #1 MegaMix Second Source	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Working Standards Storage	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A093733
Creation Date:	10/29/2014	Vendor Cat #:	567641.SEC
Container(s):	489974		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Acrylonitrile					2000.00000	ug/mL
Benzene					2000.00000	ug/mL
Bromobenzene					2000.00000	ug/mL
Bromoform					2000.00000	ug/mL
Carbon disulfide					2000.00000	ug/mL
Carbon tetrachloride					2000.00000	ug/mL
Chlorobenzene					2000.00000	ug/mL
Chlorobromomethane					2000.00000	ug/mL
Chlorodibromomethane					2000.00000	ug/mL
Chloroform					2000.00000	ug/mL
cis-1,2-Dichloroethene					2000.00000	ug/mL
cis-1,3-Dichloropropene					2000.00000	ug/mL
Cyclohexane					2000.00000	ug/mL
Dibromomethane					2000.00000	ug/mL
Dichlorobromomethane					2000.00000	ug/mL
Ethyl ether					2000.00000	ug/mL
Ethyl methacrylate					2000.00000	ug/mL
Ethylbenzene					2000.00000	ug/mL
Ethylene Dibromide					2000.00000	ug/mL
Hexachlorobutadiene					2000.00000	ug/mL
Hexane					2000.00000	ug/mL
Iodomethane					2000.00000	ug/mL
Isobutyl alcohol					50000.00000	ug/mL
Isopropylbenzene					2000.00000	ug/mL
Methyl acetate					10000.00000	ug/mL
Methyl tert-butyl ether					2000.00000	ug/mL
Methylcyclohexane					2000.00000	ug/mL

**Reagent ID: 8260MegaMixSS_00020**

Type:	ASTD	Expiration Date:	02/29/2016
Description:	8260 List 1/Std #1 MegaMix Second Source	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Working Standards Storage	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A093733
Creation Date:	10/29/2014	Vendor Cat #:	567641.SEC
Container(s):	489974		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Methylene Chloride					2000.00000	ug/mL
m-Xylene & p-Xylene					2000.00000	ug/mL
Naphthalene					2000.00000	ug/mL
n-Butylbenzene					2000.00000	ug/mL
n-Heptane					2000.00000	ug/mL
N-Propylbenzene					2000.00000	ug/mL
o-Xylene					2000.00000	ug/mL
sec-Butylbenzene					2000.00000	ug/mL
Styrene					2000.00000	ug/mL
tert-Butylbenzene					2000.00000	ug/mL
Tetrachloroethene					2000.00000	ug/mL
Tetrahydrofuran					4000.00000	ug/mL
Toluene					2000.00000	ug/mL
trans-1,2-Dichloroethene					2000.00000	ug/mL
trans-1,3-Dichloropropene					2000.00000	ug/mL
trans-1,4-Dichloro-2-butene					2000.00000	ug/mL
Trichloroethene					2000.00000	ug/mL

**Reagent ID: 8260 IS(2014)_00011**

Type: ASTD
Description: 8260 IS Mix (3-Comp.) 2500ppm
No. of Bottles: 1
Storage Location: VOA Standards Storage
Reagent Volume: 1.000 mL
Creation Date: 09/06/2014
Container(s): 444479
Comment:

Expiration Date: 05/31/2019
Laboratory: TestAmerica St. Louis
Prepared By: Hann, John D
Vendor: Restek
Vendor Lot #: A0103459
Vendor Cat #: 30241

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,4-Dichlorobenzene-d4					2500.00000	ug/mL
Chlorobenzene-d5					2500.00000	ug/mL
Fluorobenzene					2500.00000	ug/mL

**Reagent ID: 8260Gases_00078**

Type:	ASTD	Expiration Date:	09/30/2016
Description:	8260 List 1/Std #3 Gases	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Working Standards Storage	Vendor:	Restek
Reagent Volume:	1.000 mL	Vendor Lot #:	A0105755
Creation Date:	10/30/2014	Vendor Cat #:	567645
Container(s):	489981		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Bromomethane					2000.00000	ug/mL
Butadiene					2000.00000	ug/mL
Chloroethane					2000.00000	ug/mL
Chloromethane					2000.00000	ug/mL
Dichlorodifluoromethane					2000.00000	ug/mL
Dichlorofluoromethane					2000.00000	ug/mL
Trichlorofluoromethane					2000.00000	ug/mL
Vinyl chloride					2000.00000	ug/mL

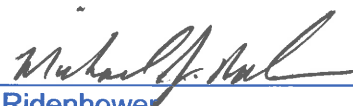
**Reagent ID: 8260_Surr_00018**

Type:	ASTD	Expiration Date:	05/31/2019
Description:	8260 Surrogate Std New Version	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Hann, John D
Storage Location:	VOA Standards Storage	Vendor:	Restek
Reagent Volume:	5.000 mL	Vendor Lot #:	A0103672
Creation Date:	09/06/2014	Vendor Cat #:	567650
Container(s):	444473		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,2-Dichloroethane-d4 (Surr)					2500.00000	ug/mL
4-Bromofluorobenzene (Surr)					2500.00000	ug/mL
Dibromofluoromethane (Surr)					2500.00000	ug/mL
Toluene-d8 (Surr)					2500.00000	ug/mL

**Title: ANALYSIS OF METALS BY INDUCTIVELY COUPLED
PLASMA/MASS SPECTROMETRY
[SW-846 6020; SW-846 6020A; SW-846 6020B; EPA 200.8]**

Approvals (Signature/Date):			
	8/8/16		8/8/16
Kristen Ely	Date	Michael Ridenhower	Date
Inorganics Manager		Health & Safety Manager / Coordinator	
	8/8/16		8/8/16
Marti Ward	Date	Elaine Wild	Date
Quality Assurance Manager		Laboratory Director	

This SOP was previously identified as SOP No. ST-MT-0001 Rev. 24

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1.0 SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA SW846 Method 6020, 6020A, 6020B and EPA 200.8.
- 1.2. This method is applicable to surface, and saline waters; soil and waste samples.
- 1.3. The aqueous sample digestion procedure is found in SOP: ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8) and the soil sample digestion procedure is found in SOP: ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS.
 - 1.3.1. The Technetium-99 soil preparation procedure is found in SOP ST-RC-0125, Determination of Technetium-99 Using Eichrom TEVA Resin,
- 1.4. The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.4.1. Additional elements may be amendable to this method provided the laboratory has established a MDL and the elements meets the QC requirements as prescribed in the associated preparation and analysis SOP.

2.0 SUMMARY OF METHOD

- 2.1. Sample digestates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a radio frequency plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

3.0 DEFINITIONS

- 3.1. See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2. MDL: Method Detection Limit
- 3.3. EPA and SW-846 methodology use different terminology. Our SOP references the SW 846 terminology:
 - 3.3.1. The ICV satisfies the QCS requirements found in method 200.8.
 - 3.3.2. The LCS satisfies the requirements of the LFB found in method 200.8.
 - 3.3.3. The MS satisfies the requirements of the LFM found in method 200.8.
 - 3.3.4. The MB satisfies the requirements of the LRB found in method 200.8.
- 3.4. Other terminology equivalents (used in SOP or LIMS)
 - 3.4.1. ICSA and ICSAB = SIC
 - 3.4.2. RL = LLOQ
 - 3.4.3. LLICV = LLC = CRI
 - 3.4.4. Dilution test = serial dilution
- 3.5. Dissolved Metals: Those elements which pass through a 0.45 µm membrane filter (Sample is filtered BEFORE preservation)
- 3.6. Suspended Metals: Those elements retained by a 0.45 µm filter
- 3.7. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion

4.0 INTERFERENCES

- 4.1. Isobaric elemental interferences: Isobaric elemental interferences associated with naturally occurring isotopes are automatically corrected by the instrument software.
- 4.2. Isobaric molecular interferences: Corrections for molecular interferences will be applied where appropriate based on known or suspected interferences. This may be done with either interference equations or collision cell technology.
- 4.3. Common molecular ion interferences are listed in [Table 3](#) of this SOP.
- 4.4. Matrix interferences: Internal standards are used to correct for some matrix interferences.
 - 4.4.1. Internal standards are added at a level to give approximately 100,000 - 10,000,000 counts of raw signal intensity. The mass of the internal standard used should ideally be within ± 50 amu of the mass of the affected analyte.
 - 4.4.2. Severe matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the initial calibration blank.

5.0 SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
 - 5.2.1. The ICP plasma emits strong UV light, harmful to vision. Analysts must avoid looking directly at the plasma.
- 5.3. PRIMARY MATERIALS USED
 - 5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

- 6.1. PerkinElmer® ELAN 6100/ PerkinElmer® ELAN 9000 /Agilent 7500/ Agilent 7700, each includes the following:
 - 6.1.1. Auto sampler
 - 6.1.2. Chiller (water cooling device)
 - 6.1.3. Peristaltic pump
 - 6.1.4. Vacuum pump
- 6.2. Helium gas: 5.5 trace analytical grade
- 6.3. Argon gas: High-purity grade (99.99%)
- 6.4. Calibrated automatic pipettes
- 6.5. Teflon® flasks
- 6.6. Instrument software: ELAN version 2.3.2 / ELAN version 3.3 / Mass Hunter version B.01.01.

7.0 REAGENTS AND STANDARD

- 7.1. All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2. Concentrated nitric acid (HNO₃), trace metal grade
- 7.3. Concentrated hydrochloric acid (HCl), trace metal grade
- 7.4. DI water from the Millipore unit (ASTM Type I)
 - 7.4.1. Water must be free of the analytes of interest as demonstrated through the analysis of method blanks. Water must be shown to have a resistivity greater than or equal to 18 Mohm-cm.
- 7.5. Standards
 - 7.5.1. Purchased as custom multi-element mixes or as single-element solutions
 - 7.5.2. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles.
 - 7.5.3. Working standards may be used for up to 6 months, with the exception of the ICV
 - 7.5.3.1. The ICV must be prepared daily, and is used to verify that the calibration standards are still viable.
 - 7.5.4. Standards should be prepared in a matrix of 2% hydrochloric and 2% nitric acid.
 - 7.5.5. Internal Standard Solution: Prepare internal standards (Li6, Sc, Ge, In, Ho, Ir) when needed.
 - 7.5.6. Tuning solution: Prepare tuning solution (Be, Ba, Ce, Co, In, Pb, Li, Mg, Rh, Tl, Y) when needed.

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8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2. Aqueous samples for total metals must be digested before analysis using an appropriate digestion procedure, ST-IP-0013.
- 8.3. Soil or waste samples are digested before analysis using an appropriate digestion procedure. Method 3050B of SW846 is the appropriate digestion procedure, ST-IP-0002.
- 8.4. Digestate holding time is 6 months from sample collection.

9.0 QUALITY CONTROL

9.1. Batch

- 9.1.1. A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
- 9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3. For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD an LCS Duplicate (LCSD) is prepared and analyzed.

9.2. Method Blank (MB)

- 9.2.1. A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2. A method blank must be prepared with every sample batch.
- 9.2.3. For water batches, 2% HNO₃ is used.
- 9.2.4. For soil batches, glass beads are used.
- 9.2.5. See Section 13 or LIMS for acceptance criteria.

9.3. Laboratory Control Sample (LCS)

- 9.3.1. An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2. An LCS must be prepared with every sample batch.
- 9.3.3. For water batches, 2% HNO₃ spiked with the analytes of interest is used.
- 9.3.4. For soil batches, a commercially available solid reference material (SRM) is used. Where an SRM is not available, or the manufacturer limits are not sufficient for client required limits, glass beads spiked with an aqueous solution of the analytes of interest is used.
- 9.3.5. See LIMS for acceptance criteria.

9.4. Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1. A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.4.2. See LIMS for acceptance criteria.

9.5. Dilution Test (SD)

- 9.5.1. A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix.
- 9.5.2. The test is performed by running a sample at a 5x (1:4) dilution.
- 9.5.3. Samples identified as field blanks cannot be used for dilution tests.

9.5.4. See Section 13 for acceptance criteria.

9.6. Post Digestion Spike (PDS)

9.6.1. A post digestion spike is a sample which has been fortified with target analytes of interest after the digestion process, with a spike concentration between 10-100 times the MDL (unless specific project/program criteria is given)

9.6.2. **200.8:** A PDS is not applicable for this method

9.6.3. **6020 and 6020B:** A PDS is analyzed with every batch

9.6.4. **6020A:** The method stipulates that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a dilution test on the sample. If the PDS is acceptable, the laboratory is not required to perform a dilution test. Since the laboratory has elected to perform the dilution test routinely, the intermediate step of a post digestion spike is not performed.

9.6.4.1. For client project or programs requiring a PDS, the laboratory will include a PDS in the batch in addition to the dilution test. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.

9.6.5. See Section 13 for acceptance criteria.

9.7. Method of Standard Addition (MSA)

9.7.1. **6020/6020A/6020B**

9.7.2. This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.

9.7.3. MSA are not required by methods 6020, 6020A or 6020B.

9.7.4. MSA are not considered standard batch QC and if required by the client, must appear in the sample comment section in LIMS.

9.7.5. MSA is required by SW846 Method 1311 when the MS/MSD recovery is less than 50%, analyte concentration is less than and within 20% of its regulatory limit.

9.7.6. See Appendix I for more information on MSA

9.8. Procedural Variations/ Nonconformance and Corrective Action

9.8.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

Follow the instrument start-up procedure outlined in the manufacturers operating manual.

10.1. Cone Conditioning

10.1.1. Aspirating a 25% tap water / 75% ICPMS rinse solution for at least 1 hour can enhance instrument performance. This procedure should be used daily after a thorough cleaning of the interface cones or the installation of new cones takes place.

10.1.2. Alternatively, the ICSA solution may be used.

10.2. Rinse Time Determination

10.2.1. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in this SOP it can be demonstrated that a shorter rinse time may be used.

10.2.1.1. To determine the appropriate rinse time, a linear range verification standard should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for the system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

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10.3. Instrument Tuning (Agilent)

10.3.1. Frequency:

10.3.1.1. Daily with each initial calibration

10.3.2. Aspirate a 10 ppb tuning solution containing all of the tuning elements. The typical tuning elements are Li, Y, Tl, Co, In, and Ce.

10.3.3. Tune Criteria:

10.3.3.1. Mass calibration and resolution checks must be documented and included as part of the raw data package.

10.3.3.1.1. Resolution

10.3.3.1.1.1 6020/6020A/6020B: peak width must be < 0.9 amu at 10% peak height

10.3.3.1.1.2 200.8: peak width of approximately 0.75 amu at 5% peak height

10.3.3.1.2. Mass calibration must be within ± 0.1 amu from the actual value for the tuning elements of interest or the mass calibration must be adjusted.

10.3.3.1.3. The tuning elements must have RSD below 5%. Doubly-charged ions and oxides must be below 3.0%.

10.3.3.1.4. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.

10.4. Instrument Tuning (Perkin Elmer)

10.4.1. Frequency:

10.4.1.1. Daily with each initial calibration

10.4.2. Aspirate a 10 ppb tuning solution containing all of the tuning elements. The typical tuning elements are Li, Mg, Rh, Ce, Pb and Ba.

10.4.3. Tune Criteria:

10.4.3.1. Mass calibration and resolution checks must be documented and included as part of the raw data package.

10.4.3.1.1. Resolution

10.4.3.1.1.1 6020/6020A/6020B: peak width must be < 0.9 amu at 10% peak height

10.4.3.1.1.2 200.8: peak width of approximately 0.75 amu at 5% peak height

10.4.3.1.2. Mass calibration must be within ± 0.1 amu from the actual value for the tuning elements of interest or the mass calibration must be adjusted.

10.4.3.1.3. Using the Tuning Solution, an Auto-lens calibration is performed to ensure that optimum voltages are being applied to the Auto-lens. The default calibration should range from 4- 12 volts

10.4.3.1.4. Mg should be greater than 20,000 counts. Pb should be at or above 100,000 counts. Rh should be at or above 150,000. The background should be less than or equal to 30 counts. These are manufacturer recommendations, not requirements.

10.4.3.1.5. The tuning elements must have RSD below 5%. Doubly-charged ions and oxides must be below 3.0%.

10.4.3.1.6. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.

10.5. Initial Calibration

10.5.1. Multi-point Calibration:

10.5.1.1. A calibration curve, consisting of 3 standards and a blank, must be analyzed daily.

10.5.1.2. Calibration criteria:

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- 10.5.1.2.1. **6020/6020A**: Correlation Coefficient of ≥ 0.998
- 10.5.1.2.2. **6020B**: Correlation Coefficient of ≥ 0.995
- 10.5.1.2.3. The low level standard in the curve must be at or below the laboratory's routine reporting limit.
 - 10.5.1.2.3.1 If a client requested reporting limit is below the laboratory's routine reporting limit and thus below the low level verification standard, the laboratory will discuss with the client, prior to sample analysis, how to proceed with this requirement.

10.6. Initial Calibration Verification/Low Level Initial Calibration Verification/Initial Calibration Blank (ICV/LLICV/ICB)

10.6.1. ICV

- 10.6.1.1. Secondary source, used to verify the initial calibration accuracy.
- 10.6.1.2. Frequency: Perform with each initial calibration
- 10.6.1.3. Criteria: $\pm 10\%$
- 10.6.1.4. Action upon failure:
 - 10.6.1.4.1. If the ICV fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
 - 10.6.1.4.2. For all other non-conformances, the samples must be re-analyzed.

10.6.2. LLICV

- 10.6.2.1. Applicable to **6020A** and **6020B** only
- 10.6.2.2. Same source as calibration
- 10.6.2.3. Frequency: Perform with each initial calibration.
- 10.6.2.4. Criteria:
 - 10.6.2.4.1. **6020A**: $\pm 30\%$
 - 10.6.2.4.2. **6020B**: $\pm 20\%$
- 10.6.2.5. Action upon failure:
 - 10.6.2.5.1. If the LLICV fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the LLICV, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.6.2.5.2. If the LLICV fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.6.2.5.3. For all other non-conformances, the samples must be re-analyzed.

10.6.3. ICB

- 10.6.3.1. Frequency: An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness.
- 10.6.3.2. Criteria:
 - 10.6.3.2.1. **200.8/6020/6020A**: $\pm$ the RL from zero.
 - 10.6.3.2.2. **6020B**: $\pm \frac{1}{2}$ the RL from zero.
- 10.6.3.3. Action upon failure:
 - 10.6.3.3.1. If the ICB fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.6.3.3.2. If the ICB fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.6.3.3.3. For all other non-conformances, the samples must be re-analyzed.

- 10.6.4. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected,

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the instrument recalibrated and the calibration re-verified.

- 10.6.5. Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.7. Continuing Calibration Verification/Low Level Continuing Calibration Verification/Continuing Calibration Blank (CCV/LLCCV/CCB)

- 10.7.1. Calibration is monitored throughout the analytical run through the analysis of a known mid-level calibration standard.

10.7.2. CCV

- 10.7.2.1. A CCV may be a second source or the same source as the calibration.
10.7.2.2. Frequency: Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run.
10.7.2.3. Criteria: 10% of true value
10.7.2.4. Action upon failure:
10.7.2.4.1. If the CCV fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
10.7.2.4.2. For all other non-conformances, the samples must be re-analyzed.

10.7.3. LLCCV

- 10.7.3.1. Applicable to **6020A** only
10.7.3.2. Same source as calibration
10.7.3.3. Frequency: Perform at a minimum at the end of the run
10.7.3.3.1. Typically analyzed every 10 samples and at the end of the run.
10.7.3.4. Criteria: $\pm 30\%$
10.7.3.5. Action upon failure:
10.7.3.5.1. If the LLCCV fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the LLICV, the potential bias has not affected the samples. Samples may be reported with an NCM.
10.7.3.5.2. If the LLCCV fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
10.7.3.5.3. For all other non-conformances, the samples must be re-analyzed.

10.7.4. CCB

- 10.7.4.1. Frequency: A CCB is analyzed immediately following each CCV.
10.7.4.2. Criteria: The CCB result must fall within $\pm$ RL from zero.
10.7.4.3. Action upon failure:
10.7.4.3.1. If the CCB fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.
10.7.4.3.2. If the CCB fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
10.7.4.3.3. For all other non-conformances, the samples must be re-analyzed.

- 10.7.5. If a CCV and/or CCB has failed and the analyst can document the reason for failure (e.g mis-injection, etc.) then a second CCV and/or CCB may be analyzed without any adjustments to the instrument. If this CCV and/or CCB meet criteria then sample analysis may continue; however the preceding 10 samples must be reanalyzed. If this second CCV and/or CCB does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-

calibration (i.e., initial calibration).

10.8. Interference Check Standard (ICSA/ICSAB)

10.8.1. Applicable to **6020,6020A** and **6020B** only

10.8.2. The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions.

10.8.3. ICSA:

10.8.3.1. The ICSA contains only interfering elements. Refer to LIMS for the details of ICSA composition.

10.8.3.2. Custom multi-element ICS solutions must be used.

10.8.3.3. Elements known to be interferences on a required analyte must be included in the ICPMS run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICPMS runs.

10.8.3.4. Frequency: The ICSA must run with each initial calibration or every 12 hours whichever is shorter.

10.8.3.5. Criteria for non-interfering analytes: $\pm 2x$ RL from zero.

10.8.3.6. Action upon failure:

10.8.3.6.1. For interfering elements:

10.8.3.6.1.1 If the ICSA fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM

10.8.3.6.2. For non-interfering elements:

10.8.3.6.2.1 If the ICSA fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.8.3.6.2.2 If the ICSA fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.8.3.6.3. For all other non-conformances, the samples must be re-analyzed.

10.8.4. ICSAB:

10.8.4.1. The ICSAB contains analytes and interferences.

10.8.4.2. Refer to LIMS for the details of ICSAB composition.

10.8.4.3. Custom multi-element ICS solutions must be used.

10.8.4.4. Frequency: The ICSAB must run with each initial calibration or every 12 hours whichever is shorter.

10.8.4.5. Criteria: The ICSAB results must fall within 80% – 120% of the true value.

10.8.4.6. Action upon failure:

10.8.4.6.1. If the ICSAB fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM

10.8.4.6.2. For all other non-conformances, the samples must be re-analyzed.

10.9. Liner Dynamic Range (LDR)

10.9.1. Prior to running the instrument, the upper limit of quantitation must be established for each analyte.

10.9.2. This upper limit is tested by running a standard containing high concentrations of the analytes against a calibration curve.

10.9.3. The concentration of the LDR standard is higher than the high calibration standard.

10.9.4. Frequency:

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- 10.9.4.1. **200.8**: as needed
- 10.9.4.2. **6020/6020A**: every 6 months
- 10.9.4.3. **6020B**: daily
- 10.9.4.4. When requested by client, the LDR is run daily
- 10.9.5. Criteria: $\pm 10\%$
- 10.9.5.1. If the LDR fails the criteria, the highest calibration standard is used as the upper limit for the linear range.

10.10. Calibration Sequence

Tuning Standard
Initial Calibration (3 standards plus a blank)
ICV
ICB
LLICV (for 6020A only)
ICSA*
ICSAB*
CCV
CCB
LDR (when necessary)
CCV
CCB
10 samples (analysis runs)
CCV
CCB
10 samples (repeat every 10 analysis runs)
LLCCV (Minimum at end of run. May be analyzed more frequently)
CCV
CCB
End

* If sequence time is longer than 12 hours, the ICSA and ICSAB standard must be re-analyzed.

11.0 PROCEDURE

- 11.1 The aqueous sample digestion procedure is found in SOP: ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8)
- 11.2 The soil sample digestion procedure is found in SOP: ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS.
- 11.3 Instrument conditions, including rinse times, must be the same for all standards and samples.
- 11.4 Internal standards are introduced to the standards and sample digestates by the instrument.
- 11.5 Load autosampler with standards and digestates in accordance with the sequence given in section 10
- 11.6 Analyze samples.
- 11.7 When analysis is completed, return unused digestate to proper storage area.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1. Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2. All measurements must fall within the defined linear range where spectral interference correction factors are valid.

12.2.1. Dilute and reanalyze all samples for required analytes that exceed the linear range

12.2.1.1. For 200.8, any sample greater than 90% of the linear range must be diluted and re-analyzed.

12.2.2. Acid strength must be maintained in the dilution of samples.

12.3. The mass ions used for determination of the element of interest is given in [Table 1](#) of this SOP

12.4. Tracer Calculations

12.4.1. Tracer Recovery: The LIMS calculates the tracer recovery using the following formula:

$$\frac{\text{Measured Tracer Concentration}}{\text{Actual Tracer Concentration}} = \text{Final Recovery}$$

12.4.1.1. Tracer Criteria: The tracer recovery must fall within 30-110%.

12.4.2. Final sample concentration (corrected for tracer recovery) is determined by the LIMS using the following formula:

$$\frac{\text{Measured Sample Concentration}}{\text{Tracer Recovery}} = \text{Final Sample Concentration}$$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1. The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process in LIMS. The NCM process is described in SOP: ST-QA-0036.

13.2. Method Blank (MB)

13.2.1. Acceptance Criteria:

13.2.1.1. **200.8/6020/6020A**: <RL

13.2.1.2. **6020B**: < ½ RL

13.2.2. Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted in the client notes.

13.2.3. Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.3.1. Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.

13.3. Laboratory Control Sample (LCS)

13.3.1. Acceptance Criteria: All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.

13.3.2. Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1. LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2. LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

13.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

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- 13.4.1. Analytes should be within control limits for accuracy (%Recovery) and precision (RPD). Control limits can be found in LIMS.
- 13.4.2. Corrective Action for MS/MSD not meeting acceptance criteria:
- 13.4.2.1. MS/MSD Spike Recovery excursion: may not necessarily warrant corrective action other than narration
- 13.4.2.1.1. If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.
- 13.4.2.1.2. If the excursion is due to physically evident matrix interference, the data is reported with an NCM.
- 13.4.2.1.3. In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with an NCM.
- 13.4.2.1.4. When the MS/MSD recoveries and the %RPD are outside criteria, if the samples are non-homogenous, the data may be reported with an NCM. Otherwise, the batch is re-prepped and re-analyzed for the affected analytes.
- 13.5. Dilution Test (SD)
- 13.5.1. A dilution test is not required for method 200.8
- 13.5.2. For methods 6020,6020A and 6020B, a dilution test is analyzed with every prep batch
- 13.5.3. Criteria:
- 13.5.3.1. **6020/6020A**: when analyte concentration $\geq 10\times$ RL, $\pm 10\%$
- 13.5.3.2. **6020B**: when analyte concentration $\geq 25\times$ RL, $\pm 20\%$
- 13.5.4. Corrective Action: Dilution Test failure is documented in an NCM and the reported data is flagged. If multiple analytes fail the Dilution Test, the analyst may re-prepare and re-analyze the samples.
- 13.6. Post Digestion Spike (PDS)
- 13.6.1. A PDS is not required for 200.8.
- 13.6.2. **6020**
- 13.6.2.1. Criteria: The acceptance criteria is 75%-125%, UNLESS, other project/program criteria is given.
- 13.6.2.2. Corrective Action: Sample must be diluted and re-analyzed to compensate for matrix effect, until the PDS is within acceptable limits.
- 13.6.3. **6020A**
- 13.6.3.1. Criteria: The acceptance criteria is 80%-120%, with a spike concentration between 10-100 times the MDL, UNLESS, other project/program criteria is given.
- 13.6.3.2. Corrective Action: There is no qualification made to the data based on the performance of the PDS, however a failed PDS is documented with an NCM and noted in the report narrative.
- 13.6.3.3. The PDS is not reported in the data package unless a client project or program requires it. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.
- 13.6.4. **6020B**
- 13.6.4.1. Criteria: The acceptance criteria is 75%-125%
- 13.6.4.2. Corrective Action: There is no qualification made to the data based on the performance of the PDS, however a failed PDS is documented with an NCM and noted in the report narrative.
- 13.7. Sample result evaluation
- 13.7.1. Dilutions
- 13.7.1.1. If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
- 13.7.2. For samples requiring dilution an NCM is created to document the reason for the dilution.
- 13.7.3. Insufficient Sample

13.7.3.1. For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, a narrative comment stating such is included in the report narrative.

13.8. Internal Recovery Standard (IS)

13.8.1. Criteria (for all samples and QC standards)

13.8.1.1. **6020:**

13.8.1.1.1. QC: 80-120%

13.8.1.1.2. Samples: 30-120%

13.8.1.2. **6020A:** 70%-140%

13.8.1.3. **6020B:** 30%-150%

13.8.1.4. **200.8:** 60%-125

13.8.2. Action Upon Failure

13.8.2.1. Samples: If the criteria is not met, the sample should be diluted and re-analyzed until the IS recoveries are within specified limits.

13.8.2.2. QC standards: If the criteria are not met, the analyst will review the data. If the sample internal standard recoveries are within control and the QC standard is within acceptable limits, it is apparent that whatever interference affected the internal standard for the QC standards has not affected the element bracketed by that internal standard based upon the criteria being met. If these specific occurrences are met then an NCM will be generated stating why the data is acceptable. Otherwise, samples linked to the QC standard will be re-analyzed

14.0 METHOD PERFORMANCE

14.1. Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS.

14.2. Demonstration of Capability

14.2.1. Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3. Training Qualification

14.3.1. The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1. Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2. Waste Streams Produced by the Method

16.2.1. The following waste streams are produced when this method is carried out.

16.2.1.1. Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.1.2. Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid Rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 6020, 6020A and 6020B

17.2. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma/Mass Spectrometry Method 200.8

17.3. PerkinElmer® ELAN 6100 Inductively Coupled Plasma Mass Spectrometer Hardware Guide

17.4. PerkinElmer® ELAN 6100 Software Kit

17.5. PerkinElmer® ELAN 9000 Hardware Guide

17.6. PerkinElmer® ELAN Version 3.0 Software Guide

17.7. Agilent 7500 Series MassHunter Workstation (G7200A) Operators Manual

17.8. Agilent 7500 Series ICP-MS Hardware Manual

17.9. Agilent 7700 Series ICP-MS Hardware Maintenance Manual

17.10. TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.11. TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.12. Associated SOPs, current revisions:

17.12.1. ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS

17.12.2. ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8)

17.12.3. ST-QA-0002, Standard and Reagent Preparation

17.12.4. ST-PM-0002, Sample Receipt and Chain of Custody

17.12.5. ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts

17.12.6. ST-QA-0016, IDL/MDL Determination

17.12.7. ST-QA-0036, Non-conformance Memorandum (NCM) Process

17.12.8. ST-RC-0125, Determination of Technetium-99 Using Eichrom Resin

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1. Method 6020A stipulates that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a serial dilution on the sample. If the PDS is acceptable, the laboratory is not required to perform a serial dilution. Since the laboratory has elected to perform the serial dilution routinely, the intermediate step of a post digestion spike is not performed. Internal standards are used to monitor matrix interferences in all samples. Post spikes are done per specific QAPP or program requirements. Post-spikes using analytes other than the internal standards may be used if an analyst encounters a new or unusual matrix.

- 18.2. Method 200.8 has more stringent blank acceptance criteria than stated in this SOP. Due to software limitations the lab is unable to evaluate and report blanks using the method criteria that “analyte concentration in the blank greater than 2.2 times the MDL, or greater than 10% of the concentration of a sample requires rep-reparation and reanalysis.” Blanks are evaluated to the MDL and flagged if they exceed that level. Re-preparation and re-analysis are required if the method blank is greater than RL and the sample is greater than the RL but less than ten times the RL.
- 18.3. Method 6020A requires the analysis of a Lower Limit Quantitation Check Sample (LLQC) on an as needed basis, to establish and confirm the lowest quantitation limit. TestAmerica St. Louis fills this requirement with the quarterly running of a MDL verification standard which is taken through the entire sample preparation procedure.
- 18.4. Method 200.8 requires that data less than 10ug/L be reported to 2 significant figures, and data greater than or equal to 10ug/L be reported to 3 significant figures. TestAmerica St. Louis uses client requested significant figures for reporting data.

19.0 CHANGES TO PREVIOUS REVISION

- 19.1. Updated formatting and spelling errors throughout SOP
- 19.2. Updated section 4.4 referring to the amount of an internal standard being used.
- 19.3. Added new instrument and gases used in section 6.0.
- 19.4. Added Lithium to section 7.0 as part of the new reagents and standards used.
- 19.5. Made reference to new instruments for calibration in section 10.0.
- 19.6. Add new list of tuning element for both instruments in section 10.5.
- 19.7. Updated the internal standard intensity throughout section 10.7 and section 10.8
- 19.8. Added new elements to [table 2](#).
- 19.9. Rev. 18;
- 19.9.1. Added LLICV to definitions in section 3.2.
- 19.9.2. Removed Hydrogen Peroxide from Safety Section (included in prep SOP)
- 19.9.3. Added tuning solution to section 7.5.5.
- 19.9.4. Updated cone conditioning solution, make up and frequency of use.
- 19.9.5. Added clarification to tuning section 10.5.
- 19.9.6. Added Low Level initial calibration verification standards plus criteria to section 10.0.
- 19.9.7. Updated tables 1 and 2, added analytes, updated concentrations.
- 19.9.8. Added method 1311 MSA requirements information to section 9.7.
- 19.9.9. Spelling and grammatical corrections.
- 19.10. Rev. 19:
- 19.10.1. Updated [Table III](#) regarding QC Criteria limits.
- 19.11. Revision 20:
- 19.11.1. Updated section 1.3 adding reference to the Technetium-99 soil procedure.
- 19.11.2. Added formulas for determining the Tracer Recovery and the Final tracer Corrected Concentration to section 12.5.
- 19.11.3. Added instrument software and hardware to section 6.0.
- 19.11.4. Updated the PDS acceptance criteria in section 9.6.
- 19.12. Rev. 21:
- 19.12.1. Removed legacy text regarding MSA from Section 18 as MSA is not required by Method 6020A.
- 19.13. Rev. 22: (8/27/2013)
- 19.13.1. Updated section 1.3 to reflect the corrected Tc-99 SOP (ST-RC-0125)
- 19.13.2. Updated section 6. Hydrogen was removed
- 19.13.3. Updated section 9.7, replace QuantIMS wording with TALS wording
- 19.13.4. Updated section 10, used a more consistent format
- 19.13.5. Updated section 12.4. Removed references to a spreadsheet, added TALS
- 19.13.6. Updated section 13
- 19.14. Rev.23: (1/16/2015)

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- 19.14.1. Made formatting and grammatical corrections
- 19.14.2. Corrected definitions in section 3 (removed IPC and CRI, added LRB)
- 19.14.3. Removed references to ASTM Method D5673-03
- 19.14.4. Added equipment and software for ICPMS 9000 to section 6
- 19.14.5. Removed the ICSA/ICSAB Table (was Table 2) and replaced references to it with instructions to look at LIMS (10.8.3.1 and 10.8.4.2)
- 19.14.6. Updated tuning criteria for Perkin Elmer ICPMS in sections 10.5.3.1.4 & 10.5.3.1.5
- 19.14.7. Updated section 10.8.3.5, replace $RL > 10\mu\text{g/L}$ with $RL \geq 10\mu\text{g/L}$
- 19.14.8. Clarified criteria in section 10, 13 and 18
- 19.14.9. Replaced "client requirement sheet" with "client notes" in section 13.2.2
- 19.14.10. Removed reference to Marginal Exceedance in section 13.3
- 19.14.11. Corrected IS criteria in section 13.8.1
- 19.14.12. Updated instrument manuals in section 17
- 19.14.13. Added affected methods to section 18
- 19.14.14. Removed section 18.3, was same as section 18.4
- 19.14.15. Added reference to method 6020
- 19.14.16. Deleted references in section 9 to batch QC criteria, referenced section 13. Instrument QC criteria is in section 10.
- 19.14.17. updated formatting, to make the wording more consistent throughout the SOP and easier to read.
- 19.14.18. section 10: separated out the tuning for Perkin Elmer vs Agilent, for clarity.
- 19.14.19. Added reference to tc-99 prep via ST-RC-0125
- 19.15. Rev. 24: (06/22/2015)
 - 19.15.1. Added Appendix 1 MSA instruction
- 19.16. Rev. 25: (08/11/2016)
 - 19.16.1. Added Appendix II – Retrieve Lost Data from Agilent 7500 or 7700
 - 19.16.2. Added method 6020B and all its requirements
 - 19.16.3. Updated Table 2
 - 19.16.4. Updated definitions
 - 19.16.5. Updated frequency needed to make standards
 - 19.16.6. Made clarifications throughout
 - 19.16.7. Changed wording from "serial dilution" to "dilution test"
 - 19.16.8. Updated criteria for ICSA
 - 19.16.9. added method modification to section 18: MB for 200.8 and sig figs for 200.8
 - 19.16.10. updated matrix for water MB and LCS

Table 1
ANALYTICAL ISOTOPES

ELEMENT	7700 Tune Step	7500 Mass	7700 Tune Step	7700 Mass	6100 Mass	9000 Mass
Li	3	7	3	7	N/A	N/A
Be	3	9	3	9	N/A	N/A
B	3	11	3	11	N/A	N/A
Na	2	23	2	23	N/A	N/A
Mg	2	24	2	24	N/A	N/A
Al	2	27	2	27	N/A	N/A
Si	3	28	3	28	N/A	N/A
P	2	31	2	31	N/A	N/A
S	2	34	2	34	N/A	N/A
K	2	39	2	39	N/A	N/A
Ca	3	44	3	44	N/A	N/A
Ti	3	47	3	47	N/A	N/A
V	2	51	2	51	N/A	N/A
Cr	2	52	2	52	N/A	N/A
Mn	2	55	2	55	N/A	N/A
Fe	2	57	2	57	N/A	N/A
Co	2	59	2	59	N/A	N/A
Ni	2	60	2	60	N/A	N/A
Cu	2	63	2	63	N/A	N/A
Zn	2	66	2	66	N/A	N/A
As	2	75	2	75	N/A	N/A
Se	2	78	2	78	N/A	N/A
Sr	3	88	3	88	N/A	N/A
Y	2	89	2	89	N/A	N/A
Zr	2	90	2	90	N/A	N/A
Nb	2	93	2	93	N/A	N/A
Mo	3	95	3	95	N/A	N/A
Ru	2	101	2	101	N/A	N/A
Rh	2	103	2	103	N/A	N/A
Pd	2	105	2	105	N/A	N/A
Ag	3	107	3	107	N/A	N/A
Cd	3	111	3	111	N/A	N/A
Sn	3	118	3	118	N/A	N/A
Sb	3	121	3	121	N/A	N/A
Te	2	125	2	125	N/A	N/A
Cs	2	133	2	133	N/A	N/A
Ba	3	137	3	137	N/A	N/A
La	2	139	2	139	N/A	N/A
Ce	2	140	2	140	N/A	N/A
Pr	2	141	2	141	N/A	N/A
Nd	2	146	2	146	N/A	N/A
Sm	3	147	3	147	N/A	N/A
Hf	2	178	2	178	N/A	N/A
Ta	2	181	2	181	N/A	N/A
W	2	182	2	182	N/A	N/A
Re	3	185	3	185	N/A	N/A
Pt	2	195	2	195	N/A	N/A
Au	3	197	3	197	N/A	N/A
Tl	3	205	3	205	N/A	N/A
Pb	3	208	3	208	N/A	N/A
Bi	2	209	2	209	N/A	N/A
Th	3	232	3	232	N/A	N/A
Tc	3	99	N/A	N/A	99	99
U	N/A	N/A	3	236	236	236
U	N/A	N/A	3	235	235	235
U	N/A	N/A	3	234	234	234
U	N/A	N/A	3	233	233	233
U	3	238	3	238	238	238

Tune Step 2: Helium

Tune Step 3: No Gas (argon only)

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Table 2
QC Criteria

Methods	6020	6020A	6020B	200.8
Corr Coeff.	>0.998	>0.998	>0.995	N/A
Tuning Res	<0.9amu 10% peak height	<0.9amu 10% peak height	<0.9amu 10% peak height	≈ 0.75amu 5% peak height
Int Std	QC: 80-120% Samples: 30-120%	70-140%	30-150%	60-125%
ICV	90-110%	90-110%	90-110%	90-110%
ICB	±RL	±RL	± ½ RL	±RL
LLICV	N/A	70-130%	80-120%	N/A
LLCCV	N/A	70-130%	N/A	N/A
CCV	90-110%	90-110%	90-110%	90-110%
CCB	±RL	±RL	±RL	±RL
MB	±RL	±RL	± ½ RL	±RL
LCS	80-120%	80-120%	80-120%	85-115%
MS	75-125%	75-125%	75-125%	70-130%
PDS	75-125%	80-120%	75-125%	N/A
SD	± 10%	± 10%	± 20%	N/A

Table 3

COMMON MOLECULAR ION INTERFERENCES IN ICP-MS
BACKGROUND MOLECULAR IONS

Molecular Ion	Mass	Element Interferences*
NH ⁺	15	
OH ⁺	17	
OH ₂ ⁺	18	
C ₂ ⁺	24	
CN ⁺	26	
CO ⁺	28	
N ₂ ⁺	28	
N ₂ H ⁺	29	
NO ⁺	30	
NOH ⁺	31	
O ₂ ⁺	32	
O ₂ H ₊	33	
³⁶ ArH ⁺	37	
³⁸ ArH ⁺	39	
⁴⁰ ArH ⁺	41	
CO ₂ ⁺	44	
CO ₂ H ⁺	45	Sc
ArC ⁺ , ArO ⁺	52	Cr
ArN ⁺	54	Cr
ArNH ⁺	55	Mn
ArO ⁺	56	
ArOH ⁺	57	
⁴⁰ Ar ³⁶ Ar ⁺	76	Se
⁴⁰ Ar ³⁸ Ar ⁺	78	Se
⁴⁰ Ar ₂ ⁺	80	Se

* Method elements or internal standards affected by the molecular ions.

Table 4

MATRIX MOLECULAR IONS* No gas Mode Only

CHLORIDE

Molecular Ion	Mass	Element Interference
$^{35}\text{ClO}^+$	51	V
$^{35}\text{ClOH}^+$	52	Cr
$^{37}\text{ClO}^+$	53	Cr
$^{37}\text{ClOH}^+$	54	Cr
$\text{Ar}^{35}\text{Cl}^+$	75	As
$\text{Ar}^{37}\text{Cl}^+$	77	Se

SULFATE

Molecular Ion	Mass	Element Interference
$^{32}\text{SO}^+$	48	
$^{32}\text{SOH}^+$	49	
$^{34}\text{SO}^+$	50	V, Cr
$^{34}\text{SOH}^+$	51	V
$\text{SO}_2^+, \text{S}_2^+$	64	Zn
Ar^{32}S^+	72	
Ar^{34}S^+	74	

PHOSPHATE

Molecular Ion	Mass	Element Interference
PO^+	47	
POH^+	48	
PO_2^+	63	Cu
ArP^+	71	

GROUP I, II METALS

Molecular Ion	Mass	Element Interference
ArNa^+	63	Cu
ArK^+	79	
ArCa^+	80	

MATRIX OXIDES*

Molecular Ion	Mass	Element Interference
TiO	62-66	Ni, Cu, Zn
ZrO	106-112	Ag, Cd
MoO	108-116	Cd

* Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

APPENDIX 1 – METHOD OF STANDARD ADDITION (MSA)

MSA is required if

- 1- Matrix spike recovery <50%, AND
- 2- Measured concentration is within 20% of the regulatory level

Regulatory Limits:

Arsenic	5mg/L
Barium	100mg/L
Cadmium	1 mg/L
Chromium	5 mg/L
Lead	5 mg/L
Mercury	0.2 mg/L
Selenium	1 mg/L
Silver	5 mg/L

How to run an MSA

- 1- Take 4 identical aliquots of test solution
- 2- Add increasing concentration of standard to 3 aliquots and add blank solution to 4th aliquot- all aliquots should be at same final volume
- 3- Perform analysis
- 4- Enter data into spreadsheet ORG-0034_TCLP_MSA
 - a. Uses unweighted least-squares linear regression curve fit
 - b. Calculates absolute value of x-intercept

Notes:

- 1- MSA spikes must come from the same analytical batch, analyzed on the same day.

The MSA curve must include the unspiked sample and 3 samples spiked with increasing concentrations of analytes

APPENDIX II- Retrieve Lost Data from Agilent 7500 or 7700

MASS HUNTER

File-> New Batch Folder (create, give new name)

File-> Import All Samples from Batch

DA Method Editor

Import DA Method and Standard Data (METALSTA – Met_2014)

Return to Batch-at-a glance

Update? YES

Process Batch

**Title: INDUCTIVELY COUPLED PLASMA-OPTICAL EMISSION
SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT
ANALYSES
[SW-846 6010B; 6010C; 6010D; EPA 200.7]**

Approvals (Signature/Date):	
 Kristen Ely Inorganics Manager	8/10/16 Date
 Michael Ridenhower Health & Safety Manager / Coordinator	8/12/16 Date
 Marti Ward Quality Assurance Manager	8/12/16 Date
 Elaine Wild Laboratory Director	8/19/16 Date

This SOP was previously identified as SOP No. ST-MT-0003 Rev. 17

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1.0 SCOPE AND APPLICATION

- 1.1 This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Optical Emission Spectroscopy (ICP-OES) using SW-846 Method 6010B, 6010C, 6010D and EPA Method 200.7.
- 1.2 This method is applicable to surface, and saline waters; soil and waste samples.
- 1.3 The aqueous sample digestion procedure is found in SOP: ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8) and the soil sample digestion procedure is found in SOP: ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS.
- 1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).
 - 1.4.1 Additional elements may be amendable to this method provided the laboratory has established a MDL and the elements meets the QC requirements as prescribed in the associated preparation and analytical SOP.

2.0 SUMMARY OF METHOD

- 2.1 This method describes a technique for the determination of multi elements in solution using simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photosensitive devices. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 MDL: Method Detection Limit
- 3.3 EPA and SW methodology use different terminology. Our SOP references the SW 846 terminology:
 - 3.3.1 The ICV satisfies the QCS requirements found in method 200.7.
 - 3.3.2 The LCS satisfies the requirements of the LFB found in method 200.7.
 - 3.3.3 The MS satisfies the requirements of the LFM found in method 200.7.
 - 3.3.4 The ICS (A and/or AB) satisfies the requirements of the SIC found in method 200.7.
 - 3.3.5 The CCV satisfies the requirements of the IPC found in method 200.7.
- 3.4. Other terminology equivalents (used in SOP or LIMS)
 - 3.4.1. RL = LLOQ
 - 3.4.2. LLICV = LLC = CRI
 - 3.4.3. Dilution test = serial dilution

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- 3.5 Dissolved Metals: Those elements which pass through a 0.45 um membrane filter. (Sample is acidified after filtration)
- 3.6 Suspended Metals: Those elements retained by a 0.45 um filter.
- 3.7 Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

4.0 INTERFERENCES

- 4.1 Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by
 - 4.1.1 Overlap of a spectral line from another element.
 - 4.1.2 Unresolved overlap of molecular band spectra.
 - 4.1.3 Background contribution from continuous or recombination phenomena.
 - 4.1.4 Stray light from the line emission of high concentration elements.
- 4.2 A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.
- 4.3 Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.
- 4.4 Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.
 - 4.4.1 Internal standards are used to correct for some matrix interferences. The use of two internal standards (one for axial view wavelengths and one for radial view wavelengths) is required.
- 4.5 Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 The ICP plasma emits strong UV light, harmful to vision. Analysts must avoid looking directly at the plasma.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide 30%	Oxidizer Corrosive Fire (increases flammability of combustible, organic, and readily oxidizable materials)	1 ppm (TWA)	Irritation to respiratory tract and burning of mucous membrane of nose and throat. Pain, redness, and blurred vision in eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Thermo iCap 6500 Duo ICP , which includes the following:

- 6.1.1 Auto sampler
- 6.1.2 Chiller (water cooling device)
- 6.1.3 ESI FAST valve
- 6.1.4 Instrument Software: iTeva version 2.4.0.81

6.2 Argon gas: High-purity grade (99.99%)

6.3 Calibrated automatic pipettes

6.4 Class A volumetric flasks

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 Concentrated nitric acid (HNO_3), trace metal grade
- 7.3 Concentrated hydrochloric acid (HCl), trace metal grade
- 7.4 DI water from the Millipore unit (ASTM Type I)
 - 7.4.1 Water must be free of the analytes of interest as demonstrated through the analysis of method blanks. Water must be shown to have a resistivity greater than or equal to 18 Mohm-cm.
- 7.5 Standards, NIST traceable
 - 7.5.1 Purchased as custom multi-element mixes or as single-element solutions.
 - 7.5.2 All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles.
 - 7.5.3 Working standards may be used for up to 6 months, with the exception of the ICV
 - 7.5.3.1 The ICV must be prepared daily, and is used to verify that the calibration standards are still viable.
 - 7.5.4 Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acid.
- 7.6 Internal Standard: 1 ppm yttrium
- 7.7 Torch Profiling solution: 2 ppm zinc

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Aqueous samples for total metals must be digested before analysis using an appropriate digestion procedure, ST-IP-0013.
- 8.3 Soil or waste samples are digested before analysis using an appropriate digestion procedure. Method 3050B of SW846 is the appropriate digestion procedure, ST-IP-0002.
- 8.4 Digestate holding time is 6 months from sample collection.

9.0 QUALITY CONTROL

- 9.1 **Batch**
 - 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
 - 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
 - 9.1.3 For this analysis, batch QC consists of a method blank (MB), a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.2 **Method Blank (MB)**

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 For water batches, 2% HNO₃ is used
- 9.2.4 For soil batches, glass beads or Teflon chips are used.
- 9.2.5 See Section 13 for acceptance criteria.
- 9.3 **Laboratory Control Sample (LCS)**
 - 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.3.2 An LCS must be prepared with every sample batch.
 - 9.3.3 For water batches, 2% HNO₃ spiked with the analytes of interest is used.
 - 9.3.4 For soil batches, a commercially available solid reference material (SRM) is used. Where an SRM is not available, or the manufacturer limits are not sufficient for client required limits, glass beads or Teflon chips spiked with an aqueous solution of the analytes of interest is used
 - 9.3.5 See Section 13 for acceptance criteria.
- 9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**
 - 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.4.2 See LIMS for acceptance criteria.
- 9.5 **Dilution Test (SD)**
 - 9.5.1 A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix.
 - 9.5.2 The test is performed by running a sample at a 5x (1:5) dilution.
 - 9.5.3 Samples identified as field blanks cannot be used for dilution tests.
 - 9.5.4 See Section 13 for acceptance criteria.
- 9.6 **Post Digestion Spike (PDS)**
 - 9.6.1 A post digestion spike is a sample which has been fortified with target analytes of interest after the digestion process, with a spike concentration between 10-100 times the MDL (unless specific project/program criteria is given)
 - 9.6.2 **200.7:** A PDS is not applicable for this method.
 - 9.6.3 **6010B and 6010D:** A PDS is analyzed with every batch
 - 9.6.4 **6010C:** The method stipulates that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a serial dilution on the sample. If the PDS is acceptable, the laboratory is not required to perform a serial dilution. Since the laboratory has elected to perform the serial dilution routinely, the intermediate step of a post digestion spike is not performed.
 - 9.6.4.1 For client project or programs requiring a PDS, the laboratory will include a PDS in the batch in addition to the serial dilution. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.
 - 9.6.5 See Section 13 for acceptance criteria
- 9.7 **Method of Standard Addition (MSA)**
 - 9.7.1 **6010B /6010C/6010D**
 - 9.7.2 This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may

enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.

- 9.7.3 MSA are not required by method 6010B, 6010C or 6010D.
- 9.7.4 MSAs are not considered standard batch QC and if required by the client, must appear on the client requirements memo or in the comment section of LIMS.
- 9.7.5 MSA is required by SW846 Method 1311 when the MS/MSD recovery is less than 50%, analyte concentration is less than and within 20% of its regulatory limit.
- 9.7.6 See section 12.4 and Appendix I for more detail on how to perform an MSA

9.8 **Procedural Variations/ Nonconformance and Corrective Action**

- 9.8.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before calibration (minimum 30 minutes of warm-up is required).
- 10.2 The instrument has a fully automated wavelength calibration that is performed when the plasma is lit.
 - 10.2.1 This is to monitor and prevent instrument spectral drift
 - 10.2.2 Criteria: ± 10
 - 10.2.3 Action upon failure: Call service engineer.
- 10.3 Rinse Time Determination
 - 10.3.1 Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in this SOP it can be demonstrated that a shorter rinse time may be used.
 - 10.3.1.1 To determine the appropriate rinse time for a particular ICP system, a linear range verification standard should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.
- 10.4 Background Correction Points
 - 10.4.1 A background correction technique is applied to compensate for variable background contribution. This is performed by setting background points during initial instrument set up and matrix matching.
- 10.5 Inter-element Corrections (IECs)
 - 10.5.1 The IECs are verified on a daily basis by analyzing the ICSA and ICSAB solutions at the beginning of every analytical run. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than $\pm$ the RL. To determine IECs, run a single element standard at the high level calibration standard. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."
 - 10.5.2 IEC factors are updated every 6 months or after significant maintenance have been performed.

- 10.6 Linear dynamic Range (LDR)
 - 10.6.1 Prior to running the instrument, the upper limit of quantitation must be established for each analyte.
 - 10.6.2 This upper limit is tested by running a standard containing high concentrations of the analytes against a calibration curve.
 - 10.6.3 The concentration of the LDR standard is equal to that of the high calibration standard.
 - 10.6.4 Frequency: Daily
 - 10.6.5 Criteria: $\pm 10\%$
 - 10.6.6 If the LDR fails criteria at the highest calibration point, the LDR will be set at the next highest verified point (typically the Cal 2/CCV)
- 10.7 Initial Calibration
 - 10.7.1 Multi-point Calibration:
 - 10.7.1.1 A calibration curve, consisting of 3 standards and a blank, must be analyzed daily.
 - 10.7.1.2 Calibration criteria:
 - 10.7.1.2.1 **6010B/6010C**: Correlation Coefficient of ≥ 0.998
 - 10.7.1.2.2 **6010D**: Correlation Coefficient of ≥ 0.995
 - 10.7.1.2.3 The low level standard in the curve must be at or below the laboratory's routine reporting limit.
 - 10.7.1.2.3.1 If a client requested reporting limit is below the laboratory's routine reporting limit and thus below the low level verification standard, the laboratory will discuss with the client, prior to sample analysis, how to proceed with this requirement.
- 10.8 **Initial Calibration Verification/Low Level Calibration Verification/Initial Calibration Blank (ICV/LLICV/ICB)**
 - 10.8.1 **ICV**
 - 10.8.1.1 Secondary source, used to verify the initial calibration accuracy
 - 10.8.1.2 Frequency: Perform with each initial calibration
 - 10.8.1.3 Criteria:
 - 10.8.1.3.1 **Method 200.7**: $\pm 5\%$ with relative standard deviation $< 3\%$ from replicate exposures.
 - 10.8.1.3.2 **Method 6010B**: $\pm 10\%$ with relative standard deviation $< 5\%$ from replicate exposures.
 - 10.8.1.3.3 **Method 6010C/6010D**: $\pm 10\%$
 - 10.8.1.4 Action upon failure:
 - 10.8.1.4.1 If the ICV fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
 - 10.8.1.4.2 For all other non-conformances, the samples must be re-analyzed.
 - 10.8.2 **LLICV**
 - 10.8.2.1 Applicable to **6010C** and **6010D** only
 - 10.8.2.2 Same source as calibration
 - 10.8.2.3 Frequency: Perform with each initial calibration.
 - 10.8.2.4 Criteria:
 - 10.8.2.4.1 **6010C**: $\pm 30\%$
 - 10.8.2.4.2 **6010D**: $\pm 20\%$
 - 10.8.2.5 Action upon failure:

- 10.8.2.5.1 If the LLICV fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the LLICV, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.8.2.5.2 If the LLICV fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.8.2.5.3 For all other non-conformances, the samples must be re-analyzed.
 - 10.8.3 **ICB**
 - 10.8.3.1 Frequency: An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness.
 - 10.8.3.2 Criteria:
 - 10.8.3.2.1 **200.7/6010B/6010C**: $\pm$ the RL from zero
 - 10.8.3.2.2 **6010D**: $\pm \frac{1}{2}$ the RL from zero.
 - 10.8.3.3 Action upon failure:
 - 10.8.3.3.1 If the ICB fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.8.3.3.2 If the ICB fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.
 - 10.8.3.3.3 For all other non-conformances, the samples must be re-analyzed.
 - 10.8.4 If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration re-verified.
 - 10.8.5 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.
- 10.9 **Continuing Calibration Verification/Low Level Continuing Calibration Verification/Continuing Calibration Blank (CCV/LLCCV/CCB)**
- 10.9.1 Calibration is monitored throughout the analytical run through the analysis of a known mid-level calibration standard.
 - 10.9.2 **CCV**
 - 10.9.2.1 A CCV may be a second source or the same source as the calibration
 - 10.9.2.2 Frequency: Analyte response factors must be verified at the beginning of each analytical run (by either an ICV or a CCV), after every 10 samples and at the end of the analysis run through the analysis of a mid-level calibration standard.
 - 10.9.2.3 Criteria:
 - 10.9.2.3.1 **Method 200.7/6010C/6010D**: $\pm 10\%$
 - 10.9.2.3.2 **Method 6010B**: $\pm 10\%$, with relative standard deviation $< 5\%$ from replicate exposures.
 - 10.9.2.4 Action upon failure:
 - 10.9.2.4.1 If the CCV fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM
 - 10.9.2.4.2 For all other non-conformances, the samples must be re-analyzed.
 - 10.9.3 **LLCCV**
 - 10.9.3.1 Applicable to **6010C** only

10.9.3.2 Same source as calibration

10.9.3.3 Frequency: Perform at a minimum at the end of the run

10.9.3.4 Criteria: $\pm 30\%$

10.9.3.5 Action upon failure:

10.9.3.5.1 If the LLCCV fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the LLICV, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.9.3.5.2 If the LLCCV fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.9.3.5.3 For all other non-conformances, the samples must be re-analyzed.

10.9.4 **CCB**

10.9.4.1 Frequency: A CCB is analyzed immediately following each CCV.

10.9.4.2 Criteria: $\pm$ RL from zero.

10.9.4.3 Action upon failure:

10.9.4.3.1 If the CCB fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the blank, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.9.4.3.2 If the CCB fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.9.4.3.3 For all other non-conformances, the samples must be re-analyzed.

10.9.5 If a CCV and/or CCB has failed and the analyst can document the reason for failure (e.g mis-injection, etc.) then a second CCV and/or CCB may be analyzed without any adjustments to the instrument. If this CCV and/or CCB meet criteria then sample analysis may continue; however the preceding 10 samples must be reanalyzed. If this second CCV and/or CCB does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require re-calibration (i.e., initial calibration).

10.10 **Interference Check Analysis (ICSA/ICSAB)**

10.10.1 The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions.

10.10.2 **ICSA:**

10.10.2.1 The ICSA contains only interfering elements. Refer to LIMS for the details of ICSA composition.

10.10.2.2 Custom multi-element ICS solutions must be used.

10.10.2.3 Elements known to be interferences on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

10.10.2.4 Frequency: Must run with each initial calibration or every 12 hours whichever is shorter.

10.10.2.5 Criteria:

10.10.2.5.1 For interfering elements: $\pm 20\%$

10.10.2.5.2 For the non-interfering elements: $\pm 1 \times$ RL from zero

10.10.2.6 Action upon failure:

10.10.2.6.1 For interfering elements:

10.10.2.6.1.1 If the ICSA fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM

10.10.2.6.2 For non-interfering elements:

10.10.2.6.2.1 If the ICSA fails high, but the concentration the associated samples is less than the RL or greater than 10X the concentration found in the blank, the potential bias has not affected the

samples. Samples may be reported with an NCM.

10.10.2.6.2.2 If the ICSA fails low, but the concentration the associated samples is 10X the RL, the potential bias has not affected the samples. Samples may be reported with an NCM.

10.10.2.6.3 For all other non-conformances, the samples must be re-analyzed.

10.10.3 ICSAB:

10.10.3.1 The ICSAB contains analytes and interferents.

10.10.3.2 Refer to LIMSLIMS for the details of ICSAB composition.

10.10.3.3 Custom multi-element ICS solutions must be used.

10.10.3.4 Frequency: Must run with each initial calibration or every 12 hours whichever is shorter.

10.10.3.5 Criteria: $\pm 20\%$

10.10.3.6 Action upon failure:

10.10.3.6.1 If the ICSAB fails high, but the sample concentrations are below the reporting limit, the potential high bias has not affected the samples. Samples may be reported with an NCM

10.10.3.6.2 For all other non-conformances, the samples must be re-analyzed.

10.11 Calibration Sequence

Initial Calibration (3 standards plus a blank)

ICV

ICB

LLICV (for 6010C only)

ICSA*

ICSAB*

CCV

CCB

LDR

CCV

CCB

10 samples (analysis runs)

CCV

CCB

10 samples (repeat every 10 analysis runs)

LLCCV** (for 6010C only)

CCV

CCB

End

* If sequence time is longer than 12 hours, the ICSA and ICSAB standard must be re-analyzed .

**Minimum at end of run. May be analyzed more frequently

11.0 PROCEDURE

11.1 The aqueous sample digestion procedure is found in SOP: ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8)

11.1.1 For 200.7 analyses, dissolved samples must be digested

11.2 The soil sample digestion procedure is found in SOP: ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, and ICP/MS.

11.3 Instrument conditions, including rinse times, must be the same for all standards and samples.

11.4 A minimum of three exposures for each standard, field sample and QC sample is required. The average of the exposures is reported.

- 11.5 Internal standards are introduced to the standards and sample digestates by the instrument.
- 11.6 Load autosampler with standards and digestates in accordance with the sequence given in section 10.
- 11.7 Analyze samples.
- 11.8 When analysis is completed, return unused digestate to proper storage area.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis LQM.
- 12.2 All measurements must fall within the defined linear range where spectral interference correction factors are valid.
 - 12.2.1 Dilute and reanalyze all samples for required analytes that exceed the linear range.
 - 12.2.1.1 For 200.7, any sample greater than 90% of the linear range must be diluted and re-analyzed.
 - 12.2.2 Acid strength must be maintained in the dilution of samples.
- 12.3 If an inter-element correction exists for an analyte which exceeds the linear range, the IEC may be applied. Even if an over-range analyte may not be required to be reported for a sample, if that analyte is an interferent for any requested analyte in that sample, the sample must be diluted.
- 12.4 The procedure for MSA is to split the sample into several even aliquots in separate volumetric flasks of the same volume. The first flask is then diluted to volume with the selected diluent. A standard containing the analyte is then added in increasing volumes to the subsequent flasks and each flask is then diluted to volume with the selected diluent. The instrument response is then measured for all of the diluted solutions and the data is plotted with volume of standards added in the x-axis and instrument response in the y-axis. Linear regression is performed and the slope (m) and y-intercept (b) of the calibration curve are used to calculate the concentration of the analyte in the sample.
 - 12.4.1 See Appendix I

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process in LIMS. The NCM process is described in SOP: ST-QA-0036.
- 13.2 Method Blank (MB)
 - 13.2.1 Acceptance Criteria:
 - 13.2.1.1 **200.7/6010B/6010C**: <RL
 - 13.2.1.2 **6010D**: < ½ RL
 - 13.2.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements memo or in the comment section of LIMS.
 - 13.2.3 Corrective Action for Method Blanks not meeting acceptance criteria:
 - 13.2.3.1 Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.
- 13.3 Laboratory Control Sample (LCS)
 - 13.3.1 Acceptance Criteria: All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).
 - 13.3.1.1 Control limits are found in LIMS.

- 13.3.2 Corrective Action for LCS not meeting acceptance criteria:
 - 13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.
 - 13.3.2.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.
- 13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
 - 13.4.1 Acceptance Criteria: Analytes should be within control limits for accuracy (%Recovery) and precision (RPD))
 - 13.4.1.1 Control limits can be found in LIMS.
 - 13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:
 - 13.4.2.1 MS/MSD Spike Recovery excursion: may not necessarily warrant corrective action other than narration
 - 13.4.2.2 If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.
 - 13.4.2.3 If the excursion is due to physically evident matrix interference, the data is reported with an NCM.
 - 13.4.2.4 In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with and NCM.
 - 13.4.2.5 When the MS/MSD recoveries and the %RPD are outside criteria, if the samples are non-homogenous, the data may be reported with an NCM. Otherwise, the batch is re-prepped and re-analyzed for the affected analytes.
- 13.5 Dilution Test (SD)
 - 13.5.1 A dilution test is not required for method 200.7
 - 13.5.2 Criteria:
 - 13.5.2.1 **6010B/6010C**: when analyte concentration $\geq 10 \times$ RL, $\pm 10\%$
 - 13.5.2.2 **6010D**: when analyte concentration $\geq 25 \times$ RL, $\pm 20\%$
 - 13.5.3 Corrective Action: Dilution test failure is documented in an NCM and the reported data is flagged. If multiple analytes fail the serial dilution test, the analyst may re-prepare and re-analyze the samples.
- 13.6 Post Digestion Spike (PDS)
 - 13.6.1.1 A PDS is not required for method 200.7
 - 13.6.1.2 Criteria:
 - 13.6.1.2.1 **6010B**: 75-125%, with a spike concentration between 10-100 times the MDL, UNLESS other project/program criteria is given.
 - 13.6.1.2.2 **6010C**: 80% – 120%, with a spike concentration between 10-100 times the MDL, UNLESS, other project/program criteria is given.
 - 13.6.1.2.3 **6010D**: 75%-125%
 - 13.6.2 Corrective Action: There is no qualification made to the data based on the performance of the PDS, however a failed PDS is documented with a Non-conformance memo (NCM) and noted in the report narrative.
 - 13.6.3 The PDS is not reported in the data package for **6010C** unless a client project or program requires it. This requirement is noted by the Project Manager in the client requirement memo and/ or in the comment section of LIMS
- 13.7 Sample result evaluation
 - 13.7.1 Dilutions
 - 13.7.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
 - 13.7.2 For samples requiring dilution an NCM is created to document the reason for the dilution.
 - 13.7.3 Insufficient Sample

13.7.3.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, a narrative comment stating such is included in the report narrative.

13.8 Internal Recovery Standard (IS)

13.8.1 Criteria:

13.8.1.1 In house internal standard recovery criteria is 50-150% of the intensity of that standard in the initial calibration standard for all samples and the QC standards.

13.8.2 Action Upon Failure:

13.8.2.1 If this criteria is not met, the sample should be diluted and re-analyzed until the IS recoveries are within specified limits.

14.0 METHOD PERFORMANCE

14.1 Method performance data, reporting limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.3 The following waste streams are produced when this method is carried out.

16.3.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.3.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 6010B, 6010C and 6010D

17.2 Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Method 200.7.

17.3 iCAP 6000 Series ICP-OES Spectrometer User Guide.

17.4 TestAmerica Laboratory Quality Assurance Manual (ST-QAM), current revision

- 17.5 TestAmerica Corporate Safety Manual CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.6 Associated SOPs, current revisions;
 - 17.6.1 ST-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, ICP/MS, and GFAA
 - 17.6.2 ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8)
 - 17.6.3 ST-QA-0002, Standard and Reagent Preparation
 - 17.6.4 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.6.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.6.6 ST-QA-0016, IDL/MDL Determination
 - 17.6.7 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 Modifications/interpretations Method 6010C
 - 18.1.1 Method 6010C recommends that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a serial dilution on the sample. If the PDS is acceptable, the laboratory is not required to perform a serial dilution. Since the laboratory has elected to perform the serial dilution routinely, the intermediate step of a post digestion spike is not performed. Internal standards are used to monitor matrix interferences in all samples. Post spikes are done per specific QAPP or program requirements. Post-spikes using analytes other than the internal standards may be used if an analyst encounters a new or unusual matrix.
- 18.2 Modifications from Method 200.7.
 - 18.2.1 TestAmerica St. Louis digests all aqueous samples. Method 200.7 allows for the digestion step to be omitted under certain conditions.
 - 18.2.2 Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength. TestAmerica labs utilize the CLP IDL definition.
 - 18.2.3 For method 200.7 the calibration blank is prepared in an acid matrix of 5% HNO₃/5% HCl instead of the specified 2% HNO₃/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.
 - 18.2.4 Method 200.7 specifies that "Analysis of the IPC (ICV/CCV) solution immediately following calibration must verify that the instrument is within $\pm 5\%$ of calibration with a relative standard deviation $< 3\%$ from replicate integrations ≥ 4 ." TestAmerica uses a minimum of three exposures.
 - 18.2.5 Method 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP deviates from the 1ppm criteria. For the analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.
 - 18.2.6 Method 200.7 states that results for the ICS should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.
 - 18.2.7 Method 200.7 states the CCB should be less than the IDL, but $>$ the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. TestAmerica has adopted an absolute control limit of $\pm$ the reporting limit for calibration blank criteria.
 - 18.2.8 Method 200.7 suggests the use of a PDS to assess the need for an internal standard. TestAmerica uses an internal standard for all samples, and so does not perform the PDS for this method
 - 18.2.9 Method 200.7 has more stringent blank acceptance criteria than stated in this SOP. Due to software limitations the lab is unable to evaluate and report blanks using the method criteria that "analyte concentration in the blank greater than 2.2 times the MDL, or greater than 10% of the concentration of a sample requires re-preparation and reanalysis." Blanks are evaluated to the MDL and flagged if they exceed that level. Re-preparation and re-analysis are required if the method blank is greater than RL and the sample is greater than the RL but less than ten times the RL

- 18.3 Modifications from Method 6010C.
 - 18.3.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.
 - 18.3.2 Method 6010C requires the analysis of a Lower Limit of Quantitation Check Sample (LLQC) to establish and confirm the lowest quantitation limit. TestAmerica St. Louis fills this requirement with the running of a MDL verification standard which is taken through the entire sample preparation procedure.
 - 18.3.3 Method 6010C stipulates that a PDS be performed. If the PDS is acceptable, the laboratory is not required to perform a serial dilution. Since the laboratory has elected to perform the serial dilution routinely, the outcome of the PDS is not critical and a PDS is only analyzed at client request.

19.0 CHANGES TO PREVIOUS REVISION

- 19.1 12/30/2010; REV 10, Modifications (Sec 18.0), removed Section 18.6 that stated that MSA requirement Method 1311 is not applied.
- 19.2 Clarified CCB requirements in section 10.11
- 19.3 Rev. 11:
 - 19.3.1 Updated section 10.12.5 ICSA Criteria: non-interfering elements with $RL < 10$ must be $\pm 2X$ the RL from zero; results for non-interfering elements with $RL > 10$ must fall within $\pm 1X$ RL from zero.
- 19.4 Rev. 12:
 - 19.4.1 Removed the exception taken to the MSA requirement in section 18.
 - 19.4.2 Added instrument software and hardware to section 6.0.
 - 19.4.3 Added MSA requirements for MS/MSD recovery in section 9.7.
 - 19.4.4 Updated new data analysis and calculations in section 12.0.
- 19.5 Rev 13:
 - 19.5.1 Updated adjustments to profile in section 10.7.
- 19.6 Rev 14:
 - 19.6.1 Added specific reference CRI recovery criteria for method 6010C in section 10.10.
- 19.7 Rev 15: (6/17/2013)
 - 19.7.1 Removed references to "Clouseau" and "QuantIMS" – replaced with "LIMS"
 - 19.7.2 Removed all references to the 61E
 - 19.7.3 Updated section 4.0 with discussion of physical interferences.
 - 19.7.4 Added internal standard discussion to section 7.7 and 12.0
 - 19.7.5 Added CCB corrective action guidance to Section 10.1
 - 19.7.6 Updated Corrective Action information in Section 13, replacing references to the Clouseau NCM database with actual corrective actions to be taken.
 - 19.7.7 Removed section 10.2.1 and 10.2.2 (information was already in section 10.7)
 - 19.7.8 Added reference to 6500 to section 10
 - 19.7.9 Updated section 9.6
 - 19.7.10 Updated section 18.3
- 19.8 Rev 16: (4/21/2015)
 - 19.8.1 Added reference to 6010B
 - 19.8.2 Removed references to 61T
 - 19.8.3 Updated supplies
 - 19.8.4 Updated instrument/software
 - 19.8.5 Removed Table 1 and 2
 - 19.8.6 Added a new Table 1 (method criteria)
 - 19.8.7 Added "actions upon failure" through out SOP
 - 19.8.8 Added dilution information to Section 11
 - 19.8.9 Formatted to match the ICPMS SOP (ST-MT-0001)
- 19.9 Rev. 17: (06/22/2015)

- 19.9.1 Added Appendix 1 MSA instructions
- 19.10 Rev. 18: (08/08/2016)
 - 19.10.1 Added method 6010D and all it's requirements
 - 19.10.2 Updated definitions
 - 19.10.3 Updated frequency needed to make standards
 - 19.10.4 Made clarifications throughout
 - 19.10.5 Changed wording from "serial dilution" to "dilution test"
 - 19.10.6 Updated criteria for ICSA
 - 19.10.7 Added method modification to section 18, method 200.7: PDS not needed, MB criteria.
 - 19.10.8 updated matrix for water MB and LCS
 - 19.10.9 updated Table 1

Table 1
QC Criteria

Methods	6010B	6010C	6010D	200.7
Corr Coeff.	≥0.998	≥0.998	≥0.995	N/A
Int Std	50-150%	50-150%	50-150%	50-150%
ICV	90-110% RSD<5%	90-110%	90-110%	95-115% RSD<3%
ICB	±RL	±RL	± ½ RL	±RL
CCV	90-110% SD<5%	90-110%	90-110%	90-110%
LLICV	N/A	70-130%	80-120%	N/A
LLCCV	N/A	70-130%	N/A	N/A
CCV	90-110%	90-110%	90-110%	90-110%
CCB	±RL	±RL	±RL	±RL
LDR	90-110%	90-110%	90-110%	90-110%
MB	±RL	±RL	± ½ RL	±RL
LCS	80-120%	80-120%	80-120%	85-115%
MS	75-125%	75-125%	75-125%	70-130%
PDS	75-125%	80-120%	75-125%	N/A
SD	± 10%	± 10%	± 20%	N/A

APPENDIX I – METHOD OF STANDARD ADDITION (MSA)

MSA is required if

- 1- Matrix spike recovery <50%, AND
- 2- Measured concentration is within 20% of the regulatory level

Regulatory Limits:

Arsenic	5mg/L
Barium	100mg/L
Cadmium	1 mg/L
Chromium	5 mg/L
Lead	5 mg/L
Mercury	0.2 mg/L
Selenium	1 mg/L
Silver	5 mg/L

How to run an MSA

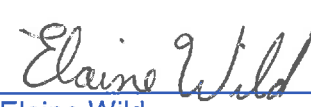
- 1- Take 4 identical aliquots of test solution
- 2- Add increasing concentration of standard to 3 aliquots and add blank solution to 4th aliquot- all aliquots should be at same final volume
- 3- Perform analysis
- 4- Enter data into spreadsheet ORG-0034_TCLP_MSA
 - a. Uses unweighted least-squares linear regression curve fit
 - b. Calculates absolute value of x-intercept

Notes:

- 1- MSA spikes must come from the same analytical batch, analyzed on the same day.

The MSA curve must include the unspiked sample and 3 samples spiked with increasing concentrations of analytes

**Title: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS
SAMPLES BY COLD VAPOR ATOMIC ABSORPTION
[SW846 7470A; MCAWW 245.1]**

Approvals (Signature/Date):			
	8/29/16		8/29/16
Kristen Ely	Date	Michael Ridenhower	Date
Inorganics Department Manager		Health & Safety Manager / Coordinator	
	8/29/16		8/29/16
Marti Ward	Date	Elaine Wild	Date
Quality Assurance Manager		Laboratory Director	

This SOP was previously identified as SOP No. ST-MT-0005 Rev. 16

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1.0 SCOPE AND APPLICATION

- 1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A and MCAWW Method 245.1.
- 1.2 CVAA analysis provides for the determination of total mercury (organic and inorganic).
- 1.3 Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, TCLP, EP and other leachates/extracts. All matrices require sample preparation prior to analysis.
- 1.4 Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, domestic and industrial wastes. All matrices require sample preparation prior to analysis.
- 1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1 This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).
- 3.3 Suspended Metals: Those elements which are retained by a 0.45 um membrane.
- 3.4 Total Metals: The concentration determined on an unfiltered sample following digestion.
- 3.5 EPA and SW methodology use different terminology. This SOP references the SW 846 terminology:
 - 3.5.1 The ICV satisfies the QCS requirements found in method 245.1.
 - 3.5.2 The LCS satisfies the requirements of the LFB found in method 245.1.
 - 3.5.3 The CCV satisfies the requirements of the IPC found in method 245.1.

4.0 INTERFERENCES

- 4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.
- 4.2 Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.
- 4.3 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.
- 4.4 Chlorides can cause a positive interference. Seawaters, brines and industrial effluents which are high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

- 4.5 Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.
- 4.6 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 Specific Safety Concerns or Requirements
- 5.2.1 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.
- 5.3 Primary Materials Used
- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limits (2)	Signs and Symptoms of Exposure
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. Contact with other material may cause fire. Liquid and mist cause severe burns to all body tissue.
Sulfuric Acid	Corrosive Oxidizer Dehydrator	1 Mg/M3- (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored

Material (1)	Hazards	Exposure Limits (2)	Signs and Symptoms of Exposure
	Poison		breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness. Sulfuric acid is extremely corrosive. Liquid and mist cause severe burn to all body tissue.
Hydrochloric Acid	Poison Corrosive	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage. Hydrochloric acid is corrosive. Liquid and mist causes severe burn to all body tissue.
Potassium permanganate	Oxidizer	5 mg/m3 (Ceiling)	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent. Contact with other materials may cause fire. Causes burns to any area of contact. Harmful if swallowed or inhaled.
Hydroxylamine Hydrochloride	Poison Corrosive	None established	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache nausea and vomiting. Skin contact may cause irritation and burns. Eye contact may cause severe irritation and corneal damage. Causes burns to any area of contact. Harmful if swallowed or inhaled.
Mercury	Poison Corrosive	0.1 mg/m3 (TWA)	Inhalation symptoms include sore throat, coughing, pain, tightness in chest, breathing difficulties, shortness of breath, headache, muscle weakness, anorexia, gastrointestinal disturbance, ringing in the ear, liver changes, fever, bronchitis and pneumonitis. Skin contact produces redness and pain. May be absorbed through skin with symptoms paralleling those of inhalation. Eye contact symptoms include redness, pain, and blurred vision. Causes burns to skin, eyes, and respiratory tract. May be fatal if swallowed or inhaled. Harmful if absorbed through skin.
Potassium Persulfate	Oxidizer	0.1 mg/m3 (TWA)	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis. Contact with other material may cause fire. Harmful if swallowed.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
STEL – Short Term Exposure Limit			
TWA – Time Weighted Average			
Ceiling – At no time should this limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Temperature controlled hot block (capable of maintaining a temperature of $95 \pm 3^{\circ}\text{C}$).
- 6.2 Mercury Analyzer - Teledyne Leeman Labs Hydra II AA
- 6.3 Hydra II AA software: Envoy Version 2.003
- 6.4 50 mL and 100mL certified class A digestion tubes
- 6.5 Argon gas supply, welding grade or equivalent.
- 6.6 Calibrated automatic pipettes
- 6.7 Class A volumetric flasks.
- 6.8 Thermometer (capable of accurate readings at $95 \pm 3^{\circ}\text{C}$).
- 6.9 Disposable sampling tubes.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
- 7.3 Nitric acid (HNO_3), concentrated, analytical reagent grade (AR) or better.
- 7.4 Hydrochloric acid concentrated, analytical reagent grade (AR) or better
- 7.5 Sulfuric acid (H_2SO_4), concentrated, analytical reagent grade (AR) or better
- 7.6 Stannous chloride: Dissolve 100g of stannous chloride into 1000 mL of 10% HCl.
- 7.7 Sodium chloride-hydroxylamine sulfate solution: Add 12 g of sodium chloride and 12 g of hydroxylamine sulfate to every 100 mL of reagent water or a certified stock reagent may be purchased from a vendor.
- 7.8 Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water or a certified stock reagent may be purchased from a vendor.
- 7.9 Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water or a certified stock reagent may be purchased from a vendor.
- 7.10 Stock (100 ppm) mercury standards (in 5% HNO_3) are purchased as custom solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.
 - 7.10.1 A primary (Cal) and secondary (QC) source is used
- 7.11 Intermediate mercury standard (0.1 ppm): Take 0.1 mL of the stock mercury standard and dilute to 100 mL with reagent water. The intermediate standard must be made daily and must be prepared in a matrix of 2% HNO_3 . This acid (2 mL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.
 - 7.11.1 A primary (Cal) and secondary (QC) source is used.

7.12

Standard	Amount (mL)	Spike Solution	Final Volume (mL)	Final Concentration (ppb)
Blank/ICB/CCB	0	Cal Int	30	0
Cal 1/LLC	0.06	Cal Int	30	0.2
Cal 2	0.15	Cal Int	30	0.5
Cal 3	0.3	Cal Int	30	1
Cal 4/CCV	1.5	Cal Int	30	5
Cal 5	3	Cal Int	30	10
ICV	1.5	QC Int	30	5
LCS/MS/MSD	1.5	QC Int	30	5

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.12.1 The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.12.2 All standards must be processed through the entire analytical procedure including sample preparation.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002. Sample collection is not performed by the laboratory for this analysis.

8.2 Sample shipments received by the laboratory will be reviewed against the Sample Acceptance Policy described in SOP ST-PM-0002, "Sample Receipt and Chain of Custody."

8.3 The sample holding time for mercury is 28 days from time of collection to the time of analysis.

8.4 Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

9.0 QUALITY CONTROL

9.1 Batch

9.1.1 Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

- 9.1.2 Conditions must be the same for all standards, samples and QC samples.
- 9.1.3 Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS), and Matrix Spike/Matrix Spike Duplicate (MS/MSD). In the event that there is insufficient sample to analyze a duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.2 **Method Blank (MB)**
 - 9.2.1 Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.2.2 A method blank must be included with each batch of samples. The matrix for aqueous is reagent (DI) water.
 - 9.2.3 A method blank must be prepared with every batch (20 or fewer samples of the same matrix).
- 9.3 **Laboratory Control Sample (LCS)**
 - 9.3.1 Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.3.2 An LCS must be prepared with every batch.
 - 9.3.3 The water LCS is reagent (DI) water fortified with mercury.
- 9.4 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)**
 - 9.4.1 Definition: Two aliquots of a field sample to which a known amount of target analyte(s) is added.
 - 9.4.2 Additional MS/MSD's do not count towards the 20 samples in an analytical batch.
 - 9.4.3 Matrix spikes are performed every 10 samples for EPA 245.1.
- 9.5 **Sample Duplicates (DU)**
 - 9.5.1 Definition: Sample Duplicate – a separate aliquot of a field sample taken through the entire analytical process.
 - 9.5.2 Additional Sample Duplicates do not count towards the 20 samples in an analytical batch.
 - 9.5.3 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed.
- 9.6 **Serial Dilution (SD)**
 - 9.6.1 Definition: a sample is diluted 1:5 and reanalyzed to assess the presence of a matrix interference.
 - 9.6.2 A serial dilution is performed with every analytical batch.
 - 9.6.3 Agreement within 10% between the concentration for the undiluted sample and five times the concentration for the diluted sample indicates the absence of interferences.
- 9.7 **Post Digestion Spikes (PDS)**
 - 9.7.1 Definition: A known amount of mercury is added to the sample chosen for serial dilution to bring the concentration of mercury to 2 to 5 times the original concentration. If the sample's mercury concentration is below the detection limit, spike at 20 times the detection limit.
 - 9.7.2 At client's request, a post digestion spike is performed when the serial dilution test fails.
 - 9.7.2.1 Post digestion spike recovery criteria: 85%-115%.
- 9.8 **Method of Standard Addition (MSA)**
 - 9.8.1 Definition: This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.
 - 9.8.1.1 Certain clients may require specific project or program QC which may supersede the SOP requirements. These are documented in comment section of LIMS.
 - 9.8.2 MSA's are not considered normal batch QC and if required by the client, must appear in comment section of LIMS

9.9 Procedural Variations/ Nonconformance and Corrective Action

- 9.9.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.9.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION**10.1 Initial Calibration**

- 10.1.1 Calibration standards must be processed through the preparation procedure.
- 10.1.1.1 Batches that are prepped at different times must each have their own calibration standards
- 10.1.2 Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.
- 10.1.3 Calibration must be performed daily (every 24 hours) or upon initial run of a method. The instrument calibration date and time must be included in the raw data.
- 10.1.4 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration.
- 10.1.5 Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the reporting limit. Analyze standards in ascending order beginning with the blank.
- 10.1.6 The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.
- 10.1.7 If the calibration curve does not meet method requirements, another calibration curve should be performed.

10.2 Initial Calibration Verification (ICV)

- 10.2.1 An ICV is a second source verification of the initial calibration.
- 10.2.2 An ICV must be performed with every initial calibration.
- 10.2.3 If the ICV fails to meet criteria $\pm 5\%$ for Method 245.1 or $\pm 10\%$ for method 7470A, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.
- 10.2.4 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).

10.3 Initial Calibration Blank (ICB)

- 10.3.1 Analyze the initial continuing calibration blank (ICB) immediately following the ICV.
- 10.3.2 The absolute value of the result must be less than or equal to the reporting limit (RL). If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument if necessary.

10.4 Low Level Check (LLC) aka CRA (When requested by client)

- 10.4.1 Can be the same source as your calibration.
- 10.4.2 Must be performed with every initial calibration.
- 10.4.3 Must fall within 30% of the true value.
- 10.4.4 Must be spiked at RL

- 10.4.5 If the LLC does not meet QC criteria, the analysis must be terminated, the problem corrected and the instrument recalibrated
- 10.5 Continuing Calibration Verification (CCV)
 - 10.5.1 A CCV is analyzed after every 10 samples and at the end of the analytical sequence run.
 - 10.5.2 The CCV for EPA 7470A must fall within 20% of the true value for that solution; for EPA 245.1, the CCV must fall within 10% of the true value.
 - 10.5.3 If the CCV does not meet QC criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.
- 10.6 Continuing Calibration Blank (CCB)
 - 10.6.1 Analyze a continuing calibration blank (CCB) immediately following the CCV.
 - 10.6.2 The absolute value of the result must be less than or equal to the reporting limit (RL). If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument if necessary.

11.0 PROCEDURE

- 11.1 All calibration and calibration verification standards (ICV, ICB, LLC, CCV, CCB) are processed through the digestion procedure as well as the field samples.
- 11.2 Calibration Standard/Verification Standard Preparation:
 - 11.2.1 Transfer 0, 0.06, 0.15, 0.3, 1.5, 3.0 mL aliquots of the Cal intermediate standard onto a series of sample digestion tubes, and bring to 30 mL final volume with DI water.
 - 11.2.1.1 The LLC is taken from the same digestion tube as the Cal 1
 - 11.2.1.2 The CCV is taken from the same digestion tube as the Cal 4
 - 11.2.2 Transfer 1.5 mL of QC intermediate for ICV, and bring to 30 mL final volume with DI water.
- 11.3 Sample and Batch QC Preparation:
 - 11.3.1 Transfer 30 mL aqueous sample, 6mLs for TCLP leachate, or 1 mL for CWET leachate to a pre-labeled sample digestion tube. If not already at 30 mL, bring up to a final volume of 30 mLs with DI water.
 - 11.3.1.1 Spike the aqueous MS/MSD aliquots with 1.5 mL of the QC intermediate mercury standard.
 - 11.3.1.2 Spike the TCLP or CWET MS/MSD aliquot with 1.5 mL of the QC intermediate mercury standard.
 - 11.3.2 For the aqueous method blank, transfer 30 mL DI water to a pre-labeled sample digestion tube. For TCLP and CWET method blanks, use 30 ml of the appropriate leaching fluid.
 - 11.3.2.1 Use 6mls of TCLP fluid diluted to 30mls with DI water.
 - 11.3.2.2 Use 1ml of CWET fluid diluted to 30mls with DI water.
 - 11.3.3 For the aqueous LCS, spike 1.5 mL of the QC intermediate mercury standard into a pre-labeled digestion tube and bring to a 30 mL final volume with DI water. For TCLP and CWET LCS, spike 1.5 mL of the QC intermediate mercury standard into a pre-labeled digestion tube and bring to a 30 mL final volume with the appropriate leaching fluid.
- 11.4 Digestion:
 - 11.4.1 Add 1.5 ml (5ml if 100 ml final volume) of concentrated H_2SO_4 and 0.75 ml (2.5 ml if 100 ml final volume) of concentrated HNO_3 mixing after each addition.
 - 11.4.2 Add 4.5 ml (15 ml if 100 ml final volume) of potassium permanganate solution.
 - 11.4.2.1 Permanganate color must persist for at least 15 minutes. Additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If additional portions of permanganate solution are needed, an equivalent portion must also be added to all QC and client samples being

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prepped to match the highest amount added to any one sample. If after the addition of up to 25 mL additional permanganate the color does not persist, sample dilution prior to reanalysis may be required.

11.4.2.2 When performing analyses using automated vs. manual techniques the sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for by the addition of the same volume of permanganate to all other associated samples and standards in the run. *In instances, where this is not feasible, the addition of additional reagent can be achieved without affecting the sample volume through the addition of small portions, 0.5g aliquots, of solid potassium permanganate.*

11.4.3 Add 2.4 mL (8 ml if 100 ml final volume) of potassium persulfate solution and heat for two hours in a hot block at $95 \pm 3^{\circ}\text{C}$.

11.4.4 Cool samples.

11.4.5 The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.5 Sample Analysis:

11.5.1 Add 2 mL of sodium chloride-hydroxylamine sulfate solution (6ml if 100ml final volume is utilized) to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains).

11.5.2 Automated determination: Follow instructions provided by instrument manufacturer.

11.5.3 Check Stannous chloride solution volume is sufficient to cover sample run.

11.5.3.1 Stannous chloride is introduced to the sample by the instrument.

11.5.4 The following analytical sequence must be used with 7470A and 245.1:

Instrument Calibration (5pt plus blank)

ICV

ICB

LLC (if requested by client)

CCV

CCB

Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run

CCV

CCB

11.5.5 If the instrument analysis is stopped for any reason, the run may be resumed. The resumed run must begin with CCV/CCB only if the run has been stopped for more than 2 hours.

11.5.6 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; re-sloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis LQM.

12.2 Appropriate factors must be applied to sample values if dilutions are performed.

12.3 Sample results should be reported with up to three significant figures in accordance with the significant figure policy.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

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- 13.2 Method Blank (MB)
- 13.2.1 Acceptance Criteria: No target analytes may be present in the method blank above the reporting limit.
- 13.2.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted in the comment section of LIMS.
- 13.2.3 Corrective Action for Method Blanks not meeting acceptance criteria:
- 13.2.3.1 Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.
- 13.3 Laboratory Control Sample (LCS)
- 13.4 Acceptance Criteria: All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Limits are in the LIMS.
- 13.5 Corrective Action for LCS not meeting acceptance criteria:
- 13.5.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.
- 13.5.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.
- 13.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
- 13.6.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD). Limits are in LIMS.
- 13.6.2 Corrective Action for MS/MSD not meeting acceptance criteria:
- 13.6.2.1 MS/MSD Spike Recovery excursion: may not necessarily warrant corrective action other than narration
- 13.6.2.1.1 If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.
- 13.6.2.1.2 If the excursion is due to a physically evident matrix interference, the data is reported with an NCM.
- 13.6.2.1.3 In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with an NCM.
- 13.6.2.1.4 When the MS/MSD recoveries and the %RPD are outside criteria, if the sample is non-homogenous, data may be reported with an NCM. Otherwise, the batch is re-prepped and re-analyzed for the affected analytes.
- 13.7 Serial Dilution (SD)
- 13.7.1 The serial dilution results shall agree within $\pm 10\%$ of the undiluted sample results, if the undiluted sample results are greater than 10 times the reporting limit. There is no criteria for sample results less than 10 times the reporting limit.
- 13.7.2 Corrective Action: Serial dilution failure is documented in an NCM and the reported data is flagged.
- 13.8 Post Digestion Spike (PDS)
- 13.8.1 The method stipulates that a PDS be performed on the sample chosen for MS/MSD.
- 13.8.2 There is no qualification made to the data based on the performance of the PDS, however a failed PDS is documented with a Non-conformance memo (NCM) and noted in the report narrative.

13.8.3 The PDS is not reported in the data package unless a client project or program requires it. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.

13.9 Sample result evaluation

13.9.1 Dilutions

13.9.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.

13.9.1.2 For samples requiring dilution an NCM is created to document the reason for the dilution.

13.9.2 Insufficient Sample

13.9.2.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis, a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATIONS OF CAPABILITY

14.1 Method performance data, reporting limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of capability

14.2.1 Initial and continuing Demonstrations of Capability requirements are established in the ST-QAM.

14.3 Training qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial Demonstration of Capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A".

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM

meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury). (Source method.)
- 17.2 “Methods for the Chemical Analysis of Water and Wastes”, EPA-600/4-79-020, U.S. EPA Method 245.1, Revision 3.0 (1994). (Source method.)
- 17.3 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision
- 17.4 Corporate Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.5 Associated SOPs, current revisions
 - 17.5.1 ST-IP-0004, Labware Preparation for Inorganic and Trace Metals Analysis
 - 17.5.2 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.5.3 ST-QA-0002, Standard and Reagent Preparation
 - 17.5.4 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
 - 17.5.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.5.6 ST-QA-0016, IDL/MDL Determination
 - 17.5.7 ST-QA-0036, Non-conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 Modifications/Interpretations from reference method.
- 18.2 Modifications from both 7470A and 245.1.
 - 18.2.1 The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
 - 18.2.2 This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, “Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology.”
- 18.3 Modifications from Method 7470A
 - 18.3.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. Due to software limitations the lab is unable to flag and report blanks using the method criteria that analyte concentration in the blank must be less than the MDL. Blanks are evaluated to the MDL and flagged if they exceed that level. Re-preparation and re-analysis are required if the method blank is greater than RL and the sample is greater than the RL but less than ten times the RL.
- 18.4 Modifications from 245.1
 - 18.4.1 Method 245.1 Section 9.3 states concentrations should be reported as follows: Between 1 and 10 ug/L, one decimal; above 10 ug/L, to the nearest whole number. TestAmerica reports all Hg results under this SOP to two significant figures.

- 18.4.2 Due to software limitations the lab is unable to flag and report blanks using the method criteria that analyte concentration in the blank must be less than 2.2 times the MDL. Blanks are evaluated to the MDL and flagged if they exceed that level. Re-preparation and re-analysis are required if the method blank is greater than RL and the sample is greater than the RL but less than ten times the RL.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Updated section 10.0 by adding Low Level Check instructions.
- 19.2 Added section 11.2.4, pre of Low Level Calibration Check.
- 19.3 Added Low Level Checks to the Instrument Calibration list in section 11.5.4.
- 19.4 Rev. 9;
- 19.4.1 Updated post digestion spike section 9.7; removed MSA requirements
- 19.4.2 Removed section 18.3.2: redundant ICV criteria.
- 19.4.3 Added Method 1311 MSA requirements, section 18.5.
- 19.5 Rev. 10;
- 19.5.1 Updated the spike recovery acceptance criteria for methods EPA 245.1 and 7470A in section 13.4.
- 19.6 Rev. 11:
- 19.6.1 Updated instrument software used in section 6.0.
- 19.6.2 Updated permanganate solution needed for method blank and LCS in section 11.4.
- 19.7 Rev. 12:
- 19.7.1 Updated requirement for prepping all client and QC samples in section 11.4.2.1
- 19.8 Rev. 13:
- 19.8.1 Updated hot block temperate in section 6.0 and 11.4.
- 19.9 Rev. 14:
- 19.9.1 Removed references to QuantIMS and Clouseau through out.
- 19.9.2 Updated section 13 with corrective actions taken
- 19.9.3 Updated section 15
- 19.9.4 Removed trouble shooting and added to ST-QA-0024
- 19.10 Rev 15:
- 19.10.1 Updated 13.2.2
- 19.10.2 Added potential NCM for MS/MSD failure in section 13.6
- 19.10.3 Minor grammatical changes
- 19.10.4 Section 13.8 (PDS) removed “if PDS fails, a SD is performed” A SD is done with every batch.
- 19.11 Rev 16: (08/28/2015)
- 19.11.1 Removed references to “client requirement” checklists and memos, replaced with “comment section of LIMS” (updated for new LIMS) throughout SOP
- 19.11.2 Made grammatical corrections throughout SOP
- 19.11.3 Section 6: clarified container types used and removed old instrumentation/software that is no longer being used
- 19.11.4 Section 7: added a table to show how to make working standards, and clarified that the stock and intermediate standards had both a primary and secondary source
- 19.11.5 Section 10: clarified wording
- 19.11.6 Section 11: updated amounts spiked and whether the Cal or QC standard was used
- 19.11.7 Section 17: removed MSA from exceptions. We will do MSA as needed.

- 19.12 Rev 17: (08/31/2016)
 - 19.12.1 Updated software in section 6
 - 19.12.2 Clarified that the cal blank is also the ICB and CCB in section 7
 - 19.12.3 Removed “245.1 only” in section 7.12.2. Both methods require that standards are digested with samples
 - 19.12.4 Added acronyms to section 9
 - 19.12.5 Clarified that the LLC and CCV are taken from the same aliquots as the calibration standards in section 11
 - 19.12.6 Added modifications to section 18 (for both 7470A and 245.1) that MB are held to criteria of <RL, instead of method criteria.

**Title: PREPARATION AND ANALYSIS OF MERCURY IN SOLID
SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY
[SW-846 7471B]**

Approvals (Signature/Date):	
 Cory Buffington Metals Manager	11/16/16 Date
 Michael Ridenhower Health & Safety Manager / Coordinator	11/16/16 Date
 Kristen Ely Quality Assurance Manager	11/17/16 Date
 Elaine Wild Laboratory Director	11/16/16 Date

This SOP was previously identified as SOP No. ST-MT-0007 Rev. 15

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1.0 SCOPE AND APPLICATION

- 1.1 This procedure describes the preparation and analysis of mercury by Cold Vapor Atomic Absorption (CVAA) Spectroscopy using SW-846 Method 7471B.
- 1.2 CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.
- 1.3 Method 7471B is applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits and sludge-type materials. All matrices require sample preparation prior to analysis.
- 1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1 This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in hydrochloric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

- 4.1 Potassium permanganate, which is used to breakdown organic mercury compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.
- 4.2 Copper has also been reported to interfere with recovery of mercury; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.
- 4.3 Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.
- 4.4 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material ⁽¹⁾	Hazards	Exposure Limit ⁽²⁾	Signs and symptoms of exposure
Mercury (1,000 ppm in Reagent)	Oxidizer Corrosive Poison	0.1 mg/m ³ for Hg Compounds (Ceiling)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 mg/m ³ for Mn Compounds (Ceiling)	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision

Material ⁽¹⁾	Hazards	Exposure Limit ⁽²⁾	Signs and symptoms of exposure
			and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time weighted average			
STEL – Short term exposure limit			
Ceiling – At no time should this exposure limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Temperature controlled hot block (capable of maintaining a temperature of 95 ± 3 °C).
- 6.2 Teledyne Leeman Hydra II Mercury Analyzer
- 6.3 Hydra II AA software: Envoy version 2.003
- 6.4 100 mL certified class A digestion tubes
- 6.5 Argon gas supply, welding grade or equivalent.
- 6.6 Calibrated automatic pipettes
- 6.7 Volumetric flasks, class A
- 6.8 Top-loading balance, capable of reading ± 0.001 g
- 6.9 Thermometer (capable of reading 95 ± 3 °C)
- 6.10 Disposable sampling tubes
- 6.11 Glass Beads
- 6.12 Boiling Chips

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 DI Water (Type II or better), obtained from the Milli-Q unit
- 7.3 Nitric acid (HNO₃), concentrated, trace grade
- 7.4 Hydrochloric Acid (HCl), concentrated, trace grade
- 7.5 Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO₃.
- 7.6 Stannous chloride - Dissolve 100 g of stannous chloride into 1000 mL of 10% HCl.
- 7.7 Hydroxylamine sulfate/12% sodium hydroxide solution, a certified stock standard is purchased
NOTE: Hydroxylamine hydrochloride (12%) may be used in place of hydroxylamine sulfate.
(120 g → 1000 mL)

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- 7.8 Potassium permanganate, 5% solution (w/v) -certified stock reagent is purchased.
7.8.1 Alternately, dissolve 5 g of potassium permanganate for every 100 mL of DI water.
- 7.9 Stock (100 ppm) mercury standards (in HNO₃) are purchased as custom solutions.
7.9.1 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.
7.9.2 A primary (Cal) and secondary (QC) source is used
- 7.10 Intermediate mercury standard (0.1 ppm)
7.10.1 Add 50 mL of DI to a 100mL volumetric flask, then 2mL of concentrated HNO₃. Add 0.1 mL of the stock mercury standard and dilute to 100 mL with DI water.
7.10.2 The intermediate standard must be made daily
7.10.3 A primary (Cal) and secondary (QC) source is used.

7.11 Working Standards

Standard	Amount (mL)	Spike Solution	Final Volume (after digestion) (mL)	Final Concentration (ppb)
Blank	0	Cal Int	100	0.0
Cal 1/LLC	0.2	Cal Int	100	0.2
Cal 2	0.5	Cal Int	100	0.5
Cal 3	1.0	Cal Int	100	1.0
Cal 4/CCV	5.0	Cal Int	100	5.0
Cal 5	10.0	Cal Int	100	10.0
ICV	5.0	QC Int	100	5.0
LCS/MS/MSD	5.0	QC Int	100	5.0

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared.

- 7.11.1 The initial calibration verification (ICV) standard must be made from a different stock solution than that of the calibration standards.
- 7.11.2 All standards must be processed through the entire analytical procedure including sample preparation.
- 7.11.3 The working standards must be made daily.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 The sample holding time for mercury is 28 days from time of collection to the time of analysis.

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- 8.3 Soil samples do not require chemical preservation but must be stored at $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ until the time of analysis.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS) and Matrix Spike Duplicate (MSD) In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.1.4 At the instrument, a Serial dilution (SD) is performed with every batch.

9.2 Method Blank

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 For Soil analyses, the method blank is comprised of either glass beads or boiling chips.
- 9.2.4 See section 13 for acceptance criteria

9.3 Laboratory Control Sample

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.
- 9.3.3 For Soil analyses, the LCS is a commercially prepared purchased solid reference material containing mercury.
- 9.3.3.1 Alternately, the LCS may be comprised of glass beads or boiling chips fortified with mercury, if client requirements request or QC criteria dictate such.
- 9.3.4 See section 13 for acceptance criteria

9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.
- 9.4.3 MS/MSD samples must be performed with every sample batch and every LIMS batch.
- 9.4.3.1 If there is insufficient sample to perform a MS and/or MSD, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.
- 9.4.4 See section 13 for acceptance criteria

9.5 Sample Duplicate (DU)

- 9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.
- 9.5.2 Certain client project requirements may request a sample duplicate in lieu of a MSD. Please check in the comment section in LIMS.

9.6 Serial Dilution (SD)

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- 9.6.1 A sample digestate is diluted 1:5 and reanalyzed to assess the present of a matrix interference.
- 9.6.2 A serial dilution is performed with every analytical batch.
- 9.6.3 Agreement within 10% between the concentration for the undiluted sample and five times the concentration for the diluted sample indicates the absence of interferences.
- 9.7 **Post Digestion Spikes (PDS)**
 - 9.7.1 A known amount of mercury is added to the sample chosen for MS/MSD to bring the concentration of mercury to 2 to 5 times the original concentration. If the sample's mercury concentration is below the detection limit, spike at a concentration between the low and mid-level standard.
 - 9.7.2 At client's request, a post digestion spike is performed when MS/MSD fails.
 - 9.7.3 See section 13 for acceptance criteria
- 9.8 **Method of Standard Addition (MSA)**
 - 9.8.1 This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.
 - 9.8.2 Method SW-846 1311 requires that the Method Standard Addition (MSA) be used when matrix spike recovery is less than 50% and the measured sample results is within the range of 80-120% of the Toxicity Characteristics Limit.
 - 9.8.3 See Appendix II for more information.
- 9.9 **Procedural Variations/ Nonconformance and Corrective Action**
 - 9.9.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
 - 9.9.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Initial Calibration
 - 10.1.1 Calibration standards must be processed through the preparation procedure, section 11.
 - 10.1.2 Due to the differences in preparation protocols, separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.
 - 10.1.3 Batches that are prepped at different times must each have their own calibration standards
 - 10.1.4 Record the prep of the calibration standard in LIMS
 - 10.1.5 Calibration must be performed daily (every 24 hours) and each time the instrument is set up.
 - 10.1.6 Set up the instrument with the operating parameters recommended by the manufacturer.
 - 10.1.7 Allow the instrument to become thermally stable before beginning calibration.
 - 10.1.8 Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank.
 - 10.1.9 One standard must be at the reporting limit. . The other standards define the working range of the detector, with the highest level standard establishing the linear range of the instrument.
 - 10.1.10 Analyze standards in ascending order beginning with the blank.

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- 10.1.11 The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.
- 10.1.12 If the calibration curve does not meet method requirements, another calibration curve should be performed.
- 10.2 Initial Calibration Verification (ICV)
 - 10.2.1 An ICV is a second source verification of the calibration.
 - 10.2.2 An ICV must be performed with every calibration.
 - 10.2.3 If the ICV fails to meet criteria ($\pm 10\%$), the analysis is terminated, the problem corrected, the instrument recalibrated and the calibration re-verified.
 - 10.2.4 If it is suspected that the failure was attributed to a poor sample introduction to the instrument, the ICV may be rerun once, provided no samples have been analyzed after the failing ICV. If the second ICV is acceptable, analysis may continue.
- 10.3 Initial Calibration Blank (ICB)
 - 10.3.1 Analyze the initial continuing calibration blank (ICB) immediately following the ICV.
 - 10.3.2 The absolute value of the ICB result must be less than or equal to the reporting limit (RL).
 - 10.3.3 If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument if necessary
 - 10.3.4 Certain client programs may require more stringent ICB criteria, please see comments in LIMS.
- 10.4 Low Level Check (LLC) aka CRA (when requested by client)
 - 10.4.1 Can be the same source as the calibration
 - 10.4.2 An LLC is analyzed at the beginning and/or end of each analytical run when requested
 - 10.4.3 LLC must be spiked at RL.
 - 10.4.4 Must fall within 30% of the known value
 - 10.4.5 If the result is not within the control level, terminate the analysis, correct the problem and recalibrate.
 - 10.4.6 If it is suspected that the failure was attributed to a poor sample introduction to the instrument, the LLC may be rerun once, provided any samples analyzed after the failing LLC are also re-analyzed. If the second LLC is acceptable, analysis may continue.
- 10.5 Continuing Calibration Verification (CCV)
 - 10.5.1 A CCV is analyzed after every 10 samples and at the end of the analytical sequence run.
 - 10.5.2 The CCV must fall within 20% of the known true value.
 - 10.5.3 If the CCV does not meet QC criteria, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.
 - 10.5.4 If it is suspected that the failure was attributed to a poor sample introduction to the instrument, the CCV may be rerun once, provided any samples bracketing the failing CCV are also re-analyzed. If the second CCV is acceptable, analysis may continue.
- 10.6 Continuing Calibration Blank (CCB)
 - 10.6.1 Analyze a continuing calibration blank (CCB) immediately following the CCV.
 - 10.6.2 The absolute value of the CCB result must be less than or equal to the reporting limit (RL).
 - 10.6.3 If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument if necessary.

- 10.6.4 Certain client programs may require more stringent CCB criteria. (see comments in LIMS).

11.0 PROCEDURE

- 11.1 All calibration and calibration verification standards (ICV, ICB, LLC, CCV, CCB) are processed through the digestion procedure as well as the field samples.
- 11.2 Calibration Standard/Verification Standard Preparation
- 11.2.1 Pipette 0.0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the Cal Intermediate standard into a series of sample digestion vessel. Bring up all to 10 mL final volume with DI water.
- 11.2.2 Pipette 5.0 mL of QC Intermediate for ICV into a digestion vessel and bring up to a 10 mL final volume with DI water
- 11.2.3 Pipette 0.2 mL of Cal Intermediate for LLC into a digestion vessel and bring up to a 10mL final volume with DI water
- 11.2.4 Pipette 5.0 mL of Cal intermediate for CCV into a digestion vessel and bring up to a 10 mL final volume with DI water.
- 11.3 Sample and Batch QC preparation
- 11.3.1 Transfer 0.6 – 0.7 g of well-mixed sample (or 1 wipe) to a clean sample digestion vessel.
NOTE: Reduced sample size can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the sample to reagent ratio.
- 11.3.2 Spike MS/MSD with 5.0 mL of the QC intermediate.
- 11.3.3 For the method blank, transfer 0.6-0.7g of glass beads or boiling chips to a sample digestion vessel
- 11.3.3.1 For wipes, enter the initial volume of the MB as “1 wipe”.
- 11.3.4 For the LCS, transfer 0.6-0.7 g of the solid reference material containing mercury to a digestion vessel
- 11.3.4.1 For wipes, enter the initial volume of the LCS as the true value (eg 0.6g)
- 11.3.5 Alternate LCS (depending on client requirements): transfer 0.6-0.7 g of glass beads or boiling chips to a digestion vessel. Spike the LCS with 5mL of the QC intermediate.
- 11.4 Sample Digestion:
- 11.4.1 To each **standard** vessel: Add 5 mL of aqua regia.
- 11.4.2 To each **sample** vessel: Add 5 mL of reagent water and 5 mL of aqua regia.
- 11.4.3 Heat for 2 minutes in a hot block at 95 ± 3 °C
- 11.4.4 Cool.
- 11.4.5 Add 50 mL of DI water.
- 11.4.6 Add 15 mL of potassium permanganate solution, mix thoroughly
- 11.4.7 Let stand until purple color persists for 15 minutes, adding additional portions of permanganate solution if needed. If additional portions of permanganate solution are needed, an equivalent portion must also be added to all QC and client samples being prepped. Amount must be equal to that of the highest amount added to any sample.
- 11.4.8 Heat for 30 minutes in the hot block at 95 ± 3 °C
- 11.4.9 Cool.
- 11.4.10 Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.
- 11.4.11 Bring sample to a final volume of 100 mL with DI water.
- 11.5 Sample Analysis:

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- 11.5.1 The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.
- 11.5.2 Follow instructions provided by instrument manufacturer.
- 11.5.3 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.
- 11.5.4 Analytical sequence:
 - Instrument Initial Calibration (5 pts plus a blank)
 - ICV
 - ICB
 - LLC (only if required by client)
 - CCV
 - CCB
 - Maximum 10 samples
 - CCV
 - CCB
 - Repeat sequence of 10 samples between CCV/CCB pairs as required
 - CCV
 - CCB

NOTE: Samples include the method blank, LCS, LCSD, MS, MSD, DU, field samples and sample dilutions.

NOTE: Analytical sequence must close with a CCV/CCB pair.

NOTE: If the instrument stops during the sequence, the instrument may be restarted. When restarting the instrument, the run must begin with an acceptable CCV/CCB only if the run has been stopped for more than 2 hours.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 Sample results are reported with up to three significant figures in accordance with the significant figure policy.
- 12.3 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.
- 13.2 Method Blank (MB)
 - 13.2.1 Acceptance Criteria: No target analytes may be present in the method blank above the reporting limit.
 - 13.2.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be in comments in LIMS.
 - 13.2.3 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.3.1 Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).

13.3.1.2 Limits can be found in LIMS.

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).

13.4.2 MS/MSD Recovery criteria: 80% – 120%

13.4.3 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.3.1 MS/MSD Spike Recovery excursion: may not necessarily warrant corrective action other than narration

13.4.3.2 If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.

13.4.3.3 If the excursion is due to a physically evident matrix interference, the data is reported with an NCM.

13.4.3.4 In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with an NCM.

13.4.3.5 When the MS/MSD recoveries and the %RPD are outside criteria, the batch is re-prepped and re-analyzed for the affected analytes.

13.5 Post Digestion Spike (PDS)

13.5.1 A PDS is only done when requested by the client.

13.5.2 The method stipulates that a PDS be performed on the sample chosen for MS/MSD.

13.5.2.1 The acceptance criteria is 80% – 120%, with a spike concentration between 10–100 times the RL UNLESS, other project/program criteria is given.

13.6 Serial Dilution (SD)

13.6.1 The serial dilution results shall agree within $\pm 10\%$ of the undiluted sample results, if the undiluted sample results are greater than 10 times the reporting limit. There is no criteria for sample results less than 10 times the reporting limit.

13.6.2 Corrective Action: Serial dilution failure is documented in an NCM and the reported data is flagged. If multiple analytes fail the serial dilution test, the analyst may re-prepare and re-analyze the samples.

13.7 Sample result evaluation

13.7.1 Analyses must fall within the calibration range.

13.7.2 Dilutions

- 13.7.2.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
 - 13.7.2.1.1 An NCM will be written for any samples that are diluted.
- 13.7.3 Carryover
 - 13.7.3.1 When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
 - 13.7.3.2 If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.
 - 13.7.3.3 If a sample analyzed after a sample with high concentrations has positive results for the same analytes, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.8 Insufficient Sample

- 13.8.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative. The insufficient sample description is included in the Clouseau NCM within the type defining the excursion.

14.0 METHOD PERFORMANCE AND DEMONSTRATIONS OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by this Method
 - 16.2.1 Acidic sample waste
 - 16.2.1.1 All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A"

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16.2.1.2 Contaminated disposable glass or plastic materials utilized in this analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste and disposed of by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Revision 2, January 1998, Method 7471B (Mercury).
- 17.2 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision.
- 17.3 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (ST-HS-0002), current revisions.
- 17.4 Associated SOPs, current revisions:
 - 17.4.1 ST-QA-0002, Standard and Reagent Preparation
 - 17.4.2 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
 - 17.4.3 ST-QA-0016, IDL/MDL Determination
 - 17.4.4 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.4.5 ST-QA-0036, Non-conformance Memorandum (NCM) Process
 - 17.4.6 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.4.7 ST-IP-0004, Labware Preparation for Inorganic and Trace Metals Analysis

18.0 CLARIFICATIONS, MODIFICATIONS AND ADDITIONS TO REFERENCE METHOD

- 18.1 Modifications from 7471B
- 18.2 Stannous chloride is used in place of Stannous sulfate.
- 18.3 TestAmerica St. Louis uses less DI water in section 11 than the method suggests. The laboratory considers the volume of 15 mL not to be significant enough to render compensation/adjustment when diluting sample to final volume. The lab has adjusted the final volume of the standards and samples by 15 mL.
- 18.4 TestAmerica uses an ERA solid reference material for the LCS. Control limits supplied by ERA are used to determine acceptability of the LCS recovery.

19.0 CHANGES TO PREVIOUS REVISION

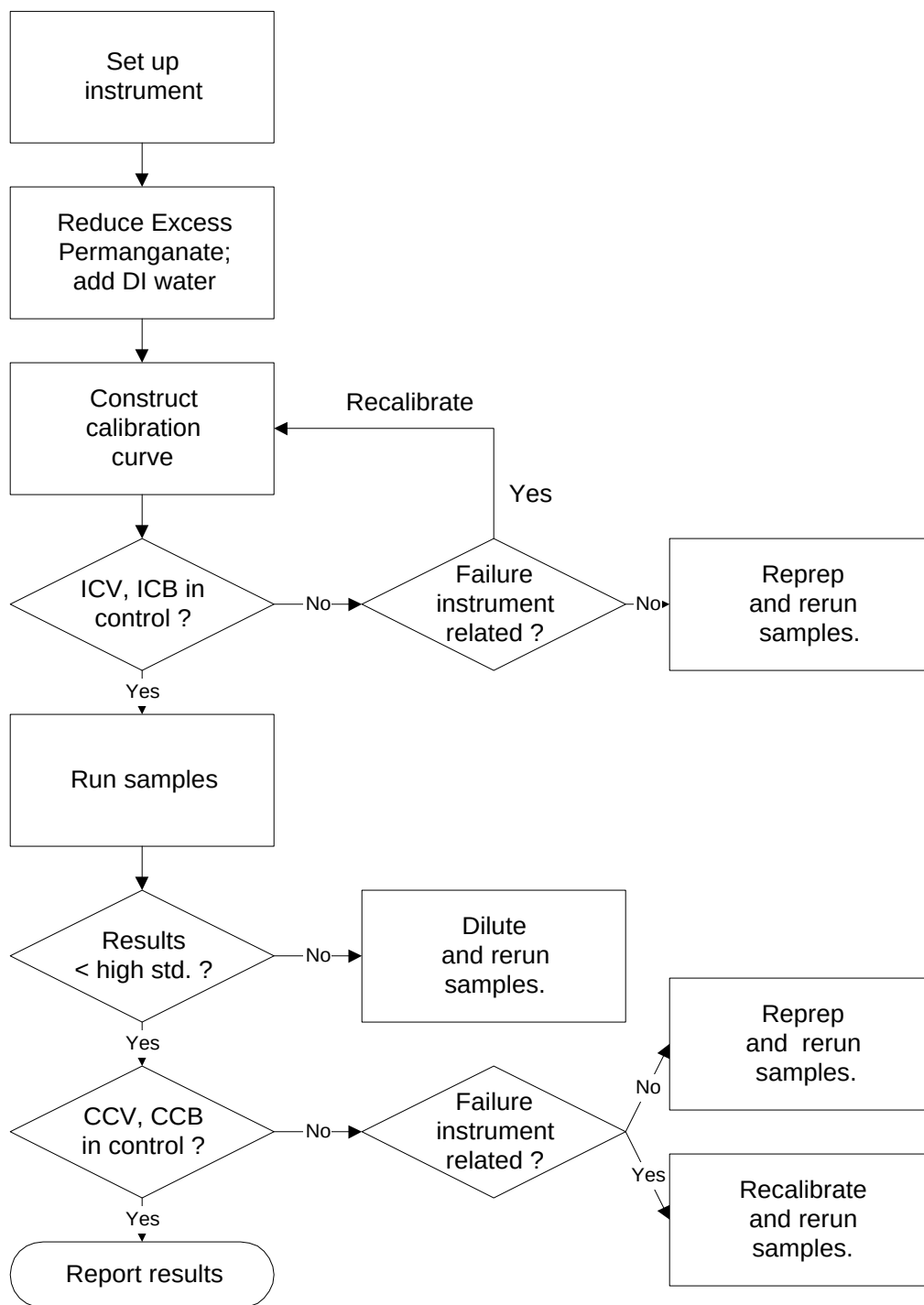
- 19.1 Removed sulfuric acid from section 5.0. It is no longer used in this method.
- 19.2 Updated section 10.0 by adding Low Level Check (LLC)
- 19.3 Added instructions on how to prepare an ICV, LLC and CCV in section 11.0
- 19.4 Added LLC, CCV and CCB to the list of analytical sequences in section 11.2.
- 19.5 Rev. 10;
 - 19.5.1 Updated post digestion spike section 9.7; removed serial dilution requirements.
 - 19.5.2 Updated MSA section 9.8; removed suggested criteria for MSA analysis.
 - 19.5.3 Added Method 1311 MSA requirements, section 18.4.
- 19.6 Rev. 11:
 - 19.6.1 Added instrument hardware and software to section 6.0.
 - 19.6.2 Added requirement to document preparation of calibration standard to section 10.1.
 - 19.6.3 Clarified instructions on addition of permanganate solution in section 11.4.

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- 19.7 Rev. 12:
 - 19.7.1 Updated requirement for prepping all client and QC samples in section 11.4.3.8.
- 19.8 Rev. 13:
 - 19.8.1 Removed references to QuantIMS and Clouseau
 - 19.8.2 Section 6, updated top-loading balance and DI water requirements
 - 19.8.3 Section 9, recovery criteria and updated post digestion spike recovery criteria
 - 19.8.4 Section 10, updated Low Level Check and Continuing Calibration Blank
 - 19.8.5 Section 11, added sample amount for CCV preparation
 - 19.8.6 Section 13 updated
 - 19.8.7 Section 15 updated
 - 19.8.8
- 19.9 Rev 14
 - 19.9.1 Added glass beads and boiling chips to section 6, added reference to boiling chips throughout SOP
 - 19.9.2 Formatting changes throughout, particularly section 7.9, 7.10, and 11
 - 19.9.3 Removed references to Client Requirement Sheet
 - 19.9.4 Added Hydra II to Equipment section.
 - 19.9.5 Removed exception to MSA in Section 18.
- 19.10 Rev 15 (11/16/2015)
 - 19.10.1 Formatting changes throughout, particularly section 10 and 11
 - 19.10.2 Updated section 6
 - 19.10.2.1 removed reference to old Hg equipment and software
 - 19.10.2.2 corrected type of sample and digestion tubes used
 - 19.10.3 Section 7: revised for clarification purposes, and to mirror SOP ST-MT-0005 (Hg water)
 - 19.10.4 Section 9.1: removed references to old LIMS system
 - 19.10.5 Section 10: revised for clarification of when new standards must be prepped
 - 19.10.6 Section 11:
 - 19.10.6.1 Clarified that standards must go through prep process
 - 19.10.6.2 Changed ICV, LCS, and MS/MSD spike to 5mL
 - 19.10.6.3 Changed CCV parent spike from QC to Cal
- 19.11 Rev 16 (11/16/2016)
 - 19.11.1 Section 6.3: corrected software version
 - 19.11.2 Section 7.10: clarified procedure to make intermediate
 - 19.11.3 Section 11.3: Added instructions for digesting wipes.

APPENDIX I – CVAA MERCURY ANALYSIS



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APPENDIX II – METHOD OF STANDARD ADDITION (MSA)

MSA is required if

- 1- Matrix spike recovery <50%, AND
- 2- Measured concentration is within 20% of the regulatory level

Regulatory Limits:

Arsenic	5mg/L
Barium	100mg/L
Cadmium	1 mg/L
Chromium	5 mg/L
Lead	5 mg/L
Mercury	0.2 mg/L
Selenium	1 mg/L
Silver	5 mg/L

How to run an MSA

- 1- Take 4 identical aliquots of test solution
- 2- Add increasing concentration of standard to 3 aliquots and add blank solution to 4th aliquot- all aliquots should be at same final volume
- 3- Perform analysis
- 4- Enter data into spreadsheet ORG-0034_TCLP_MSA
 - a. Uses unweighted least-squares linear regression curve fit
 - b. Calculates absolute value of x-intercept

Notes:

- 1- MSA spikes must come from the same analytical batch, analyzed on the same day.

The MSA curve must include the unspiked sample and 3 samples spiked with increasing concentrations of analytes

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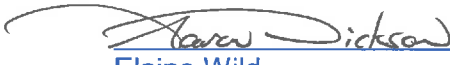
**Title: EXTRACTION AND CLEANUP OF ORGANIC COMPOUNDS FROM
WATERS AND SOILS**

Approvals (Signature/Date):

 9/20/16
Dennis Konopka
Extractable Dept. Manager

 9/20/16
Michael Ridenhower
Health & Safety Manager / Coordinator

 9/20/16
Marti Ward
Quality Assurance Manager

 9/20/16
Elaine Wild
Laboratory Director

This SOP was previously identified as SOP No. ST-OP-0002 Rev. 26

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1.0 SCOPE AND APPLICATION

- 1.1. This SOP describes procedures for extraction preparation of semivolatiles, pesticide, PCB, and hydrocarbon organic analytes in aqueous, TCLP leachate, and soil matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS).
- 1.2. This SOP is based on SW-846 Methods 3500C, 3510C, 3550C, 3600C, 3620C, 3650B, 3660B, AND 3665A.
- 1.3. Sample homogenization is not covered in this SOP. Refer to SOP ST-QA-0038. This SOP also handles subsampling and information on stick and rock removal for soils.
- 1.4. The clean up methods: GPC , florisil, sulfuric acid, sulfur removal are described.
- 1.5. The procedures are based on SW-846 and 600 series methodology and are acceptable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.
- 1.6. See individual analysis SOPs: ST-GC-0005, ST-GC-0013, ST-GC-0015, ST-GC-0016, and ST-MS-0001 for analytical requirements.

2.0 SUMMARY OF METHOD

- 2.1. Separatory Funnel Extraction
 - 2.1.1. A measured volume of sample, typically 1 liter, is adjusted, if necessary, to a specified pH and serially extracted with appropriate solvent using a separatory funnel.
 - 2.1.2. For an LVI extraction, a measured volume of sample, typically 250 mL is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a 500 mL separatory funnel.
- 2.2. Sonication Extraction
 - 2.2.1. A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent extracted three times using an ultrasonic horn.
- 2.3. Waste Dilution
 - 2.3.1 This method is used for materials that are soluble in an organic solvent.
- 2.4. Cleanup
 - 2.4.1. Procedures are presented for removing interferents from sample extracts, and for drying and concentrating of the extract to final volume for analysis.
- 2.5. Wipes
 - 2.5.1. This method is listed under the 3550 ext. but is used for sample matrices that are wipes, and not soils.], The autoshaker is used for extraction of wipes instead of sonication..

3.0 DEFINITIONS

- 3.1. See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2. Lower Volume Initiative (LVI): procedures which provide for the collection of reduced volume of water samples while maintaining standard and achievable reporting limits.

4.0 INTERFERENCES

- 4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.
- 4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5.0 SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2. Specific Safety Concerns or Requirements
- 5.2.1 The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and must be performed in a ventilated hood and safety glasses or goggles must be worn.
- 5.2.2 Nitrile gloves should be used when performing this procedure. Latex and vinyl gloves provide no significant protection against organic solvents.
- 5.2.3 During Kuderna-Danish (KD) concentration technique (if used), do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard. The KD apparatus has ground glass joints which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints. Broken glassware must be disposed of or repaired.
- 5.2.4 The addition of Sulfuric Acid or Potassium Permanganate to the sample extract has the potential to create an exothermic reaction. Both can cause severe burns.
- 5.2.5 Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection.
- 5.3 Primary Materials Used
- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limits (2)	Signs of Exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/m3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can

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	Carcinogen		occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Sodium Hydroxide	Corrosive Poison	2 ppm (TWA)	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methanol (Methyl Alcohol)	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Acetonitrile	Flammable Poison	40 ppm (TWA)	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Methylene chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

- 6.1. Labware is cleaned to remove any residual organics. Please refer to SOP ST-OP-0001 for detailed glassware cleaning procedures.
- 6.2. Separatory Funnel: 2 L; 500 mL
- 6.3. Balance:
 - 6.3.2 For Waters - greater than 1400 g capacity, accurate +/- 0.1 g
 - 6.3.3 For Soils – balance accurate to +/- 0.01 g

- 6.4. pH indicator paper, wide-range, peroxide test strips, chlorine test strips
- 6.5. Graduated cylinder: 1 liter. (other sizes may be used)
- 6.6. Solvent Dispenser Pump or 100 mL Graduated Cylinder
- 6.7. Boiling Chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).
- 6.8. Heating Mantle: Rheostat controlled
- 6.9. Beakers: 250, 400 & 1000 mL, graduated (Note: used as backup equipment)
- 6.10. Mason Jars
- 6.11. 250 mL and 500 mL chem. Bottles
- 6.12. Sonicator (at least 300 watts)
- 6.13. Sonicator horn, 3/4 inch
- 6.14. Kuderna-Danish (K-D) Apparatus: 500 mL, 250mL- precleaned
- 6.15. Concentrator Tip: 10 mL, attached to K-D with clips Class A
- 6.16. Snyder Column: Three ball macro
- 6.17. Water Bath: Heated, with concentric ring cover, capable of temperature control ($\pm 5^{\circ}\text{C}$) up to 95°C .
The bath must be used in a hood or with a solvent recovery system.
- 6.18. Vials: Glass, 2 mL, 4 mL, and 40 mL capacity with Teflon®-lined screw cap
- 6.19. Nitrogen Blowdown Apparatus
- 6.20. Syringes: 10 μl – 1000 μl
- 6.21. Phase Separation Paper (filter paper)
- 6.22. Poly Funnel: 75 X 75 mm
- 6.23. Transfer Pipettes
- 6.24. Muffle ovens
- 6.25. Drying Ovens

EQUIPMENT AND SUPPLIES FOR CLEANUP

Gel permeation chromatography system (OI Analytical Autoprep 2000 with OI Analytical 1096TB Autosampler). Bio Beads: (S-X3) -200-400 mesh, 70 gm (Bio-Rad Laboratories, Richmond, CA, Catalog 152-2750 or equivalent). Chromatographic column: 700 mm x 25 mm ID glass column. Flow is upward. Ultraviolet detector: Fixed wavelength (254 nm) and a semi-prep

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EQUIPMENT AND SUPPLIES FOR CLEANUP

flow-through cell.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 DI water, organic free
- 7.3 Sodium hydroxide (NaOH), Pellets: Reagent Grade.
 - 7.3.1 Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 1000 mL. Depending on purity of NaOH pellets, 37% KOH is needed.
- 7.4 Sulfuric acid (H₂SO₄), Concentrated: Reagent Grade.
 - 7.4.1 Sulfuric acid (1:1): Carefully add 500 mL of H₂SO₄ to 500 mL of reagent water. Mix well.
- 7.5 90:10 Hexane:Acetone
- 7.6 Sodium sulfate (Na₂SO₄), J.T. Baker Catalog No. 3375-07-5345200, Granular, Anhydrous (ST certified).
 - 7.6.1 Purify by heating at 400°C a minimum of four hours.
- 7.7 Sand
 - 7.7.1 Muffle for 2 hours at 400°C
- 7.8 Extraction/Exchange Solvents: Methanol, Methylene chloride, Hexane, , Acetone, and 1:1 Methylene chloride/Acetone mixed solvent, pesticide quality or equivalent
- 7.9 Nitrogen: reagent grade.
- 7.10 Supelco Superclean ENVI Florisil Cartridges – precertified [Other vendors may be used if they pass the lab certification criteria: < 5% trich remaining and 80 – 100% recovery of pesticide target analytes.]
- 7.11 Organic standards are purchased as certified solutions or prepared from neat.
 - 7.11.1 Refer to SOP ST-QA-0002, “Standards and Reagent Preparation” for the required storage conditions and shelf lives of the standards used in this procedure. All stock standards must be protected from light. Standard solutions must be replaced by the manufacturer’s expiration date or 6 months after opening the ampoule (or making the standard if from neat materials), whichever is shorter.
 - 7.11.2 Standards must be allowed to come to room temperature before use.
 - 7.11.3 Surrogate spiking standards are prepared as dilutions of the stock standards. See recipe in the LIMS Reagent program.
 - 7.11.3.1 The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Spiking standards are purchased or prepared as dilutions of the stock standards. See recipe in the LIMS Reagent program.
 - 7.11.3.2 Standard preparation information is included in Appendix 1.
- 7.12 5% Aqueous Potassium Permanganate – Reagent grade. This chemical is a powerful oxidizer and should be handled with care. Consult MSDS for more information if using for the first time.

- 7.13 GPC calibration solution - prepare or purchase a solution in methylene chloride that contains the following analytes in the concentrations listed below:

Analyte	mg/mL
Corn Oil	25.0
Bis (2-ethylhexyl) phthalate	1.0
Methoxychlor	0.2
Perylene	0.02
Sulfur	0.08

8.0 SAMPLE COLLECTION PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Water samples are unpreserved and stored at $4 \pm 2^{\circ}\text{C}$.
- 8.3 Soil samples are refrigerated at $4 \pm 2^{\circ}\text{C}$.
- 8.4 Holding Times
- 8.4.1 Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.
- 8.4.2 For TCLP leachates, the extraction is initiated within seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step. If the filtration step requires extended time, then this time counts as part of the seven day holding time.
- 8.4.3 Analysis of the extracts is completed within forty days of extraction.
- 8.4.4 Holding times do not apply to PCB method 8082A unless otherwise specified by a client or agency.. 608 Holding times for PCBs is one year from the sample date unless otherwise specified by a client or agency.

9.0 QUALITY CONTROL

9.1. Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 Batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS) and Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.1.4 A method blank and LCS may be shared across QC programs provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed, as required by clients, for each separate QC program.

9.2 Method Blank

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 Surrogates are spiked into the method blank at the same level as the samples.

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- 9.2.4 Aqueous Method Blanks use 1000 mL of DI water spiked with the surrogates. Solid method blanks use 30 g of sodium sulfate spiked with the surrogates.
 - 9.2.4.1 South Carolina requires the use of Ottawa sand for Method Blanks.
- 9.2.5 LVI Method blank uses 250 mL of DI water spiked with the surrogates.
- 9.2.6 TCLP method blanks use 100 mL of leachate fluid (200 mL for BNA) spiked with the surrogates. Use the leaching fluid type (1 or 2) that was used in leaching the samples. If both fluid types are used in a batch, a method blank with each fluid is prepared.

9.3 Laboratory Control Sample

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.
- 9.3.3 Aqueous LCS use 1000 mL of DI water fortified with the organic analytes of interest.
- 9.3.4 LVI LCS uses 250 mL of DI water fortified with the organic analytes of interest.
- 9.3.5 Solid LCS use 30 g of sodium sulfate fortified with the organic analytes of interest.
 - 9.3.5.1 South Carolina requires the use of Ottawa sand for the LCS.
- 9.3.6 TCLP LCS use 100 mL (or 200 mL for BNA) of leachate fluid. Use the leaching fluid type (1 or 2) that was used in leaching the samples. If both fluid types were used in a batch, a LCS with each fluid is prepared.

9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.
- 9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.
- 9.4.4 If there is insufficient sample to perform an MS/MSD, a duplicate LCS is analyzed. An NCM is written to document the insufficient volume and the utilization of an LCS to demonstrate precision.

9.5 Surrogates

- 9.5.1 Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.
- 9.5.2 Each applicable sample, blank, LCS and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.6 Procedural Variations/ Nonconformance and Corrective Action

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Balance calibration must be checked daily when used. Refer to SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipette".
- 10.2 Thermometers must be checked as prescribed in SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipette".

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- 10.3 If Class A concentrator tips are not available the Concentrator tip calibration is checked gravimetrically at the 1, 5 and 10 mL marks when new lots are received.
- 10.3.1 Place the empty tip into a suitable container to allow tip to remain upright, place on balance and tare.
- 10.3.2 Add DI water exactly to the 5 mL/10 mL mark and record weight. Criteria is $\pm 3\%$ (9.7 to 10.3 mL).
- 10.3.3 This is not required for Class A concentrator tips.
- 10.4 Sonicators are calibrated (tuned) each day of use.
CAUTION: Hearing protection must be worn when tuning the sonicators.
- 10.4.1 See work instruction ST-WI-0010 for tuning instructions for the different horns.
- 10.5 Florisil cartridge performance check – required only if pre-certified cartridges are not used)
- 10.5.1 If not certified every lot number of Florisil must be tested before use.
- 10.5.1.1 Check with the lab to ensure lot check was performed for the lot in use.
- 10.5.1.2 If check was not performed by another TestAmerica location
- 10.5.1.2.1 Add 0.5 ug/mL of 2,4,5-trichlorophenol solution and 0.5 mL of GC Standard Mix A (midpoint concentration) to 9 mL hexane.
- 10.5.1.2.2 Add the concentrate to a pre-conditioned Florisil cartridge and elute with 9 mL hexane/acetone [(90:10)(v/v)].
- 10.5.1.2.3 Rinse cartridge with 1.0 mL hexane two additional times.
- 10.5.1.2.4 Concentrate eluate to 1.0 mL final volume and transfer to vial.
- 10.5.1.2.4.1 Concentration may be done by any of the techniques given in section 11.
- 10.5.1.2.5 Analyze the solution by GC/EC.
- 10.5.1.2.5.1 The test sample must show 80 to 110% recovery of all pesticide analytes with <5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected.
- 10.5.1.2.5.2 The trichlorophenol standard has a lifetime of six months. The cartridge has a lifetime of one year or the manufacturer's expiration date, whichever is shorter.
- 10.5.1.2.5.3 Alternatively, this standard may be purchased as a stock solution.
- 10.6 See individual analysis SOPs: ST-GC-0005, ST-GC-0013, ST-GC-0015, ST-GC-0016, and ST-MS-0001 for instrument calibration requirements.

11.0 PROCEDURE

- 11.1 Aqueous Samples – Preparation (SW846 3500C)
- 11.1.1 Weigh full sample bottle, without the cap.
- 11.1.2 The normal sample volume is 1 liter.
- 11.1.3 Pour sample aliquot into a Separatory Funnel. Details available in section 11.2
- 11.1.4 For all analyses (except SIM PAH or TCLP BNA), add 0.5 mL surrogate solution (Table 3) to samples and QC.
- 11.1.5 Spike LCS and MS/MSD with spiking mix applicable to the requested analysis.
- 11.1.5.1 Document spiking volumes and standard numbers on the bench sheet.
- 11.1.5.2 Change the caps on the standards and return spiking solutions to the refrigerator as soon as possible.

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- 11.1.6 Take pH of sample
 - 11.1.6.1 For all tests record pH on extraction log. Initial pH must be between 5 and 9 for all tests except for TPHs.
 - 11.1.6.2 As necessary, adjust sample pH as indicated in Table 1.
 - 11.1.6.3 For Base/Neutral/Acids the typical order of extraction is as follows: reduce pH to < 2 with 1:1 H₂SO₄. Then, perform the extraction with 60 mL of methylene chloride. Repeat the extraction in acid twice more using 60 mL methylene chloride. Adjust pH to 12-14 with NaOH. Add 100 mLs of methylene chloride and perform extraction. Repeat the following last two base extractions with 50 mLs of methylene chloride. (NOTE: for SIM analysis, only the base portion of the extraction needs to be performed.)
 - 11.1.6.4 Check each sample with pH paper after each pH adjustment. Record adjusted pH.
 - 11.1.6.5 For method EPA 608, check for residual chlorine and record.
- 11.1.7 After the sample bottle is emptied, rinse the container with approximately 60 mL of the extraction solvent. Gently swirl the solvent in the container, rinsing the sides of the container thoroughly and add solvent rinse to the extraction vessel.
 - 11.1.7.1 If the entire sample volume is not used the container cannot be solvent rinsed. Organic materials adhering to the container walls will be absent in the extraction process. An NCM is written documenting that the container rinse could not be performed.
- 11.1.8 Weigh the empty sample bottle, without the cap.
 - 11.1.8.1 Volume is calculated by gravimetric determination assuming a sample density of 1. Samples that are not aqueous or are suspected to have a density greater than 1.2 will have aliquots taken for density analysis to correct volume for density.
- 11.1.9 Using a graduated cylinder, measure 100 mL of leachate for TCLP pesticides and PCBs , 200 mL of leachate for TCLP semivolatiles For TCLPs dilute to about 1 liter with DI water. For BNA, make acidic after dilution.

Refer to Table 4 for the LVI amounts. All steps are the same as a 3510C extraction, only the amounts differ: initial volume, final volume, spike/surrogate and solvent amounts. LVI is not to be used for South Carolina work. This state requires certification for this method that St. Louis does not currently hold.

11.2. Aqueous Water - Separatory Funnel Liquid/Liquid Extraction (SW846 3510C)

- 11.2.1. Weight full sample bottle, without the cap.
- 11.2.2. If a sample is known or suspected (based upon client knowledge, project scope, or site history) to have a high density (<1.2 g/mL, e.g. a brine or waste) or a low density (<0.98 g/mL, e.g. mixed solvent), the sample density will be measured and the volume determined arithmetically (sample mass divided by the density equals the volume).
- 11.2.3. Thoroughly mix the liquid sample in accordance with SOP ST-QA-0038. The Normal Sample liter is 1mL
 - 11.2.3.1. Other sample volumes may be used to obtain specific reporting limits, and reduce sample volumes. All samples are diluted to 1L with DI water even if reduced sample volume is used for dirty samples or matrix issues. See manager/supervisor for instructions.
- 11.2.4. Take the PH of the sample using a glass pipette and placing the sample on the strip. Do not put the strip directly into the sample. Record the ph.
- 11.2.5. Spike and surrogate directly into the client containers then proceed to Pour the sample aliquot into the sep funnel, rinse the containers with the extraction solvent, then weigh the empty bottles and dispose of them.
- 11.2.6. Adjust the ph of the sample if needed AFTER the spike and surrogate have occurred.
 - 11.2.6.1. As necessary, adjust sample PH as indicated in Table 1.

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- 11.2.6.2. 1:1 sulfuric:di water is used to make sample ph acidic and 10N sodium hydroxide is used to make sample ph base.
- 11.2.6.3. Make sure to record each adjustment (initial, first, second, etc).
- 11.2.7. For EPA 608 check for residual chlorines and record.
- 11.2.8. NOTE; for SIM analysis, only the base portion of the 8270 extraction needs to be performed.
- 11.2.9. Add 60 mL methylene chloride to the Separatory funnel..
- 11.2.10. Seal and shake or rotate the separatory funnel vigorously for 1 minute with periodic venting to release excess pressure.
- Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.**
- 11.2.11. Allow the organic layer to separate from the water phase until complete visible separation has been achieved.
- 11.2.11.1. If the emulsion interface between layers is more than one third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. If the emulsion cannot be broken (recovery of <80% of the methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in continuous liquid-liquid extraction.
- 11.2.11.1.1. The sample must be extracted as part of a valid 3510C batch.
- NOTE:** 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 40 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.
- 11.2.12. Line a funnel with filter paper or place glass wool in the funnel stem.
- 11.2.13. Add 10-20 g of anhydrous sodium sulfate to the funnel.
- 11.2.14. Place the prepared funnel on a pre-rinsed K-D flask or amber jar.
- 11.2.14.1. Condition each sodium sulfate filled funnel with methylene chloride before each drain and rinse with methylene chloride after each drain.
- 11.2.15. Drain solvent extract from the separatory funnel (Methylene chloride is the bottom layer) through the prepared filtration funnel into a clean glass container or K-D flask.
- 11.2.16. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.
- 11.2.16.1. If concentration does not begin immediately, place extract in labeled amber jars and secure the lids.
- 11.2.17. Repeat the extraction steps 11.3.5-11.3.10 two more times with a fresh 60 mL of methylene chloride. Use the same funnel and sodium sulfate for all three extractions.
- 11.2.18. Combine the three solvent extracts in the K-D flask or labeled amber jar.
- 11.2.18.1. For **Semivolatiles analysis**, a second extraction at a basic pH is required (see Table 1).
- 11.2.18.2. Adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimal amount of 10 N NaOH.
- 11.2.18.3. Measure with pH paper and record the adjusted pH on the bench sheet.
- 11.2.18.4. Serially extract with three portions of methylene chloride for semi-volatiles using 100 mL, 50 mL and 50 mL and rinsing before and after each drain with the extraction solvent.
- 11.2.19. Dispose of solvent and water remaining in the extractor in the appropriate waste container.
- 11.2.20. Proceed to Section 11.6 for concentration.

11.3. Solids and Waste Sonication Extraction (SW846 3550C)

- 11.3.1. Decant and discard any water layer on a sediment/soil sample.
 - 11.3.1.1. Document if a water layer was discarded.
- 11.3.2. Homogenize the sample by mixing thoroughly. See SOP ST-QA-0038 for details.
- 11.3.3. Weigh approximately 30 g of sample into a 250 or 400 mL beaker or Mason Jar.
- 11.3.4. Record the weight to the nearest 0.01g in the appropriate column on the bench sheet.
- 11.3.5. Use approximately 30 g of sodium sulfate for the method blank and the LCS.
- 11.3.6. Spike samples and QC with surrogate.
- 11.3.7. Spike LCS and MS/MSD with spiking mix applicable to the requested analysis. (spike and surrogate do not have to be done in any special order- spike may come first).
 - 11.3.7.1. Document spiking volumes and standard numbers on the bench sheet.
 - 11.3.7.2. Return spiking solutions to the refrigerator as soon as possible.
- 11.3.8. Mix sample with a spatula adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)
- 11.3.9. Add a minimum of 100 mL of solvent to the mason jar.

Semivolatile GC/MS**1:1 Methylene Chloride / Acetone****Organochlorine Pesticides, PCBs****1:1 Methylene Chloride / Acetone****TPH Hydrocarbons by FID (DRO, etc.)****Methylene Chloride**

- 11.3.10. Place the bottom surface of the disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.
- 11.3.11. Set sonicator between 7 and 8 (to achieve the required 300 watt power level) with mode switch on pulse, and percent- duty cycle knob set at 50% and record the sonicator ID.
- 11.3.12. Sonicate for 3 minutes, making sure the entire sample is agitated.
- 11.3.13. Line a funnel with filter paper.
- 11.3.14. Add 10-20 g of anhydrous sodium sulfate to the funnel. Condition funnel with methylene chloride before each drain.
- 11.3.15. Place the prepared funnel on an amber bottle or K-D flask.
Decant and filter extract through the prepared funnel into the amber bottle or K-D flask,.
If concentration is not to begin immediately, place extract in labeled amber jars and secure the lids.
- 11.3.16. Repeat the extraction steps 11.3.9-11.3.16 two more times with fresh a 100 mL amount of solvent each time. Use the same funnel and sodium sulfate for all three extractions. Decant extraction solvent after each sonication, combining all three extractions.
 - 11.3.16.1. Dump remaining soil from mason jar on top of sodium sulfate filled funnel, rinsing the jar with methylene chloride into KD flask or amber jar.
- 11.3.17. Rinse funnel with an additional 10 – 20 mL of the methylene chloride.
- 11.3.18. Proceed to Section 11.5 for concentration.
- 11.3.19. Wipes
 - 11.3.19.1. Wipes are done when a client sends in a wipe matrix. Sample is spikes and surrogates added to the client's container, and then enough extraction solvent is added to fully submerge the wipe. The extraction solvent for wipes is what is used for the final solvent for the test it is in for. No solvent exchanges occur.

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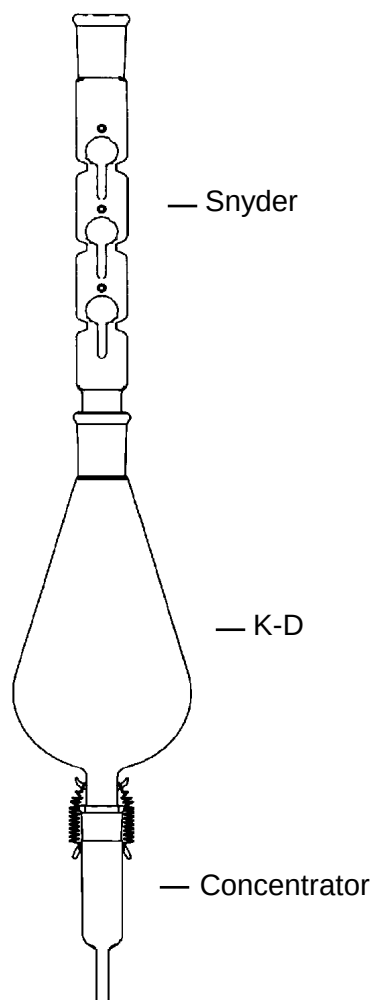
- 11.3.19.2. QC for wipes contain a clean wipe placed in a container like that used for the sample and taken through the entire process. If the client sends in their own QC a note must be made incase contamination may have occurred outside the lab when the QC was prepped.
- 11.3.19.3. Samples are then placed on a shaker table and shaken for a minimum of 2 hours. They are then poured up through a filter lined funnel with a small amount of sodium sulfate in it. The funnel is pre-wet with the final solvent before and after the draining. The client container is also rinsed into the funnel.
- 11.3.19.4. Samples are then placed on the hot bath and taken through the normal process of the test they are in for. No synder columns need to be used because no solvent exchanges happen for wipes since they are already in their final solvents.

11.4. Waste Dilution (SW846 3580)

- 11.4.1. This method is used for non-aqueous liquid or solid materials (e.g. oils, organic product) that are soluble in an organic solvent or samples known to contain high concentrations of target analytes.
 - 11.4.1.1. Waste dilutions are reported in ug/L for liquids and ug/kg for soils.
 - 11.4.1.2. For liquids, sample density must be performed to reflect volume of sample.
 - 11.4.1.2.1. Aliquot 10 mL of sample and deliver aliquot to Wet Chemistry Supervisor or designee for density determination or NCM that the density is based on 1mL determination of 1mL=xg.
- 11.4.2. Pre-weigh empty Class A concentrator tip.
- 11.4.3. Add 1g of sample to the tip. Record the weight.
- 11.4.4. Use 1g of mineral oil for Method Blank and LCS.
- 11.4.5. For Semivolatiles, add 2 mL spike solution to QC samples and 1 mL surrogate solution (Table 3) for all samples. For all other analyses, add 0.5 mL of surrogate solution (Table 3) to samples and 0.5 mL of spike solution to QC samples.
- 11.4.6. Bring final volume to 10 mL with appropriate solvent.
 - 11.4.6.1. Methylene Chloride for GC/MS Semivolatiles, TPH and Phenol analysis
 - 11.4.6.2. Hexane for Pesticide and PCB analysis.
- 11.4.7. Mix thoroughly approximately 1 minute.
- 11.4.8. 11.5.8 Proceed to cleanup if method specified.
- 11.4.9. 11.5.9 Vial samples.

11.5. Initial Concentration

- 11.5.1. Different final extract volumes are required, depending on analysis, matrix and cleanup. Refer to Table 2 for the appropriate final volumes and concentrations.
- 11.5.2. Kuderna-Danish (KD) Method:
 - 11.5.2.1. Assemble a KD concentrator by attaching a 10 mL Class A concentrator tip to the 500 mL KD flask.
 - 11.5.2.2. Transfer the sample to the KD flask.
 - 11.5.2.3. Rinse extract container with methylene chloride and add rinse to KD flask.
 - 11.5.2.4. Add one or two boiling chips to KD flask.
 - 11.5.2.5. Attach a three ball Snyder Column.
 - 11.5.2.5.1. Ensure that the balls are not stuck and that the column is working properly.



11.5.2.6. Set water bath at 85 - 95 °C depending on the analysis.

11.5.2.6.1. For Semivolatiles, Phenols, PAHs, and TPH, set temperature at approximately 90°C.

11.5.2.6.2. For Pesticides and PCBs, set temperature at approximately 95°C.

11.5.2.7. Record water bath temperature on the extraction log.

11.5.2.8. Place the KD apparatus in water bath so that the tip of the concentrator tip is submerged. The water level should not reach the joint between the concentrator and the KD flask.

11.5.2.8.1. The balls should actively chatter but the chambers should not flood.

11.5.2.9. Semivolatiles, Phenols and PAH Initial Concentration

11.5.2.9.1. Concentrate to 10 mL.

11.5.2.9.2. Remove extract from water bath.

11.5.2.9.3. Let cool and remove the concentrator tip from the KD flask.

11.5.2.10. Pesticide and PCB Initial Concentration

11.5.2.10.1. Concentrate to 5-10 mL.

11.5.2.10.1.1. If the KD flask is removed from the water bath prior to solvent exchange, a new boiling chip must be added to the flask to prevent “bumping” of the solvent.

11.5.2.10.2. Add approximately 60 mL hexane to the top of the Snyder Column, remove column after solvent exchange.

11.5.2.10.3. Return extract to water bath.

11.5.2.10.4. Concentrate to 5-8 mL.

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- 11.5.2.10.5. Remove extract from water bath.
- 11.5.2.10.6. Let cool and remove the concentrator tip from the KD flask.
- 11.5.3. Concentrating extracts to dryness can cause the loss of analytes.
 - 11.5.3.1. Remove the KD apparatus from the water bath and allow it to cool for a minimum of 10 minutes. The level of the extract should be below the level of the tip joint. If the level of the extract is above the level of the concentrator tip joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes. Remove the Snyder column,, rinse the flask and it's lower joints into the concentrator tube with 0.2 mL of methylene chloride or the exchange solvent.
 - 11.5.3.2. If necessary, dilute the extract to the desired final volume in the concentrator tip.
 - 11.5.3.3. If the desired final volume is less than the remaining extract, nitrogen evaporation (see next section) may be used to further concentrate the sample to 5 or 10 mL.
 - 11.5.3.4. Transfer the extract to a vial for refrigerated storage and document the final volume.
- 11.6. Final Concentration
 - 11.6.1. Nitrogen Evaporators are used for the final concentration of extract.
 - 11.6.2. Nitrogen Evaporation Concentration Method
 - 11.6.2.1. Semivolatiles, Pesticides, PCB, TPH, Phenol Final Concentration
 - 11.6.2.1.1. Place the pre-rinsed concentrator tip in the rack of the nitrogen evaporation apparatus.
 - 11.6.2.1.2. Lower the nitrogen tip to approximately ½" above the surface of the solvent.
 - 11.6.2.1.3. Turn on the nitrogen gas and observe the solvent in the concentrator tip.
 - 11.6.2.1.4. The nitrogen flow should be a gentle stream forming a slight depression on the surface of the solvent and should not create splattering of the extract.
 - 11.6.2.1.5. Gas flow can be adjusted using the flow control knobs located at the top of each gas tip.
 - 11.6.2.1.6. Place the concentrator tip in a warm water bath at approximately 35°C.
 - 11.6.2.1.6.1. If during the course of the evaporation sample residue appears to be adhering to the sides of the tip, rinse the sides of the evaporation tip with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1 mL.
 - 11.6.2.1.7. Concentrating extracts to dryness can cause analytes to be lost.
 - 11.6.2.1.8. Concentrate the solvent slightly below the required final volume and then draw the extract into a syringe. (keep in mind the boiling chip with disperse the volume in the tip)
 - 11.6.2.1.9. Rinse the evaporation tip with a small amount of solvent and draw additional solvent into the syringe to make up the required final volume.
 - 11.6.2.1.10. transfer the extract to a vial for storage and document the final volume.
- 11.7. Cleanup Techniques [SW846 3620C(Florist Cleanup), SW846 3660B (Sulfur Cleanup)]
 - 11.7.1. The following techniques may be used to remove interferences that may cause column deterioration and/ or loss of detector sensitivity.
 - 11.7.2. Cleanup procedures may be done in combination. If done in combination, they must be

performed in the order listed.

11.7.2.1. Florisil column cleanup is particularly useful for cleanup of pesticides analysis and is applied to these samples. It separates compounds with a different polarity from the target analytes.

11.7.2.2. Sulfur cleanup is generally applied to samples for analysis by ECD since the detector responds strongly to sulfur. It is performed after Florisil cleanup. Sulfur cleanup is performed by the analyst. See SOP: ST-GC-0015 for instruction.

11.7.2.3. Sulfuric acid cleanup is applied to samples requiring analysis for Polychlorinated Biphenyls (PCBs) only. Most organic matter is destroyed by sulfuric acid.

11.7.2.4. A copper cleanup or potassium permanganate cleanup may be employed by the analyst if the above cleanups do not work. This is documented in an NCM.

11.7.3. Florisil Cartridge Cleanup

11.7.3.1. Florisil cleanup is used for all soil pesticides extracts and may be applied to Pesticide water extract or other method extracts, at the discretion of the Manager/Supervisor or request of the client. See Client Requirement sheet.

11.7.3.1.1. If semivolatile, TPH or Phenol extracts require Florisil Clean up, the extract must be exchanged into Hexane. Consult Manager/Supervisor for instruction.

11.7.3.2. Place one Florisil cartridge into a vacuum manifold for each sample and associated QC extract.

11.7.3.3. Pre-elute each cartridge with 5 mL of hexane/acetone (90:10).

11.7.3.4. Using a pipette bulb, apply pressure to each cartridge so that the flow is a constant, continual drip. Do not allow the cartridges to go dry.

11.8.3.4.1 Stop applying pressure to the cartridge and remove the manifold top, before the cartridge goes dry.

11.7.3.5. Label 10 mL concentrator tips or comparable vessel (disposable vial) and place into the manifold. Tips and vials are conditioned with hexane.

11.7.3.6. Replace the manifold top.

11.7.3.7. Make sure that the solvent line from each cartridge is placed inside the appropriate tip.

11.7.3.8. Add 1.0 mL of the extract to the appropriate Florisil cartridge and let the sample drip through the column.

11.7.3.9. Before the column goes dry add another 6-8 mLs of 90:10 hexane/acetone to the column. Let the remaining solvent flow through the column. When the column is done dripping the cartridge can be removed and the port is then rinsed to ensure all the liquid has fully drained.

11.7.3.10. Collect into the labeled vessel held in the rack inside the vacuum manifold.

11.7.3.11. Concentrate the extract to 1 mL using the Nitrogen Evaporator See 11.6.2

11.7.4. Sulfuric Acid Cleanup

11.7.4.1. All PCB (only) extracts are taken through the sulfuric acid cleanup procedure.

11.7.4.2. Use an equal volume of concentrated sulfuric acid to sample extract volume

11.7.4.2.1. Add concentrated sulfuric acid to empty, labeled vial.

11.7.4.2.2. Carefully add the sample extract to the vial and cap.

11.7.4.3. QC extracts are also taken through this clean up procedure.

11.7.4.4. There must be no water present in the extract or the reaction may shatter the sample container. A face shield, splash guard, or hood sash must be used when performing acid clean-up.

11.7.4.5. Shake or vortex for about thirty seconds and allow to settle.

11.7.4.5.1. If extract will not settle, centrifuge.

11.7.4.6. Remove the sample extract (top layer) from the sulfuric acid using a pipette and transfer to a clean, labeled vial being careful not to transfer the sulfuric acid (bottom layer).

11.7.4.6.1. It is not necessary to remove all the extract since the final volume has already been determined.

- 11.7.4.6.2. Transfer small amounts of sulfuric acid along with the extract will destroy the chromatographic column.
- 11.7.4.7. If the hexane layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 3 acid cleanups. Document it if this is required.
- 11.7.4.8. If extract color persists after completion of the sulfuric acid cleanup, a potassium permanganate cleanup *may* be employed if deemed necessary by the analyst or requested by the client. Document it if this procedure is performed.
- 11.7.4.9. Slowly add 5 mL of 5 percent aqueous potassium permanganate solution to the hexane fraction. Caution: Permanganate is a powerful oxidizer and a violent reaction, while uncommon, may occur
- 11.7.4.10. Seal the vial tightly and shake or vortex for 30 second
- 11.7.4.10.1. If extract does not form two distinct layers within a few minutes, centrifuge.
- 11.8 Gel Permeation Chromatography - A generally applicable technique which can be used to prepare extracts for analysis. It is capable of separating high molecular weight material from the sample analytes, and so is particularly useful if tissue or vegetable matter is part of the sample, and for many soil samples.
- 11.8.1 Gel Permeation Chromatography (GPC)
- Note: GPC system (OI Analytical Autoprep 2000 with OIA 1096TB Autosampler)
- 11.8.1.1 GPC Column Preparation
- 11.8.1.2 Weigh out 70 g of Bio Beads (SX-3) into a 400-mL beaker.
- 11.8.1.3 Add approximately 300 mL of methylene chloride and stir gently.
- 11.8.1.4 Cover with aluminum foil and allow the beads to swell for a minimum of 12 hours. Maintain enough solvent to sufficiently cover the beads at all times.
- 11.8.1.5 Position and tighten the outlet bed support (top) plunger assembly in the tube by inserting the plunger and turning it clockwise until snug. Install the plunger near the column end but no closer than 5 cm (measured from the gel packing to the collar).
- 11.8.1.6 Turn the column upside down from its normal position with the open end up. Place the tubing from the top plunger assembly into a waste beaker below the column.
- 11.8.1.7 Swirl the bead/solvent slurry to get a homogeneous mixture and pour the mixture into the open end of the column. Transfer as much as possible with one continuous pour trying to minimize bubble formation. Pour enough to fill the column. Wait for the excess solvent to drain out before pouring in the rest. Add additional methylene chloride to transfer the remaining beads and to rinse the beaker and the sides of the column. If the top of the gel begins to look dry, add more methylene chloride to rewet the beads.
- 11.8.1.8 Wipe any remaining beads and solvent from the inner walls of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.
- CAUTION:** Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.
- 11.8.1.9 Push the plunger until it meets the gel, then compress the column bed about 4 cm.
- 11.8.1.10 Connect the column inlet to the solvent reservoir and place the column outlet tube in a waste container. Pump methylene chloride through the column into a glass beaker at a rate of 5 mL/min. for one hour. Inspect the glass beaker to ensure that gel beads do not escape from the column.

- 11.8.1.11 If observing beads in the beaker, continue pumping for several more minutes and check again for bead leakage. If gel beads are still present in the column effluent after 30 minutes, then disassemble, clean, inspect, and repack the column.
- 11.8.1.12 Closely monitor the column pressure. High pressure occurs occasionally while rewetting the gel, especially for the 100% methylene chloride column. If pressure continues to rise above 15 psi, reverse the flow direction through the column by exchanging the input and output column lines at the GPC column bypass ports until pressure begins to fall. Replace the connections in their original configuration. The pressure should begin to decline to between 7 and 10 psi as the gel uniformly saturates and swells. If pressure remains high, check for any restrictions or blockages.
- 11.8.1.13 After washing the column for at least one hour, connect the column outlet tube to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. Placing a restrictor (made from a piece of capillary tubing of 1/16"OD x 10/1000"ID x 2") in the outlet tube from the UV detector will prevent bubble formation which causes a noisy UV baseline. The restrictor will not effect the flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 6-10 psi back-pressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.
- 11.8.1.14 When the GPC column is not to be used for several days, connect the column inlet and outlet lines to prevent column drying and/or channeling. If channeling occurs, the gel must be removed from the column, re-swelled, and re-poured as described above. If drying occurs, pump methylene chloride through the column until the observed column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify that retention volumes have not changed.

11.8.2 Initial Calibration of the GPC Column

- 11.8.2.1 Before use, the GPC must be calibrated based on monitoring the elution of standards with a UV detector connected to the GPC column.
- 11.8.2.2 Pump solvent through the GPC column for 2 hours. Verify that the flow rate is 4.5-5.5 mL/min. Corrective action must be taken if the flow rate is outside this range. Record the column pressure (should be 6-10 psi) and room temperature (22°C is ideal).

Note: Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared.

- 11.8.2.3 Inject the calibration solution, for which the component concentrations can be found in section 7.1.1, and retain a UV trace that meets the following requirements (See resolution calculation in section 11.8.7.):

Peaks must be observed and should be symmetrical for all compounds in the calibration solution.

Corn oil and phthalate peaks must exhibit >85% resolution.

Phthalate and methoxychlor peaks must exhibit >85% resolution.

Methoxychlor and perylene peaks must exhibit >85% resolution.

Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

- 11.8.2.4 A UV trace that does not meet the criteria in paragraph 11.8.3.2 indicates the need for system maintenance and/or the need for a new column.

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11.8.2.5 Determine appropriate dump and collect cycles by following calculations 11.8.7.2 and 11.8.7.3, respectively.

11.8.2.6 The calibrated GPC program for pesticides/PCB should dump >85% of the phthalate and should collect >95% of the methoxychlor and perylene. Use a wash time of 10 minutes.

11.8.2.7 For semivolatile extracts, initiate a column eluate collection just before the elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the elution of perylene. Stop collection before sulfur elutes. Use a wash time of 10 minutes after the elution of sulfur.

11.8.2.8 Reinject the calibration solution after appropriate dump and collect cycles have been set.

11.8.2.9 Measure and record the volume of collected GPC eluate in a graduated cylinder.

11.8.2.10 The retention times for both bis(2-ethylhexyl) phthalate and perylene must not vary more than +/- 5% between calibrations.

11.8.3 GPC calibration check

11.8.3.1 Check the calibration of the GPC immediately after the initial calibration and at least every 7 days thereafter, while the column is in use.

11.8.3.2 Inject the calibration solution, and obtain a UV trace. If the retention times of bis(2-ethylhexyl)phthalate or perylene have changed by more than $\pm 5\%$ use this run as the start of a new initial calibration. Otherwise, proceed with the recovery check. Excessive retention time shifts may be caused by poor laboratory temperature control or system leaks, an unstabilized column, or high laboratory temperature causing outgassing of methylene chloride. Pump methylene chloride through the system and check the retention times each day until stabilized.

11.8.4 GPC Recovery Check for Pesticides/ PCBs

11.8.4.1 The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Two recovery check solutions are used. The first mixture is prepared by diluting 1.0 mL of the pesticide matrix spiking solution (Table 6) to 10 mL in methylene chloride. The second mixture is prepared by diluting 1 mL of the PCB only matrix spiking solution (Table 6) to 10 mL with methylene chloride.

11.8.4.2 Load the pesticide matrix spike mixture, the PCB mixture, and a methylene chloride blank onto the GPC using the GC dump and collect values.

Note: If the analysis is for PCBs only, then the pesticide recovery check is not necessary.

11.8.4.3 After collecting the GPC calibration check fraction, concentrate, solvent exchanging to hexane. Adjust the final volume to 5.0 mL, and analyze by GC/EC. Refer to concentration, section 11.5.

11.8.4.4 The methylene chloride blank may not exceed more than one half the reporting limit of any analyte. And if the recovery of each of the single component analytes

is 80-110% and if the Aroclor pattern is the same as previously run standards, then the analyst may use the column. If the above criteria are not met, there may be a need for system maintenance.

11.8.5 GPC Recovery Check for Semivolatiles

11.8.5.1 The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Dilute 1.0 mL of the semivolatiles matrix spiking solution to 10 mL in methylene chloride.

11.8.5.2 Load the matrix spike mixture and a methylene chloride blank onto the GPC using the semivolatiles dump and collect values.

11.8.5.3 After collecting the GPC recovery check fraction, concentrate to 0.5 mL, and analyze by GC/MS. Refer to the concentration section 11.5.

11.8.5.4 Recovery of the matrix spike analytes should be at least 60%. The blank should not contain any analytes at or above the reporting limit. If these conditions are met the column may be used for sample analysis. Otherwise correct the contamination problem, or extend the collect time to improve recovery of target analytes.

11.8.6 Sample Extract Cleanup

11.8.6.1 Reduce the sample extract volume to 1-2 mL, then adjust to 10 mL with methylene chloride prior to cleanup. This reduces the amount of acetone in the extract.

11.8.6.2 Start the pump and let the flow stabilize for 2 hours. The solvent flow rate should be 4.5-5.5 mL/min. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 22°C. The normal backpressure is 6-10 psi.

11.8.6.3 In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of a 1:1 glycerol:water solution (by visual comparison) must be diluted and loaded into several loops.

11.8.6.4 Samples being loaded onto the GPC should be filtered with a 5 micron (or less) filter disk. Attach a filter to a 10 mL Luerlok syringe and filter the 10 mL sample extract into the sample tube.

11.8.6.5 Load the filtered samples into the proper sample tubes and place on the GPC.

11.8.6.6 Set the collect, dump, and wash times determined by the calibration procedure.

11.8.6.7 Switch to the run mode and start the automated sequence. Process each sample using the collect and dump cycle times established by the calibration procedure.

11.8.6.8 Collect each sample in a suitable glass container. Monitor sample volumes collected.

11.8.6.9 Any samples that were loaded into 2 or more positions must be recombined.

11.8.6.10 Concentrate semivolatile sample extracts to 0.5 mL in methylene chloride.

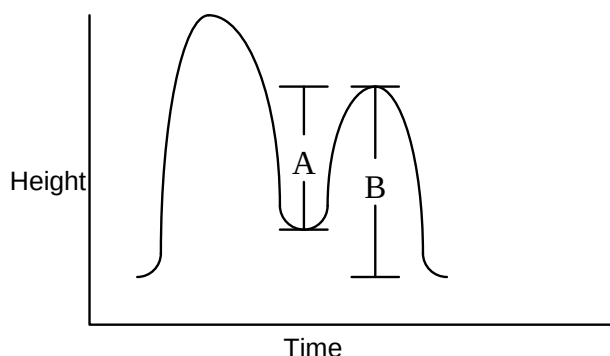
11.8.6.11 Solvent exchange the pesticide/PCB sample extracts into hexane and concentrate to 5.0 mL.

11.8.7 Calculations

11.8.7.1 Resolution

To calculate the resolution between two peaks on a chromatograph, divide the depth of the valley between the peaks by the peak height of the smaller peak being resolved and multiply by 100.

Resolution Calculation



$$\% \text{ Resolution} = \frac{A}{B} \times 100$$

Where: A = depth of valley to height of smaller peak

B = peak height of smaller peak

11.8.7.2 Dump Time

11.8.7.2.1 Mark on the chromatograph the injection point and the point where collection is to begin. Measure the distance from the injection point to the point of collection. Divide the distance by the chart speed.

$$\text{Dump time (min)} = \frac{\text{Distance (cm) from injection to collection start}}{\text{Chart speed (cm / min)}}$$

11.8.7.3 Collection Time

$$\text{Collection time (min)} = \frac{\text{Distance (cm) between collection start and stop}}{\text{Chart speed (cm / min)}}$$

11.9 Deliver extract and extraction log paperwork to the analyst.

11.10 See individual analysis SOPs: ST-GC-0005, ST-GC-0013, ST-GC-0015, ST-GC-0016, ST-LC-0001, and ST-MS-0001 for extract analysis.

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11.11 If not immediately analyzed, extracts are stored in designated refrigerators.

12 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 See individual analysis SOPs: ST-GC-0005, ST-GC-0013, ST-GC-0015, ST-GC-0016, and ST-MS-0001 for specific calculations.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 Data assessment does not pertain to this sample preparation procedure.
- 13.2 Samples requiring re-preparation are submitted to the preparation lab with an NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in ST-QAM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out:

16.2.1.1 Sulfuric acid waste from the clean up will be disposed of in the acid waste barrel after removal of the hexane layer.

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash.

16.2.1.3 Solid materials (soil, gloves, soiled paper products) are collected in the container labeled "Non-Regulated Material" for disposal.

16.2.1.4 If the labware was used for the analysis of radioactive samples and contains radioactivity at level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

16.2.1.5 Methylene chloride saturated water and sample volume remaining after extraction is disposed of in the base waste barrel.

16.2.1.6 Solvent rinses from the labware cleaning procedure are disposed of in the flammable solvent waste stream.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996) and Update IV, February 2007. Sections 3500C, 3510C, 3580A, 3550C, 3600C, 3620C, 3650B, 3660B, AND 3665A.

17.2 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision

17.3 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.4 Associated SOPs:

17.4.1 Corporate White Paper CA-Q-W-010 LVI Technique for Organic Prep & Analysis

17.4.2 ST-OP-0001, Labware Preparation for Organic analysis.

17.4.3 ST-PM-0002, Sample Receipt and Chain of Custody

17.4.4 ST-QA-0002, "Standards and Reagent Preparation."

17.4.5 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes"

17.4.6 ST-QA-0016, IDL/MDL Determination

17.4.7 ST-QA-0036, "Non-Conformance Memorandum (NCM) Process"

17.4.8 ST-QA-0038, "Procedure for Compositing and Sub-sampling"

17.4.9 ST-GC-0005, "Extractable Total Petroleum Hydrocarbons Iowa OA-2 and Method 8015 by GC"

17.4.10 ST-GC-0015, "PCB Gas Chromatography Analysis Method SW-846 8000C/8082A"

17.4.11 ST-GC-0016, "Pesticide Gas Chromatography Analysis Method SW-846 8000C/8081B and EPA 608"

17.4.12 ST-MS-0001, "GC/MS Analysis Based on Methods 8270C and 625"

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

18.2 For method 625, the acid fraction is performed first and then followed by the base/neutral extraction. It has been demonstrated that this procedure yields acceptable LCS recoveries.

18.3 Aqueous sample volumes are determined by weight.

18.4 The lab uses a modified 3665A sulfuric acid clean up. The permanganate step is not routinely used.

18.5 Method 3510C states that water bath temperatures may be elevated so that concentration is

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completed with 10-20 minutes. TestAmerica St. Louis prefers to keep the water bath temperature lower for semivolatiles extracts and thus concentrating time is greater than 20 minutes.

- 18.6 The solvent rinsing of the extraction glassware when transferring the extract to the KD flask is not performed. Once the organics are extracted from the water and in the solvent layer, adhesion to the glassware is no longer a significant concern.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1. Clarified pH requirements and BNA extraction order in Section 11.1.5
- 19.2. Added Section 11.9: GPC cleanup procedure.
- 19.3. Rev. 15:
- 19.3.1. Added ovens to equipment in Section 6
 - 19.3.2. Added Class A description to Concentrator tips in Section 6
 - 19.3.3. Updated Section 10.3 to include Class A concentrator tips and calibration requirement
 - 19.3.4. Updated Sonicator settings in Section 11.4 (from 10 to ~7.5)
 - 19.3.5. Updated the use of methylene chloride/acetone blend in section 11.3.1 and 11.3.10.
 - 19.3.6. Added volume of spike solution added to QC samples in Section 11.5.5
 - 19.3.7. Updated the procedure steps in section 11.7.2.2.9.
 - 19.3.8. Added SOP ST-QA-0039 to Reference Section 17
- 19.4. Rev 16:
- 19.4.1. Updated GPC section 11.9.
 - 19.4.2. Added documentation of sonicator in section 11.4.
 - 19.4.3. Added residual chlorine check to section 11.1.
- 19.5. Rev 17:
- 19.5.1. Added Methanol to solvents in Section 7
 - 19.5.2. Updated Table 2: removed hexane as solvent for Pest/PCBs; replaced with methanol
- 19.6. Rev, 18:
- 19.6.1. Removed acetone blend for separatory funnel in section 11.3.
 - 19.6.2. Updated PAH analysis extract blend throughout section 11.3.
- 19.7. Rev, 19:
- 19.7.1. Added Potassium Permanganate to the list of reagents in section 7.0.
 - 19.7.2. Updated instructions for added Potassium Permanganate throughout section 11.8.4.
 - 19.7.3. Updated Sep Funnel Liquid Extraction in section 11.3.
- 19.8. Rev 20:
- 19.8.1. Updated Section 11.4 to read the same as Method 3550C regarding the amount of sample weighed up and the requirements for recording of the amount used.
- 19.9. Rev 21:
- 19.9.1. 11.1.1.1.: Addition of density limits
- 19.10. Rev 22 (5/14/14)
- 19.10.1. Removed references to Method 8310, PAHs and SOP ST-LC-0001. Method no longer supported by laboratory.
 - 19.10.2. Added information on Lower Volume Initiative (LVI) to SOP.
 - 19.10.3. Updated balance and syringe information in Section 6.
 - 19.10.4. Added chlorine and peroxide test strips to Section 6.
 - 19.10.5. Added acidic sodium sulfate and Hexane:Acetone mix to Section 7
 - 19.10.6. Removed references to Standards Log program; replaced with LIMS reagent program.
 - 19.10.7. Added info on PCB holding time to Section 8.
 - 19.10.8. Clarified QC batching and requirements in Section 9.1.
 - 19.10.9. Updated TCLP volumes in Sections 9.2 and 9.3.
 - 19.10.10. Updated BNA extraction procedure in Section 11.1.5.
 - 19.10.11. Added funnel conditioning information to Section 11.3.
 - 19.10.12. Removed Turbovap procedure from Concentration Section 11.7.
 - 19.10.13. Updated Clean-up information in Section 11.8.
 - 19.10.14. Updated Section 14 to indicate performance and QC data for methods is maintained in

LIMS.

- 19.10.15 Updated tables.
- 19.11 Revision 23 (9/16/14):
 - 19.11.1 Added note to Section 11.1.3 that LVI requires South Carolina certification if to be used for South Carolina compliance samples.
- 19.12 Revision 24 (12/01/2014)
 - 19.12.1 Added standards prep info to Section 7 and Appendix 1
- 19.13 Revision 25 (05/11/2015)
 - 19.14.1 Removed all references to SW846 Method 3520C (CLLE)
 - 19.14.2 Add a 1 mL check for the concentrator tip check
 - 19.14.3 Clarified source for florisil cartridge in Section 7
 - 19.14.4 Updated LVI volumes throughout SOP (from 200 to 250mL)
 - 19.14.5 Updated recovery criteria for florisil checks from 80 – 120% to 80 – 110% per method 3620
 - 19.14.6 Corrected order of steps in the extraction in Section 11.1– adding surrogates and spikes prior to taking sample pH
 - 19.14.7 Added NCM option to Section 11.5 for density correction
 - 19.14.8 Added table containing volume and spiking amounts for LVI
 - 19.14.9 Added Section 11.6.3.5 to document handling of extracts waiting for concentration step
 - 19.14.10 Updated steps in florisil clean up Section 11.8.3
 - 19.14.11 Corrected Section references and spelling errors
- 19.15 Revision 26: (10/26/15)
 - 19.15.1 Removed section 11.5.3.5 which had allowed storage time between extraction and concentration.
- 19.16 Revision 27: (9/20/16)
 - 19.16.1 Added reference to SOP ST-QA-0038 (Sub-sampling) to Section 1.
 - 19.16.2 Added process for extracting Wips to Section 2 and 11
 - 19.16.3 Updated Sections 6 and 7.
 - 19.16.4 Clarified PCB holding times in Section 8
 - 19.16.5 Updated Sections 11.1, 11.2; added 11.3 (Wipes)
 - 19.16.6 Added rinse requirement to Section 11.5.3
 - 19.16.7 Updated Reference Section (17)
 - 19.16.8 Removed reference to 3520 in Section 18.

TABLE 1

Liquid/Liquid Extraction Conditions		
Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA 8270/625	< 2	> 12
PAH 8270	> 11	None
Pest/PCB 8081/8082	5 – 9	None
TPH 8015	As received	none

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Table 2

Final Volume and Exchange Solvents if no cleanup is used			
Type	Exchange Solvent for Analysis	Final Volume for Analysis (mL)	Final Volume for LVI Analysis (mL)
Semivolatiles: 8270, 625	N/A	1.0	1.0
PAHs: 8270	N/A	1.0	
Pesticides/PCBs: 8081/8082 & 608	Hexane	10.0 (routine waters and soils)	2.0
Hydrocarbons (TPH): 8015	N/A	1.0	

Final Volumes and exchange solvents if Florisil cleanup is used			
Type	Exchange Solvent for Florisil	Final Volume for Florisil	Final Volume and solvent for Analysis
Pesticides	90:10 Hexane/Acetone	10 mL (1 mL aliquot used)	1 mL, hexane

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

Table 3

Determinative Method	Surrogate Standard ID	Amount	Spike Standard	Amount
Semivolatiles: 8270, 625	BNA SURR	500 µL	BNA/TCLP	1 mL
8270 SIM	BNA SURR	10 µL	8270 SIM	1 mL
Pesticides: 8081 & 608	PEST SURR	500 µL	PEST	500 µL
PCBs: 8082 & 608 (if PCB only)	PCB SURR	500 µL	PCB	500 µL
Hydrocarbons (TPH): 8015	TPH SURR	500 µL	TPH	500 µL

Table 4

Method	LVI Amounts Initial/final	Surrogate/Spike	Solvent amt and reps
TPH	250uL/1mL	125/125 uL	15uL x 3
BNA	250uL/1mL	400/200 uL	Acid: 15uL x 3 Base; 25uL x 1, then 15uL x 2
	250uL/2mL	100/100 uL	15uL x 3
	250uL/2mL	100/100 uL	15uL x 3

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APPENDIX 1

**Reagent ID: Pest_Surr_00007**

Description:	Pesticide Surrogate	Expiration Date:	02/10/2015
No. of Bottles:	1	Laboratory:	TestAmerica St. Louis
Storage Location:	Organic Prep Standards Storag	Prepared By:	Agu, Kelli K
Reagent Volume:	500.000 mL	Solvent:	Methanol
Creation Date:	08/13/2014	Solvent Lot:	399910
Container(s):	425501		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
DCB Decachlorobiphenyl (Surr)	8081 surr_00007	08/01/2016	200.00000	ug/mL	0.40000	ug/mL
Tetrachloro-m-xylene	8081 surr_00007	08/01/2016	199.92000	ug/mL	0.39984	ug/mL

Source Reagents

Reagent	Description	Type	Expiration	Vendor	Vendor Lot #	Vendor Cat Lot #	Volume Used	Volume Units
8081 surr_00007	8081 surr custom mix (PEST)	ASTD	08/01/16	Restek	A097672	553428	1.00000	mL

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**Reagent ID: BNASpike_00073**

Type:	ASTD	Expiration Date:	03/29/2015
Description:	Custom TCL BNA Spike Mix	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Saulters, Camme N
Storage Location:	Organic Prep Standards Storag	Vendor:	Restek
Reagent Volume:	50.000 mL	Vendor Lot #:	A0105191
Creation Date:	09/30/2014	Vendor Cat #:	564240
Container(s):	463332		
Comment:	reference ncm#06-0201042 for expiration date solvent methanol 99%		

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
1,2,4-Trichlorobenzene					100.00000	ug/mL
1,2-Dichlorobenzene					100.00000	ug/mL
1,3-Dichlorobenzene					100.00000	ug/mL
1,4-Dichlorobenzene					100.00000	ug/mL
2,2'-oxybis[1-chloropropane]					100.00000	ug/mL
2,4,5-Trichlorophenol					100.00000	ug/mL
2,4,6-Trichlorophenol					100.00000	ug/mL
2,4-Dichlorophenol					100.00000	ug/mL
2,4-Dimethylphenol					100.00000	ug/mL
2,4-Dinitrophenol					100.00000	ug/mL
2,4-Dinitrotoluene					100.00000	ug/mL
2,6-Dinitrotoluene					100.00000	ug/mL
2-Chloronaphthalene					100.00000	ug/mL
2-Chlorophenol					100.00000	ug/mL
2-Methylnaphthalene					100.00000	ug/mL
2-Methylphenol					100.00000	ug/mL
2-Nitroaniline					100.00000	ug/mL
2-Nitrophenol					100.00000	ug/mL
3 & 4 Methylphenol					100.00000	ug/mL
3,3'-Dichlorobenzidine					100.00000	ug/mL
3-Nitroaniline					100.00000	ug/mL
4,6-Dinitro-2-methylphenol					100.00000	ug/mL
4-Bromophenyl phenyl ether					100.00000	ug/mL
4-Chloro-3-methylphenol					100.00000	ug/mL
4-Chloroaniline					100.00000	ug/mL
4-Chlorophenyl phenyl ether					100.00000	ug/mL

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**Reagent ID: BNASpike_00073**

Type:	ASTD	Expiration Date:	03/29/2015
Description:	Custom TCL BNA Spike Mix	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Saulters, Camme N
Storage Location:	Organic Prep Standards Storag	Vendor:	Restek
Reagent Volume:	50.000 mL	Vendor Lot #:	A0105191
Creation Date:	09/30/2014	Vendor Cat #:	564240
Container(s):	463332		
Comment:	reference ncm#06-0201042 for expiration date solvent methanol 99%		

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
4-Methylphenol					100.00000	ug/mL
4-Nitroaniline					100.00000	ug/mL
4-Nitrophenol					100.00000	ug/mL
Acenaphthene					100.00000	ug/mL
Acenaphthylene					100.00000	ug/mL
Anthracene					100.00000	ug/mL
Benzo(a)anthracene-d12					100.00000	ug/mL
Benzo(a)pyrene-d12					100.00000	ug/mL
Benzo(b)fluoranthene-d12					100.00000	ug/mL
Benzo[a]anthracene					100.00000	ug/mL
Benzo[a]pyrene					100.00000	ug/mL
Benzo[b]fluoranthene					100.00000	ug/mL
Benzo[g,h,i]perylene					100.00000	ug/mL
Benzo[k]fluoranthene					100.00000	ug/mL
Bis(2-chloroethoxy)methane					100.00000	ug/mL
Bis(2-chloroethyl)ether					100.00000	ug/mL
Bis(2-ethylhexyl) phthalate					100.00000	ug/mL
Butyl benzyl phthalate					100.00000	ug/mL
Carbazole					100.00000	ug/mL
Chrysene					100.00000	ug/mL
Dibenz(a,h)anthracene					100.00000	ug/mL
Dibenzofuran					100.00000	ug/mL
Diethyl phthalate					100.00000	ug/mL
Dimethyl phthalate					100.00000	ug/mL
Di-n-butyl phthalate					100.00000	ug/mL
Di-n-octyl phthalate					100.00000	ug/mL

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**Reagent ID: BNASpike_00073**

Type:	ASTD	Expiration Date:	03/29/2015
Description:	Custom TCL BNA Spike Mix	Laboratory:	TestAmerica St. Louis
No. of Bottles:	1	Prepared By:	Saulters, Camme N
Storage Location:	Organic Prep Standards Storag	Vendor:	Restek
Reagent Volume:	50.000 mL	Vendor Lot #:	A0105191
Creation Date:	09/30/2014	Vendor Cat #:	564240
Container(s):	463332		
Comment:	reference ncm#06-0201042 for expiration date solvent methanol 99%		

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Diphenylamine					100.00000	ug/mL
Fluoranthene					100.00000	ug/mL
Fluorene					100.00000	ug/mL
Hexachlorobenzene					100.00000	ug/mL
Hexachlorobutadiene					100.00000	ug/mL
Hexachlorocyclopentadiene					100.00000	ug/mL
Hexachloroethane					100.00000	ug/mL
Indeno[1,2,3-cd]pyrene					100.00000	ug/mL
Isophorone					100.00000	ug/mL
Naphthalene					100.00000	ug/mL
Nitrobenzene					100.00000	ug/mL
N-Nitrosodi-n-propylamine					100.00000	ug/mL
N-Nitrosodiphenylamine					100.00000	ug/mL
Pentachlorophenol					100.00000	ug/mL
Phenanthrene					100.00000	ug/mL
Phenol					100.00000	ug/mL
Pyrene					100.00000	ug/mL

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**Reagent ID: 150ppmBNASurr_00019**

Description:	150 ppm BNA Surrogate Working Std	Expiration Date:	03/03/2015
No. of Bottles:	1	Laboratory:	TestAmerica St. Louis
Storage Location:	Organic Prep Standards Storag	Prepared By:	Saulters, Camme N
Reagent Volume:	500.000 mL	Solvent:	Methanol
Creation Date:	10/21/2014	Solvent Lot:	440990
Container(s):	478528		
Comment:	combined 3 stds due to new spike amounts using more surrogate.		

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
2,4,6-Tribromophenol	8270 Surr Std_00041	05/01/2019	5000.00000	ppm	150.00000	ppm
2-Fluorobiphenyl (Surr)	8270 Surr Std_00041	05/01/2019	5000.00000	ppm	150.00000	ppm
2-Fluorophenol	8270 Surr Std_00041	05/01/2019	5000.00000	ppm	150.00000	ppm
Nitrobenzene-d5	8270 Surr Std_00041	05/01/2019	5000.00000	ppm	150.00000	ppm
Phenol-d5	8270 Surr Std_00041	05/01/2019	5000.00000	ppm	150.00000	ppm
Terphenyl-d14	8270 Surr Std_00041	05/01/2019	5000.00000	ppm	150.00000	ppm
2,4,6-Tribromophenol	8270 Surr Std_00042	05/01/2019	5000.00000	ppm	150.00000	ppm
2-Fluorobiphenyl (Surr)	8270 Surr Std_00042	05/01/2019	5000.00000	ppm	150.00000	ppm
2-Fluorophenol	8270 Surr Std_00042	05/01/2019	5000.00000	ppm	150.00000	ppm
Nitrobenzene-d5	8270 Surr Std_00042	05/01/2019	5000.00000	ppm	150.00000	ppm
Phenol-d5	8270 Surr Std_00042	05/01/2019	5000.00000	ppm	150.00000	ppm
Terphenyl-d14	8270 Surr Std_00042	05/01/2019	5000.00000	ppm	150.00000	ppm
2,4,6-Tribromophenol	8270 Surr Std_00043	05/01/2019	5000.00000	ppm	150.00000	ppm
2-Fluorobiphenyl (Surr)	8270 Surr Std_00043	05/01/2019	5000.00000	ppm	150.00000	ppm
2-Fluorophenol	8270 Surr Std_00043	05/01/2019	5000.00000	ppm	150.00000	ppm
Nitrobenzene-d5	8270 Surr Std_00043	05/01/2019	5000.00000	ppm	150.00000	ppm
Phenol-d5	8270 Surr Std_00043	05/01/2019	5000.00000	ppm	150.00000	ppm

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**Reagent ID: 150ppmBNASurr_00019**

Description:	150 ppm BNA Surrogate Working Std	Expiration Date:	03/03/2015
No. of Bottles:	1	Laboratory:	TestAmerica St. Louis
Storage Location:	Organic Prep Standards Storag	Prepared By:	Saulters, Camme N
Reagent Volume:	500.000 mL	Solvent:	Methanol
Creation Date:	10/21/2014	Solvent Lot:	440990
Container(s):	478528		
Comment:	combined 3 stds due to new spike amounts using more surrogate.		

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Terphenyl-d14	8270 Surr Std_00043	05/01/2019	5000.00000	ppm	150.00000	ppm

Source Reagents

Reagent	Description	Type	Expiration	Vendor	Vendor Lot #	Vendor Cat Lot #	Volume Used	Volume Units
8270 Surr Std_00041	8270 Surrogate parent standard	ASTD	05/01/19	Restek	A0103615	567685	5.00000	mL
8270 Surr Std_00042	8270 Surrogate parent standard	ASTD	05/01/19	Restek	A0103615	567685	5.00000	mL
8270 Surr Std_00043	8270 Surrogate parent standard	ASTD	05/01/19	Restek	A0103615	567685	5.00000	mL

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**Reagent ID: PCB_Spike_00014**

Description:	Aroclor 1016/1260 Mix in Hexane	Expiration Date:	12/16/2014
No. of Bottles:	1	Laboratory:	TestAmerica St. Louis
Storage Location:	Organic Prep Standards Storag	Prepared By:	Saulters, Camme N
Reagent Volume:	250.000 mL	Solvent:	Hexane
Creation Date:	08/19/2014	Solvent Lot:	413629
Container(s):	429775		
Comment:	cycletainer vendor exp date is 12/16/14		

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
PCB-1016	8082 Spike_00012	04/01/2019	1000.00000	ug/mL	10.00000	ug/mL
PCB-1260	8082 Spike_00012	04/01/2019	1000.00000	ug/mL	10.00000	ug/mL
PCB-1016	8082 Spike_00013	06/01/2019	1000.00000	ug/mL	10.00000	ug/mL
PCB-1260	8082 Spike_00013	06/01/2019	1000.00000	ug/mL	10.00000	ug/mL

Source Reagents

Reagent	Description	Type	Expiration	Vendor	Vendor Lot #	Vendor Cat Lot #	Volume Used	Volume Units
8082 Spike_00012	8082 spike aroclhor 1060/1260 (PCB)	ASTD	04/01/19	Restek	A092844	32039	1.25000	mL
8082 Spike_00013	8082 spike aroclhor 1060/1260 (PCB)	ASTD	06/01/19	Restek	A094177	32039	1.25000	mL

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**Reagent ID: PCB Surr_00006**

Description:	PCB Surrogate	Expiration Date:	12/16/2014
No. of Bottles:	2	Laboratory:	TestAmerica St. Louis
Storage Location:	Organic Prep Standards Storag	Prepared By:	Saulters, Camme N
Reagent Volume:	500.000 mL	Solvent:	Hexane
Creation Date:	08/06/2014	Solvent Lot:	413629
Container(s):	421310, 421311		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
DCB Decachlorobiphenyl (Surr)	8082SurrDeca_00009	02/28/2015	1004.00000	ug/mL	0.40160	ug/mL

Source Reagents

Reagent	Description	Type	Expiration	Vendor	Vendor Lot #	Vendor Cat Lot #	Volume Used	Volume Units
8082SurrDeca_00009	Decachlorobiphenyl Solution	ASTD	02/28/15	Ultra Scientific	CC-3147Z	PPS-150	200.00000	uL

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Reagent ID: PestSpike_00007

Description:	Pesticide Spike	Expiration Date:	03/03/2015
No. of Bottles:	1	Laboratory:	TestAmerica St. Louis
Storage Location:	Organic Prep Standards Storag	Prepared By:	Saulters, Camme N
Reagent Volume:	200.000 mL	Solvent:	Methanol
Creation Date:	09/08/2014	Solvent Lot:	440990
Container(s):	444962		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
4,4'-DDD	8081 Spike_00010	11/01/2016	200.01400	ug/mL	1.00007	ug/mL
4,4'-DDE	8081 Spike_00010	11/01/2016	200.20000	ug/mL	1.00100	ug/mL
4,4'-DDT	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL
Aldrin	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL
alpha-BHC	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL
alpha-Chlordane	8081 Spike_00010	11/01/2016	200.01400	ug/mL	1.00007	ug/mL
beta-BHC	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL
delta-BHC	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL
Dieldrin	8081 Spike_00010	11/01/2016	200.20000	ug/mL	1.00100	ug/mL
Endosulfan I	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL
Endosulfan II	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL
Endosulfan sulfate	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL
Endrin	8081 Spike_00010	11/01/2016	200.20800	ug/mL	1.00104	ug/mL
Endrin aldehyde	8081 Spike_00010	11/01/2016	200.11600	ug/mL	1.00058	ug/mL
Endrin ketone	8081 Spike_00010	11/01/2016	200.26000	ug/mL	1.00130	ug/mL
gamma-BHC (Lindane)	8081 Spike_00010	11/01/2016	200.20000	ug/mL	1.00100	ug/mL
gamma-Chlordane	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL

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**Reagent ID: PestSpike_00007**

Description:	Pesticide Spike	Expiration Date:	03/03/2015
No. of Bottles:	1	Laboratory:	TestAmerica St. Louis
Storage Location:	Organic Prep Standards Storag	Prepared By:	Saulters, Camme N
Reagent Volume:	200.000 mL	Solvent:	Methanol
Creation Date:	09/08/2014	Solvent Lot:	440990
Container(s):	444962		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
Heptachlor	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL
Heptachlor epoxide	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL
Methoxychlor	8081 Spike_00010	11/01/2016	200.00000	ug/mL	1.00000	ug/mL

Source Reagents

Reagent	Description	Type	Expiration	Vendor	Vendor Lot #	Vendor Cat Lot #	Volume Used	Volume Units
8081 Spike_00010	8081 Spike(PEST)organochlorine mix AB#1	ASTD	11/01/16	Restek	A092002	32291	1.00000	mL

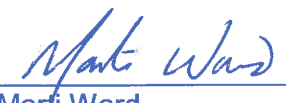
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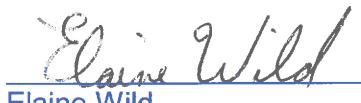
**Title: EXTRACTION OF HERBICIDES FROM WATERS AND SOILS
8151A**

Approvals (Signature/Date):

 9/15/16
Dennis Konopka Date
Extraction Dept. Manager

 9/15/16
Michael Ridenhower Date
Health & Safety Manager / Coordinator

 9.16.16
Marti Ward Date
Quality Assurance Manager

 9/15/16
Elaine Wild Date
Laboratory Director

This SOP was previously identified as SOP No. ST-OP-0007 Rev. 16

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Facility Distribution No.: 0

Distributed To: See Electronic Distribution Sheet

1.0 SCOPE AND APPLICATION

- 1.1 This method is applicable to the extraction of chlorinated herbicides in waters, solids, oils, and leachates (e.g. TCLP, SPLP), based on Method 8151A.
- 1.2 The reporting limits and QC criteria are maintained in the LIMS

2.0 SUMMARY OF METHOD

- 2.1 This method is based on SW846 method 8151A. . After acidifying the sample the free acids are extracted into diethyl ether. Solids are extracted into ether by sonication. If esters and acids are to be determined, the extract is hydrolyzed and extracted into diethyl ether. For both soils and aqueous samples, the free acid herbicides in the ether extract are esterified. The final volume is adjusted to prepare the extract for gas chromatography.

3.0 DEFINITIONS

- 3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

- 4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.
- 4.2 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
 - 5.2.1 Ether and methylene chloride creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.
 - 5.2.2 Ethyl ether must be periodically checked for the formation of peroxides. The minimum frequency shall be each time a new container is opened.
- 5.3 PRIMARY MATERIALS USED
 - 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the

information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Compound (1)	Hazards	Exposure Limits (2)	Signs of Exposure
Potassium hydroxide	Poison Corrosive	2 mg/m ³ (Ceiling)	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. Contact with skin can cause irritation or severe burns. Eye contact causes irritation with tearing, redness and swelling.
Sulfuric Acid	Corrosive Poison Cancer Hazard	1 mg/m ³ (TWA)	Inhalation may cause irritation of the nose and throat, and labored breathing. Skin contact symptoms include redness, pain, and severe burning. Eye contact can cause blurred vision, redness, pain, and severe tissue burns.
(Trimethyl-silyl) diazomethane (2.0M Solution in Hexanes)	Flammable	500 ppm (TWA) for hexane	Inhalation symptoms include headache, dizziness, drowsiness, incoordination, slowed reaction time, slurred speech, and giddiness. Skin contact can cause irritation. Eye contact can cause redness, tearing, and blurred vision.
Methanol (Methyl Alcohol)	Poison Flammable	200 ppm (TWA)	Inhalation symptoms may include headache, drowsiness, nausea, vomiting, blurred vision and blindness. Symptoms of skin contact may parallel inhalation exposure and can cause skin to become dry and cracked. Eye contact can cause irritation.
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
Ethyl ether	Flammable	400 ppm (TWA)	General anesthesia by inhalation can occur. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. Skin contact is irritating. Eye contact may cause irritation, redness, and pain.
1 – Always add acid to water to prevent violent reactions			
2 – Exposure limit refers to OSHA regulatory exposure limit			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling - At no time should this exposure limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Labware is cleaned to remove any residual organics. Please refer to SOP STL-OP-0001 for detailed glassware cleaning procedures.
- 6.2 Separatory Funnel: 2 L, 250ml
- 6.3 Separatory Funnel Rack
- 6.4 Balance: >2000 g capacity, accurate +/- 1% for waters
- 6.5 Balance :>300g capacity, accurate +/-1% for soils (100-300g +/-0.1%)
- 6.6 pH indicator paper, wide-range: covers extraction pH
- 6.7 Graduated cylinder: 1 liter. (other sizes may be used)
- 6.8 Solvent Dispenser Pump or 100 mL Graduated Cylinder
- 6.9 Mason Jars
- 6.10 Sonicator (at least 300 watts)
- 6.11 Sonicator horn, 3/4 inch
- 6.12 Kuderna-Danish (K-D) Apparatus: 500 mL
- 6.13 Concentrator Tube: 10 mL, Class A
- 6.14 Vials: Glass, 2 mL, and 4 mL, capacity with Teflon®-lined screw cap
- 6.15 Nitrogen Blowdown Apparatus
- 6.16 Syringe: 100uL through 1 mL
- 6.17 Phase Separation Paper
- 6.18 Teflon Funnel: 75 X 75 mm
- 6.19 Disposable Pipettes
- 6.20 Peroxide test strips

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 DI water, organic free
- 7.3 Potassium hydroxide solution, 37% aqueous solution, (w/v)
 - 7.3.1 Dissolve approximately 370 g of potassium hydroxide pellets (this may change based on the purity of the KOH pellets – a minimum of 37% KOH must present) in reagent water and dilute to 1 L. Other volumes of solution may be made up as convenient.

Considerable heat will be generated.

- 7.4 Sulfuric acid, 1:1
 - 7.4.1 Slowly add 500 mL concentrated sulfuric acid to 500 mL water. **Considerable heat will be generated. The acid must be added to the water.**
- 7.5 Sodium sulfate, Na₂SO₄, Anhydrous, granular, acidified
 - 7.5.1 Heat sodium sulfate in a shallow tray at 400°C for a minimum of 4 hours to remove phthalates and other interfering organic substances. In a large beaker, acidify by slurring 1000 g sodium sulfate with just enough diethyl ether to cover. Add 15 mL of concentrated sulfuric acid and mix thoroughly. Allow the ether to evaporate overnight. Work with the ether in a fume hood, making sure not to have any possible ignition sources nearby (i.e., steam bath, hot plate, etc.). Do not place mixture on a steam bath as this may lead to auto-ignition of the ether. Larger or smaller batches of acidified sodium sulfate may be prepared using the reagents in the same proportions.
- 7.6 Sodium Chloride, NaCl
- 7.7 Acidified 5% sodium sulfate
- 7.8 Diethyl ether, reagent grade.
- 7.9 Methanol, HPLC grade.
- 7.10 Silicic acid
- 7.11 TMS Diazomethane
- 7.12 Extraction/Exchange Solvents: Hexane and Ether solvent, pesticide quality or equivalent.
- 7.13 Stock Herbicide Standards, NIST traceable
 - 7.13.1 Semivolatile stock standards are stored at $\leq 6^{\circ}\text{C}$. Standards must be allowed to come to room temperature before use.
 - 7.13.2 Refer to SOP ST-QA-0002, "Standards and Reagent Preparation" for the required storage conditions and shelf lives of the standards used in this procedure. All stock standards must be protected from light. Standard solutions must be replaced by the manufacturer's expiration date or 6 months after opening the ampoule (or making the standard if from neat materials), whichever is shorter.
 - 7.13.3 Surrogate spiking standards are prepared as dilutions of the stock standards. See recipe in the Standards Log program.
 - 7.13.4 The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Spiking standards are purchased or prepared as dilutions of the stock standards. See recipe in the Standards Log program.

8.0 SAMPLE COLLECTION PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Water samples are unpreserved and stored between 0 and 6°C.
- 8.3 Soil samples are refrigerated between 0 and 6°C.

- 8.4 Holding Times
- 8.4.1 Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.
- 8.4.2 For TCLP leachates, the extraction is initiated within seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step. If the filtration step requires extended time, this time counts as part of the seven day holding time.
- 8.4.3 Analysis of the extracts is completed within forty days of extraction.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), Matrix Spike (MS) and Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.1.4 A method blank and LCS may be shared across TALS projects provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each project as specified by the client.

9.2 Method Blank

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 For Water analyses, the method blank is comprised of DI water.
- 9.2.4 For Soil analyses, the method blank is comprised of sodium sulfate.
- 9.2.4.1 South Carolina requires the use of Ottawa sand for the Method Blank.

9.3 Laboratory Control Sample

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.
- 9.3.3 For Water analyses, the LCS is comprised of DI water fortified with Herbicide acids.
- 9.3.4 For Soil analyses, the LCS is comprised of sodium sulfate fortified with Herbicide acids.
- 9.3.4.1 South Carolina requires the use of Ottawa sand for the LCS.

9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.
- 9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every TALS batch.

9.5 Surrogate

- 9.5.1 A surrogate is a non-target analyte similar in chemical composition and behavior, which mimics the target analytes during preparation, extraction and analysis.

- 9.5.2 Surrogate(s) is added to every field sample, method blank, LCS and MS/MSD for analysis at the beginning of the sample preparation process.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Balance calibration must be checked daily when used. Refer to SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipette".
- 10.2 Thermometers must be checked as prescribed in SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipette".
- 10.3 Sonicators are calibrated (tuned) each day of use.
CAUTION: Hearing protection must be worn when tuning the sonicators.
- 10.3.1 Place the power switch to the "Off" position.
- 10.3.2 Switch to the "Continuous" position.
- 10.3.3 Turn the output control knob fully counter-clockwise, below setting "1", for minimum amplitude.
- 10.3.4 Push and hold the power switch down in the "Tune" position; it is spring loaded in this position and must be held during tuning.
- 10.3.5 Slowly turn the output knob to setting "10", maximum amplitude, taking care not to exceed 70-80% on the meter. Should the meter approach 80%, lower the control knob setting to reduce the meter reading to about 10 and continue through section 10.4.9, then raise the setting and retune. If the generator has been thrown far off resonance, several increments may be required.
- 10.3.6 Turn the tuning knob in whichever direction will cause the meter needle to deflect towards zero (0).
- 10.3.7 Continue turning the tuning knob in the same direction until the needle stops deflecting and reverses direction.
- 10.3.8 Reverse the tuning knob very slowly to return the meter needle to its lowest reading, that "null" point at which any motion of the tuning knob in either direction will cause the needle to rise (to deflect away from zero).
- 10.3.9 Return the output control knob to minimum setting (fully counter-clockwise) and release the power switch. The sonicator is now tuned properly.
- 10.4 See analysis SOP: ST-GC-0017 for instrument calibration requirements.

11.0 PROCEDURE

11.1 Aqueous Samples - Preparation

- 11.1.1 Weigh full sample bottle without the cap.
- 11.1.1.1 If a sample is known or suspected (based upon client knowledge, project scope, or site history) to have a high density (>1.2 g/mL, e.g. a brine or waste) or a low density (<0.98 g/mL, e.g. mixed solvent), the sample density will be measured

and the volume determined arithmetically (sample mass divided by the density equals the volume).

- 11.1.1.2 Volume is calculated by gravimetric determination assuming a sample density of 1. Samples that are not aqueous, or suspected to have a density greater than 1.2, will have aliquots taken for density analysis to correct volume for density.
- 11.1.2 Thoroughly mix the liquid sample in accordance with SOP ST-QA-0038.
- 11.1.3 The normal sample volume is 1 liter.
 - 11.1.3.1 Other sample volumes may be used to obtain specific reporting limits, and reduced sample volumes, diluted to 1 liter with reagent water, may be used for very dirty samples. See manager/supervisor for instruction.
- 11.1.4 Pour sample aliquot into a Separatory Funnel
- 11.1.5 Spike each sample, blank LCS and MS with 1000 μ L of DCAA surrogate solution. Spike matrix spikes and LCS with 1000 μ L of herbicide matrix spiking solution.
 - 11.1.5.1 Document spiking volumes and standard numbers on the bench sheet.
 - 11.1.5.2 Return spiking solutions to the refrigerator as soon as possible.
- 11.1.6 After the sample bottle is emptied, rinse the container with 60 mls of the extraction solvent. Gently swirl the solvent in the container, rinsing the sides of the container thoroughly and add solvent rinse to the extraction vessel. Weight the empty sample bottle without the cap and record.
 - 11.1.6.1 If the entire sample volume is not used the container cannot be solvent rinsed. Organic materials adhering to the container walls will be absent in the extraction process. An NCM is written documenting that the container rinse could not be performed.
- 11.1.7 Add 250 g of NaCl to each sample and shake to dissolve the salt.

11.2 Hydrolysis

- 11.2.1 Add 5 mL of 37% KOH to each sample, seal and shake.
- 11.2.2 Check the pH of the sample with pH paper and document.
 - 11.2.2.1 If the pH of the sample is not ≥ 12 adjust to ≥ 12 by adding more KOH.
- 11.2.3 Let the sample sit at room temperature for 2 hours to complete the hydrolysis.
 - 11.2.3.1 Add 60 mL of methylene chloride to sample Separatory Funnel.
 - 11.2.3.2 Rotate for five minutes.
 - 11.2.3.3 Allow methylene chloride layer to separate.
 - 11.2.3.4 Drain methylene chloride layer and discard.
- 11.2.4 Add approximately with 17mL of cold (4°C) 1:1 sulfuric acid to the sample.
- 11.2.5 Seal, and shake to mix.
- 11.2.6 Check the pH of the sample with pH paper and document.
 - 11.2.6.1 If the pH is not ≤ 2 , add more acid to adjust the pH to ≤ 2 .
- 11.2.7 Add 120 mL diethyl ether to each sample and extract by shaking or rotating vigorously for 2 minutes, venting as necessary.
- 11.2.8 Allow the organic layer to separate from the aqueous layer.
 - 11.2.8.1 If an emulsion layer greater than one third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation.
- 11.2.9 Drain the aqueous layer (bottom layer) into the original sample container.
- 11.2.10 Collect the ether phase in a labeled amber bottle containing approximately 10g of acidified anhydrous sodium sulfate.
- 11.2.11 Return the aqueous phase to the separatory funnel, add 60 mL diethyl ether and repeat the extraction procedure a second time, combining the ether extracts.
- 11.2.12 Repeat the extraction a third time with 60 mL diethyl ether.
- 11.2.13 Discard the aqueous phase after the third extraction.
- 11.2.14 Allow the extract to remain in contact with the sodium sulfate for at least 2 hours, shaking periodically.

11.2.14.1 If the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free flowing crystals are visible when the bottle is swirled or shaken.

11.2.15 Proceed to section 11.4, Extract Drying

11.2.16 If drying does not commence immediately, store all extracts in freezer designed for explosion prevention.

11.3 Soil and Sediment Samples Preparation

11.3.1 Homogenize the sample by mixing thoroughly. See SOP: ST-QA-0038 for details.

11.3.2 Weigh 50.0 g of moist solid sample into a glass jar.

11.3.2.1 Use 50 g of sodium sulfate for the Method Blank and the LCS.

11.3.3 Acidify sample with 2-5 mL of concentrated H₂SO₄.

11.3.3.1 Use 2ml for Method Blank and LCS samples. This is because the method blank and LCS do not contain the carbonates naturally found in soil.

11.3.4 There should be a small amount of free flowing liquid phase.

11.3.4.1 If not, add DI water until there is.

11.3.5 Stir well with a **metal** (do not use wood) spatula.

11.3.6 Wait 15 minutes.

11.3.7 Stir with a spatula and check the pH of the liquid phase.

11.3.7.1 Add more acid if necessary to bring the pH to <2, repeating the stirring and standing time after each acid addition.

11.3.8 Add 50g of sodium sulfate and mix well. The sample should be free flowing.

11.3.8.1 If not, add more sodium sulfate.

11.3.9 Spike each sample, blank LCS and MS with 500 µL of DCAA surrogate solution. Spike matrix spikes and LCS with 250 µL of herbicide matrix spiking solution. (Refer to tables A1 and A2)

11.3.10 Add a minimum of 100 mL of ether to the mason jar.

11.3.11 Place the bottom surface of the appropriate disrupter horn tip (clean with acetone during this method) approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.3.12 Sonicator is set at 10 with mode switch on pulse, and percent-duty cycle knob set at 50%.

11.3.13 Sonicate for 3 minutes, making sure the entire sample is agitated.

11.3.14 Line funnel with filter paper, and acidify with 10-20 g of acidic sodium sulfate.

11.3.15 Place the prepared funnel on a collection sep funnel. The collection sep funnel is a sep funnel suitable for the hydrolysis step, typically a 2L sep funnel, which contains:

11.3.15.1 500mL of DI water, 100g of NaCL, 5mLs KOH. Thoroughly shake to make sure the salt has dissolved before addition of the sample decanting.

11.3.16 Decant and filter extracts through the prepared funnel into the collection apparatus.

11.3.17 Repeat the extraction two more times with additional 100 mL minimum portions of **ether**.

11.3.18 Decant off extraction solvent after each sonication.

11.3.18.1 On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the ether.

11.3.18.2 If drying does not commence immediately, store all extracts in freezer designed for explosion prevention.

11.3.19 Check pH ≥ 12 if not, add 37% KOH until a pH ≥12 is achieved.

11.3.20 rotate base portion of samples for 15mins then allow to sit.

11.3.21 Make samples acidic with 1:1 DI:sulfuric at a pH ≤2

11.3.22 Adjust the pH of the solution to ≤2 with 1:1 sulfuric acid. **(Slowly or heat will hurt Dinoseb recovery.)**

11.3.23 Rotate acidic portion of samples for 15mins then allow to sit and separate.

11.3.24 Proceed to Section 11.4, Extract Drying.

11.4 Extract Drying

- 11.4.1 Pour the extracts into an amber bottle containing approximately 10 g acidified sodium sulfate.
- 11.4.2 Allow the extract to remain in contact with the sodium sulfate for at least 2 hours. If the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free flowing crystals are visible when the flask or bottle is swirled or shaken. Proceed to section 11.5 or 11.6 concentration.

11.5 KD setup w/Concentrator tip attached.(pre-rinsed)

- 11.5.1 Transfer the ether extract through a filter paper lined funnel into a hot bath set at 60°C.. Use a stirring rod to crush the caked sodium sulfate during transfer. Rinse the flask or bottle with 20-30 mL ether to complete transfer.
- 11.5.2 Concentrate ether extract to approximately 5 - 8 mL in KD.
- 11.5.3 Transfer extract into a 10 mL concentrator tube. Rinse Turbovap tube with ether and add this rinse to concentrator tube to a final volume of 10 mL.
- 11.5.4 Add 2 mL hexane into a fresh concentrator tip, then add 5mL of ether extract.

11.6 Concentration (Hot Bath Version)

- 11.6.1 Transfer the ether extract through a filter paper lined funnel into a K-D flask w/ attached concentrator tip and add a boiling chips. Bath set at 60 - 65 °C, rinse the flask or bottle with 20-30mL ether to complete transfer.
- 11.6.2 Concentrate ether extract to 5-8mL on hot bath.
- 11.6.3 Disassemble the contractor tip from K-D and bring the extract to 10 mL by rinsing the sides of the tip with ether; If the sample level is too high, use the n-evap to blow the sample down to 10mL.
- 11.6.4 Add 2 mL hexane into a clean concentrator tip, then transfer 0.5 mL of ether extract into the tip.

NOTE: South Carolina requires the lab follow SW 846 Method 8151A Section 7.4.5 for South Carolina compliance samples.

11.7 Esterification (Trimethylsilyl Diazomethane Solution)

- 11.7.1 Add 200 µL methanol and 100 µL of trimethylsilyl diazomethane solution to the extract.
- 11.7.2 Extract should turn a yellow color. If this doesn't occur, add additional 100 µL aliquots of (trimethylsilyl)diazomethane until the yellow color persists. (Swirling with a glass pipette helps the process.)
- 11.7.3 Allow the extract to sit for 1 hour at room temperature to allow the methylation reaction to proceed.
- 11.7.4 Dilute the extract to a final volume of 10 mL with hexane
- 11.7.5 After 1 hour the reaction is halted and the diazomethane removed by the addition of 1-2 g silicic acid.
 - 11.7.5.1 Sufficient silicic acid must be added to remove all remaining diazomethane. This is indicated by the change from a yellow to a clear solution.
 - 11.7.5.2 Color change from yellow to clear is not effective with soil extracts which are usually colored. Silicic acid is to be added until gas generation (bubbling) has nearly ceased.
- 11.7.6 Submit to GC lab for analysis.

NOTE: Be careful, do not transfer silicic acid into final extract as silicic acid will destroy G.C. column.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 There are no calculations pertaining to this sample preparation procedure.

- 12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analytical SOP.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 Data assessment does not pertain to this sample preparation procedure.
- 13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS.
- 14.2 Demonstration of Capability
- 14.2.1 Initial and continuing demonstrations of capability requirements are established in the QAM.
- 14.3 Training Qualification
- 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
- 16.2.1 The following waste streams are produced when this method is carried out.
- 16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."
- 16.2.1.2 Sodium Sulfate/filter paper contaminated with Ether will be stored in designated container within fume hood until such time solvent has evaporated. At such time container is to be emptied into proper waste accumulation container in 90-day storage area, labeled as Drum Type "C".

- 16.2.1.3 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D."
- 16.2.1.4 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 SW846, Test Methods for Evaluating Solid Waste, Chlorinated Herbicides, Method 8151A.
- 17.2 TestAmerica Quality Assurance Manual (ST-QAM), current revision
- 17.3 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.4 Associated SOPs current revisions:
 - 17.4.1 ST-OP-0001, Labware Preparation for Organic Analysis
 - 17.4.2 ST-PM-002, Chain of Custody and Sample Receipt.
 - 17.4.3 ST-QA-0002, Standards and Reagent Preparation.
 - 17.4.4 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.
 - 17.4.5 ST-QA-0016, IDL/MDL Determination
 - 17.4.6 ST-QA-0036, Non-Conformance Memorandum (NCM) Process
 - 17.4.7 ST-GC-0017, Herbicide Gas Chromatography Analysis by SW-846 8000C/8151A

18.0 CLARIFICATIONS, MODIFICATIONS TO REFERENCE METHOD

- 18.1 Directions to add sufficient DI water to the soil sample so that the pH can be measured have been added.
- 18.2 Method 8151A describes esterification either by the bubbler method or by diazomethane generation by the use of a diazald kit. This procedure utilizes (trimethylsilyl)diazomethane for esterification.
- 18.3 50g soil used instead of EPA suggestion of 30g. In house control charts of QC data and passing PT results show this to be accurate and acceptable.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Revised section 5.
- 19.2 Updated to TestAmerica format.
- 19.3 Revised text in section 11.6 from 400 μ L methanol to 200 and revised 200 μ L diazomethane to 100.
- 19.4 Rev. 8: No Changes, Annual Review.
- 19.5 Rev 9: No Changes, annual Review
- 19.6 Rev. 10: *(corrections to Section 19 made 11-14-11 M. Ward)*
 - 19.6.1 Added additional size separatory funnel to section 6.2.
 - 19.6.2 Updated section 11.3: soil and sediment sample preparation.
 - 19.6.3 Removed of section 11.6.6.
- 19.7 Rev. 11:
 - 19.7.1 Added reference for use of explosion proof freezer for extracts in section 11.2 and 11.3.

- 19.7.2 Updated amount of sulfuric acid used in sample in section 11.2.
- 19.8 Rev. 12:
 - 19.8.1 Updated the concentration process by added a hot bath version in section 11.6.
 - 19.8.2 Added peroxide checks and test strip requirements to Sections 5 and 6.
- 19.9 Rev. 13:
 - 19.9.1 § 11.1.1.1.: Addition of density limits
 - 19.9.2 § 11.1.1.2.: Addition of volume calculation by gravimetric determination
- 19.10 Rev. 14:
 - 19.10.1 Equipment and supplies updated in Section 6.
 - 19.10.2 Requirements for the Potassium hydroxide solution were clarified in Section 7. Sea sand was removed from the reagents information.
 - 19.10.3 References to Quantims were removed throughout SOP.
 - 19.10.4 Removed calibration steps for Concentrator tips (former section 10.3) as the lab utilizes Class A tips and calibration is not required.
 - 19.10.5 Removed all references to turbo vaps from the SOP – no longer used by the laboratory
 - 19.10.6 Clarified Sections 11.6 and 11.7
 - 19.10.7 Updated Section 14.1 to indicate that limits and QC criteria are maintained in LIMS
- 19.11 Revision 15
 - 19.11.1 Added South Carolina requirement to perform SW846 Method 8151A Section 7.4.5 to SOP Section 11.6
- 19.12 Revision 16 (10/01/2015)
 - 19.12.1 Updated section 6, removing 250 and 400 mL beakers
 - 19.12.2 Updated section 11.4 and 11.6
- 19.13 Revision 17 (9/19/16)
 - 19.13.1 Section 11.4.1 – removed funnel portion
 - 19.13.2 Added South Carolina requirement to use Ottawa sand for solid Method Blank and LCS

Herbicide Surrogate Spiking Solutions		
Analyte Group	Surrogate Spike Solution ID	Volume (μL)
Herbicides	Herbicides SS	500
Herbicide Matrix Spike and LCS Solutions		
Analyte Group	Matrix Spike Solution ID	Volume (μL)
Herbicides	Herbicides MS	250

Herbicide Surrogate Spike Components			
Type	Compounds ¹	Solvent	Conc. (ug/mL)
Herbicides SS	2,4-DCAA	Acetone	32

¹The surrogate is spiked as the free acid

Herbicide LCS and Matrix Spike Components			
Type	Compounds ¹	Solvent	Conc. (ug/mL)
Herbicides MS	2,4-D	Methanol	100
	2,4-DB		100
	Dinoseb		50
	Dicamba		10
	Dichloroprop		100
	2,4,5-TP (Silvex)		10
	Dalapon		250
	2,4,5-T		10

¹The herbicide spiking solution contains the herbicides as the free acids.

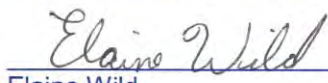
**Title: EXTRACTION of NITROAROMATIC and NITRAMINE
EXPLOSIVES [3535A] for WATERS and SOILS [8330B]**

Approvals (Signature/Date):

 5/23/16
Aaron Dickson Date
Operations Manager

 5/23/16
Michael Ridenhower Date
Health & Safety Manager / Coordinator

 5-23-16
Tony Byrd Date
Quality Assurance Manager

 5/23/16
Elaine Wild Date
Laboratory Director

This SOP was previously identified as SOP No. ST-OP-0008 Rev. 11

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1.0 SCOPE AND APPLICATION

- 1.1. This SOP describes procedures for the preparation of nitroaromatic and nitramine explosives by high performance liquid chromatography (HPLC) with UV detection and HPLC with atmospheric pressure, chemical ionization (APCI) mass spectrometry/mass spectrometry (MS/MS), in water, soil and sediment samples. Sample homogenization is not covered in this SOP. Refer to SOP ST-QA-0038.
- 1.2. This SOP is based on SW-846 Methods 8000C, 8330B, and 3535A.
- 1.3. The laboratory target analytes supported by this method, the reporting limit, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1. Nitroaromatic and nitramine analytes are prepared for analysis using one of the following procedures:
 - 2.1.1 Low-level solid phase extraction method [3535A]: Aqueous samples of low concentration are extracted and concentrated using solid phase extraction (SPE). Analytes from a 500 mL sample are adsorbed onto the sorbent material in a SPE cartridge, and then eluted with acetonitrile. The concentrated extract is diluted 1:1 with H₂O (HPLC grade) prior to analysis for HPLC and LCMSMS.
 - 2.1.2 Sonication method [3550C]: Soil and sediment samples are extracted using acetonitrile in a cooled ultrasonic bath. The concentrated extract is diluted 1:1 with H₂O (HPLC grade) prior to analysis.
 - 2.1.3 Orbital Shaker Table Method: Soil, sediment and wipe samples are extracted into acetonitrile on an orbital shaker table. The concentrated extract is diluted 1:1 with H₂O (HPLC grade) for HPLC and LCMSMS.

3.0 DEFINITIONS

- 3.1. See the St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2. There are no specific definitions for this procedure.

4.0 INTERFERENCES

- 4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.
- 4.2. Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain Tetryl should be diluted with acetonitrile prior to filtration. SAMPLES EXPECTED TO CONTAIN TETRYL SHOULD NOT BE EXPOSED TO TEMPERATURES ABOVE ROOM TEMPERATURE.

5.0 SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2. Specific Safety Concerns or Requirements
- 5.2.1 2,4,6-TNT is the analyte most often detected in high concentrations in soil samples. Soil samples as high as 2% 2,4,6-TNT can be safely ground. Samples containing higher concentrations should not be ground. The project manager or client must provide information as to whether the samples are suspected to contain explosives at a level greater than 2%. Visual observation of soil samples taken from a site expected to contain explosives is also important. Lumps of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.
- 5.3 Primary Materials Used
- The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: **This list does not include all materials used in the method.** The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limits (2)	Signs of Exposure
Acetonitrile	Flammable Poison	40 ppm (TWA)	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Methanol (Methyl Alcohol)	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hydrochloric Acid	Corrosive Poison	5 ppm- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.
TWA – Time Weighted Average

6.0 EQUIPMENT AND SUPPLIES

- 6.1. Labware is cleaned to remove any residual organics. Please refer to SOP ST-OP-0001 for detailed glassware cleaning procedures.
- 6.2. Temperature controlled ultrasonic bath
- 6.3. Vortex mixer
- 6.4. Muffle Oven
- 6.5. Analytical balance
- 6.6. 10- μ L, 25- μ L, 50- μ L, 100- μ L, 500- μ L, 1000- μ L, 0.2-mL, 0.3-mL Hamilton liquid syringe or equivalent
- 6.7. Disposable cartridge filters – 0.45 μ m Teflon filter
- 6.8. SPE Cartridges (Resprep RDX, 500 mg/6 mL or Waters Porapak RDX, 500 mg/6mL) or equivalent
- 6.9. Volumetric flasks (Class A), 100-mL, 10-mL, 1000-mL with screw tops
- 6.10. Scintillation, 12-mL, or volatile vials, 40-mL, screw top with Teflon liners.
- 6.11. Mortar and Pestle
- 6.12. Puck Mill
- 6.13. Orbital shaker table.
- 6.14. Wipes- 4-ply rayon/polyester 4"x4" Avant Gauze PRM25444

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 HPLC grade water is used both for the instrumentation mobile phase as well as at the extraction level.
- 7.3 Acetonitrile, HPLC grade
- 7.4 Methanol, HPLC Grade
- 7.5 Sand, purified (JT Baker)
- 7.6 0.1M HCL

- 7.6.1 Add 10 mL of 12 M HCL to 1200 mL of HCLP grade water
- 7.7 Stock Standards
- 7.7.1 It is recommended that diluted standards be purchased as stock solution with a concentration no greater than 1000 mg/L (ppm).
- 7.7.2 Standard solutions may also be prepared using Standard Analytical Reference Materials (SARMs).
- 7.7.2.1 **NOTE: IF USING NEAT STANDARDS PLEASE NOTE: HMX, RDX, Tetryl, and 2,4,6-TNT are explosives and the neat material must be handled carefully. Drying at ambient temperature requires several days. DO NOT DRY AT HEATED TEMPERATURES.**
- 7.7.2.2 Place about 10 mg (weighed to the nearest 0.1 mg) of a single analyte into a 10 mL volumetric flask and dilute to volume with acetonitrile. Invert the flask several times until dissolved. Calculate the concentration of the stock solution from the actual weight used (nominal concentration = 1,000 mg/L).
- 7.7.2.3 Store at $< 6^{\circ}\text{C}$ and protect from light.
- 7.7.2.4 Stock standards must be replaced after six months or sooner if comparison with quality control indicates a problem.
- 7.7.2.5 Standard solution may also be purchased as prepared certified solutions. The vendor specified expiration dates will apply until ampoules are opened and mix is used.
- 7.8 Intermediate Stock Standard Solutions
- 7.8.1 Prepare an intermediate stock standard solution at 2000 $\mu\text{g/L}$ for low level and 20,000 for high level per analyte in acetonitrile.
NOTE: If the analytical column cannot achieve separation of 2,4-DNT and 2,6-DNT, it will be necessary to prepare a separate intermediate stock solution of 2,6-DNT.
- 7.8.2 Intermediate stock standards must be replaced after 30 days or sooner if comparison with quality control samples indicated a problem.
- 7.9 Surrogate Standards
- 7.9.1 1,2-Dinitrobenzene is the surrogate used for this method. The surrogate standards are prepared and stored in the same way as the calibration standards.

8.0 SAMPLE COLLECTION PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Aqueous samples should be collected in 1-L glass bottles and soil samples should be collected in 4-oz glass jars. Samples are protected from light and stored at $4 \pm 2^{\circ}\text{C}$. Sample extracts are stored at a temperature $< 0^{\circ}\text{C}$, protected from light and isolated from all potential contaminants and all standards.
- 8.3 Holding Times
- 8.3.1 Extraction is initiated within 7 days of the sampling date for aqueous samples, and 14 days of the collection date for solid and waste samples.

- 8.3.2 In the case, where drying/homogenization techniques and/or incremental sampling techniques are employed, the hold time is assessed from when the sample was collected in the field, . Homogenization and incremental sampling techniques are diverse and vary depending on the nature of the material, methodologies and project objectives. Please reference the project's Client Requirements Memorandum regarding compositing and hold times.

9.0 QUALITY CONTROL

9.1. Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS) and Matrix Spike Duplicate (MSD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2 Method Blank

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 Surrogates are spiked into the method blank at the same level as the samples.
- 9.2.4 Aqueous method Blanks use 500 mL of HPLC water spiked with the surrogates.
- 9.2.5 Solid method blanks use either 2 or 10 g of muffled sand spiked with the surrogates depending on method variation.
- 9.2.6 Wipe method blanks use blank wipes.

9.3 Laboratory Control Sample

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.
- 9.3.3 Aqueous LCS use 500 mL of HPLC water fortified with the organic analytes of interest, including surrogates.
- 9.3.4 Solid LCS use either 2 or 10 g of muffled sand fortified with the organic analytes of interest, including surrogates, depending on method variation.
- 9.3.5 Wipe LCS use blank wipe fortified with the organic analytes of interest, including surrogates, depending on method variation.

9.4 Matrix Spike (MS) /Matrix Spike Duplicate (MSD)

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.
- 9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.
- 9.4.4 If there is insufficient sample to perform an MS/MSD, a duplicate LCS is

analyzed. An NCM is written to document the insufficient volume and the utilization of an LCSD to demonstrate precision.

- 9.4.5 There is no MS/MSD for wipe sample batches due to the nature of the sample. A LCS/LCSD will be performed to demonstrate precision.

9.5 Surrogates

- 9.5.1 Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.
- 9.5.2 Each applicable sample, blank, LCS and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.6 Procedural Variations/ Nonconformance and Corrective Action

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Balance calibration must be checked daily when used. Refer to SOP ST-QA-0005.
- 10.2 Thermometers must be checked as prescribed in SOP ST-QA-0005.
- 10.3 See the analysis of SOPs: ST-LC-0002 for instrument calibration requirements.

11.0 PROCEDURE

- 11.1 Solid phase extraction (SPE) for aqueous samples
- 11.1.1 Measure 500 mL of sample gravimetrically and record the pH.
- 11.1.2 Add the appropriate amount of surrogate based on the middle range of the initial calibration. If the sample is an LCS or MS/MSD, add the appropriate amount of spike and surrogate based on the mid level of the ICAL. Do not agitate the contents of the container while pouring the 500 mL.
- 11.1.2.1 If a sample is known or suspected (based upon client knowledge, project scope, or site history) to have a high density (>1.2 g/mL, e.g. a brine or waste) or a low density (<0.98 g/mL, e.g. mixed solvent), the sample density will be measured and the volume determined arithmetically (sample mass divided by the density equals the volume).
- 11.1.2.2 Volume is calculated by gravimetric determination assuming a sample density of 1. Samples that are not aqueous, or suspected of having a density greater than 1.2, will have aliquots taken for density analysis to correct volume for density.
- 11.1.3 Prior to conditioning the cartridges, the connector caps, straws and PTFE tubing thoroughly rinsed with methanol and then acetonitrile.
- 11.1.4 Adjust the flow rate through the solid-phase extraction cartridge to approximately 10 mL/min with the vacuum pressure. It is important not to allow the cartridge to go dry at any point during the conditioning stage or during the addition of sample to the cartridge.
- 11.1.5 Precondition the RDX cartridge with acetonitrile.

- 11.1.6 Condition the cartridge with **HPLC grade** water.
 - 11.1.7 Begin dripping the sample through the solid phase extraction cartridge at a rate of approximately 10 mL/min. Do not pre-filter the sample unless the sample is suspected to cause clogging. Do not let the solid phase extraction tube go completely dry during the running of the sample through the cartridge. If the tube clogs after sample loading, measure the amount of the sample that was successfully filtered and use that volume for the extraction constant. A cartridge is considered clogged when there can be no more sample drawn through it.
 - 11.1.8 When there is 10 to 20 mL of sample left in the container, use 5 to 10 mL **HPLC grade** water to rinse the interior of the cartridge in order to insure that all the contents have been removed.
 - 11.1.9 If the tube is not clogged, rinse the column with 10 mL HPLC grade water. After rinsing the cartridge allow any excess water to be drawn through. The cartridge is then dried with nitrogen.
Note: After extraction is finished the cartridge must be dry in order to insure that no water is being carried over into the final extract.
 - 11.1.10 Put the sample collection vials in the rack and slowly elute with acetonitrile into a 4mL amber vial by pulsing the pipette bulb once and adjust eluent to a final volume of 2.5 mL allowing the acetonitrile to be pulled by gravity (this may take several minutes).
- 11.2 Extraction of Soil/Sediment Samples Method Modified 8330B
- 11.2.1 Allow sample to air dry at least 18 hours. After drying, weigh the sample until constant mass is achieved. The period in between measurements may not be less than one hour.
 - 11.2.2 See SOP ST-QA-0038 for the procedure for sub-sampling.
 - 11.2.3 Place a 2.0 g sub-sample of each soil sample in a 40 mL glass vial. Add the appropriate amount of surrogate based on the middle range of the initial calibration. If the sample is an LCS or MS/MSD, add the appropriate amount of spike and surrogate based on the mid level of the ICAL. Bring to 10.0mL with acetonitrile. Vortex swirl for one minute. Place in an ultrasound bath for 18 ± 2 hours or an orbital shaker table for 18 ± 2 hours.
 - 11.2.4 After extraction remove from the bath or shaker table and centrifuge for 15 minutes if necessary.
 - 11.2.5 Place extract in a syringe and filter through a 0.45 μ m Teflon filter. Discard the first 3 mL of filtrate and retain the remainder in a Teflon capped vial for HPLC analysis.
- 11.3 Extraction of Soil/Sediment Samples Method 8330B (ISM and Pantex)
- 11.3.1 See SOP ST-QA-0038 for the procedure for sub-sampling.
 - 11.3.2 Place a 10.0 g sub-sample of each soil sample in a 2 oz wide mouth bottle. Add the appropriate amount of surrogate based on the middle range of the initial calibration. If the sample is an LCS or MS/MSD, add the appropriate amount of spike and surrogate based on the mid level of the ICAL. Bring to 20.0 mL with acetonitrile, cap with Teflon-lined cap, vortex swirl for one minute, and place in a cooled ultrasonic bath for 18 ± 2 hours or on an orbital shaker table for 18 ± 2 hours.
 - 11.3.3 After extraction remove from the bath or shaker table and centrifuge for 15 minutes if necessary.
 - 11.3.4 Place extract in a syringe and filter through a 0.45 μ m Teflon filter. Discard the first 3 mL of filtrate and retain the remainder in a Teflon capped vial for HPLC analysis.
- 11.4 Extraction of Wipe Samples Method 8330B

- 11.4.1 Place wipe into a 50-mL polypropylene centrifuge tube (or appropriately sized jar). Add about 10 mL of acetonitrile (or enough to submerge the wipe). Add the appropriate amount of surrogate, based on the middle range of the initial calibration (for an LCS or MS/MSD, add the appropriate amount of spike). Vortex samples for half of an hour.
- 11.4.2 Place in an orbital shaker for 1 hour. For each sample, squeeze the wipe three times.
- 11.4.3 Repeat step 11.4.2 one more time.
- 11.4.4 Vortex each sample for 10 seconds.
- 11.4.5 Transfer the extract into a syringe and filter through a 0.45 µm Teflon filter. Discard the first 3 mL of filtrate and retain the remaining sample in a Teflon capped vial for HPLC analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 Calculations are not applicable for this SOP.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 Data assessment does not pertain to this sample preparation procedure.
- 13.2 Samples requiring re-preparation are submitted to the preparation lab with an NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1. Method performance data, Reporting Limits, and QC acceptance limits, are given in the associated analytical SOP
- 14.2. Demonstration of Capability
 - 14.2.1. Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3. Training Qualification
 - 14.3.1. The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for

laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
The following waste streams are produced when this method is carried out:
- Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash.
 - Solid materials (soil, gloves, soiled paper products) are collected in the container labeled "Non-Regulated Material" for disposal.
 - If the labware was used for the analysis of radioactive samples and contains radioactivity at level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.
 - Solvent rinses from the labware cleaning procedure are disposed of in the flammable solvent waste stream.

17.0 REFERENCES

- 17.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW846 February 2007 Method 8330A.
- 17.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, March 2003 Section 8000C.
- 17.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, October 2006 Method 8330B.
- 17.4 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, February 2007 Method 3535A.
- 17.5 St. Louis Quality Assurance Manual (ST-QAM), current revision
- 17.6 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.7 Associated SOPs, current revisions:
- 17.7.1 ST-OP-0001, Labware Preparation for Organic Analysis
 - 17.7.2 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.7.3 ST-QA-0002, "Standards and Reagent Preparation."
 - 17.7.4 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes"
 - 17.7.5 ST-QA-0036, "Non-Conformance Memorandum (NCM) Process"
 - 17.7.6 ST-LC-0002, "Analysis of Nitroaromatic and Nitroamine Explosives by HPLC."

17.7.7 ST-QA-0038, "Procedure for Compositing and Sub sampling."

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.
- 18.2 Aqueous sample volumes are determined by weight.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1. Updated section 9.2 regarding amounts of reagent water used with surrogates and also, updated the amount muffled sand used with surrogates.
- 19.2. 12/23/2010; REV5, Updated section 11.2.3 regarding ultrasound times.
- 19.3. Rev 6:
 - 19.3.1. Updated dilution information in section 2.1.
 - 19.3.2. Added shaker table to equipment list.
 - 19.3.3. Updated spike and surrogate amounts in section 11.
 - 19.3.4. Added cartridge conditioning information to section 11.1.
 - 19.3.5. Added constant weight determination to section 11.2 and 11.3.
- 19.4. Rev 7:
 - 19.4.1. Through-out: removed references to Clouseau and Quantims – replaced with LIMS
 - 19.4.2. Updated volume of intermediate stock standard solution from 5000ug/L to 2000 ug/L in Section 7.11
 - 19.4.3. Removed references to tables no longer in SOP from Section 7.12
 - 19.4.4. Added MDNA stock standard information to Section 7.13
 - 19.4.5. Updated size of vial used in soil prep (Section 11.2.3)
 - 19.4.6. Removed specific amount of surrogate to add in Section 11.4.4 as amount depends upon concentration
 - 19.4.7. grammatical and spelling errors corrected
 - 19.4.8. added grinding information for 8330B – altered language
 - 19.4.9. § 11.1.1.1.: Addition of density limits
 - 19.4.10. § 11.1.1.2.: Addition of volume calculation by gravimetric determination
- 19.5. Rev 8:
 - 19.5.1. Grammatical errors corrected through out
 - 19.5.2. Added §2.1.3, reference to wipe samples
 - 19.5.3. Added §7.0, wipes
 - 19.5.4. Added §9.0, material used for MB, LCS and MS/MSD
 - 19.5.5. Added §11.0, procedure for processing wipes
- 19.6. Rev 9:
 - 19.6.1. Updated §8.0
- 19.7. Rev. 10 (05/21/2014)
 - 19.7.1. Annual review, no changes.
- 19.8. Rev.11 (06/22/2015)
 - 19.8.1. Updated Method Summary. Sec. 2.1
 - 19.8.2. Updated Reagents, Sec. 7.0
 - 19.8.3. Added method references to title and section 2.
- 19.9. Rev. 12 (5/23/16)
 - 19.9.1. Updated title, Sec. 11.2.
 - 19.9.2. Updated Sec. 11.3, to continue after ST-QA-0083.
 - 19.9.3. Removed references to LCMSMS analysis and Method 8321A

19.9.4. Clarified holdintimes in Section 8.3

**Title: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)
SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP)
AND THE CALIFORNIA WET LEACH PROCEDURE (CWET)**

Approvals (Signature/Date):

 10/25/16

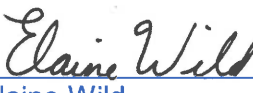
Sarah Bernsen Date
Pre-prep Manager

 10/24/16

Michael Ridenhower Date
Health & Safety Manager / Coordinator

 10/25/16

Kristen Ely Date
Quality Assurance Manager

 10/24/16

Elaine Wild Date
Laboratory Director

This SOP was previously identified as SOP No. ST-OP-0009 Rev. 6

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes the application of the Toxicity Characteristic Leaching Procedure (TCLP), SW-846 Method 1311.
 - 1.1.1 The Toxicity Characteristic (TC) of a waste material is established by determining the levels of 8 metals and 31 organic chemicals in the aqueous leachate of a waste. The TC is one of four criteria in 40 CFR Part 261 to determine whether a solid waste is classified as a hazardous waste. The other three are corrosivity, reactivity and ignitability. The TC Rule utilizes the TCLP method to generate the leachate under controlled conditions which were designed to simulate leaching through a landfill. EPA's "worst case" waste disposal model assumes mismanaged wastes will be exposed to leaching by the acidic fluids generated in municipal landfills. The EPA's model also assumes the acid/base characteristics of the waste will be dominated by the landfill fluids. The TCLP procedure directs the testing laboratory to use a more acidic leaching fluid if the sample is an alkaline waste, again in keeping with the model's assumption that the acid fluids will dominate leaching chemistry over time.
- 1.2 This SOP also describes the application of the Synthetic Precipitation Leaching Procedure (SPLP) which was designed to simulate the leaching that would occur if a waste was disposed in a landfill and exposed only to percolating rain water. The procedure is based on SW846 Method 1312.
 - 1.2.1 The list of analytes for SPLP may extend beyond the toxicity characteristic compounds shown in Appendix A. With the exception of the use of a modified extraction fluid, the SPLP and TCLP protocols are essentially equivalent. Where slight differences may exist between the SPLP and TCLP they are distinguished within this SOP.
- 1.3 This SOP describes the procedure for obtaining a leachate of solid, liquid, or multiphasic samples, as provided by the California Title 22, Waste Extraction Test (WET).
 - 1.3.1 The leachate procedure is intended to determine the mobility of both organic and inorganic analytes. However, this SOP covers only the application of this procedure for inorganic analytes.
- 1.4 Results obtained are highly dependent on the pH of the extracting solution, the length of time that the sample is exposed to the extracting solution, the temperature during extraction, and the particle size/surface area of the sample. These parameters must be carefully controlled.
- 1.5 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1 For liquid wastes that contain less than 0.5% dry solid material, the waste, after filtration through 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP leachate.
- 2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solids and stored for later analysis. The particle size of the remaining solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. For TCLP, the extraction fluid employed for extraction of non-volatile analytes is a function of the alkalinity of the solid phase of the waste. For SPLP, the extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater the extraction fluid employed is a pH 4.2 solution. Two leachates may be generated: a) one for analysis of non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals and/or b) one from a Zero Headspace Extractor (ZHE) for analysis of volatile organic constituents. Following extraction, the liquid leachate is separated from the solid phase by filtration through a 0.6 to 0.8 μm fiber filter.

- 2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid leachate and these are prepared and analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (QAM) a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 “Leachate” is used to refer to the TCLP solution generated from this procedure.
- 3.3 “Percent Wet Solids” is that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

4.0 INTERFERENCES

- 4.1 Oily wastes may present unusual filtration and drying problems. As recommended by EPA (see Figure 3), oily wastes that are unfilterable will be assumed to be 100% liquid and *with client approval* the laboratory will perform a total constituent analysis in lieu of the TCLP extraction. Note that per Section 1.2 of method 1311 the results of the total analysis may be divided by 20 to convert the total results into the “maximum leachable concentration.” This applies specifically to samples containing viscous non-aqueous liquids that would be difficult to filter.

NOTE: Discussion with client regarding the substitution of total analysis for TCLP and the conversion factor of 20 to determine “maximum leachable concentration” must be documented and included in the job’s Case Narrative.

If total analysis is performed, a density measurement is needed in order to convert results from a weight basis (mg/Kg) to a volume basis (mg/L) in order to compare results to the limits that are expressed in mg/L. The laboratory should explain to the client that the total results divided by 20 represent the “maximum leachable concentration,” and those are the values that should be compared to the Toxicity Characteristic Limits.

- 4.2 Wastes containing free organic liquids (i.e., those with separable non-aqueous liquid phases) will be assumed to be 100% liquid and totals analysis will be performed to determine if the oil exceeds TCLP limits.
- 4.3 Solvents, reagents, glassware and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.
- 4.4 Glassware and equipment contamination may result in analyte degradation. Soap residue on glassware and equipment may contribute to this. All glassware and equipment should be rinsed very carefully to avoid this problem.
- 4.5 Phthalates may be eliminated by proper glassware cleanup and by avoiding plastics. Only glass, Teflon or Type 316 stainless steel tumblers may be used for leachates to be analyzed for organics. Plastic tumblers may be used for leachates to be analyzed for the metals.
- 4.6 Overexposure of the sample to the environment will result in the loss of volatile components.

- 4.7 Glass fiber filters must be pre-cleaned with 1:1 nitric acid before use with leachates to be analyzed for the metals.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

- 5.1.1 None.

5.2 PRIMARY MATERIALS USED

- 5.2.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid	Corrosive Poison Flammable	10 ppm (TWA)	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Sodium Hydroxide	Corrosive	2 Mg/M3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
STEL – Short Term Exposure Limit			
TWA – Time Weighted Average			
Ceiling – At no time should this limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Extraction vessels

6.1.1 For volatile analytes - zero-headspace extraction (ZHE) vessel, gas-pressure actuated, Millipore YT3009OHW or equivalent (see Figure 2).

6.1.1.1 ZHE Cleaning Procedure

6.1.1.1.1 Clean all parts, thoroughly, in warm, soap water using a brush and/or steel wool. Rinse completely with tap water followed by DI water. Set aside any plastic parts.

6.1.1.1.2 If the ZHE is suspected to be highly contaminated based on visual examination, screening information and/or technical judgment, proceed with these additional steps.

6.1.1.1.2.1 Completely disassemble the ZHE including all valves, pressure gauge and Teflon tape.

6.1.1.1.2.2 Place all stainless steel parts in a 200°C oven for 3 hours, minimum.

6.1.1.1.2.3 Remove parts from oven and allow to air cool for 1 hour, minimum, and rinse with DI water.

6.1.1.1.2.4 Reassemble using new O-rings and Teflon tape.

6.1.1.2 ZHE Leak Testing

6.1.1.2.1 The ZHE should be checked for leaks after every extraction.

6.1.1.2.1.1 If device has a built in pressure gauge, pressurize the device to 50 psi and allow to stand for one hour. Recheck the pressure.

6.1.1.2.1.2 If the device does not have a built in gauge, pressurize to 50 psi and submerge in water. Check for the presence of escaping air bubbles.

6.1.1.2.1.3 If pressure is lost, check all fittings and inspect or replace O-rings as needed.

6.1.1.2.1.4 Re-test the device.

6.1.2 For metals - 2 L HDPE (Nalgene or equivalent) bottles may be used.

6.2 Vacuum filtration apparatus

6.2.1 Cleaning procedure

6.2.1.1 Clean all parts, thoroughly, in warm, soap water using a brush. Rinse completely with tap water followed by DI water.

6.3 Borosilicate glass fiber filters, 0.6 - 0.8 µm (Whatman GF/F 14.2 cm, 0.7 µm or equivalent). When analyzing for metals, wash the filters with 1 N nitric acid and de-ionized water prior to use, or purchase filters that are pre-washed with acid. Glass fiber filters are fragile and should be handled with care.

- 6.4 Rotary agitation apparatus, multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent (see Figure 1). The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.
- 6.5 ZHE Extract Collection Devices are used to collect the initial liquid phase and the final extract of the waste from the ZHE device, either of the following may be used:
 - 6.5.1 Gas-tight syringes, 25 mL, Hamilton 0158330 or equivalent, or Tedlar bags
- 6.6 Top loading balance (all measurements are to be within ± 0.1 grams).
- 6.7 pH meter and probe capable of reading to the nearest 0.01 unit, and with automatic temperature compensation; pH strips
- 6.8 Magnetic stirrer/hotplate and 1.5 inch magnetic stirring bars.
- 6.9 VOA vials, 20 mL, with caps and septa
- 6.10 HDPE plastic bottles, 2 liter.
- 6.11 Glass fiber filters (pre-filters), acid washed with 1:1 nitric, then rinsed with copious amounts of deionized water.
- 6.12 Sieve, No. 10
- 6.13 Aluminum Weigh Dishes (200g)

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 Reagent water for non-volatile constituents must be produced by a Millipore DI system or equivalent. For volatile constituents, water must be passed through an activated carbon filter bed (Milli-Q or tap water passed through activated carbon). Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
- 7.3 Hydrochloric acid, 1 N: Carefully add 83 mL concentrated reagent grade HCl to 800 mL reagent water, cool and dilute to 1 liter with reagent water. Cap and shake to mix well.
- 7.4 Nitric acid, 1 N: Carefully add 64 mL concentrated reagent grade HNO₃ to 800 mL of reagent water, cool and dilute to 1 liter with reagent water. Cap and shake to mix well.
- 7.5 Sodium hydroxide, 5N and 10 N
- 7.6 Acetic acid, glacial: concentrated, reagent grade liquid (HOAc).
- 7.7 pH calibration solutions: buffered to a pH of 2, 4, 7, and 10. Commercially available.
- 7.8 Citric acid, anhydrous, ACS reagent grade
- 7.9 TCLP Leaching Fluids

- 7.9.1 The pH of both solutions listed below shall be monitored daily and the pH probes are to be calibrated on a daily basis.
- 7.9.1.1 The leaching fluids MUST be prepared carefully. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching. If the pH is not within the specifications, the fluid must be discarded and fresh extraction fluid prepared.
- 7.9.1.2 Additional volumes of extraction fluids listed above may be prepared by multiplying the amounts of acetic acid and NaOH by the number of liters of extraction fluid required.
- 7.9.2 TCLP Fluid #1: Carefully add 250 mL glacial acetic acid and 290 mL of 10 N NaOH to 45 L reagent water in a 45 liter carboy. Cap and shake to mix well. The pH of this solution is 4.93 ± 0.05 .
- 7.9.2.1 Alternatively, add 11 mL of acetic acid and 26.8 mL of 5N NaOH to 2000 g of reagent water.
- 7.9.2.1.1 Fill the extraction vessel to approximately 1900 g of reagent water, add 26.8 mL 5N NaOH and 11 mL of acetic acid then bring to a 2000 g final weight with reagent water.
- 7.9.3 TCLP Fluid #2: Carefully add 250 mL glacial acetic acid to 45 L reagent water in a 45 liter carboy. Cap and shake to mix well. The pH of this solution is 2.88 ± 0.05 .
- 7.9.3.1 Alternatively, add 11 mL acetic acid to 2 L reagent water.
- 7.9.3.1.1 Fill the extraction vessel to approximately 1900 g of reagent water, add 11 mL of acetic acid then bring to a 2000 g final weight with reagent water.
- 7.10 Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated HNO_3 to 500 mL reagent water. Cap and shake to mix well.
- 7.11 Sulfuric acid / nitric acid (60/40 weight percent mixture) $\text{H}_2\text{SO}_4/\text{HNO}_3$. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid. A more dilute acid mixture may be prepared, if desired. (1000X dilution is recommended)
- 7.12 SPLP Leaching fluids
- 7.12.1 SPLP solutions are unbuffered and exact pH may not be attained. The pH of TCLP and SPLP fluids should be checked prior to use. If not within specifications, the fluid should be discarded and fresh fluid prepared.
- 7.12.2 SPLP fluid #1: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 4.20 ± 0.05 . This fluid is used for soils from a site that is east of the Mississippi River and for wastes and wastewaters.
- 7.12.3 SPLP fluid #2: Add 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05 . This fluid is used for soils from a site that is west of the Mississippi River.
- 7.12.4 SPLP fluid #3: This fluid is reagent water and is used for leaching of volatiles. Additionally, any cyanide-containing waste or soil is leached with fluid #3 because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.
- 7.13 CWET Leaching fluid
- 7.13.1 0.2 M Sodium Citrate Extraction Fluid. Add 18L of DI water to a 20 liter carboy. Carefully add 924 g of anhydrous citric acid and 1950 ml of 5N NaOH. Place on a magnetic stirrer and stir until all the solids dissolve. Measure the pH and adjust until the pH is 5.0 ± 0.1 . Alternatively, the fluid may be purchased. The pH of the fluid must be checked and recorded daily prior to use.

8.0 SAMPLE COLLECTION, PRESERVATION, STORAGE AND SHIPMENT

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Samples being analyzed for non-volatile organic compounds should be collected and stored in glass containers with Teflon lid liners. Chemical preservatives shall NOT be added UNTIL AFTER leachate generation.
- 8.3 Samples being analyzed for metals only can be collected in either glass or polyethylene containers.
- 8.4 When the waste is to be evaluated for volatile analytes, care should be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon lined septum capped vials with minimal headspace and stored at 4 ± 2 °C). Samples should be opened only immediately prior to extraction. Samples being analyzed for volatile organics do not require preservation.
- 8.5 Samples should be refrigerated to 4 ± 2 °C unless refrigeration results in irreversible physical changes to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.
- 8.6 The minimum TCLP sample collection size is determined by the physical state or states of the waste and the analytes of concern. The amount of waste required varies with the percent solids. The lower the percent solids, the more waste will be required for preliminary and final testing. For aqueous samples containing between 0.5 and 10% solids, several kilograms of sample are required to complete the analyses. The general minimal requirements when the samples are 100% solids include: 1 - 32 oz jar for semi-volatile organic analysis and metals, and 1 - 4 oz jar for volatile organic analysis. Low density sample materials, such as rags or vegetation, will require larger volumes of sample. For liquid samples (less than 50% solids) the minimum requirements are 2 - 32 oz jars for semi-volatile organic analysis and metals, and 2 - 8 oz jars for volatile organic analysis. If volatile organic analysis is the only requested parameter, 2 separate jars are required. If matrix spike or duplicate control samples are requested, additional sample volume is required. If sufficient sample volumes were not received, analyses cannot be started and the client should be notified as soon as possible.
- 8.7 TCLP leachates should be prepared for analysis and analyzed as soon as possible following extraction. Leachates or portions of leachates for metallic analyte determinations must be acidified with nitric acid to a pH less than 2, unless precipitation occurs. **If precipitation occurs upon addition of nitric acid to a small aliquot of the leachate, then the remaining portion of the leachate shall not be acidified** and the leachate shall be analyzed as soon as possible. All other leachates should be stored under refrigeration (4 ± 2 °C) until analyzed.
- 8.8 Samples are subject to appropriate treatment within the following time periods:

Table 1 – Holding Times (days)

Parameter	Collection to Leach	Leach to Preparation	Preparation to Analysis	Total Elapsed Time
Volatiles:	14	N/A	14	28
Extractable Organics:	14	7	40	61
Mercury:	28	N/A	28	56
Other Metals:	180	N/A	180	360

NOTE: The initial holding time is measured from date of collection to date extraction started, and subsequent holding times are measured from the date extraction starts. If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding holding times is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory limit.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 A batch consists of environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
- 9.1.2 Each LEACHATE batch may contain up to 20 environmental samples, a method blank for each fluid.

9.2 Method Blank

- 9.2.1 A Method Blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.2.2 One leachate (method) blank must be prepared and leached for each leachate batch.
- 9.2.3 One leachate blank is prepared for each leachate fluid used. The leachate blank must consist of the same extraction fluid as used for the samples or reagent water if only filtration of the sample(s) was required.

9.3 LCS and MS/MSD

- 9.3.1 For metals analysis, a MS/MSD is spiked after leaching but prior to preservation and submittal for metals sample preparation. See [Table 1](#) and [Table 2](#).
- 9.3.2 All Laboratory Control Samples (LCS) and Organic MS/MSD samples are initiated in inorganic and organic sample preparation procedures and not included in this SOP. See SOPs: ST-IP-0013 (ICP metals digestion), ST-MT-0005 (mercury), ST-OP-0002 (pesticides and semivolatile extraction), and ST-OP-0007 (herbicide extraction)

9.4 Procedural Variations

- 9.4.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

9.5 Nonconformance and Corrective Action

- 9.5.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Sample analysis instrumentation calibration requirements are found in the respective analytical SOPs: ST-MT-0003 (metals by ICP), ST-MT-0005 (mercury), STL-GC-0016 (pesticides), ST-GC-0017 (herbicides), ST-MS-0001 (Semivolatiles) and ST-MS-0002 (Volatiles).

- 10.2 The rotation devices must be checked once a month for proper rotation speed. Count the number of rotations in exactly 60-seconds. The number of rotations must be between 28 and 32, or corrective action is required. Record the result in a logbook.
- 10.3 Calibrate the pH meter daily when used to bracket the pH being measured. The pH meter is calibrated in accordance with SOP: ST-WC-0011.
- 10.4 Calibrate the top loading balance with weights bracketing the targeted weights. See SOP, ST-QA-0005, for details regarding balance calibration and documentation.

11.0 PROCEDURE

- 11.1 Preliminary evaluations (percent solids, particle size, selection of extraction fluid, and fluid/leachate compatibility)
- 11.1.1 Preliminary evaluations are required to be done using a minimum of a 100 gram aliquot of waste. This aliquot may also undergo the actual leachate extraction for Non-volatiles ONLY IF it has NOT been oven dried. If the solid portion is oven dried, a separate aliquot must be used for the actual leaching procedure.
- 11.1.2 Determine the total volume of leachate (solid phase leachate + liquid filtrate) that needs to be generated for analysis according to the following:

Minimum Required Leachate Volume

Analysis	Required Volume (mL)
Volatiles	2 x 20
Semi-volatiles	200
Pesticides/ PCBs/Herbicides	100 each
Metals	100

- 11.1.2.1 If extractable organics are required on the same sample, a full 1 L volume must be prepared for each test requested.
- 11.1.2.2 For samples used for matrix spike and matrix spike duplicate analysis, three times the listed volumes are required.
- 11.1.3 Check the number of phases observed in the sample. It is common that when more than one container of multi-phasic materials is received from the field, each container will show different amounts of each phase.
- 11.1.3.1 If the sample has multiple phases and is received in more than 1 bottle then the contents of each bottle should be combined in a single larger container prior to processing the sample further. If this is not possible, then the alternate procedure described in the following section should be used.
- 11.1.4 Properly record the relative amounts of each phase by measuring the depth of the layers in each container after the contents have been allowed to settle. Determine the combined volume of each phase for all containers. Then mark the phase composition on a single container, mix thoroughly to obtain a representative subsample, and accurately measure the phase composition according to the following procedure. The two sets of values (combined volumes per phase and phase composition for one container) are used to determine the correct volume/mass adjustments on the final result. This procedure is not appropriate if testing will be done for volatile organic compounds.
- 11.2 Percent Solid Phase
- 11.2.1 **If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., it is 100% solid), then proceed to Section 11.4 (Particle-size Reduction).**

- 11.2.2 If the waste will obviously yield no solid when subjected to pressure filtration (i.e. it is 100% liquid), then proceed to Section 11.3.7.**
- 11.2.3 For TCLP and SPLP determinations:
- 11.2.3.1 Percent Solids and ZHE Extractions - The ZHE filtration apparatus cannot accurately determine percent **solids less than 5%**. If an extraction is to be performed solely for volatile organic compounds and the percent solids concentration is apparently greater than 5%, proceed to Section 11.7. (Procedure: ZHE Extraction Procedure, Volatile Constituents). Otherwise, continue with the steps in this section. The aliquot of sample used here cannot be used again for the ZHE extraction.
- 11.2.3.2 Weight of filter - Measure and record this value on the extraction record before loading the filter into the filter holder.
- 11.2.3.3 Assemble the filtration apparatus.
- 11.2.3.3.1 Homogenize the waste, transfer a minimum of a 100 g subsample to the weighing vessel. Measure and record the gross weight.
- 11.2.3.3.2 Determine the exact amount of sample transferred by weighing a beaker containing the sample before and after transfer of the sample.
- 11.2.3.4 Transfer the sample to the filtration device attempting to spread the waste sample evenly over the surface of the filter. Measure and record the tare weight of the empty weighing vessel and any residual sample.
- 11.2.3.5 Calculate and record the net weight of sample used for testing.
- 11.2.4 For CWET determinations
- NOTE:** Percent solids for WET is not the same as the percent solids normally referred to in other extractions and is not at all related to the percent moisture. Here, the percent solids is the dried fraction of the sample from which no liquid may be forced by applied pressure.
- 11.2.4.1 If visual inspection indicated that the sample will yield no liquid upon filtration, the sample is declared to be 100% solids.
- 11.2.4.2 If the sample is liquid or multiphasic, filtration must be used to determine the percent solids.
- 11.2.4.3 Record the weight of the filter and pre-filter in the laboratory notebook. Securely assemble the filter holder, screen, pre-filter, filter, and head onto the filtration unit. Leave off the screw-on cap on the filtration head.
- 11.2.4.4 Weigh a minimum 50-gram aliquot of the sample into a 250-ml beaker, after homogenizing the sample. If the percent solids are expected to be low, it may be advantageous to use a larger amount of the sample in order to obtain enough filtrate for subsequent metals analysis. Record the weight to the nearest 0.1 gram. Quantitatively transfer (>99%), the aliquot into the filter unit through the opening at the top of the filtration head using physical means only. Do not rinse with water. Tightly screw the cap onto the opening.
- 11.2.4.5 Collect the filtrate into a clean collection flask. Filtration stops when no additional filtrate in two-minute interval, is collected.
- 11.2.4.6 After filtration is complete, if liquid remains, it is considered part of the solids content since it would not filter.
- 11.2.4.7 Remove the screen with the pre-filter, filter and solids on it. If the solids are obviously greater than 0.5% by weight, it is not necessary to dry the filter and the weight of the pre-filter, filter and solids can be immediately determined. If it is not obvious transfer the screen, pre-filter, filter, and solids to the oven. Dry in the oven at 100 +/- 20 degrees C for approximately 1-hour. Remove from the oven and allow too cool. Using forceps transfer the pre-filter and filter onto the balance. Record the weight in the logbook. **Note:** if no liquid was obtained from the filtration, the sample is considered 100% solids, and it is not necessary to continue with drying the filter, etc.

11.2.4.8 Calculate and record the percent solids in the logbook. If the percent solid is < 0.5%, the sample is considered 100% liquid, otherwise, the sample is multiphasic. If the solids were not dried, they may be used to obtain the leachate, which must be combined with the original filtrate (if the liquids are compatible). If the solids were dried, and the percent solids is less than or equal to 0.5%, then a fresh aliquot of waste must be filtered to obtain solids to be leached.

11.3 Filtration for percent solids

11.3.1 Slowly apply gentle pressure of 10 psi to the filtration apparatus. Allow the sample to filter until no SIGNIFICANT additional liquid has passed through the filter during a 2 minute period.

11.3.2 Repeat previous step by increasing the pressure in 10 psi increments until a maximum of 50 psi is reached. Stop the filtration when no additional filtrate is generated within a 2 minute period.

NOTE: Some samples will contain liquid material that does not filter (e.g., oil). Do not attempt to filter the sample again by exchanging filters. Viscous oils or any wastes which does not pass through the filter is classified as a solid.

11.3.3 Weight percent of dry solids (skip this step for oily samples).

NOTE: These steps are required only if it is noticed that a small amount of the filtrate is entrained in wetting of the filter and the percent wet solids (if measured) is $\geq 0.5\%$.

11.3.3.1 Remove the filter with the wet solids from the filtration apparatus.

11.3.3.2 Dry the filter and solid phase at $100 \pm 20^\circ \text{C}$.

11.3.3.3 Remove the filter from the oven and allow to cool in a desiccator.

11.3.3.4 Weigh and record the gross dry weight.

11.3.4 If not dried > 18 hours, repeat the drying step. Weigh and record the second gross dry weight. If the two weightings do not agree within 1%, perform additional drying and weighing until successive weightings agree within 1%.

11.3.5 Calculate and record the weight percent of dry solids.

11.3.6 If the dry solids result is $\geq 0.5\%$ and the sample will be extracted for non-volatile constituents, proceed to Section 11.4 (Particle Size Reduction) using a fresh wet portion of waste.

11.3.7 If the percent solids result is less than 0.5%, discard the solid phase. No leaching will be necessary; the filtrate is the TCLP leachate. Proceed to Section 11.6.11 for non-volatile constituents and Section 11.7.9.2 for volatile constituents.

11.4 Particle-size Reduction for Fluid Selection

11.4.1 For TCLP and SPLP determinations:

11.4.1.1 The subsample used for fluid selection must consist of particles less than 1 mm in diameter (versus the less than 1 cm requirement for the material used for the actual extraction). The method requires a smaller particle size to partially compensate for the shorter duration of contact time with the leachate solution as compared to the full extraction. Inappropriate use of coarser materials could result in the selection of the wrong fluid type.

11.4.1.2 Surface area exclusion - size reduction is not required if the sample surface area is greater than or equal to 3.1 cm^2 per gram., such as might be the case with pieces of paper, cloth, etc.

11.4.1.3 If the sample contains particles greater than 1 mm in diameter, crush, cut, or grind the solids to the required size. Note the type of particle size reduction performed on the extraction record.

11.4.2 For CWET determinations:

11.4.2.1 Determine if particle-size reduction is required by sieving the solids through a No. 10 sieve (unless it is obvious that all material will pass through the sieve). Material that does not pass through the sieve must be crushed, ground, etc. in order to pass through the sieve.

11.4.2.2 Perform any particle size reduction on the solid portion of the sample as necessary, for sample to pass through a no. 10 sieve.

11.4.2.2.1 For a multiphasic sample, quantitatively transfer the entire solids portion obtained, including filter, into an extraction bottle.

11.5 Determination of Appropriate Extraction Fluid

11.5.1 If the solid content is greater than or equal to 0.5%, and if the sample is being analyzed for metals or nonvolatile organic compounds, the type of leaching solution must be determined.

11.5.2 For SPLP, refer to Section 7.12 for fluid selection. Matrix type must be specified by the client. Check special instructions or see the project manager, then put a check mark by the fluid type selected.

11.5.3 For CWET use the equation in section 12.1.5, to determine the appropriate volume of 0.2-M sodium citrate solution to add to the extraction bottle. Add this volume to the extraction bottle.

11.5.4 The TCLP leaching fluid for all volatiles is Fluid #1.

11.5.5 For TCLP leach fluid determination for non-volatile analytes, continue with the following steps.

11.5.5.1 Weigh out a 5.0 ± 0.1 g subsample (less than 1 mm particle size) of the solid phase into a 150-mL disposable plastic bottles.

11.5.5.2 Add 96.5 ± 1.0 mL of reagent water, cover with a disposable watchglass, and stir with a 1.5 inch stirring bar for 5 minutes.

11.5.5.3 Measure and record the pH

11.5.5.4 If the pH is less than or equal to 5.0, use Fluid #1 and proceed to Section 11.5.6.

11.5.5.5 If the fluid pH is greater than 5.0, add 3.5 mL 1 N HCl, cover with a disposable watchglass. Slurry the sample briefly then transfer to a hotplate. Place a thermometer in sample, heat at 50°C - 55°C for 10 minutes.

NOTE: The heating cycle is a critical step. If the solid waste does not remain in contact with the acidic solution under specified time and temperature conditions, an erroneous pH may be measured.

11.5.5.6 Allow sample to cool until it reaches between 25°C (room temperature) and 30°C .

11.5.5.7 Slurry the sample briefly and let the solids settle.

11.5.5.8 Measure and record the pH using a pH meter.

11.5.5.9 If the pH is less than or equal to 5.0, use Fluid #1.

11.5.5.10 If the pH is greater than 5.0, use Fluid #2.

11.5.6 For samples requiring analysis for semi-volatile organics, pesticides, herbicides or metals proceed to Section 11.6.

11.5.7 For samples requiring analysis for volatile organics (ZHE), proceed to Section 11.7.

11.6 TCLP/SPLP Bottle Extraction Procedure: Non-Volatile Constituents

11.6.1 All masses should be recorded to the nearest 0.1 g.

11.6.2 The aliquot used in the Preliminary Evaluation MAY be used for this procedure ONLY if it was not oven dried. If the sample is 100% solid or if the preliminary aliquot was not oven dried proceed directly to Section 11.6.8 (Particle Size Reduction). If the Preliminary Evaluation aliquot was oven dried then, using a fresh aliquot of sample, continue as described in Sections 11.6.3 through 11.6.5.

11.6.3 Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 11.1.2.

11.6.4 Divide the total volume of solid phase leachate required by 20 to determine the mass of solid phase required for leaching. Round this mass UP to the nearest 5 g.

11.6.5 Repeat the steps outlined in Sections 11.2.3 through 11.3.2. (skipping CWET section inserts)

11.6.6 Determine and record the volume (mass) of the initial filtrate. Cover with aluminum foil or retain in a glass bottle for use.

11.6.7 Determine and record the "solid" phase mass by subtracting the mass of the liquid filtrate from the mass of the subsample.

- 11.6.8 Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e., capable of passing through a 9.5 mm, 0.375 inch, standard sieve). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm² per gram. If particle size reduction was required, record the type of reduction used onto the extraction record.
- 11.6.8.1 Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal. Note that size reduction to fine powder is not appropriate, and could invalidate results. If necessary, consult client for guidance.
- 11.6.9 Weigh the required mass of solid phase into an appropriate plastic bottle and **slowly** add 20 times its mass of appropriate leaching fluid as determined under Section 11.5 (e.g., 20 g of sample would require 400 g of leaching fluid). Record the weight of the sample aliquoted for the extraction and the amount of extraction fluid added.
- 11.6.10 Ensure any effervescence has stopped before capping the bottle tightly. Secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours. The temperature of the room should be 23 ± 2°C. The room temperature and time should be checked at both the start and end of the extraction and recorded on the extraction record.
- NOTE:** As agitation continues, pressure may build up within the bottle for some types of wastes. To relieve excessive pressure, the bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.
- 11.6.11 Remove the bottle and filter the sample using pressure filtration by filtering through a new glass fiber filter. For final filtration of the TCLP leachate, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filters must be acid washed if metals are to be determined (see Section 6.3). The entire sample need not be filtered unless the sample yielded an initial liquid phase that needs to be combined with this filtrate; however, sufficient volume should be generated to support the required analyses.
- 11.6.12 If the waste contained no initial filtrate, this solution is defined as the leachate.
- 11.6.13 If the waste did yield an initial filtrate, and if this filtrate and the initial filtrate are compatible, they are to be combined in the correct proportions (see Section 12.1.4) and mixed well. This combined solution is defined as the TCLP leachate.
- 11.6.14 If the individual phases are NOT miscible, they are to be prepared and analyzed separately and the results combined mathematically. See Section 12.1.5.
- 11.6.15 Measure and record the pH of the TCLP leachate using a pH meter.
- 11.6.16 Prepare subsamples for metals for MS/MSD quality control testing using the appropriate TCLP spiking solution (do not spike for organics). Refer to the appropriate determinative SOPs for further guidance on the spike components, levels and action criteria.
- 11.6.17 Immediately preserve the leachate as follows:
- 11.6.17.1 Metals to pH < 2 w/concentrated HNO₃ for non-oils (do not acidify oils)
- 11.6.17.1.1 If precipitate is observed upon addition of nitric acid to a small aliquot of the extract, the remaining portion of the extract for metals shall not be acidified and the extract shall be analyzed as soon as possible.
- 11.6.17.2 All others: Refrigerate to 4 ± 2 °C.
- NOTE:** Refer to Section 8.6 if precipitation occurs upon preservation.
- 11.6.18. Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis with copies of the TCLP preparation worksheets.
- 11.7. TCLP/SPLP ZHE Extraction Procedure: Volatile Constituents
- 11.7.1 Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g. metals, pesticides, etc.).
- 11.7.2 Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized. Charge the ZHE with sample only once and do not open the device until the final extract has

been collected. Do not allow the waste, the initial liquid phase or the extract to be exposed to the atmosphere any longer than necessary.

- 11.7.3 If the TCLP extraction is for volatile components only, refer to Section 11.2.3.1 before proceeding.
- 11.7.4 All masses should be recorded to the nearest 0.01 g.
- 11.7.5 Partially assemble the ZHE apparatus.
- 11.7.6 Adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid or DI water if necessary).
- 11.7.7 If the preliminary evaluations indicated the need for particle size reduction, homogenize the waste, weigh out a sufficient size subsample and prepare for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size as measured with a ruler (Do NOT sieve the sample). Size reduction is not required if the sample surface area is greater than or equal to 3.1 cm² per gram. If particle size reduction was required record the type of reduction used on the extraction record.
NOTE: To minimize loss of volatiles, samples for volatiles that require particle size reduction should be kept in sample storage (at 4 °C) until immediately before size reduction. Aggressive reduction which would generate heat should be avoided and exposure of the waste to the atmosphere should be avoided to the extent possible. Size reduction to a fine powder is not appropriate.
 - 11.7.7.1 Scissors or shears may be used to cut cloth, plastic or sheet metal. Saws may be used for wood or solid metal.
- 11.7.8 Place a 200g Aluminum weigh dish on the balance and tare the balance.
- 11.7.9 Determine the appropriate size subsample to weigh using the percent solids information from Section 11.2
- 11.7.10 Homogenize and transfer an appropriate size sub-sample of the waste into the weigh dish and record the weigh in the extraction log.
- 11.7.11 If the waste is 100% solid, pour TCLP fluid number 1, in the amount of 20 times the aliquot's weights into the ZHE. **NOTE: Reserve approximately half the aliquot to wash off the weigh dish.**
- 11.7.12 Transfer the entire sample from the weigh dish into the ZHE.
- 11.7.13 Using the remainder of the ZHE fluid, rinse off the weigh dish into the ZHE.
- 11.7.14 Immediately place the glass fiber filter between the support screens, place the lid on the ZHE and to secure the ZHE tighten all the fittings.
 - 11.7.14.1 For wastes that are 100% solids, a 25 g sample is used.
 - 11.7.14.2 For wastes containing < 0.5% solids, the liquid portion of the waste, after filtration, is defined as the TCLP leachate. Filter enough of the sample to support all of the volatile analyses required.
 - 11.7.14.3 For wastes containing ≥ 0.5% and < 5.0% solids, a 500 g subsample of waste is recommended.
 - 11.7.14.4 If the sample has ≥ 5.0% solids, a smaller sample size can be determined using the equation in Section 12.1.2.
- 11.7.15 Homogenize and transfer an appropriate size subsample of the waste into the weigh dish and record the weight in the extraction log.
- 11.7.16 If the waste is 100% solid, pour TCLP fluid number 1, in the amount of 20 times the aliquot's weight, into the ZHE.
Note: Reserve approximately half the aliquot to wash off the weigh dish.
- 11.7.17 Transfer the entire sample from the weigh dish into the ZHE.
- 11.7.18 Using the remainder of the ZHE fluid, rinse off the weigh dish into the ZHE.
- 11.7.19 Immediately place the glass filter between the support screens, place the lid on the ZHE and to secure the ZHE tighten all the fittings.
- 11.7.20 Place the ZHE in an upright position; open both the gas AND liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.
- 11.7.21 If the waste is 100% solid, carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the

liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure. Proceed to Section 11.7.16.

- 11.7.22 If the waste is < 100% solids, proceed as follows:
- 11.7.22.1 Carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure.
 - 11.7.22.2 Attach a pre-weighed Tedlar bag to the liquid inlet/outlet valve and open the valve. Record the tare weight of the bag. A tared gas-tight syringe may also be used to collect the liquid phase.
 - 11.7.22.3 Carefully apply gas pressure of no more than 10 psi to force out the liquid phase. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a 2 minute period.
 - 11.7.22.4 Repeat previous step increasing the pressure in 10 PSI increments until 50 psi is reached and no significant liquid has passed in a 2 minute period. Remove the Tedlar bag or syringe and record the total weight of the container with filtrate. Close the gas-inlet valve and discontinue gas pressure. Retain the Tedlar bag or syringe with filtrate at 4° C for recombination with the leachate.
 - 11.7.22.5 If the original waste contained less than 0.5% solids (Section 11.2), this filtrate is defined as the TCLP leachate. Proceed to 11.7.23.
 - 11.7.22.6 If the original waste contained greater than 0.5% solids (Section 11.2), the material remaining in the ZHE is defined to be the “solid” phase. Calculate the weight of the solid phase using Equation 4.
 - 11.7.22.7 Based on the information from Sections 11.2, and using the formula in 12.1.3, determine the weight of fluid to load into the ZHE onto the “solid” phase.
 - 11.7.22.7.1 Load ZHE with 500 mL of Fluid #1
- 11.7.23 Check the ZHE to make sure all valves are closed and manually rotate the ZHE (end-over-end) 2 or 3 times. Reposition the ZHE in the vertical position.
- 11.7.24 Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.
- 11.7.25 Pressurize the ZHE to 30 psi. Allow to stand for 10 minutes, and then recheck the pressure. If the ZHE appears to be leaking, follow the corrective action protocols recommended by the manufacturer and repeat the analysis.
- 11.7.26 Place the ZHE in the rotary agitator. Record the initial ZHE pressure on the extraction record. Rotate at 28-32 rpm for 16-20 hours. Room temperature should be 23 ± 2 °C. The room temperature and time should be checked at both the start and end of the extraction and recorded on the extraction record.
- 11.7.27 Confirm that the pressure of 30 psi was maintained throughout the leaching. Record the final ZHE pressure on the extraction record. If it was NOT maintained, return to Section 11.7.1 and repeat the leachate with a new aliquot of sample.
- 11.7.28 Attach a syringe or Tedlar bag and open the liquid inlet/outlet valve to collect the aqueous leachate. If the filtrate is to be combined with an initial filtrate, record the volume/mass of the leachate and any oil phase.
- 11.7.28.1 If the waste contained an initial liquid phase, and the leachate is miscible with the initial liquid, the liquid may be filtered directly into the same collection device holding the initial liquid phase of the waste.
 - 11.7.28.2 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.
 - 11.7.28.3 A separate filtrate collection container must be used if combination would create multiple phases or there is not enough volume left within the filtrate collection container.

- 11.7.29 If the waste contained an initial filtrate (Section 11.7.15) that is miscible with the leachate, the leachate and the initial filtrate are directly recombined in the correct proportions. If the individual phases are NOT compatible, they are to be collected and analyzed separately.
- 11.7.30 Following collection, store the TCLP leachate in 2 20-mL unpreserved VOA vials with minimal headspace at 4 ± 2 °C and prepare for analysis as soon as possible using the appropriate organic extraction procedure.
- 11.7.31 If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.1.4.
- 11.8 CWET Leaching Procedure for the Solid Fraction
- 11.8.1 For a 100% solids sample, weigh out and quantitatively transfer a minimum of 50-grams of the sample into an extraction bottle. The extraction bottles shall be fitted with covered air scrubbers extended well into the extraction solution and flushed vigorously with nitrogen gas for 15 minutes so as to remove and exclude atmospheric oxygen from the extraction medium.
- 11.8.2 Secure the lid on the bottle and place the bottle in the rotator. Rotate the sample for a minimum of 48 hours (and a maximum of 50 hours) at 20-40 degrees C at 30 +/- 2 rpm. Record the initial room temperature in the logbook.
- NOTE:** It may be necessary to vent the extraction bottle periodically throughout the extraction (samples that contain carbonates may react with the acid to yield carbon dioxide gas). It is recommended to check the bottles for built-up gas after 15-30 minutes. If a build-up was observed, the bottle should be vented occasionally. If no build-up is observed, it is safe to let it rotate without further venting. Failure to vent the bottles may result in breakage of the bottle, with subsequent loss of the sample, and possible widespread radioactivity contamination.
- 11.8.3 Following the rotation period, record the room temperature, then stop the rotator and remove the bottle. If using a 1-liter plastic bottle, the bottle can be centrifuged at 2300 rpm for about 10-minutes, otherwise it is recommended to stand to allow the solids to settle in order to facilitate the filtering process. The method makes no specification on the maximum amount of time the bottle can stand before beginning the filtration process.
- 11.8.4 Filter the leachate using disposable 0.45 um filter or vacuum filtration apparatus. Record the lot number of the filter used on the TCLP prep-sheet.
- 11.8.5 If the leachate is from a 100% solids, the leachate is ready for preservation. If the leachate is from a multiphasic sample, the leachate should be physically recombined with the filtrate obtained from the pre-rotation filtration if the two are miscible. If the two are not miscible, record the volumes of each liquid, and report to the analytical labs that both must be analyzed separately. The results from the analyses must then be mathematically combined. Proceed with preservation.
- 11.8.6 CWET Preservation
- 11.8.6.1 Leachates to be analyzed for the metals must be preserved with nitric acid, so as to add an equivalence of 5% by volume concentrated nitric acid (*e.g.* for 100-mls of leachate, add 5-mls of concentrated nitric acid). Matrix spiking is performed prior to acidification. Measure out the appropriate volumes of leachate. Record the standard number, volume added and standard expiration date on the extraction record.
- 11.8.7 Analysis of the leachate proceeds according to the standard analytical procedures.
- NOTE:** Non-aqueous samples would normally be reported on a weight by weight basis; however, the WET regulatory limits are stated on a weight by volume basis. Therefore, aliquots on non-aqueous liquids for WET analysis should be measures by volume instead weight. For example, if the procedure calls for 1-gram of sample, measure 1-ml of sample instead.
- 11.9 TCLP Procedure for Determination of Major Cations
- 11.9.1 Record physical description of sample.
- 11.9.2 Mix sample thoroughly prior to taking aliquot.
- 11.9.3 Weigh 100g +/- 0.5g.

- 11.9.4 Add volume of DI water in mL to 20x weight in grams (100g - 2000mL).
- 11.9.5 Process a blank comprised of DI water only.
- 11.9.6 Agitate continuously for 18 hours (+/- 0.25hrs), tumbler must rotate at a rate of 29 r/mins.
- 11.9.7 Maintain room temperature at 18-27c°.
- 11.9.8 Record any physical changes in the sample and or leaching solution.
- 11.9.9 Allow sample to settle.
- 11.9.10 Filter through TCLP filter (7µm)
- 11.9.11 Filter again through 0.45µm filter.
- 11.9.12 Record pH
- 11.9.13 Preserve with HNO₃.
- 11.9.14 Sample is ready to be analyzed by ICP or ICPMS.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1. Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis QAM.

- 12.1.1 Calculation of Percent Wet Solids:

$$\text{Percent Wet Solids} = 100 \left(\frac{\text{Mass, "solid" phase}}{\text{Mass, initial subsample}} \right)$$

- 12.1.2 Calculation of weight of waste to charge to ZHE:

$$\text{Weight of waste to add to ZHE} = 100 \left(\frac{25}{\% \text{ wet solids}} \right)$$

- 12.1.3 Calculation of weight of extraction fluid to use:

$$\text{Weight of Extraction fluid} = \frac{20 \times \% \text{ wet solids} \times \text{weight of waste to be extracted}}{100}$$

TCLP/SPLP Calculation of volume of initial filtrate phase to recombine with solid phase leachate:

$$\text{Vol. filtrate for recombination} = \left(\frac{\text{Wt solids leached}}{\text{Total Wt solids}} \right) \left(\frac{\text{Leachate recovered}}{\text{Fluid added}} \right) (\text{Vol. of initial aq. filtrate})$$

$$\text{Vol. filtrate for recombination} = \left(\frac{\text{Wt solids leached}}{\text{Total Wt solids}} \right) \left(\frac{\text{Leachate recovered}}{\text{Fluid added}} \right) (\text{Vol. of initial aq. filtrate})$$

- 12.1.4 Mathematical recombination of analytical results:

$$\text{Final Analyte Concentration} = \frac{(V_1 \times C_1) + (V_2 \times C_2)}{V_1 + V_2}$$

V₁ = total volume of the initial filtrate phase (L).

C₁ = analyte concentration in initial filtrate phase (mg/L).

V₂ = volume of the theoretical solid phase leachate (L).

C₂ = analyte concentration in solid phase leachate (mg/L).

- 12.1 For CWETs Extraction Fluid Volume Requirement:

Volume = 0.10 x %solids x Wtw

Where:

Volume = volume of the extraction fluid to be added to the extraction vessel (mls)

%solids = Percent Solids (%)

Wtw = weight the waste filtered (grams)

12.2 REPORTING REQUIREMENTS

12.2.1 Follow these reporting conventions for multi-phase samples:

12.2.2 If both phases have positive results, use the values from each phase to calculate the recombined result. Use the reporting limit for each phase to calculate the recombined reporting limit.

12.2.2.1 If both phases are “ND,” not detected, the recombined result is “ND,” and the reporting limit is calculated from the reporting limit for each phase.

12.2.2.2 If one phase is “ND” and the other phase has a positive result, use the reporting limit for the “ND” phase and the positive value for the other phase to calculate the combined result. The combined reporting limit is based on the reporting limit for both phases. If the combined result is less than the combined reporting limit, then supply a footnote to indicate that “a positive result was detected below the calculated detection limit.”

12.2.3 Units - regardless of the nature of the sample, all results are reported in units of mg/L.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMs Nonconformance Memorandum (NCM) process. The NCM process is described in SOP ST-QA-0036.

13.2 Method Blanks: Leachate blanks which show contamination for the analytes of interest may require reextraction of the leachate batch (if positive detects were encountered), if the contamination cannot be traced to the analytical method’s preparatory procedure.

13.3 A NCM shall be generated for the following anomalies:

13.3.1 Sample weights less than the method requirement

13.3.2 Temperature excursions outside $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

13.3.3 Multiphasic samples, single phase oils, or samples that contain liquids that didn’t filter under test conditions.

13.4 All quality control measures described in the appropriate analytical SOPs shall be followed. Sample QC requirements are found in the respective analytical SOPs: ST-MT-0003 (metals by ICP), ST-MT-0005 (mercury), ST-GC-0016 (pesticides), ST-GC-0017 (herbicides), ST-MS-0001 (Semivolatiles) and ST-MS-0002 (Volatiles).

14.0 METHOD PERFORMANCE

14.1 Method performance data, Reporting Limits, MDLs, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the QAM.

- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 WASTE STREAMS PRODUCED BY THE METHOD
- 16.2.1 The following waste streams are produced when this method is carried out.
- 16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
- 16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992, SW-846 Final Update II. (Source method.)
- 17.2 Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, September 1994, SW-846 Final Update II. (Source method.)
- 17.3 Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990.
- 17.4 Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990.
- 17.5 Technical Background Document and Response To Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April, 1989.
- 17.6 TestAmerica Quality Assurance Manual (QAM), current revision
- 17.7 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.8 ASTM D3987-06, Standard Test Method for Shake Extraction of Solid Waste with Water1.
- 17.9 Associated SOPs
- 17.9.1 ST-PM-0002, Sample Receipt and Chain of Custody
- 17.9.2 ST-QA-0002, Standard and Reagent Preparation
- 17.9.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
- 17.9.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process

- 17.9.5 ST-OP-0002, Extraction and Cleanup of Organic Compounds from Water and Soils, Based on SW-846 3500 Series, 3600 Series, and 600 Series
- 17.9.6 ST-OP-0007, Extraction and Cleanup of Herbicides from Water and Soils, Based on SW-846 8151A
- 17.9.7 ST-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A)
- 17.9.8 ST-MT-0003, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis, SW-846 Method 6010B and EPA Method 200.7
- 17.9.9 ST-MT-0005, Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW846 7470A and MCAWW 245.1
- 17.9.10 ST-MS-0001, GC/MS Analysis Based on Methods 8270C and 625
- 17.9.11 ST-MS-0002, Determination of Volatile Organics by GC/MS Based on Method 8260B, 624, and 524.2
- 17.9.12 ST-GC-0016, Pesticide Gas Chromatography Analysis Method SW-846 8000C/8081B and EPA 608
- 17.9.13 ST-GC-0017, Herbicide Gas Chromatography Analysis Method SW-846 8000C/8151A
- 17.9.14 ST-WC-0011, Analysis of pH in Water

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 Preliminary Evaluations. Section 7.1 of the source method states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction." Section 7.1.5 of the source method indicates that the portion used for the preliminary evaluation may be used for either the ZHE or non-volatile extraction if the sample was 100% solid. Section 7.1.5 further indicates that if the sample was subjected to filtration (i.e., < 100% solid) that this aliquot may be used for the non-volatile extraction procedure only as long as sufficient sample is available (minimum 100 g). Samples which have been subjected to the oven drying step may not be used for TCLP extraction because solid phase degradation may result upon heating.
- 18.2 Percent Solids Determination. Section 7.1.2 of the source method indicates that "if the percent wet solids is $\geq 0.5\%$ and it is noticed that a small amount of the filtrate is entrained in wetting of the filter" that the filter should be oven dried to determine percent dry solids ". Drying of oil or organic matrices can both be hazardous and inappropriate. Additionally, it may be impossible to achieve a constant weight when performing this step. Due to safety concerns, if obviously oily or heavy organic matrices are entrained on the filter, the filter is not oven dried.
- 18.3 TCLP Extraction Blanks. Section 8.1 of the source method states that a minimum of one blank for every 20 extractions "...that have been conducted in an extraction vessel." TestAmerica has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e., Bottle or ZHE) per leach fluid used.
- 18.4 Determination of Appropriate Extraction Fluid. Method 1311 does not address the appropriate approach to take if the pH equals 5.0. This SOP requires that Fluid #1 must be used if the pH is less than or equal to 5.0.
- 18.5 QA/QC - Matrix Spikes. Section 8.2 of the source method states, "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch." Further, Section 8.2.3 of the source method also states, "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist." A minimum of one MS will be prepared for each TCLP leachate batch. The MS results are used to determine the effect of a matrix on the accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS results have immediate bearing only on the specific sample spiked and not all samples in the batch.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Adding section 11.9; TCLP Procedure for Determination of Major Cations.
- 19.2 01/07/2011, Rev 0, SOP name changed from ST-IP-0016 to ST-OP-0009.
- 19.3 Revision 1:
 - 19.3.1 Updated section 11.6.15 by replacing the use of pH strips with a pH probe.
 - 19.3.2 Updated section 11.7.22 by replacing preserved VOA vials with unpreserved ones to store TCLP leachate samples.
- 19.4 Revision 2;
 - 19.4.1 Removed reference for storing ZHE leachates in HCL persevered VOA vials in section 8.7.
- 19.5 Rev 3:
 - 19.5.1 Updated section equipment in Section 6 (added aluminum weigh pans).
 - 19.5.2 Updated the ZHE weighing procedure throughout section 11.0.
- 19.6 Revision 4 (4/2/14):
 - 19.6.1 Updated Section 4.1 to include requirement to discuss and get documented client approval before running a total analysis on an oily sample and to include information on converting the total analysis to a “maximum leachable concentration.”
 - 19.6.2 Added example/reference to form ORG-0034 TCLP MSA spreadsheet.
- 19.7 Revision 5 (6/5/15)
 - 19.7.1 Removed left over reference to Quantims (Section 9.1.3)
 - 19.7.2 Added note to Section 10.3 that the pH meter needs to be calibrated to bracket the pH level being measured.
 - 19.7.3 Updated Section 11.5.5.8 regarding measuring the final pH.
- 19.8 Revision 6 (7/29/16)
 - 19.8.1 Revised Section 11.7.28, adding requirement to discard first 5 mL of liquid from the device.
- 19.9 Revision 7 (10/28/16)
 - 19.9.1 Updated Appendix (Table 1 and Table 2). Removed concentrations, and added volumes.

Table 1
Metals and Hg MS/MSD Volumes

	Metals Initial Volume (Sample)	Metals Final Volume (bring up with DI)	Hg Initial Volume (Sample)	Hg Final Volume (bring up with DI)
TCLP	20 mL	50 mL	6 mL	30 mL
CWET	10 mL	50 mL	1 mL	30 mL
SPLP	50 mL	50 mL	30 mL	30 mL

Table 2
Metals and Hg MS/MSD Spike Amounts

Spike Volume	Solution	Metals
0.25 mL	ICP Spike #1	Ca, Mg, K, Na
		Al, As, Ba, Se, Tl
		Fe, Sr
		Co, Pb, Mn, Ni, V, Zn
		Cu
		Cr (trivalent)
		Be, Cd, Ag
0.25 mL	ICP Spike #2	Mo
		Sb
0.05 mL	Hg Spike	Hg

Figure 1 & 2 - Rotary Agitation Apparatus and Zero Headspace Extraction Vessel (ZHE)

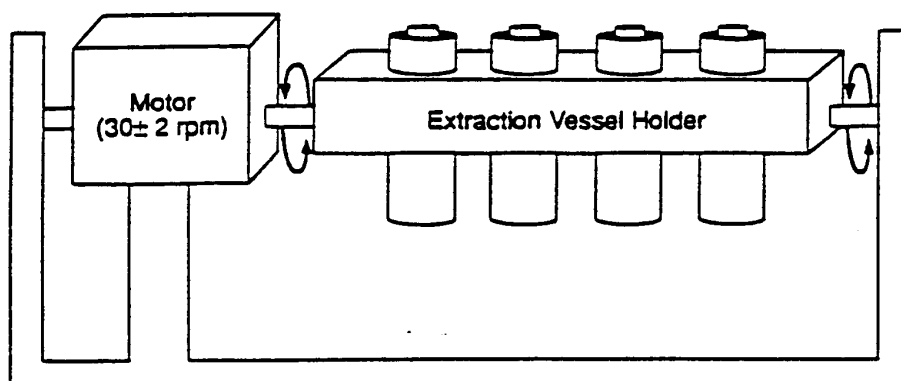


Figure 1. Rotary Agitation Apparatus

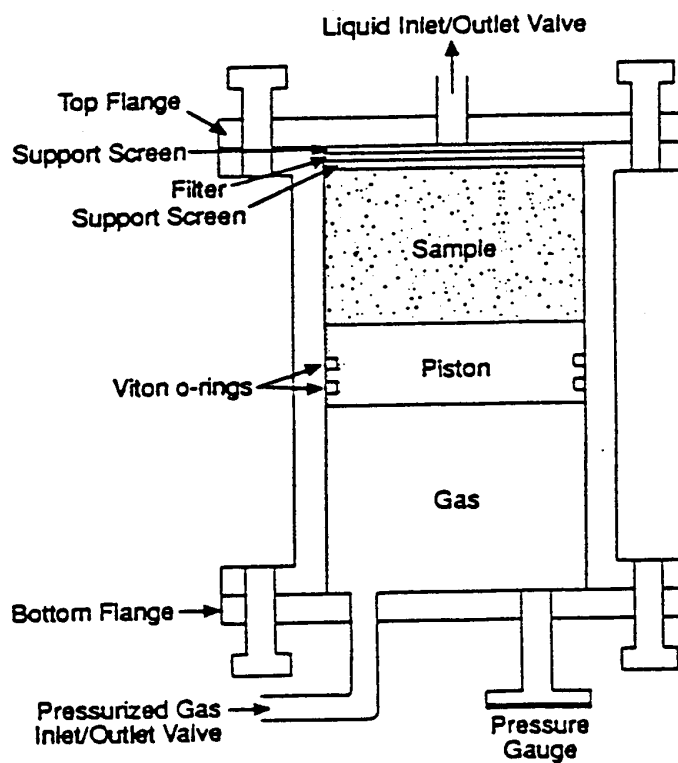


Figure 3 - US Environmental Protection Agency Memorandum #35, Page 1



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
SOLID WASTE AND EMERGENCY RESPONSE

MEMORANDUM # 35

DATE: June 12, 1992
SUBJECT: Notes on RCRA Methods and QA Activities
From: Gail Hansen, Chief *Gail Hansen*
Methods Section (OS-331)

This memo addresses the following topics:

- 1992 Symposium on Waste Testing and Quality Assurance
- SW-846 Update
 - Final Rule for January 23, 1989 Proposed Rule
 - Notice, Proposed Rulemaking for the Second Update to the Third Edition
- Chlorofluorocarbon 113 (CFC-113) Solvent Replacement Update
- Environmental Monitoring Methods Index (EMMI)
- Sampling Work Group Formation
- MICE Update
- Oily Waste Analysis
- Electronic SW-846 Availability.

Figure 3 - US Environmental Protection Agency Memorandum #35, Page 10Oil Waste Analysis

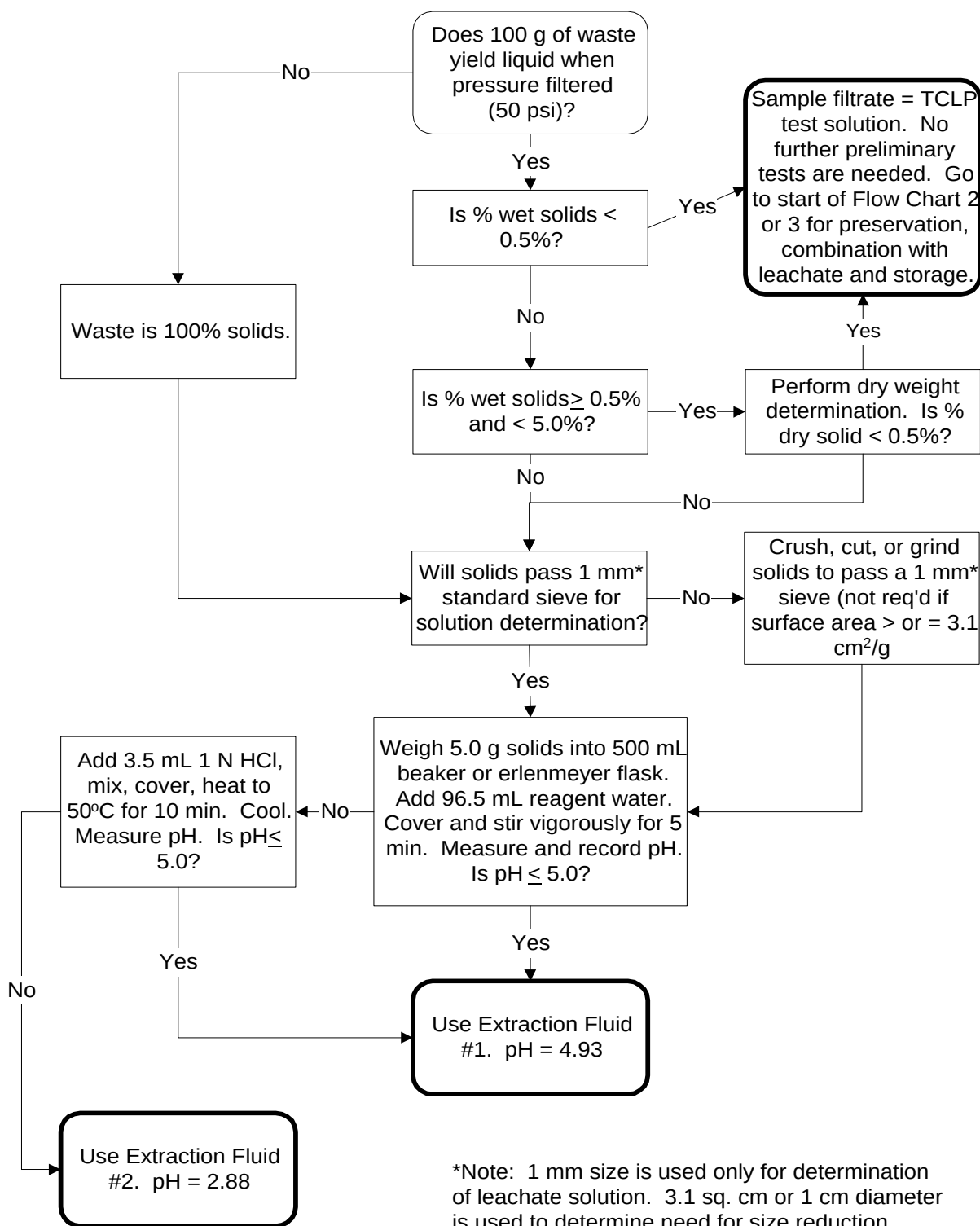
One of the most frequently asked questions on the MICE Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

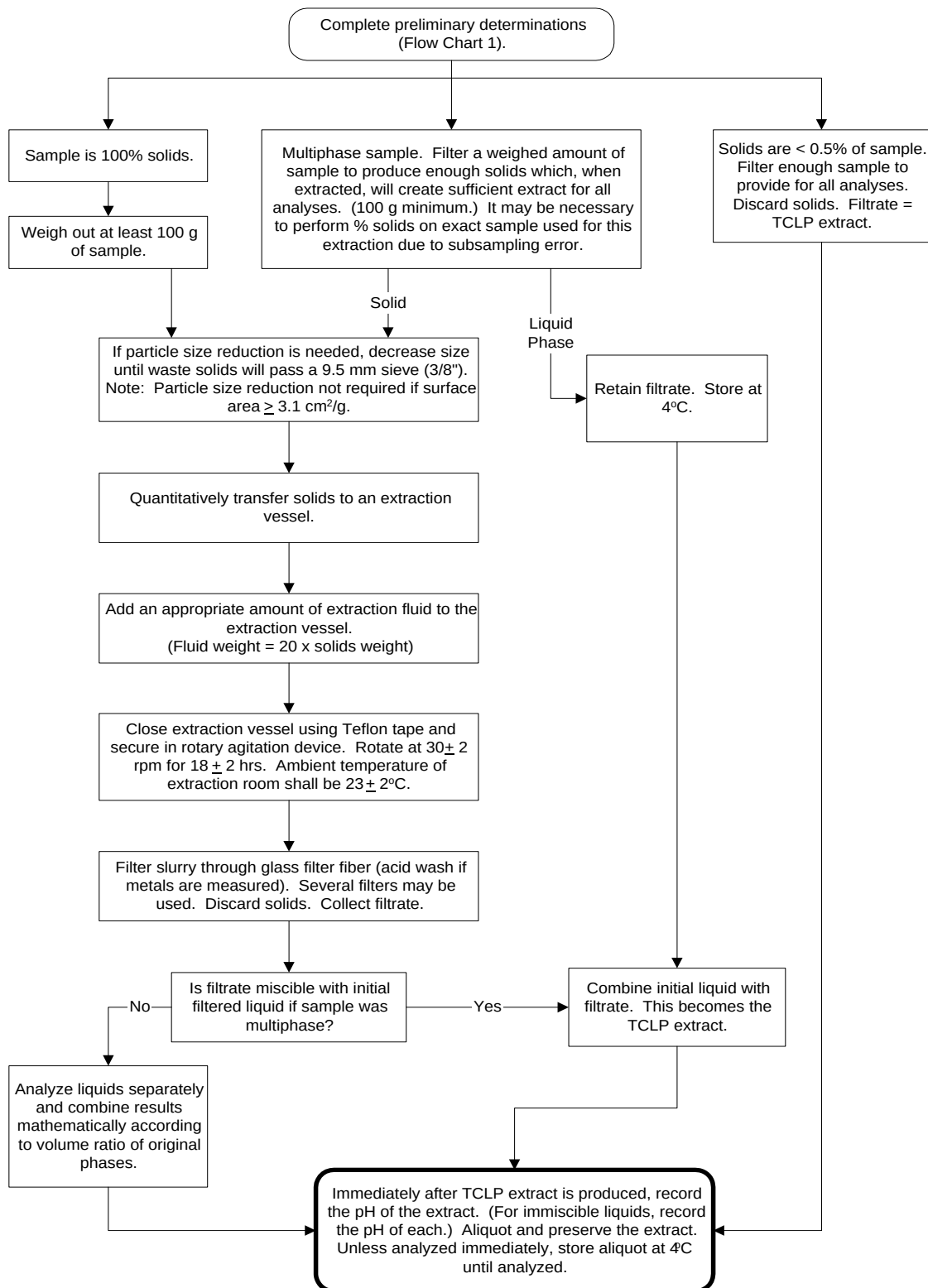
Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

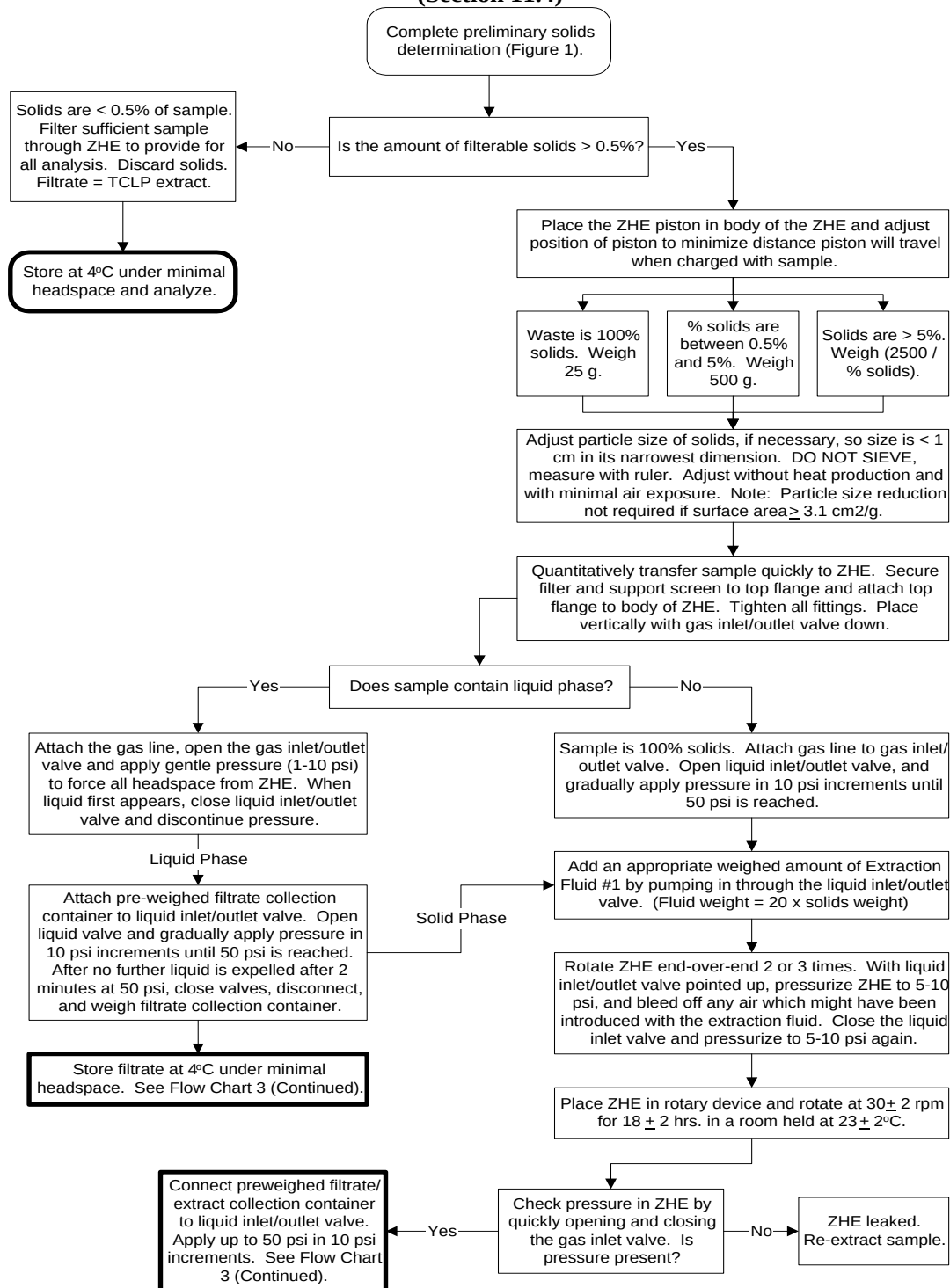
**Flow Chart 1. Preliminary Sample Evaluation
(Section 11.2)**



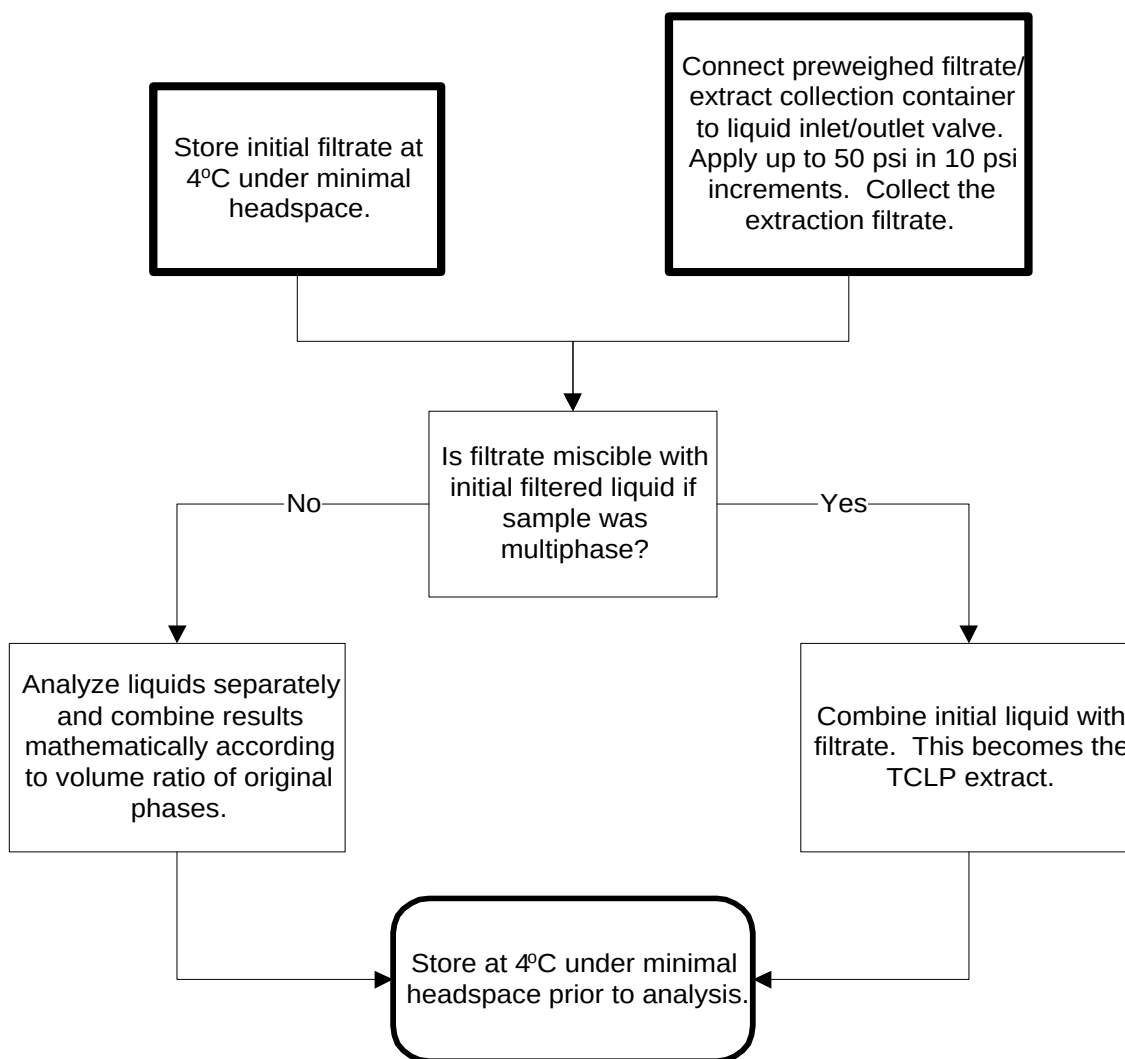
Flow Chart 2. Bottle Extraction, Non-Volatile Constituents (Section 11.3)



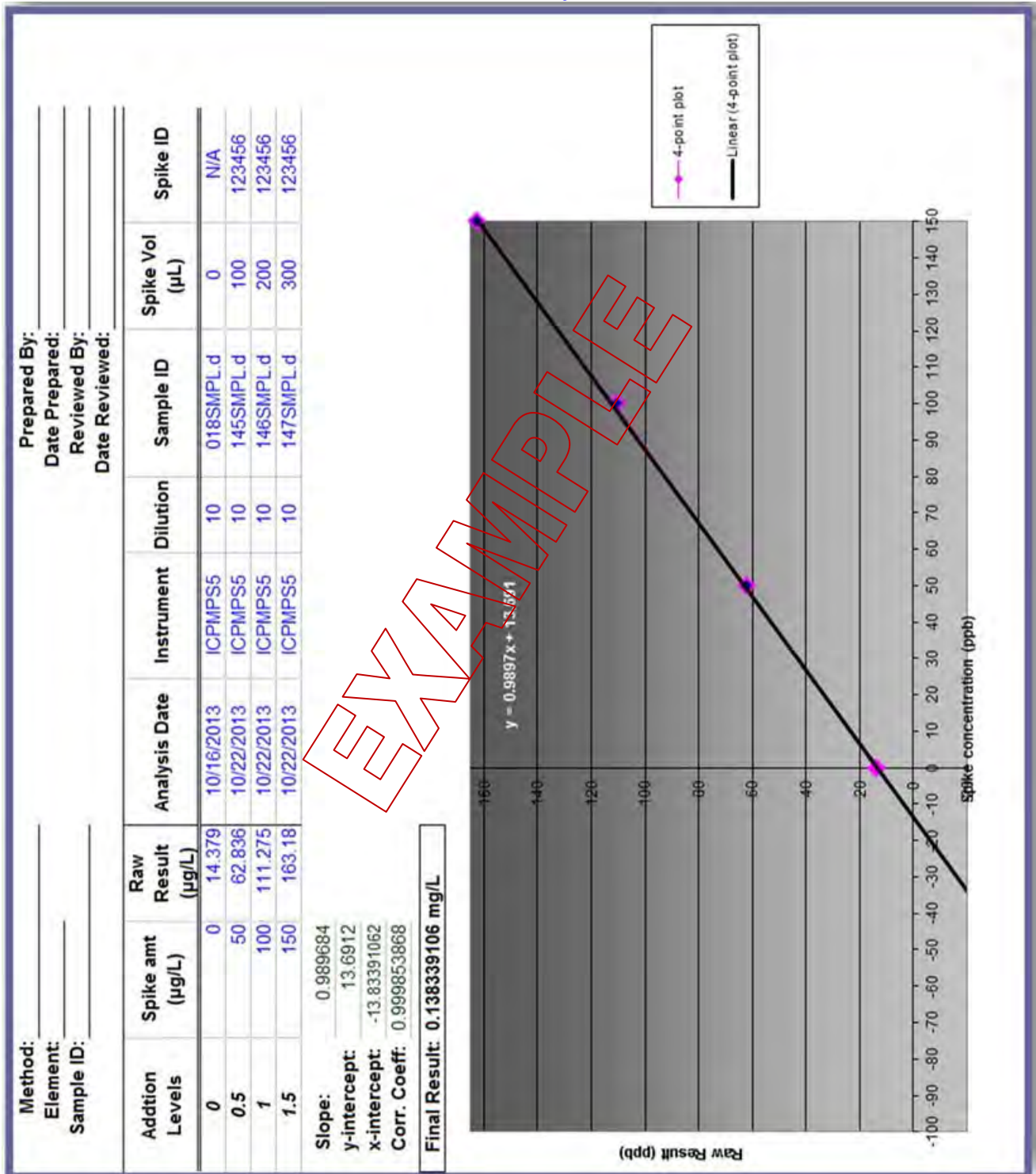
**Flow Chart 3. ZHE Extraction, Volatile Constituents
(Section 11.4)**



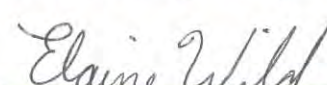
**Flow Chart 3. ZHE Extraction
(Continued)**



ORG-0034 TCLP MSA Spreadsheet Example
[TAFS\Lab\St Louis\Public\QA\FORMS\ORG](#)



Title: SAMPLE RECEIPT AND CHAIN OF CUSTODY

Approvals (Signature/Date):	
 Brian Daniels Sample Control Supervisor	6/23/16 Date
 Michael Ridenhower Health & Safety Manager / Coordinator	6/23/16 Date
 Marti Ward Quality Assurance Manager	6/23/16 Date
 Elaine Wild Laboratory Director	6/23/16 Date

This SOP was previously identified as SOP No. ST-PM-0002 Rev. 19

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1.0 SCOPE AND APPLICATION

- 1.1 The purpose of this procedure is to describe the receipt of samples by the Sample Control Department and to describe the intra-laboratory custody transfer of samples.
- 1.2 This procedure applies to all samples arriving at the TestAmerica St. Louis laboratory.
- 1.3 Radiological screening of samples received is an integral part of the sample receipt process. The SOPs on the screening and classifying of samples must be used in conjunction with this procedure, ST-RC-0010.
- 1.4 NELAC (National Environmental Laboratory Accreditation Conference) specifies requirements under which any NELAC accredited laboratory will accept samples.

2.0 SUMMARY

- 2.1 All samples received by the TestAmerica St. Louis laboratory, will be received by the Sample Control Department or authorized designees. Upon receipt, samples will be checked for completeness of associated paperwork, leakage/breakage, proper preservation and sample integrity.
- 2.2 All discrepancies will be noted on the Condition Upon Receipt ([CUR](#)) Form. This form must be completed at the time the items are being checked. If any item is completed by someone other than the initiator, then that person is required to apply his/her initials and the date next to that item.
- 2.3 Samples are entered into the laboratory information management system. Internal sample ID numbers are assigned by the LIMS.
- 2.4 Custody documentation and proper storage conditions are maintained until samples are returned to client or disposal authorization is granted
- 2.5 All associates involved in the sample receipt process must read, understand and perform according to this Standard Operating Procedure. Any questions should be brought to the attention of the Sample Control Department or the QA Department.
- 2.6 Responsibilities
 - 2.6.1 Sample Control Department: Accepts initial custody of samples received; responsible for the integrity and security of samples while in storage; follow the sample login initiation procedures stipulated in this SOP; responsible to indicate and/or initiate any precautions associated with a sample based on matrix type and/or analysis requested.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers
- 3.2 Chain of Custody (COC): Documentation of physical possession of a sample. The hardcopy form that begins the documentation process and is initiated by client. The signature of the laboratory Sample Control personnel or authorized designee denotes release of custody by the client and possession by the laboratory [Attachment 1](#).
- 3.3 Condition Upon Receipt (CUR) Form: Generated at time of sample receipt (Documents the items checked during the receipt of samples prior to login) [Attachment 2](#).
- 3.4 LIMS: The laboratory information management system that tracks sample information.

4.0 INTERFERENCES

- 4.1 Not Applicable

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow

appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

- 5.2.1 The preparation of reagents for the sample receiving area will be conducted in a fume hood with the sash closed as far as the operations will permit. If an operation requires the hood sash to be raised to above face level, a face shield shall be used.
- 5.2.2 All employees receiving samples shall be trained in accordance with the applicable Department of Transportation (DOT) regulations.
- 5.2.3 Exposure to chemicals will be maintained as low as reasonably achievable. Sample coolers/shipping containers shall be opened in a well-ventilated area (i.e., sample receiving area). If a broken container is found, the cooler/shipping container shall be transferred to fumehood and carefully unpacked. All sample coolers/shipping containers which show signs of damage will be opened in front of or in an operating fume hood.
- 5.2.4 Cut resistant gloves must be worn while initially inspecting coolers that are received or while cleaning coolers for re-use.

5.3 PRIMARY MATERIALS USED

- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limits (2)	Signs of Exposure
Nitric Acid	Corrosive Poison Oxidizer	2 ppm, (TWA) 4 ppm, (STEL)	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns. Notes: Nitric acid is a strong oxidizer. Contact with other material may cause fire. CORROSIVE. Liquid and mist cause severe burns to all body tissue.
Sodium Hydroxide	Corrosive Poison	2 mg/m ³ (TWA)	Nose irritation; pneumonitis; eye, skin burns; temporary loss of hair. Notes: Sodium hydroxide is corrosive. Causes burns to any area of contact. Reacts with water, acids and other materials.
Sulfuric Acid	Corrosive Poison Cancer Hazard	1 mg/m ³ , (TWA)	Inhalation may cause irritation of the nose and throat, and labored breathing. Skin contact symptoms include redness, pain, and severe burning. Eye contact can cause blurred vision, redness, pain, and severe tissue burns. Notes: Sulfuric acid is extremely corrosive. Liquid and mist cause severe burns to all body tissue.
Hydrochloric Acid	Poison Corrosive	5 ppm, (Ceiling)	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns. Notes: Hydrochloric acid is a corrosive. Liquid and mist causes severe burns to all body tissue.
1 - Always add acid to water to prevent violent reactions.			
2 - Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
Ceiling - At no time should exposure limit be exceeded.			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 pH paper, low range (0 - 2.3) and wide range (0.0 - 14)
- 6.2 Disposable pipettes for sampling liquids
- 6.3 Tongue depressors
- 6.4 Thermometers, electronic, calibrated
- 6.5 Survey Meter, sensitive to Alpha and/or Beta/Gamma; calibrated
- 6.6 Protective clothing, safety glasses, gloves (including cut resistant), lab coats
- 6.7 Swipes for surveying for loose surface contamination

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 Reagent water, obtained from the Milli-Q system.
- 7.3 Sulfuric acid (H_2SO_4), concentrated (36 N), ACS grade.
 - 7.3.1 Sulfuric acid (, 18 N (1:1) – Carefully add 500 mL of concentrated H_2SO_4 to 500 mL of reagent water while stirring. Mix well.
- 7.4 Nitric acid (HNO_3), concentrated (16 N), Trace grade.
 - 7.4.1 Nitric acid (ACS or trace metals grade), 8 N (1:4) – Carefully add <50 mL of concentrated HNO_3 to 1000 mL of reagent water while stirring. Mix well.
- 7.5 Hydrochloric acid (HCl), concentrated (12 N), ACS grade, STL certified.
 - 7.5.1 Hydrochloric acid, 6 N (1:1) – Carefully add 500 mL of concentrated HCl to 500 mL of reagent water while stirring. Mix well.
- 7.6 Sodium hydroxide (NaOH), approximately 10 N (50% w/w), reagent grade. CAUTION - Sodium hydroxide is corrosive. Causes burns to any area of contact. Reacts with water, acids and other materials.
- 7.7 Cleaning solution for used shipping containers (one of the following):
 - 7.7.1 Bleach solution
 - 7.7.2 Quaternary ammonia
 - 7.7.3 70% alcoholol solution

8.0 SAMPLE COLLECTION PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures.

9.0 QUALITY CONTROL

- 9.1 Matrix Spike/Matrix Spike Duplicates and/or Sample Duplicates are logged-in on samples as designated by the client on the COC, or instruction from the Project Manager.
- 9.2 Method Blanks and LCS QC samples are initiated by the sample preparation analysts and are not part of the log-in process.
- 9.3 Percent moisture determination is logged-in for all inorganic and organic soil samples, unless wet weight, “as is,” results are requested by the client, there is limited sample volume, or performing percent moisture is determined to be hazardous on the given matrix. Radiological samples are dried and ground as part of the routine procedure (Exception: Tritium, Carbon-14 and Tc-99 analysis in soil, see [Attachment 3](#)) and percent moisture is performed upon request.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Thermometers used for sample storage cooler and sample arrival cooler measurement will be calibrated as described in SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."
- 10.2 Survey meters used for the detection of radiological contamination are calibrated as described in SOP ST-RP-0032, "Instrumentation and Surveillance."

11.0 PROCEDURE

NOTE: Thermal preservation of samples shall be considered during sample receiving and login. Care should be given to minimize, as best as possible, the length of time that samples are "on the bench" during the receiving and login process. As general practice, the Sample Control staff should leave samples in their shipping containers until they can be actively processed. The laboratory shall document any thermal preservation that is not maintained during sample receiving and login. An NCM will be written and clients will be notified in writing if thermal preservation is not maintained.

11.1 Sample Acceptance Policy

- 11.1.1 NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. TestAmerica St. Louis will review sample shipment against those requirements listed below, and will communicate any discrepancies to the client. The project manager will assist the client in the appropriate resolution of any issues related to sample receipt.
 - 11.1.1.1 NELAC requirements are as follows:
 - 11.1.1.1.1 Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
 - 11.1.1.1.2 Each sample shall be labeled with unique, durable and indelible identification.
 - 11.1.1.1.3 The samples shall be collected in the appropriate sample containers.
 - 11.1.1.1.4 The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
 - 11.1.1.1.5 Sufficient sample volume must be available to perform the requested analyses.
 - 11.1.1.1.6 The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation.
- 11.1.2 When completing the chain of custody form, the client signs in the "relinquished by" box.
- 11.1.3 Department of Defense (DoD) has its own sample acceptance policy ([Attachment 5](#)). Clients must be notified if requirements are not met.

11.2 Sample Handling and Storage

- 11.2.1 Prior to performing radiological surveys each day, the Sample Control Department must calibrate the radiological survey instrument that will be used to perform the surveys. The calibration shall consist of checking the performance of the instrument using a radiological source, performing a battery check, checking the reset button and testing the audible response of the instrument. The nominal radiological background will be measured and recorded. All performance checks shall be documented in a logbook.
- 11.2.2 Upon receipt, laboratory samples shall be handled and stored in such a manner as:
 - 11.2.2.1 to ensure the safety of all personnel,
 - 11.2.2.2 to maintain the integrity of the samples in accordance with the requested analytical methods, and
 - 11.2.2.3 to prevent potential cross contamination of samples.
- 11.2.3 Coolers received from Department of Energy (DOE) sites are opened in a hood.
- 11.2.4 Coolers received from TestAmerica Service Centers will contain a bar code label affixed to the inside of the cooler lid's upper left hand corner. The label is scanned into the LIMs "Service

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Center Revenue Tracking” field in the LIMs. Once scanned, the label is removed and discarded. (See [Attachment 6.](#))

11.2.5 Sample Segregation

11.2.5.1 Samples are stored in locations remote from standard reference materials, calibration standards and any other chemicals or reagents. These locations are designated for sample storage only and include walk-in type coolers located in Sample Control or other designated areas within the laboratory.

11.2.5.2 To prevent contamination from other samples, samples to be analyzed for volatile organics (VOAs) are stored in locations designated for these samples only. VOA refrigerator/cooler storage blanks are analyzed to monitor air contaminants. Refer to SOP STL-QA-0031, “VOA Holding Blank Analysis.”

11.2.5.3 Samples received for volatiles analysis are stored in cooler 4, until retrieval by the volatiles lab and placed in refrigerators in the volatiles laboratory.

11.2.5.3.1 Actual drinking water samples submitted to the laboratory for analysis by EPA Method 524.2 are stored separate from EPA Method 8260 samples.

11.2.5.4 Radioactive samples may be stored in any refrigerator or walk in cooler.

11.2.5.5 Coolers or refrigerators designated for sample storage are maintained just above 0°C to 6°C. Samples that have been submitted to the laboratory that do not require cold preservation are stored in ambient storage areas.

11.2.5.6 Litigation samples require locked storage. The laboratory would have to acquire a lockable refrigeration unit for the storage of such samples. The lockable unit would be under the control of the Sample Control Department.

11.2.5.7 Samples that have been submitted to the laboratory that need quarantine (e.g. quarantine due to foreign or known-regulated domestic soil) will be stored on a segregated shelf marked as “Quarantined Soil”, within a walk-in cooler or RAD shelf. Each sample must be labeled as regulated soil under USDA permit. A sign must be placed on the outside of the walk-in cooler which states: “Foreign soil and/or regulated domestic soil to be used in accordance with USDA, APHIS, PPQ Soil Permit and Compliance Agreement.”

11.2.5.8 Samples requiring screening for radioactivity have their activities reviewed by the Radiation Safety Officer (RSO) or designee. Based on the activity present, samples will be categorized as Rad Category 1, 2, 3, or 4 (Categories 2, 3 or 4 require a Radiation Work Permit (RWP)). This radioactivity level designation is entered into the LIMs after sample login.

11.2.5.9 Protective clothing is worn at all times when handling sample containers. Gloves must be changed when necessary to prevent cross contamination.

11.2.5.10 Samples are handled to ensure that labels or markings on each container remain intact.

11.2.5.11 Samples are kept securely capped except when checking pH.

11.3 Sample Receipt Condition

11.3.1 Airbills will be checked upon sample arrival with the time and date of arrival noted as verified by the signature of a Sample Control Associate or designee. Airbill numbers and the courier name will be listed on the CUR Form. Receipt of the shipment is documented in the sample receipt log maintained in Sample Control. Airbills or shipping papers, if available, are maintained in the Project Files. If the samples have been designated a Proper Shipping Name, listed below, or DOT class 7 primary or subsidiary hazard code, the samples shall be received per ST-RP-0050, “Purchase, Receipt, Handling, and Identification of Radioactive Material.”

11.3.1.1 Radioactive Material, Excepted Package, Limited Quantity of Material

11.3.1.2 Radioactive Material, n.o.s.

11.3.1.3 Radioactive Material, Low Specific Activity (LSA), n.o.s.

11.3.1.4 Radioactive Material, Surface Contaminated Object (SCO)

11.3.2 When the sample cooler is opened and a Limited Quantity warning is in the package or the samples have radiation stickers/labels, the shipment shall be received per ST-RP-0050, “Purchase, Receipt, Handling, and Identification of Radioactive Material.”

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NOTE: No packing material shall be discarded until the completion of step 11.3.9.

- 11.3.3 Evidential (custody) seals on shipping container(s) are inspected. Any evidence of tampering (i.e., broken seals), or if the seals are of a non-tamper evident type, is noted on the CUR Form.
- 11.3.4 The temperature of the coolers is checked upon arrival. This is done by quickly opening the cooler, inserting the probe of a calibrated electronic thermometer, closing the cooler, and leaving it until the thermometer reaches a constant temperature. The probe shall be placed in close proximity to the samples (i.e., between the sample container and its surrounding bubble wrap). The temperature as displayed on the probe without rounding, is then noted and documented on the CUR Form and in TALS. The probe number is documented on the CUR and in TALS. (Note: When multiple coolers are received in a shipment, the specific contents of a cooler that was outside acceptance criteria must be listed.) The Project Manager is notified of unacceptable temperatures. The acceptable temperature is 0° to 6° C. Alternatively, the temperature of a cooler blank, if provided, will be recorded.
- 11.3.5 If the shipment is from a known or suspected radiological site, the sample cooler/shipping container is surveyed for loose surface contamination. Refer to ST-RP-0032, "Instrumentation and Surveillance," for instructions on the proper use of radiacs. Coolers from DOE sites require frisking of the entire cooler, including the bottom.
- 11.3.6 If the shipment is of a foreign or known-regulated domestic soil, all soil residue on packaging must be treated with one of the schedules below:
- | | |
|---|-----------------|
| 11.3.6.1 Dry Heat Temperatures | Exposure Period |
| 110.0 – 120.5 degrees Celsius (230-240 degrees F) | 16 hours |
| 121.0 – 154.0 degrees Celsius (250-309 degrees F) | 2 hours |
| 154.4 – 192.5 degrees Celsius (310-379 degrees F) | 30 minutes |
| 193.0 – 220.0 degrees Celsius (380-429 degrees F) | 4 minutes |
| 221.0 – 232.0 degrees Celsius (430-450 degrees F) | 2 minutes |
- Do not start counting time until entire mass reaches the required temperature.
- 11.3.7 Used shipping containers must be decontaminated by one of the treatments approved for soil or destroyed by incineration. The coolers used to ship soil samples must be generously sprayed with an approved disinfectant such as bleach, quaternary ammonia, or 70% alcohol solution to the point of runoff, allowed to drain into a municipal water system and air dried.
- 11.3.8 Sample coolers/shipping containers with loose surface contamination greater than 100 cpm above background on a beta/gamma frisker require the immediate notification of the RSO or designate. The shipment shall be received per ST-RP-0050, "Purchase, Receipt, Handling, and Identification of Radioactive Material."
- 11.3.9 Samples are checked against the Client COC. All discrepancies are noted on the Client COC and detailed on the CUR Form. The Client COC should indicate:
- 11.3.9.1 Client Name
- 11.3.9.2 Unique sample number for each sample
- 11.3.9.3 Date of sampling
- 11.3.9.4 Time of sampling
- 11.3.9.5 Matrix sampled
- 11.3.9.6 Chemical preservatives added
- 11.3.9.7 Analyses requested
- 11.3.9.8 Numbers of containers collected for each analysis
- 11.3.9.9 Pertinent observations (odor, hazards, etc.)
- 11.3.10 COCs will be checked for any requested tests that may have short hold times as indicated by [Attachment 3](#), (ex. BOD, pH, etc.).
- 11.3.11 Samples determined to have short holding times will be removed from cooler/shipping container and will be placed in pass through window to wet chem.
- 11.3.11.1 Stickers/labels are placed on the sample container indicating what test to perform. The current date and time is recorded on the sticker and placed in the short hold window.
- 11.3.11.2 Specific short hold internal COC forms will be provided upon client request.
- NOTE:** it is the responsibility of Wet Chem department to check for short hold time samples.

- 11.3.12 COCs will be checked for any requested tests on soil matrix that require sample(s) to **not be dried** and a "Do Not Dry" sticker/label is affixed to said container(s) and noted on the CUR.
- 11.3.12.1 Test that require Do Not Dry sticker/label (also see Attachment 3):
- 11.3.12.1.1 C-14 (carbon-14)
 - 11.3.12.1.2 H-3 (tritium)
 - 11.3.12.1.3 Tc-99 (technetium-99)
 - 11.3.12.1.4 I-129/131
 - 11.3.12.1.5 Cl-36
- 11.3.13 COCs will be checked for any requested tests that require sample(s) to **not be preserved** and a "Do Not Preserve" sticker/label is affixed to said container(s) and appropriate box checked on the CUR.
- 11.3.13.1 Test that require sample to **not be preserved** (also see Attachment 3) :
- 11.3.13.1.1 C-14 (carbon-14)
 - 11.3.13.1.2 H-3 (tritium)
 - 11.3.13.1.3 I-129/131 (iodine-129/131)
 - 11.3.13.1.4 Cl-36
- 11.3.14 The pH is taken on all samples (with the exception of volatile organics samples, Oil and Grease and TOX samples) for preservation verification. All discrepancies are noted on the CUR Form. The pH is recorded in the appropriate field in the LIMs.
- 11.3.15 A scant amount of sample is removed by dipping a clean pipette into the sample and applying a drop to pH paper.
- 11.3.15.1 Acid preservation checked with low range pH paper (0 - 2.3)
 - 11.3.15.2 Base preservation checked with 0 - 14 pH paper
 - 11.3.15.3 When aqueous samples from DOE-Albuquerque SMOs are received, the pH of both the preserved and nonpreserved samples must be taken. The pH readings must be documented and communicated, via the CUR, to the Project Manager as soon as possible so that the client may have the opportunity to request corrective action, if necessary.
 - 11.3.15.3.1 When samples are received with improper pH, Sample Control will preserve the samples with the appropriate preservative. If pH remains incorrect the project manager will be notified, and client must be contacted before adding additional acid or base.
 - 11.3.15.3.2 Sufficient acid or base is added to the sample container to adjust the sample to the required pH. The following guidelines should be used for the initial pH adjustment:
 - 11.3.15.3.2.1 Sulfuric acid: 1 mL (1:1) for a 1 liter container.
 - 11.3.15.3.2.2 Nitric acid: 1 mL (1:4) for a 1 liter container.
 - 11.3.15.3.2.3 Hydrochloric acid: 7.8 mL (1:1) for a 1 liter container.
 - 11.3.15.3.2.4 Sodium hydroxide: 1.25 mL (50% w/v) for a 500 mL container.
 - 11.3.15.3.3 Verify pH after addition of acid or base. For metals analysis, sample aliquots may not be removed for analysis until 24 hours after preservation. For radiological analyses, sample aliquots may not be removed for analysis until 24 hours after preservation.
 - 11.3.15.3.4 Preservation by the lab must be documented on both the Condition Upon Receipt (CUR) Form and on the sample container itself. Documentation on the CUR Forms consists of the date and time of preservation and the lot number of the acid used. Required documentation on the container is the date and time of preservation.
- 11.3.16 The pH of samples to be analyzed for volatile organics or TOX is measured by the analyst after removing the aliquot for analysis.
- 11.3.17 After completion by the Sample Control Department, the CUR Form is forwarded to the applicable Project Manager for review and client notification, if necessary. Client notification and any required corrective action will be documented.

- 11.4 Sample Log in
 - 11.4.1 Samples are logged into the LIMS which assigns a “Job” number to the group of samples being entered. Individual samples within the job are given unique sample ID numbers for each test to be performed. Multiple containers for a single test are given unique identifiers.
 - 11.4.2 Projects specifying the required analyses and any specific instructions are created in the LIMS by the Project Manager and are used by the Sample Control Department during login.
 - 11.4.2.1 When entering a composite sample which lists multiple collection times, the latest time will be used as the time of sample collection.
 - 11.4.2.2 All collection times logged into the LIMs are designated to the time zone from where sample(s) originated.
 - 11.4.3 Composite Samples
 - 11.4.3.1 There are instances where the client requests the laboratory to perform sample compositing after collection/receipt. The instructions for how compositing is to be performed (namely what containers to put together) should be communicated to Sample Control **prior** to the time of receipt.
 - 11.4.3.1.1 Project Managers will request the client to denote “compositing” on the COC instructions.
 - 11.4.3.1.2 The Project Manager will put Composite Notification and Instructions in the specific client Project comments. These comments will appear first to highlight it for Sample Control.
 - 11.4.3.1.3 If the PM receives advanced notice of samples or copies of COCs, the PM will inform their Sample Control contact.
 - 11.4.3.2 Samples are logged in for all requested analyses and put in a separate storage location to await compositing. This location is identified as being “**on hold**” until compositing is complete.
 - 11.4.3.3 The samples awaiting compositing are stored in Cooler 3.
 - 11.4.4 PM will include a rad screening test in the Project, if samples are from a potential radioactive site. Sample control logs in a rad screen. The Rad department performs the aliquoting for the rad screen.
 - 11.4.5 Samples are placed into the appropriate refrigerated or ambient sample storage area. The storage location is entered into the LIMS.
 - 11.4.6 Upon completion of the login, the login is reviewed for accuracy by the Project Manager.
- 11.5 Subsampling
 - 11.5.1 In the case when a sample must be subsampled, it will be done in accordance with ST-QA-0038.
 - 11.5.2 Subsamples created in accordance with ST-QA-0038 for the tests listed below must be labeled with “Do Not Preserve” labels. The aliquots derived from the sub-sampling are then returned to Sample Control for routine login procedures.
 - 11.5.2.1.1 C-14 (carbon-14)
 - 11.5.2.1.2 H-3 (tritium)
 - 11.5.2.1.3 I-129 (iodine-129)
 - 11.5.3 Subsamples created in accordance with ST-QA-0038 for the tests listed below must be labeled with “Do Not Dry” labels. The aliquots derived from the sub-sampling are then returned to Sample Control for routine login procedures.
 - 11.5.3.1.1 C-14 (carbon-14)
 - 11.5.3.1.2 H-3 (tritium)
 - 11.5.3.1.3 Tc-99 (technetium-99)
- 11.6 Receipt of Samples after normal working hours
 - 11.6.1 Associates other than those in the Sample Control Department may receive non-radiological samples after normal working hours provided they have been trained in DOT awareness by the EH&S Coordinator, except for when only filling out After Hours Sample Receipt Checklist
 - 11.6.2 The following actions will be performed when samples are received after normal working hours: After Hours Sample Receipt Checklist to be completed and left in Sample Control Department office

- 11.7 Internal Custody Transfer [See SOP-QA-0045 for details on how to use the program]
- 11.7.1 The laboratory is required to document the history of all samples through use of the internal chain of custody (ICOC). The purpose of the Chain of Custody is to provide a legal record of the handling of samples from collection through disposal. Each change of custody must be documented in the TALS ICOC program as they occur. When an analyst receives a sample, from storage or from a prep department, the sample must be immediately scanned via the ICOC program. When an analyst is finished with the sample or it is the end of that analysts work shift, it must be returned to its storage location and be scanned back into the ICOC program.
- 11.7.2 Custody transfer to the individual laboratories is accomplished using the Internal Chain of custody module in the LIMS. Alternate methods of documenting custody transfers may be utilized for short hold time samples which have not yet been logged into the LIMS.
- 11.7.3 When one of the individual laboratories is prepared to perform an analysis, an analyst will retrieve the necessary sample(s) from the applicable walk-in cooler or ambient storage area. The analyst is directed to the correct storage location by the cooler/shelf number listed on their backlog. After retrieval, the analyst will accept custody of the sample(s) using the Internal Chain of custody.
- 11.7.4 After aliquots are removed for analysis, sample containers are returned to the assigned storage location for disposal or return to the client. The Internal Chain of custody module documents this custody transfer. If sample disposal is to be performed by the lab analyst (i.e., for metals water samples), the analyst retains final custody of the sample.
- 11.7.5 If specific project plans require a person-to-person transfer of sample custody, an Internal Chain of Custody Form can be printed. In this case, a member of Sample Control and the analyst will sign the “Relinquished” and “Received” boxes upon custody transfer. ([Attachment 4](#))
- 11.7.6 For samples submitted to the laboratory for specific projects requiring person-to-person custody transfer, samples must be returned to the special storage area immediately after the sample aliquot is taken.

12.0 APPENDICES

- 12.1 [Attachment 1, Chain of Custody Form.](#)
- 12.2 [Attachment 2, Condition Upon Receipt Form.](#)
- 12.3 [Attachment 3, Sample Container, Volumes and Preservation Requirements.](#)
- 12.4 [Attachment 4, TestAmerica St. Louis Internal Chain-of-Custody.](#)
- 12.5 [Attachment 5, DoD QSM Sample Acceptance Policy](#)
- 12.6 [Attachement 6, Instructions for Service Center Source Tracking](#)

13.0 REFERENCES

- 13.1 TestAmerica Quality Assurance Manual (ST-QAM), current revision
- 13.2 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 13.3 National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards, EPA 600/R-98/151, US EPA Office of Research and Development, May 2001.
- 13.4 TNI Standard, 2009
- 13.5 Department of Defense/Department of Energy Quality Systems Manual, current version
- 13.6 Errata Document, QSM Revision of Rev 5.0; October 2015
- 13.7 United States Dept. of Agriculture, Foreign and Known-Regulated Domestic Soil Compliance Agreement, Agreement No. SOIL BRD 05 04
- 13.8 TALS Training Brief, CW-I-T-090, “Internal Chain of Custody”, 12/07/2011
- 13.9 Associated SOPs:
- 13.9.1 ST-HS-0004, Hazardous Waste Management Plan
- 13.9.2 ST-HS-0006, Quarantine Soil Procedure
- 13.9.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
- 13.9.4 ST-QA-0031, VOA Holding Blank Analysis

- 13.9.5 ST-QA-0038, Procedure for Compositing and Subsampling
- 13.9.6 ST-QA-0045, Internal Chain of Custody
- 13.9.7 ST-RC-0010, Screening Samples for the Presence of Radioactive Material
- 13.9.8 ST-RP-0031, Radiation Work Permits
- 13.9.9 ST-RP-0032, Instrumentation and Surveillance
- 13.9.10 ST-RP-0050, Purchase, Receipt, Handling, and Identification of Radioactive Material

14.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 14.1 None.

15.0 CHANGES FROM PREVIOUS REVISION

- 15.1 Added Internal Chain of Custody Form, attachment 4.
- 15.2 Revision 15
 - 15.2.1 Updated Condition Upon Receipt Form, attachment 2.
 - 15.2.2 Section 9.3, "(Exception: Tritium analysis in soil)" added
 - 15.2.3 Section 11.1.2 and subsections, formatting changed
 - 15.2.4 Section 11.3, updated to contain instructions for labeling samples with "Do Not Preserve" label
 - 15.2.5 Section 11.3.13, "requiring acid/base preservation" was removed
 - 15.2.6 Section 11.4.3.1, "must" changed to "should" in last sentence of paragraph
 - 15.2.7 Section 11.5.2, added to indicate tests for subsampling samples that require a "Do Not Preserve" label to be affixed to container(s).
- 15.3 Revision 16
 - 15.3.1 Grammatical error and formatting corrections
 - 15.3.2 Section 7: added cleaning solution for used shipping containers
 - 15.3.3 Section 9.3: added C-14 & Tc-99
 - 15.3.4 Section 11.5.3, added to indicate test for subsampling samples that require a "Do Not Dry" label to be affixed to containers
 - 15.3.5 Attachment 3: updated analysis that are "Do Not Dry" and "Do Not Preserve"
- 15.4 Revision 17:
 - 15.4.1 Removed reference to QuantIMs; replaced with LIMS.
 - 15.4.2 Updated Section 11.4 to reflect current sample identification process.
 - 15.4.3 Changed reference to "Quote" to "Project" following nomenclature of new LIMS.
 - 15.4.4 Added instructions for Service Center Source Tracking to sections 11.2 and 12 and added [Attachment 6](#).
- 15.5 Revision 18 (02/08/2013):
 - 15.5.1 Added instruction for calibration of radiological survey instrumentation to Section 11.
 - 15.5.2 Updated storage temperature for samples from 4°C ± 2°C to "just above 0°C to 6°C" per NELAC and DOECAP instructions.
 - 15.5.3 Added Reference to TALS internal chain of custody training brief to Section 17.
- 15.6 Revision 19 (01/31/2015):
 - 15.6.1 Updated disposition of CUR in Section 2
 - 15.6.2 Updated acid concentrations in Section 7 and 11.3
 - 15.6.3 Updated locked storage information in Section 11.2
 - 15.6.4 Updated temperature recording in Section 11.3
 - 15.6.5 Updated requirements for maintaining internal chain of custody in Section 11.7
 - 15.6.6 Added requirement to record pH in LIMS
 - 15.6.7 Replaced "log-in" with "Job" in Section 11.4
 - 15.6.8 Added references to SOP ST-QA-0045, TNI and DoD to Section 13
 - 15.6.9 Updated Table 3
- 15.7 Revision 20: 6/24/2016)
 - 15.7.1 Removed Plastic vials from Section 6.0
 - 15.7.2 Added the probe number is documented on the CUR and in TALS. Section 11.3.4

- 15.7.3 Removed rad screen process from 11.4.4
- 15.7.4 Added After Hours Sample Receipt Checklist in 11.6
- 15.7.5 Changed Sample Control Department to assigned storage location in 11.7.4
- 15.7.6 Removed SOP ST-QA-0025 from Section 13
- 15.7.7 Updated attachment 3 – replaced 4°C with $\leq 6^{\circ}\text{C}$.

Attachment 1

(Example: Chain of Custody Form)

(Example: Condition Upon Receipt Form)

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Attachment 3

Sample Container, Volumes, and Preservation Requirements

Analysis	Method	Matrix	Volume* / Container	Preservative	Hold Time	QC (Default)
Wet Chemistry						
Alkalinity: Total, Carbonate, Bicarbonate	2320B	Water	200 mL P,G	Cool from > 0° to ≤ 6° C	14 days	Dup
	310.1	Soil	20 g P,G	Cool from > 0° to ≤ 6° C	14 days	MS/Dup
Ammonia	350.1	Water	20 mL P,G	H ₂ SO ₄ (pH < 2), Cool	28 days	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
BOD	405.1; 5210B	Water	1 L P,G	Cool from > 0° to ≤ 6° C	48 hrs	MS/Dup
Bromide	300.0; 9056A	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
CBOD	5210B	Water	1 L P,G	Cool from > 0° to ≤ 6° C	48 hrs	Duplicate
Chloride	300.0; 9056A	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
Cyanide	9010A; 9012; 335.2	Water	50 mL P,G	NaOH (pH > 12), Cool	14 days	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C	14 days	MS/Dup
COD	410.4	Water	50 mL P,G	H ₂ SO ₄ (pH < 2), Cool	28 days	MS/Dup
Conductivity	120.1; 9050	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
		Soil	20 g P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
Ferrous Iron	SM 3500D	Water	100 mL P,G	Cool from > 0° to ≤ 6° C	24 hrs	MS/Dup
		Soil	10 g P,G	Cool from > 0° to ≤ 6° C	24 hrs	MS/Dup
Fluoride	300.0; 9056A (IC)	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup

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Analysis	Method	Matrix	Volume* / Container	Preservative	Hold Time	QC (Default)
	340.2 (probe)	Water	50 mL	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
Flashpoint (Ignitability)	1010	Water	100 mL P,G	Cool from > 0° to ≤ 6° C	28 days	Duplicate
		Soil	100 g P,G	Cool from > 0° to ≤ 6° C	28 days	Duplicate
Hardness	2340C 130.2	Water	100 mL	Cool from > 0° to ≤ 6° C C HNO ₃ (pH < 2)	180 days	MS/Dup
Hexavalent Chromium	7196A	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	24 hrs	MS/Dup
		Soil	20 g P,G	Cool from > 0° to ≤ 6° C	30 days	MS/Dup
Iodide	300	Water	50 mL	Cool from > 0° to ≤ 6° C	7 days	MS/Dup
Nitrate	300.0; 353.1; 9056A	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	48 hrs	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C	48 hrs	MS/Dup
Nitrate/Nitrite	353.1; 4500NO ₃ - H	Water	50 mL P,G	H ₂ SO ₄ (pH < 2), Cool	28 days	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
Nitrite	300.0; 353.1; 9056A	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	48 hrs	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C	48 hrs	MS/Dup
Oil & Grease	9070; 413.1; 5520B; 1664	Water	1000 mL G	HCl (pH < 2), Cool	28 days	MS/Dup
	9071	Soil	50 g G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
Orthophosphate	300.0; 365.1; 9056A; 4500 PE	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	48 hrs	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C	48 hrs	MS/Dup
Paint Filter	9095	Soil	100 g P,G	Cool from > 0° to ≤ 6° C	28 days	Duplicate

Analysis	Method	Matrix	Volume* / Container	Preservative	Hold Time	QC (Default)
Perchlorate	314	Water	50 mL P,G	Cool from > 0° to ≤ 6° C store with headspace	28 days	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C store with headspace	28 days	MS/Dup
	6850	Water	50 mL P	Cool from > 0° to ≤ 6° C store with headspace	28 days	MS/Dup
		Soil	5 g G	Cool from > 0° to ≤ 6° C store with headspace	28 days	MS/Dup
pH	9040; 150.1; 4500H+B	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	immediate	Duplicate
	9045	Soil	20 g P,G	Cool from > 0° to ≤ 6° C	immediate	Duplicate
Phenols	9066; 420.2	Water	50 mL P,G	H ₂ SO ₄ (pH < 2), Cool	28 days	MS/Dup
		Soil	5 g G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
Phosphorus	365.1	Water	50 mL P,G	H ₂ SO ₄ (pH < 2), Cool	28 days	MS/Dup
		Soil	10 g P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
Reactive Cyanide	SW846 Chapter 7	Water	50 mL P,G	NaOH (pH > 12), Cool	14 days	MS/Dup
		Soil	10 g P,G	Cool from > 0° to ≤ 6° C	None	MS/Dup
Reactive Sulfide	SW846 Chapter 7	Water	50 mL P,G	NaOH,Zn Ac. (pH > 9), Cool	7 days	MS/Dup
		Soil	10 g P,G	Cool from > 0° to ≤ 6° C	None	MS/Dup
Residual Chlorine	330.3	Water	200 mL AP, AG	Light Resistant Container	24 hrs	MS/Dup
Settleable Solids	160.5	Water	1000 mL P,G	Cool from > 0° to ≤ 6° C	7 days	Duplicate
Sulfate	300.0; 9056A	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
		Soil	5 g P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
Sulfide	4500S ² -F 9030; 376.1	Water	200 mL P,G	NaOH,Zn Ac. (pH > 9), Cool	7 days	MS/Dup

Analysis	Method	Matrix	Volume*/ Container	Preservative	Hold Time	QC (Default)
	9030; 376.1	Soil	25 g P,G	2 N Zinc Acetate Cool, ≤ 6° C	7 days	MS/Dup
Sulfite	377.1	Water	50 mL P,G	Cool from > 0° to ≤ 6° C	24 hrs	MS/Dup
		Soil	25 g P,G	Cool from > 0° to ≤ 6° C	24 hrs	MS/Dup
Surfactants	425.1	Water	1000 mL P,G	Cool from > 0° to ≤ 6° C	48 hrs	MS/Dup
TDS	160.1, 2540C	Water	100 mL P,G	Cool from > 0° to ≤ 6° C	7 days	Duplicate
TOC	415.1; 9060; 5310B	Water	10 mL G	H ₂ SO ₄ (pH <2), Cool	28 days	MS/Dup
		Soil	5 g G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
TKN	351.1	Water	20 mL P,G	H ₂ SO ₄ (pH <2), Cool	28 days	MS/Dup
		Soil	1 g P,G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
Total Solids	160.3; 2540B	Water	100 mL P,G	Cool from > 0° to ≤ 6° C	7 days	MS/Dup
TOX (EOX)	450.1; 9020	Water	500 mL AG	H ₂ SO ₄ (pH <2), Cool	28 days	MS/Dup
		Soil	1 g G	Cool from > 0° to ≤ 6° C	28 days	MS/Dup
TSS	160.2; 2540D	Water	100 mL P,G	Cool from > 0° to ≤ 6° C	7 days	Duplicate
Turbidity	180.1	Water	100 mL P,G	Cool from > 0° to ≤ 6° C	48 hrs	MS/Dup
Microbial						
Chlorophyll A		Water	500 mL AG	Light resistant container, Cool, ≤ 6° C	24 hrs	Duplicate
VOA Organics						
BTEX	8020/8021; 8260; OA- 1	Water	2 X 40 mL G	HCl (pH<2), Cool, ≤ 6° C	14 days	MS/MSD
		Soil	5 g AG	Cool from > 0° to ≤ 6° C	14 days	MS/MSD
TCLP ZHE Volatiles	1311/ 8260	Solid	2 X 120 mL AG	Cool from > 0° to ≤ 6° C	14 days	MS/MSD
TPH, Gasoline	8015; OA- 1	Water	2 X 40 mL G	HCl (pH <2), Cool, 4 deg. C	14 days	MS/MSD

Analysis	Method	Matrix	Volume* / Container	Preservative	Hold Time	QC (Default)
		Soil	5 g AG	Cool, $\leq 6^{\circ}$ C	14 days	MS/MSD
Volatiles	624; 8260 (5mL purge)	Water	2 X 40 mL G	HCl (pH<2), Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	14 days/ 7 days, if not preserved w/ HCl	MS/MSD
	524.2; 624; 8260 (25mL purge)	Water	2 X 40 mL G	HCl (pH <2), Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	14 days/ 7 days, if not preserved w/ HCl	MS/MSD
	8260 (5030)	Soil	5 g AG	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	14 days	MS/MSD
	8260 (5035)	Soil	Encore Sampler x 2	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	14 days/ 48 hrs (if not rec'd in Sodium Bisulfate preservative)	MS/MSD
MDNA	8321A	Water	2X40 mL	HCL(pH<2)	14 days	MS/MSD
Extractable Organics						
Dioxin	8280; 8290; 613	Water	1 L AG	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	30 days	MS/MSD
		Soil	30 g G	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	30 days	MS/MSD
Explosives	8330	Water	1 L AG	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	7 days	MS/MSD
		Soil	30 g G	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	14 days	MS/MSD
PAHs	8310	Water	1 L AG	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	7 days	MS/MSD
		Soil	30 g G	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	14 days	MS/MSD
Herbicides	8151	Water	1 L AG	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	7 days	MS/MSD
		Soil	50 g G	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	14 days	MS/MSD
Pesticides	608; 8081	Water	1 L AG	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	7 days	MS/MSD
	8081	Soil	30 g G	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	14 days	MS/MSD
PCBs	608; 8082	Water	1 L AG	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	608: 7 days- 8082: none	MS/MSD
	8082	Soil	30 g G	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	none	MS/MSD
Phenol	8040	Water	1 L AG	Cool from $> 0^{\circ}$ to $\leq 6^{\circ}$ C	7 days	MS/MSD

Analysis	Method	Matrix	Volume*/ Container	Preservative	Hold Time	QC (Default)
		Soil	30 g G	Cool from > 0° to ≤ 6° C	14 days	MS/MSD
Semivolatiles	625; 8270	Water	1 L AG	Cool from > 0° to ≤ 6° C	7 days	MS/MSD
	8270	Soil	30 g G	Cool from > 0° to ≤ 6° C	14 days	MS/MSD
TCLP Herbicide	1311/ 8151	Solid	100 g G	Cool from > 0° to ≤ 6° C	14 days	MS/MSD
TCLP Pesticide	1311/ 8081	Solid	100 g G	Cool from > 0° to ≤ 6° C	14 days	MS/MSD
TCLP Semivolatile	1311/ 8270	Solid	100 g G	Cool from > 0° to ≤ 6° C	14 days	MS/MSD
TPH, Diesel	8015; OA- 2	Water	1 L AG	Cool from > 0° to ≤ 6° C	7 days	MS/MSD
		Soil	30 g G	Cool from > 0° to ≤ 6° C	14 days	MS/MSD
Metals						
Mercury	7470	Water	30 mL P,G	HNO ₃ (pH <2)	28 days	MS/MSD
	7471	Soil	0.6 g G	Cool from > 0° to ≤ 6° C	28 days	MS/MSD
Metals	200.7; 200.8; 6010; 6020	Water	50 mL P,G	HNO ₃ (pH <2)	180 days	MS/MSD
		Soil	0.5 g G	Cool from > 0° to ≤ 6° C	180 days	MS/MSD
TCLP Metals	1311/ 6010; 7470	Solid	100 g G	Cool from > 0° to ≤ 6° C	28 days (mercury), 180 days	MS/MSD
TCLP						
CWET, TTLC, and SPLP are the same containers and preservatives as TCLP.						
TCLP ZHE Volatiles	1311/ 8260	Solid	2 X 120 mL G	Cool from > 0° to ≤ 6° C	14 days	
Full Extraction	1311	Solid	100 g G	Cool from > 0° to ≤ 6° C	14 days	
TCLP ZHE Volatiles	1311/ 8260	Liquid	3 X 40 mL G	Cool from > 0° to ≤ 6° C	14 days	
Full Extraction	1311	Liquid	2 L G	Cool from > 0° to ≤ 6° C	14 days	

TCLP Notes:

1. Clients are required to do the phase separation when a liquid sample is in multiple phases.

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Analysis	Method	Matrix	Volume*/ Container	Preservative	Hold Time	QC (Default)

2. When a liquid sample has a % solid content of less than 25 % of the volume, more sample volume will be required to provide an adequate amount of solids for extraction.

3. For samples requiring Matrix QC, 3 times the volume is required.

Radiochemistry						
Carbon-14 (C-14)	EERF C-01	Water	500 mL P,G	DO NOT PRESERVE	180 days	MS/Dup
		Soil	5 g P,G	DO NOT DRY	180 days	MS/Dup
Chlorine-36 (Cl-36)		Soil	2 g P	DO NOT PRESERVE DO NOT DRY	180 days	Duplicate
Gross Alpha/Beta**	900.0; 9310	Water	200 mL P,G	HNO ₃ (pH < 2)	180 days	MS/Dup
		Soil	1 g P,G	None	180 days	MS/Dup
Iodine-129/131 (I-129/131)	GA-01-R MOD	Water	1 L P,G	DO NOT PRESERVE	180 days	Duplicate
		Soil	650 g P,G	DO NOT PRESERVE DO NOT DRY	180 days	Duplicate
(Carrier analysis)	Liquid Scint	Water	2 L P,G	None	180 days	Duplicate
Iron-55 (Fe-55) (Carrier analysis)		Water	500 mL	None	180 days	Duplicate
		Soil	5 g	None	180 days	Duplicate
Gamma Scan	901.1; HASL 300	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
		Soil	650 g P,G	None	180 days	Duplicate
Americium-241/Curium-243 244 (Tracer analysis)	HASL 300 A-R-01	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
		Soil	5 g P,G	None	180 days	Duplicate
Americium-243/Curium-245,246,247,248 (Tracer analysis)	HASL 300 A-R-01	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
		Soil	5 g P,G	None	180 days	Duplicate
Isotopic Plutonium (Tracer analysis)	HASL 300 A-R-01	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
		Soil	5 g P,G	None	180 days	Duplicate

Analysis	Method	Matrix	Volume* / Container	Preservative	Hold Time	QC (Default)
Isotopic Thorium	HASL 300 A-R-01	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Tracer analysis)		Soil	5 g P,G	None	180 days	Duplicate
Isotopic Uranium	HASL 300 A-R-01; DOE U-02	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Tracer analysis)		Soil	5 g P,G	None	180 days	Duplicate
Lead-210 (Pb-210)	EERF PB- 01	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Carrier analysis)		Soil	5 g P,G	None	180 days	Duplicate
Nickel-59/63 (Ni-59/63)		Soil	5 g P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Carrier analysis)		Water	500 mL P,G	None	180 days	Duplicate
Polonium-210 (Po-210)	HASL 300 PO-01	Water	500 mL P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Tracer analysis)		Soil	5 g P,G	None	180 days	Duplicate
Radium-226 and 228 (Ra-226/228)	903.0 / 904.0	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Carrier analysis)	HASL 300	Soil	5 g P,G	None	180 days	Duplicate
Radium-226 (Ra-226)	903	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Carrier analysis)	HASL 300	Soil	5 g P,G	None	180 days	Duplicate
Radium-228 (Ra-228)	904	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Carrier analysis)	HASL 300	Soil	5 g P,G	None	180 days	Duplicate
Total Alpha Radium (Ra-223/224/226)	903	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Carrier analysis)	HASL 300	Soil	5 g P,G	None	180 days	Duplicate
Strontium-89/90 (Sr-89/90)	DOE Sr-02	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Carrier analysis)		Soil	5 g P,G	None	180 days	Duplicate
Strontium-90 (Sr-90)	DOE Sr-02	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate

Analysis	Method	Matrix	Volume*/ Container	Preservative	Hold Time	QC (Default)
(Carrier analysis)		Soil	5 g P,G	None	180 days	Duplicate
Strontium-89 (Sr-89)	DOE Sr-02	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Carrier analysis)		Soil	5 g P,G	None	180 days	Duplicate
Technetium-99 (Tc-99)	HASL 300 TC-02	Water	1 L P,G	HNO ₃ (pH < 2)	180 days	Duplicate
(Tracer analysis)		Soil	10 g P,G	DO NOT DRY	180 days	Duplicate
Total Uranium	ASTM 5174-91	Water	5 mL P,G	HNO ₃ (pH < 2)	180 days	MS/Dup
		Soil	1 g P,G	None	180 days	MS/Dup
Tritium (H-3)	906.0 (distilled)	Water	120 mL G	DO NOT PRESERVE	180 days	MS/Dup
		Soil	100 g G	DO NOT DRY	180 days	MS/Dup

* Sample volumes are based on dry weights; volumes need to be increased if soil is wet/moist.

For samples requiring Matrix QC, 3 times the volume is required.

For normal samples, 2 or more times the volume may be required for re-extracts/digestions.

** Gross Alpha MDA is achievable only when solids are less than 500 ppm.

Container Key

P – Plastic
G – Glass
AG – Amber Glass
AP – Amber Plastic

This table should be used for guidance only. If there is **any** doubt or question, please consult the appropriate group leader. Some analyses may be able to be combined, such as the anions, with no additional volume required. Check with group leader if there are changes in the method or the manner in which the lab performs an analysis.

Attachment 4
(Example: TestAmerica Internal Chain-of-Custody)



TestAmerica St. Louis
Internal Chain-of-Custody

The “✓” (checked) samples on the attached copy of the original chain-of-custody were transferred internally as indicated below:

	Department/Individual	Date
Relinquished by:	Sample Control	
Received by:		

		Date
Relinquished by:		
Received by:		

Client ID	Test(s)	Client ID	Test(s)

Note: Samples not returned to Sample Control Custody within 24 hours, were placed into a sample cooler for interim storage.

Form No.: QA-0063, Revised 06/06/11
SOP References: STL-PM-0002

Attachment 5
(DoD QSM SAMPLE ACCEPTANCE POLICY)

Sample Acceptance Policy

NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. TestAmerica St. Louis will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When completing the chain of custody form, sign your name in the "relinquished by" box.

NELAC requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
- Each sample shall be labeled with unique, durable and indelible identification.
- The samples shall be collected in the appropriate sample containers.
- The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.

The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation. Samples shall be considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservative.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it must be documented on a Condition Upon Receipt Form (CUR) for the project records and the client must be contacted for instructions. If the client decides to proceed with analysis, the project report shall clearly indicate any of the above conditions and the resolution.

If the conditions listed on the Acceptance Policy are not satisfactory and when lacking direction from the client to the contrary, the sample will be rejected.

For DoD QSM project work, sample containers must be certified to meet the "less than" ½ the RL criteria for the analytes of concern. Analytes for which this certification can not be obtained will be noted in the Case Narrative. Upon DoD project approval, the laboratory will analyze method blanks prepared in the containers of concern, qualify and narrate the sample analytes which do not meet the criteria, or take other appropriate action as determined by the DoD project site.

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Attachment 6**Lab / Sample Receiving Responsibility (Beginning 7/1/2012)**

For every log in; sample receiving personnel are responsible to scan the label from the inside of the cooler lid, which will enter the "Service Center Revenue Tracking" into TALs.

If there is no label present on the inside of the cooler lid, sample receiving will select their home location by using the "Default Loc" button.

The screenshot shows the 'Login' window in the TALs software. The window has a menu bar with options like Save, Cancel, Deactivate, Scan, Doc's, NCM, Settings, Delete, All Notes, Project, and Audit. Below the menu bar, there are tabs for Receipt, Login, Job, Info, Containers, Check List, Tasks, Assets, and Comments. The 'Login' tab is active. The form contains the following fields and buttons:

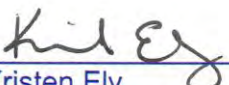
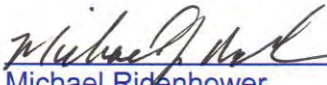
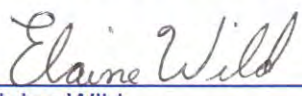
- Number of Coolers:** 0
- Number of Containers:** 12
- Date/Time Received:** 12:25, 02/06/2012
- Received Via:** (dropdown menu)
- Received By:** Langille, Alan D
- Company:** 324th Estate Partner
- State/Province:** California
- Country:** United States of America
- Service Center Revenue Tracking:** 280, TestAmerica Denver
- Default Loc:** (button)
- Chain of Custody:** (Add, Remove buttons)
- Coolers:** (Add, Remove buttons)
- Table:** A table with columns: Temp (°C), Cor Temp (°C), ID, Cooler Description, Relinquished By, Client Cooler, Ice Type, Cooler Condition, and Packing 1.

If the scanning process is not successful (scanning failure, or other reason) the service center location must be selected using the list feature in TALs.

As of July 1, 2012 this data will be considered mandatory in TALs. The Login cannot be saved successfully until the Service Center Tracking field information has been completed.

Once the label from the cooler is scanned, the label must be removed from the cooler and thrown away.

**Title: ANALYSIS OF CYANIDE IN WATER AND SOIL
[SW-846 9010C, 9012B; EPA 335.4]**

Approvals (Signature/Date):	
 Kristen Ely Inorganics Manager	<u>5/23/16</u> Date
 Michael Ridenhower Health & Safety Manager / Coordinator	<u>5/23/16</u> Date
 Tony L. Byrd Quality Assurance Manager	<u>5-23-16</u> Date
 Elaine Wild Laboratory Director	<u>5/23/16</u> Date

This SOP was previously identified as SOP No. ST-WC-0002 Rev. 17

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP is applicable to the determination of cyanide in drinking and surface waters, domestic and industrial wastes, soils/sediments and other solids.
- 1.2 This SOP is based on EPA method 335.4 and SW-846, 9010C and 9012B.
- 1.3 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1 During the distillation, cyanide is converted to hydrogen cyanide vapor which is trapped in a trapping solution containing sodium hydroxide solution.
- 2.2 The cyanide is then converted to cyanogen chloride by reactions with chloramine-T which subsequently reacts with pyridine and barbituric acid to give a red colored complex.

3.0 DEFINITIONS

- 3.1 See the St. Louis TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 CN, Cyanide - Defined as cyanide ion and complex cyanides converted to hydrocyanic acid by reaction in a reflux system of a mineral acid in the presence of magnesium ions.
- 3.3 CN, amenable- Defined as cyanides that are chlorinated which decompose at a pH less than 11.

4.0 INTERFERENCES

- 4.1 Most interference is removed by distillation prior to sample analysis.
- 4.2 The presence of organics may cause the sample to cake the distillation membrane. If this occurs, the addition of Bio-Beads.
- 4.3 Thiocyanates may be an interference when present at high levels (> 10 mg/L).
- 4.4 Sulfides adversely affect the colorimetric procedures. Check for the presence of sulfides in the sample with lead acetate test paper. The paper will turn dark if sulfides are present. Notify PM before proceeding.
 - 4.4.1 If sulfides are detected, treat the sample by using the NaOH/PbCO₃ trapping solution detailed in section 7.0.
 - 4.4.2 NOTE: Samples treated in this manner to remove sulfides must be accompanied by calibration standards that are distilled in the same manner.
- 4.5 Check for the presence of chlorine using KI-starch paper.
 - 4.5.1 If the paper turns dark, notify PM before proceeding. Samples are treated by adding ascorbic acid crystals a few at a time until strip produces no color. Then add an additional 0.06g ascorbic acid for each liter of sample volume.
- 4.6 Nitrate and/or nitrite may form oximes that will generate HCN, therefore all samples are treated for nitrate and/or nitrite.
 - 4.6.1 Add 0.25 mL of the micro sulfamic acid solution just prior to the addition of the releasing agent. Note on the cyanide distillation prep sheet that this step was done.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 **SAFETY CONCERNS OR REQUIREMENTS**

- 5.1.1 Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.2 **PRIMARY MATERIALS USED**

- 5.2.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.
- 5.2.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as potential health hazard; do not ingest.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m ³ (Ceiling)	Inhalation symptoms may include coughing, sneezing, damage to the nasal and respiratory tract. High concentration can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting and diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causing irritation of eyes with tearing, redness and swelling. Greater exposure can cause burns with possible blindness.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Cyanide	Poison Corrosive	5 Mg/M3 (TWA) as CN	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Pyridine	Flammable Irritant	5 ppm (TWA)	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3- (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – time Weighted Average			
Ceiling – at no time should this limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Reflux distillation apparatus (Figure 1)
- 6.2 Lachat hot block
- 6.3 Boiling chips
- 6.4 Glass beads
- 6.5 Lead acetate paper/KI starch indicator paper
- 6.6 Beakers 50 or 100ml

- 6.7 Digestion Tubes, 130mL
- 6.8 High range pH paper
- 6.9 Suitable sample container, 15 ml centrifuge tubes
- 6.10 Pipette, 10 ml, 1 ml and 100 μ l
- 6.11 Graduated cylinders- 100 ml and 25 ml
- 6.12 Stir plate
- 6.13 Lachat collector tubes, user fill option or pre-filled.
- 6.14 Re-pipettor
- 6.15 Tube Press
- 6.16 Sysma Easychem Plus Discrete Analyzer, consisting of:
 - 6.16.1 Sample tray, using 2mL sample cups.
 - 6.16.2 Reagent tray, capable of holding up to 18 reagents, temperature controlled.
 - 6.16.3 Reagent cuvettes, temperature controlled cells where precise mixing of sample and reagents is performed.
 - 6.16.4 Reagent and sample dosing station/dispensing syringe.
 - 6.16.5 Colorimeter – 10mm path length flow cell with a 578nm optical filter.
 - 6.16.6 Peristaltic pump.
 - 6.16.7 PC for data collection, using Sysmedia Easychem software, ver.. 1.9.18
- 6.17 Printer
- 6.18 Analytical Balance
- 6.19 Scoopula
- 6.20 Heat resistant gloves

7.0 REAGENTS AND STANDARDS

- 7.1 All reagents and standards preparation, documentation and labeling must follow the requirements of ST-QA-0002.
- 7.2 NaOH, 1.0N: Dissolve 100 ml 10N of NaOH in distilled water and dilute to 1 liter or purchase commercially.
- 7.3 NaOH, 0.25N: Dissolve 25ml 10N of NaOH in distilled water and dilute to 1 liter or purchase commercially
- 7.4 NaOH/PbCO₃ Trapping Solution (for sulfide positive samples): Dissolve 0.8g of lead carbonate (PbCO₃) into 1L of 1.0N NaOH
- 7.5 Potassium Cyanide, reagent grade
- 7.6 Potassium Hydroxide, reagent grade

- 7.7 Sulfuric acid, concentrated
- 7.8 Stock QC Cyanide Standard: purchase commercially or prepare a 1000 mg/L CN by dissolving 2.51 g of potassium cyanide and 2.0 g of potassium hydroxide in one liter of DI water. Store under refrigeration. Stability is six (6) months or depends upon the manufacturer's recommendations.
- 7.9 Intermediate QC Cyanide Standard: for LCS/Low, High, and MS (10 mg/l): Dilute 1.0 ml of stock cyanide to 100 ml with 0.25 N NaOH. This standard should be preserved with 1 ml of 1.25 NaOH if the standard is prepared in advance. Stability is six (6) months or until parent stock expires.
- 7.10 Stock Cal Cyanide Standard: purchase commercially or prepare a 1000 mg/L CN by dissolving 2.51 g of potassium cyanide and 2.0 g of potassium hydroxide in one liter of DI water. Store under refrigeration. Stability is six (6) months or depends upon the manufacturer's recommendations. The standards must be from a different source than the QC stock.
- 7.11 Intermediate Cal Cyanide Standard (10 mg/l CN): To a 100 ml volumetric flask containing approximately 90 ml of 0.25N NaOH, add 1.0 ml of stock standard. Bring to volume with 0.25 N NaOH and mix until homogeneous. Stability is six (6) months or until parent stock expires
- 7.11.1 Cyanide Calibration Curve: Using the intermediate cyanide standard and 0.25N NaOH for dilution, prepare the following working standards. The lowest standard must be at the RL or lower than the RL. Stability for each Calibration Curve point is 1 week.

Concentration of Working Standard (µg/L)	Aliquot of Intermediate (10 ppm) Standard (ml)	Dilution Volume (ml)
0	0	100
5	0.05	100
20	0.2	100
100	1.0	100
250	2.5	100
300	3.0	100
400	4.0	100
500	5.0	100

NOTE: If the samples were treated for sulfides before distillation, for method 9010, the calibration standards must be treated by the same procedure.

NOTE: The above intermediate cyanide standard is only used for calibration standards and CCV preparation.

- 7.12 ICV (200ug/L): Pipette 2ml of QC intermediate and bring to 100ml with 0.25 N NaOH, stability 1 week.
- 7.13 QC for Micro Distillation
- 7.13.1 High check standard (HLCS) (400ug/L or 4.8mg/kg): Pipette 6ml of the 400ug/L curve solution into a sample cup (0.5g of glass beads to be included if soil prep). This will be used as a distilled check standard (high) for Method 9010C, 9012B and Method 335.4.
- 7.13.2 LCS/Low check standards (200ug/L or 2.4mg/kg): Pipette 6ml of the 200ug/L ICV solution into a sample cup (0.5g of glass beads to be included if soil prep). This will be used as a distilled check standard (low) for Method 9010C, 9012B and Method 335.4.

- 7.13.3 MS for waters (200ug/L): Pipette 0.12mL of the QC intermediate solution into the sample cup containing 6mL of sample.
- 7.13.4 MS soils (2.4mg/kg): Pipette 0.12mL of the QC intermediate solution into 0.5g of sample and 5ml of DI water.
- 7.14 Magnesium Chloride ($\text{MgCl}_2 \times 6\text{H}_2\text{O}$), Reagent Grade.
- 7.15 Sulfamic acid, Reagent Grade
- 7.16 Sulfamic acid solution: Dissolve 9.6g of sulfamic acid in DI water. Dilute to 100mls.
- 7.17 Deionized water, ASTM Type II.
- 7.18 Calcium Hypochlorite
 - 7.18.1 Calcium Hypochlorite solution: Dissolve 5 g into 100 mL of DI water.
- 7.19 Ascorbic acid crystals.
- 7.20 Barbituric acid, purified
- 7.21 Pyridine, reagent grade
- 7.22 Hydrochloric acid, concentrated
- 7.23 Deionized (DI) water, obtained from the Milli-Q unit.
- 7.24 Sodium Phosphate Monobasic ($\text{NaH}_2\text{PO}_4 \bullet \text{H}_2\text{O}$)
- 7.25 Cadmium carbonate, powdered.
- 7.26 Micro Distillation Releasing Agent
 - 7.26.1 In a hood, place 110.8g of DI water into a 500ml beaker. Add and completely dissolve 32.2g of magnesium chloride ($\text{MgCl}_2 \times 6 \text{H}_2\text{O}$). Add 139g (approximately 80ml) concentrated sulfuric acid SLOWLY (in roughly 20ml increments) while stirring. Let cool and add to the repipettor
- 7.27 Discrete Reagent Preparation
 - 7.27.1 Phosphate Buffer – add 13.8g sodium dihydrogen phosphate to a 130mL digestion tube and dilute to 100mL with DI. Stable for one month.
 - 7.27.2 Chloramine – T Reagent – add 0.4g chloramine-t to 130mL digestion tube and dilute to 100mL with DI. Stable for one day.
 - 7.27.3 Pyridine Barbituric Acid Reagent – in a fume hood, add 1.5g barbituric acid to a 130mL digestion tube, add about 50mL DI and begin to mix. Add 7.5mL pyridine and mix some more. Add 1.5mL concentrated HCl and dilute to 100mL. Stable for one month.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

- 8.2 Water samples must be preserved with 2 ml of 10 N sodium hydroxide per liter of sample (pH > 12) at the time of collection.
- 8.3 Oxidizing agents (chlorine) – Test a drop of sample with KI-starch paper. A blue color indicates treatment is needed. Add ascorbic acid (a few crystals at a time) until a drop of sample produces no color on the indicator paper. Add 0.6g of additional ascorbic acid for each liter of sample. This must be done by the sampler immediately after collection. Water samples must be preserved at the time of collection with 2ml of 10 N NaOH per liter (pH>12).
- 8.4 Method 335.4 requires the sample be checked for sulfide in the field and appropriate preservation be performed by the samplers. TestAmerica St. Louis does not collect samples. The laboratory will check for sulfide in the lab (Section 11). The project manager will notify the client if sulfide is found in the sample and request re-sampling.
- 8.5 Samples should be analyzed as soon as possible after collection. The maximum holding time for soil and water is 14 days from collection. If storage is required, the samples should be stored in a refrigerator or in an ice chest filled with water and ice to maintain temperature at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), a High Level Laboratory Control Sample (HLCS), Matrix Spike (MS) and Sample Duplicate (SD). In the event that there is insufficient sample to analyze a MS, an LCS Duplicate (LCS D) is prepared and analyzed.
- 9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual “sample batch” does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 Method Blank

- 9.2.1 A Method Blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 For Water analyses, the method blank is organic free water.
- 9.2.4 For Soil analyses, the method blank is comprised of boiling chips or glass beads.

9.3 Laboratory Control Sample

- 9.3.1 A LCS/HLCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS and HLCS must be prepared with every sample batch.
- 9.3.3 For Water analyses, the LCS and HLCS is comprised of DI water fortified with Cyanide.
- 9.3.4 For Soil analyses, the LCS and HLCS is comprised of glass beads fortified with Cyanide.

9.4 Sample Duplicates

- 9.4.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision. For this method there should be one sample duplicate run per every ten analytical samples.

- 9.4.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.
- 9.4.3 Additional Sample Duplicates do not count towards the 10 samples in an analytical batch.
- 9.5 **Matrix Spike/Matrix Spike Duplicate (Matrix Spike/Sample Duplicates)**
 - 9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. For this method there should be one MS run per every ten analytical samples.
 - 9.5.2 MS/MSD samples do not count towards the 10 environmental samples in a sample batch.
 - 9.5.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.
- 9.6 **Procedural Variations/ Nonconformance and Corrective Action**
 - 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
 - 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
 - 9.6.3 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Calibrate the balance daily when used. The balance is calibrated in accordance with SOP ST-QA-0005.
- 10.2 The Systema instrument must be calibrated every day before it is used for sample analysis. Include the instrument standardization date and time in the raw data.
- 10.3 The instrument is calibrated by analyzing a calibration blank and the calibration standards described in ascending order of concentration.
- 10.4 A linear calibration curve is plotted and a correlation coefficient calculated by the TRAACS 800 software by comparing the standard concentration to the instrument response.
- 10.5 The calibration curve must have a correlation coefficient of 0.995 or greater.
 - 10.5.1 Evaluate whether the problem is related to the analytical range. The low standard or the high standard having a response that is out of line with the others typically expresses this. In such situations, the analyst should determine whether the analytical range should be decreased by deleting the high or low standard. If the low standard is deleted, adjust the reporting limit. If the high standard is deleted, dilute samples appropriately. If the high standard shows saturation of the response, the calibration range must be adjusted. |
 - 10.5.2 Evaluate whether the problem was related to a misinjection of one of the standards. This must be confirmed by reanalyzing the suspect standard. If the reanalyzed standard is in line with the others, the misinjection of the original standard is confirmed. At this point, the analyst has the choice of deleting the offending calibration point, or replacing it with the new run. The minimum number of calibration points must be five.
- 10.6 Analyze an ICV immediately after the last calibration standard.

- 10.6.1 If the measured value of the ICV is not within 10% of the true value, for 300 series methods, $\pm 15\%$ for SW846 methods, the analysis must be stopped, the problem corrected, the instrument recalibrated, and the calibration re-verified.
- 10.7 Analyze an ICB immediately after the ICV.
 - 10.7.1 If the ICB or CCB result exceeds the reporting limit, stop analysis, correct the problem, recalibrate, verify the calibration and reanalyze all analytical samples analyzed since the last compliant calibration blank.
- 10.8 Analyze a CCV at the beginning of the run, every 10 samples during the sequence and after the last analytical sample.
 - 10.8.1 The CCV and ICV must be $\pm 10\%$ of true value. The high and low distilled check standard must be $\pm 10\%$ for 300 series methods and $\pm 15\%$ for SW846 methods.
 - 10.8.2 If the CCV value is not within $\pm 10\%$ of the true value for 300 series methods, and $\pm 15\%$ for SW846 methods, stop analysis, correct the problem, recalibrate, verify the calibration and reanalyze all analytical samples analyzed since the last compliant CCV.
- 10.9 Analyze a CCB immediately after each CCV.

11.0 PROCEDURE

- 11.1 Pretreatment of samples for cyanide amenable to chlorination (perform in a hood):
 - 11.1.1 Two sample aliquots are required to determine cyanide amenable to chlorination. To one aliquot of 50 ml add Calcium hypochlorite solution drop-wise while agitating and maintaining the pH between 11 and 12 with NaOH solution.
 - 11.1.2 Test to ensure residual chlorine is present using KI-starch paper. A blue color on the paper indicates sufficient chlorine level.
 - 11.1.3 After maintaining the chlorine level for one hour excess chlorine is removed by small additions of ascorbic acid (0.1g) until KI-starch indicator paper fails to give blue result. A small addition of ascorbic acid (0.1g) is then added to ensure chlorine removal.
- 11.2 MICRO DISTILLATION
 - 11.2.1 Warm Lachat hot block to 120 degrees C.
 - 11.2.2 Place as many collector tubes as needed for the batch in the rack with the "D" end down and the "M" end up.
 - 11.2.3 Pipette approximately 1.51 mL of 1.0N NaOH in M end of User-Fill collector tubes. Center a 30mm membrane over the "M" end and seat a cap firmly on the end. If using pre-filled collector tubes, put in rack with "M" end up.
 - 11.2.4 Put sample tubes into the sample tube rack.
 - 11.2.5 Place 6.0 mL of sample (or standard) into each sample tube
 - 11.2.5.1 For soil/sludge add between 0.5 and 1.0 g of soil or sludge to 5.0 mL of water.
 - 11.2.6 Test all samples for the presence of sulfides and chlorine. (see section 4.6, 04.7 and 4.8)
 - 11.2.7 In LIMS, record the following information: date and time distilled, sample weights and volumes, QC batch number for prep blank and LCS, comments describing any sample reactions during preparation, and test strips results.
 - 11.2.8 Treat all samples for nitrates by adding 0.25mL of the sulfamic acid solution.
 - 11.2.9 Prime the re-pipettor
 - 11.2.10 Add 0.75 mL of the releasing agent to sample tubes.
 - 11.2.11 Immediately push the "D" end of a Cyanide-1 collector tube over each sample tube and use a "tube-press" until the stop ring on the sample tubes hits the "D" end of the collector tube.
 - 11.2.11.1 Grip collector tube at breakaway point to keep tube from shifting during pressing procedure. Use smooth constant pressure to avoid damaging the tubes.

- 11.2.12 Wearing heat resistant gloves, push the sample tube and “D” end of each tube completely down in the preheated block.
 - 11.2.13 Set timer for 30 minutes.
 - 11.2.14 After 30 minutes, wearing heat resistant gloves, remove the first tube from the block and immediately twist off the sample tube. **This must be done within 4 seconds to avoid “suck back”.**
 - 11.2.15 Dispose of acid solution in appropriate waste container. Discard sample tube into trash container.
 - 11.2.16 Repeat this procedure for the rest of the sample tubes.
 - 11.2.17 Invert all collector tubes and place them in the collector tube rack with the “D” end up and allow to cool for ten minutes.
 - 11.2.18 Hold each tube horizontally and rinse the walls with the distillate. Slowly roll the tube to make the drops clinging to the walls settle down into the bulk of the distillate.
 - 11.2.19 With the “D” end up, flick the tube with a finger to cause stubborn drops to fall into the “M” end.
 - 11.2.20 With the “D” end up, break the collection tube by pulling the “D” end hard toward yourself. Twist off and discard the “D” end.
 - 11.2.21 Place the collector tube rack with the “M” end down.
 - 11.2.22 Dilute up to the 6.0 mL mark with DI water in the remaining “M” end, resulting in the original sample volume in 0.25M NaOH.
 - 11.2.22.1 Mix by shaking with a gently whipping motion, and transfer to a labeled 15mL centrifuge tube.
 - 11.2.22.2 Samples are ready for analysis.
- 11.3 Instrument Startup and Analysis – Systea Instrument
- 11.3.1 Power on the Easychem unit. Select the Easychem icon to open the data acquisition software. Login to the software. When the system has been properly initialized, a message box displaying “Instrument ready” will appear.
 - 11.3.2 Fill the rinse water container with new reagent grade water. Tighten the peristaltic pump winding.
 - 11.3.3 Empty the waste container.
 - 11.3.4 Through the “User Service” tab, have the instrument perform 15 washes, followed by the filter’s autozero function. New values for all filters must match within 5% of the old values. If they do not meet the criteria, the instrument requires maintenance.
 - 11.3.5 Through the “Operations” tab, and “Worklist Editor,” create a sample worklist with the appropriate method applied to each sample. Load the samples, calibrants, QC, LCS, MB, etc., into the autosampler tray.
 - 11.3.6 Prepare the working reagents. Fill the R1 (Phosphate Buffer), R2 (Chloramine-T), and R3 (Pyridine Barbituric Acid) reagent containers and place in the correct cartridge position as specified in the instrument method (R1 is position R8, R2 is position R9, and R3 is position R10). Place the cartridge into the reagent tray on the analyzer.
 - 11.3.7 Through the “Operations” tab select “Start Analysis”. Check to ensure that the current worklist is loaded. Change both the analysis file (.slz file) and the analysis text file (.txt file) locations and name to the type and date of the analysis (i.e. CN 041111).
 - 11.3.7.1 Verify that there are sufficient reaction cuvettes to complete the run. Replace if needed, and change the starting location.
 - 11.3.7.2 Verify that reagent and cuvette temperatures are within control. (Instrument will monitor temp and alert analyst to excursions.) Start the analysis.
 - 11.3.7.3 To export results, click “File,” “Export Results,” then “Continue.” Save the file as the analysis name, followed by the date (ex: CN051414). At the top of the window, under “Save In” select “Easychem,” followed by “TALS Export” and click save. Then exit out of the program and open the desktop folder “TALS Export,” right click on the file that was just saved, and select “Send To,” and select the appropriate method being uploaded. The data is now uploaded to LIMS.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the St. Louis ST-QAM.
- 12.2 The EasyChem Plus program will prepare a standard curve by plotting peak heights of standards against concentration values. It will then compute the concentration of samples by comparing sample peak heights with standards.

12.3 Final Concentration (water)

$$\text{Final Concentration } (\mu\text{g/L}) = \frac{(\text{RV} \times \text{D})}{V_O} \times V_D$$

where:

RV = Raw value ($\mu\text{g/L}$) from instrument

D = Dilution factor, if any

V_D = Distillate Volume (L)

V_O = Original volume (L)

12.4 Final Concentration (soil)

- 12.4.1 If the concentration for soil is on dry weight basis, determine the solid fraction of the sample. Refer to SOP STL-WC-0005, Percent Solids Determination.

$$\text{Final Conc } (\mu\text{g/g}) = \frac{(\text{RV} \times \text{D})}{W \times \text{SF}} \times V_D$$

where:

W = Weight of sample (g)

SF = Solid Fraction (%Solids/100)

And remaining terms are as defined in 12.1

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.
- 13.2 Method Blank
- 13.2.1 Acceptance Criteria: No target analytes may be present in the method blank above the reporting limit.
- 13.2.1.1 Project specific requirements, if more stringent than the lab routine procedure (e.g. no target analytes present above $\frac{1}{2}$ RL), will be noted in the comments section of LIMS.
- 13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:
- 13.2.2.1 Method Blank Contamination – If the Method Blank concentration exceeds the applicable criteria the samples must be re-prepped/re-analyzed unless their concentration is less than the RL or greater than ten times the concentration found in the blank.
- 13.3 Laboratory Control Sample (LCS)
- 13.3.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in the LIMS.
- 13.3.2 Corrective Action for LCS not meeting acceptance criteria:

- 13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM, (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.
- 13.3.2.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.
- 13.3.2.3 RPD excursion for LCS/LCSD – If spike recoveries are acceptable, data is evaluated for reportability and an NCM generated. If spike recoveries and RPD are outside limits, batch is re-prepped/re-analyzed for affected analytes.
- 13.4 Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/SD)
 - 13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in LIMS.
 - 13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:
 - 13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration.
 - 13.4.2.1.1 If the affected analyte concentration in the original sample is greater than four times the amount spiked, recovery information is ineffective and the data is reported with an NCM.
 - 13.4.2.1.2 If the excursion is due to a physically evident matrix interference, the data is reported with an NCM.
 - 13.4.2.1.3 In cases where the MS and/or MSD don't meet criteria, but the RPD is in control, data may be reported with an NCM.
 - 13.4.2.1.4 When the MS/MSD recoveries and the %RPD are outside criteria, the batch is re-prepped and re-analyzed for the affected analytes.
 - 13.4.2.2 % RPD excursion for MS/MSD - sample/sample duplicate – If there is physical evidence of non-homogeneity or matrix interference, the data is reported with an NCM describing the physical evidence; otherwise the sample should be re-prepped and re-analyzed
- 13.5 Sample result evaluation
 - 13.5.1 Dilutions
 - 13.5.1.1 If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range.
 - 13.5.1.1.1 An NCM is written to document the reason for the dilution.
 - 13.5.1.1.2 If the dilution causes the Spike(s) to be diluted out an NCM is written to document the excursion.
- 13.6 Insufficient Sample
 - 13.1.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification

- 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Wastewater contaminated with Pyridine shall be collected in a 5-gallon poly waste accumulation container, labeled as "Waste Pyridine". Please refer to SOP ST-HS-0001 and ST-HS-0004 for proper waste handling.

17.0 REFERENCES

- 17.1 Standard Methods for the Examination of Water and Wastewater, 17th Edition, Method 4500-CN.
- 17.2 EPA Manual 600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983. Method, 335.4.
- 17.3 EPA SW-846, Update IV, Method 9010C and Method 9012B November 2004\
- 17.4 "Micro Distillation Operations and Applications", Lachat Instruemts Catalog 01304, HACH Co. 2004 – 2006
- 17.5 "Easy Chem Methodology", Systea Scientific, LLC, February 2009
- 17.6 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.7 St. Louis Quality Assurance Manual (ST-QAM), current revision.
- 17.8 Associated SOPs (current revisions):
 - 17.8.1 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.8.2 ST-QA-0002, Standards Preparation.
 - 17.8.3 ST-QA-0011, Data Review, Verification and Reporting.
 - 17.8.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process.
 - 17.8.5 ST-WC-0005, Percent Solids Determination.

- 17.8.6 ST-IP-0004, Glassware Preparation for Inorganic and Trace Metals Analysis
- 17.8.7 ST-QA-0005, Calibration and Verification Procedure for Laboratory Support Equipment
- 17.8.8 ST-HS-0001, Waste Minimization Plan
- 17.8.9 ST-HS-0004, Hazardous Waste Management Plan

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCED METHODS

- 18.1 The SW846 9000 series methods for Cyanide analysis require that the Method of Standard Addition (MSA) be used for samples with matrix interferences. TestAmerica St. Louis does not perform an MSA. For samples demonstrating matrix interference, the sample is diluted to try to eliminate the interference and an NCM is included in the case narrative.
- 18.2 Amounts of standards and reagents used may vary from the method guidance. QC check samples, MDL studies and PT sample results demonstrate that the changes have not adversely impacted the results of the analysis.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Updated section 4.0 regarding PM notification.
- 19.2 Rev. 14:
 - 19.2.1 Updated title of Sop from "Cyanide Analysis by the Technicon Traacs 800 Auto Analyzer" to "Analysis of Cyanide in Water and Soil".
 - 19.2.2 Updated the Method procedural numbers to 9010C and 9012B throughout SOP.
 - 19.2.3 Added instructions for Micro Distillation treatment of sulfide interferences to Section 4.
 - 19.2.4 Removed Phosphoric Acid and Silver Nitrate from the safety table in section 5.0.
 - 19.2.5 Added Systea Easychem Plus Discrete Analyzer and its supplies to section 6.0.
 - 19.2.6 Added a stock cal standard along with instructions to section 7.0.
 - 19.2.7 Inserted a Traacs Reagent section, 7.44.
 - 19.2.8 Inserted a Discrete Reagent Preparation section, 7.45.
 - 19.2.9 Corrected SOP reference for balance calibration procedure from ST-WC-0005 to ST-QA-0005 in section 10.0.
 - 19.2.10 Removed the requirement for the undistilled ICVS from Section 10.
 - 19.2.11 Noted that the pH check is done at time of receipt by the Sample Control Dept in section 11
 - 19.2.12 Updated Section 11.5 with new NAP instructions.
 - 19.2.13 Created Section 11.6 for start-up and analysis using the Systea Instrument
 - 19.2.14 Updated Reference Section (17) to include method revisions and new instrument documentation.
 - 19.2.15 Updated the Cyanide Distillation Log worksheet (Attachment #2).
- 19.3 Rev. 15:
 - 19.3.1 Updated section 7.0 regarding the amounts of NaOH used.
 - 19.3.2 Updated the amount of NaOH used during pipetting for micro-distillation in section 11.3.
 - 19.3.3 Updated information for pre-filled collection tubes in section 11.3.23.
- 19.4 Rev. 16: (6/18/2013)
 - 19.4.1 Removed references to "Clouseau" and "QuantIMS" replacing with "LIMS".
 - 19.4.2 Removed Structure and Analysis Codes from SOP and referenced LIMS as the new source to recover that information in section 1.0.
 - 19.4.3 Updated supplies in section 6.0.
 - 19.4.4 Removed the use of Sodium Hypochlorite throughout the entire SOP and replaced it with Calcium Hypochlorite.
 - 19.4.5 Updated section 13.0.
 - 19.4.6 Updated section 15.0.
- 19.5 Rev 17: (1/16/2015)
 - 19.5.1 Updated Inorganics Manager on cover page
 - 19.5.2 Added Glass Beads to section 6
 - 19.5.3 Reorganized Cal and QC stocks and their intermediates in section 7.

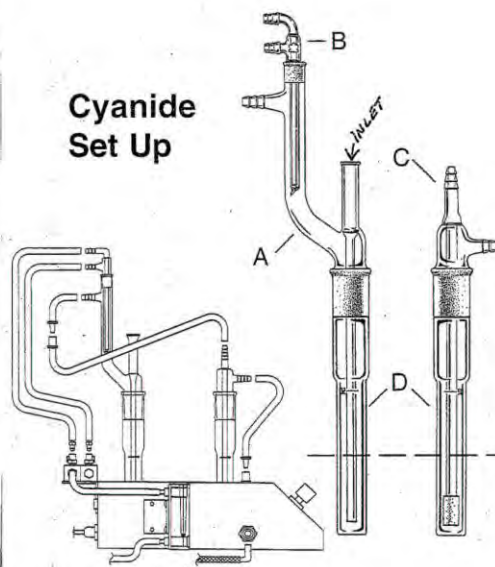
- 19.5.4 Clarified throughout the SOP that the High Check Standard or HCS is now called the HLCS in LIMS.
- 19.5.5 Removed redundancies throughout the SOP
- 19.5.6 Removed references to the distillation log from section 11 and also removed the excel spreadsheet for this log from the last page of the SOP.
- 19.5.7 Updated the correct upload procedures for the TRAACS and Systea in sections 11.5 and 11.6.
- 19.5.8 Added three Associated SOPs to section 17.9
- 19.6 Rev. 18 (05/23/2016)
 - 19.6.1 Removed references to TRAACS
 - 19.6.2 Removed references to Midi distillation

Attachment 1

Figure 1 -



Kible - Konte Glassware



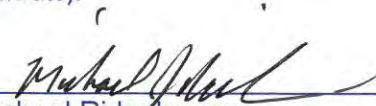
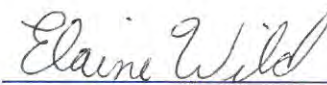
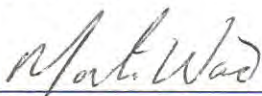
Cyanide Distillation Apparatus, Complete, MIDI-VAP™ 2000

Fisher Article No.	Description	Case Qty.	Case Price
K479200-2000	MIDI-VAP™ 2000, Cyanide Complete Includes the following:	1	6,789.51K
K479160-0000	Midi-Cyanide Glassware (479200-2000 supplied with 10 sets)	1	246.14
K720140-2000	MIDI-VAP™ 2000 Manifold Heater, 115-V	1	4,841.53
Component Parts			
K479161-0000	(A) Head, 3/29/42 Lower, 3/14/20 Upper	1	96.90
K282000-0000	(B) Cold Finger Condenser, 3/14/20, #1 Hose Connection	1	47.11
K479162-0023	(C) Dispersion Tube, Coarse Porosity, 3/29/42, #1 Hose Connection	1	70.45
K479155-0050	(D) Purge Tube, 3/29/42 (2 Supplied)	1	42.10

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Title: PERCENT SOLIDS DETERMINATION

Approvals (Signature/Date):			
			
Sarah Bernsen Pre-prep Department Manager	Michael Ridenhower Health & Safety Manager / Coordinator	Elaine Wild Laboratory Director	Marti Ward Quality Assurance Manager
Date	Date	Date	Date
6/29/16	6/29/16	6/29/16	6/29/16

This SOP was previously identified as SOP No. ST-WC-0005 Rev. 12

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1.0 SCOPE AND APPLICATION

- 1.1 The purpose of this standard operating procedure is to define the method used at TestAmerica St. Louis for determination of percent solids of an environmental sample.
- 1.2 This SOP is based on the Percent Solids Determination Procedure for EPA CLP SOW ILM05.3.
- 1.3 This procedure is applicable to solid samples (soil, sand, clay, etc.) which are received for analysis. The determination is performed when client requested and for the purpose of correcting concentrations of compounds found in organic and inorganic analyses to reflect the amount present in dry weight of samples.
- 1.4 This SOP is **not** intended to be used for solid determination methodologies (TSS, TDS, TVS, TS). These method procedures are found in SOP, ST-WC-0036.
- 1.5 Due to the potential of explosion, this procedure is not to be performed on samples submitted for nitroaromatic analysis when there is the possibility that the samples contain a high level of explosives. If samples are identified as having the potential of containing a high level of explosives, sample drying must follow the procedure described in SOP ST-LC-0002.
- 1.6 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1 A weighed amount of a solid sample is dried overnight in an oven at 105 °C to drive off water. The cooled sample is reweighed to determine the percent moisture or percent solid of the sample as received.

3.0 DEFINITIONS

- 3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

- 4.1 None

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

- 5.2.1 Due to the potential of **explosion**, this procedure is not to be performed on samples submitted for nitroaromatic analysis when there is the possibility that the samples contain a high level of explosives. If samples are identified as having the potential of containing a high level of explosives, the procedure for sample drying must follow the procedure described in SOP ST-LC-0002.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

5.3.2 There are no hazardous reagents used in this procedure.

6.0 EQUIPMENT AND SUPPLIES

6.1 Analytical balance capable of weighing accurately to the nearest 0.0001 g.

6.2 Aluminum pans capable of holding approximately 10 g of solid samples for drying.

6.3 Desiccator

6.4 Oven, capable of maintaining a temperature from 103 to 105 °C.

7.0 REAGENTS AND STANDARDS

7.1 None

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 No specific holding time is required for this procedure.

9.0 QUALITY CONTROL**9.1 Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a Sample Duplicate (SD).

9.2 Sample Duplicate

9.2.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.2.2 If there is insufficient sample to perform a Sample Duplicate an NCM is written to document the insufficient volume.

9.3 Procedural Variations/ Nonconformance and Corrective Action

9.3.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

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- 9.3.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Balance calibration must be checked daily when used. Refer to SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."

11.0 PROCEDURE

- 11.1 Remove one lettered shelf from the oven and allow to cool to room temperature on a large cart. (Shelves are used in alphabetical order)
- 11.2 Weigh a clean labeled aluminum pan on a calibrated analytical balance. Record the weight to 0.01 g. as the "pan weight" on the appropriate spreadsheet. Record the number of the balance used on the data spreadsheet.
- 11.3 Document the letter of the batch in the spreadsheet in the space labeled as "letter", this letter corresponds to the side of the shelf where the batched samples will be placed as well as the batch document title.
- 11.4 Add approximately 10 g. of solid sample to each pan, and record the weight to 0.01 g. as the "Wet Soil + Pan" on the spreadsheet.
- 11.4.1 Samples with a standing water layer should be homogenized as much as possible before removing the aliquot for % solid determination.
- 11.4.2 After all samples have been weighed and documented in the spreadsheet confirms all the applicable data blanks on the spreadsheet have been filled in to document time, date, analyst, etc.
- 11.5 Place entire shelf containing the samples in back in the oven heated to 103 - 105°C for at least 12 hours, or overnight, (but no more than 24 hours). Monitor the temperature at beginning and end of drying time, to assure that the sample was dried at the correct temperature for the required length of time.
- 11.6 Remove entire oven shelf and place on large cart to cool to room temperature.
- 11.7 Reweigh cooled samples and pan after zeroing balance and record as "dry soil plus pan" weight to the nearest 0.01g.
- 11.7.1 If a drying time of less than 12 hours is required by contractual data delivery restraints, constant weight must be demonstrated in the following manner:
- 11.7.1.1 Perform steps 11.3 through 11.5 with a drying time no less than one hour.
- 11.7.1.2 Repeat the drying cycle at least once.
- 11.7.1.3 Constant weight is achieved when there is a loss in weight of no greater than 0.01 g between the start and final weights of the last cycle.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the St. Louis ST-QAM.
- 12.2 Enter the sample weights into the Percent Total Solid/Percent Moisture Logsheet or upload into TALS.
- 12.3 Calculation of percent solid of the sample is as follows:

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$$\% \text{ solid} = \frac{\text{dry soil plus pan} - \text{pan}}{\text{wet soil plus pan} - \text{pan}} \times 100$$

12.4 Percent moisture is calculated as follows:

$$\% \text{ Moisture} = 100 - \% \text{ Solid}$$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Sample Duplicates

13.2.1 Sample duplicate criteria is 30% RPD.

13.2.1.1 % D excursion for sample/sample – If there is physical evidence of non-homogeneity or matrix interference, the data is reported with an NCM describing the physical evidence; otherwise the sample should be re-prepped and re-analyzed.

13.3 Insufficient Sample

13.3.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

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- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
 - 16.2.1 The following waste streams are produced when this method is carried out.
 - 16.2.1.1 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 EPA CLP Program SOW ILM05.3, Exhibit D, Section 1.6
- 17.2 TestAmerica Quality Assurance Plan (ST-QAM), current revision
- 17.3 TestAmerica Corporate Environmental Health & Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002) current revisions.
- 17.4 Associated SOPs, current revisions:
 - 17.4.1 ST-QA-0002, Standard and Reagent Preparation
 - 17.4.2 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.
 - 17.4.3 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.4.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process.
 - 17.4.5 ST-IP-0004, Labware Preparation for Inorganic and Trace Metal Analysis.
 - 17.4.6 ST-WC-0036, Determination of Solids in Water and Wastes
 - 17.4.7 ST-LC-0002, Analysis of Nitroaromatic and Nitroamine Explosives by HPLC.

18.0 MODIFICATIONS TO CITED METHOD

The method calls for the weighing dishes to be processed with "the cover tipped to allow for moisture escape".
The laboratory does not cover the aluminum weighing dishes.

19.0 CHANGES TO PREVIOUS REVISION

- 19.1 Updated section 11.0, referring to the use of lettered shelf to cool samples and documenting this information in the proper spreadsheet.
- 19.2 Revision 10:
 - 19.2.1 Added cooling period before handling oven shelf in section 11.6.
- 19.3 Revision 11:
 - 19.3.1 No Changes, Annual Review.
- 19.4 Revision 12: (07/15/2013)
 - 19.4.1 Clarified the procedure for cooling samples after removing from the oven in section 11.6.
 - 19.4.2 Updated section 13 with corrective actions
 - 19.4.3 Removed references to QuantIMS and Clouseau
- 19.5 No changes, annual review (05/06/2015)
- 19.6 Revision 13 (6/30/16)
 - 19.6.1 Changed Quantims to TALS in section 12.2

TestAmerica St. Louis Laboratory
Constant Weight Determination Form

*to be used when drying time is less than 12 hours.
drying cycles must be at least one hour*

Date: _____

Time in: _____ Temp in: _____ Analyst _____

Time out: _____ Temp out: _____

Time in: _____ Temp in: _____ Batch _____

Time out: _____ Temp out: _____

Time in: _____ Temp in: _____

Time out: _____ Temp out: _____

Pan Number	Sample Id	pan weight g	wet + pan g	dry + pan g	2nd cycle final Dry+Sam+Pan g	3rd Cycle final Dry+Sam+Pan wt	Constant Weight Achieved (for 2nd cycle)	Constant Weight Achieved (for 3rd cycle)	Results % Solids	Results % Moisture
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										
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16										
17										
18										
19										
20										
21										

File: Slivr01\Public\INORGANICS\Wet Chemistry\spreadsheets\Percent Solids.xls, modified 06/14/07

Title: pH ANALYSIS IN WATER AND SOIL
[SW-846 9040C, 9045D; EPA 150.1; Standard Methods 4500-H⁺B]

Approvals (Signature/Date):

 7/27/16
Kristen Ely
Inorganics Manager
Date

 7/27/16
Michael Ridemower
Health & Safety Manager / Coordinator
Date

 7/27/16
Marti Ward
Quality Assurance Manager
Date

 7/27/16
Elaine Wild
Laboratory Director
Date

This SOP was previously identified as SOP No. ST-WC-0011 Rev. 16

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes a method for the measurement of pH in aqueous samples, those multiphase wastes where the aqueous phase constitutes at least 20% of the total volume of the waste, and soils
- 1.2 pH determination in aqueous samples is performed by SW-846 9040C, EPA 150.1 and Standard Methods 4500-H⁺B.
- 1.3 pH determination in soil samples is performed by SW-846 9045D.
- 1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1 The pH of a sample is determined electrometrically using a combination electrode. The pH electrode is calibrated using a series of standard solutions of known pH.

3.0 DEFINITIONS

- 3.1 See the TestAmerica Quality Assurance Manual for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

- 4.1 Samples with a pH lower than 2 may give falsely high readings by pH meter.
- 4.2 Samples with a pH above 12 may give falsely low readings.
- 4.3 Sodium error at pH levels > 10 can be reduced by using a low-sodium-error electrode.
- 4.4 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by distilled water rinsing. An additional treatment with hydrochloric acid (1:9) may be necessary to remove any remaining film.
- 4.5 Temperature effects on the electrometric measurement of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source is the change of pH inherent in the sample at various temperatures. This error is sample dependent and cannot be controlled. It should therefore be noted by reporting both the pH and temperature at the time of analysis.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under

the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 None

5.3 PRIMARY MATERIALS USED

5.3.1 There are no materials used in this method that have a serious or significant hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Poison Corrosive	5 ppm (Ceiling)	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit. If an OSHA limit is unavailable, the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Value (TLV) is listed.			
Ceiling – At no time should this limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 pH meter – ThermoScientific Orion 5 Star Plus or equivalent.
- 6.2 Combination pH electrode with temperature sensor probe for automatic compensation
- 6.3 Analytical balance capable of weighing to 0.1 g
- 6.4 Disposable cups, 100-mL
- 6.5 Shaker Table
- 6.6 50-mL centrifuge tube
- 6.7 Volumetric DI Water dispenser pipet

7.0 REAGENTS AND STANDARDS

- 7.1 All reagents and standards preparation, documentation and labeling must follow the requirements of ST-QA-0002.
- 7.2 DI Water - Obtained from the laboratory's commercial ion-exchange deionizing unit which includes a polishing unit (Milli-Q System).
- 7.3 Calibration Standards (pH 2, 4, 7, and 10) - Standard Buffer solutions.
- 7.4 Calibration Check Standard (pH 7) - Standard Buffer solution.

7.5 The pH 7 calibration check standard must be from a different lot or manufacturer than the calibration standards. Second source from another manufacture is preferred; only use different lot when another vendor cannot be found.

7.6 HCl (1:9)

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples for pH analysis must be transported and stored at 4 ± 2 °C.

8.3 pH samples are not to be chemically preserved.

8.4 Water and soil samples should be analyzed as soon as possible after collection, preferably in the field.

8.4.1.1 pH methods for soil and water matrices require that samples be analyzed immediately. As the samples are being shipped to the lab for analysis, meeting this holding time is not feasible. Where possible, samples are to be analyzed within 24 hours of receipt by the laboratory. Samples received on Fridays, weekends or holidays will be analyzed the morning of the next business day. Samples will be flagged on the data as outside of hold, and a narrative note will be included in the data report.

8.4.2 Waste samples should be analyzed within 7 days of collection

8.5 South Carolina requires that:

8.5.1 all water pH samples analyzed outside the fifteen minute hold time be flagged with a qualifier.

8.5.2 the pH of soil samples is to be determined immediately after allowing the soil suspension to stand for one hour.

9.0 QUALITY CONTROL

9.1 Batch

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a Laboratory Control Sample (LCS) and a Sample Duplicate (SD) In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2 Laboratory Control Sample (LCS)

9.2.1 An LCS is a standard with a known pH. The LCS must come from a second source (7 pH buffer) that was not used for instrument calibration.

9.2.2 An LCS must be analyzed with every sample batch.

9.2.2.1 LCS consists of pH 7 buffer solution for waters and soils.

9.3 Sample Duplicate (SD)

9.3.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.4 Procedural Variations/ Nonconformance and Corrective Action

9.4.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.4.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Initial Calibration

10.1.1 A three point calibration consisting of standard buffer solutions which are at least three pH units apart.

10.1.2 Buffer solutions 4, 7 and 10 are used.

10.1.2.1 A pH buffer of 2 is to be included in the calibration when a sample analysis results in a pH that is less than 4. The pH meter is re-calibrated with the buffer of 2 included and the affected samples re-analyzed.

10.1.3 Frequency

10.1.3.1 Calibration is performed each day of use.

10.1.4 Calibration Criteria

10.1.4.1 Each buffer standard reading must be within ± 0.05 from the true value

10.1.4.2 If the pH meter calibration standards are not within 0.05 pH units of the true values, the analyst must take corrective action that may require one or more of the following:

10.1.4.2.1 Replace standard buffers with fresh solutions to assure that they are uncontaminated.

10.1.4.2.2 Perform preventive maintenance on pH meter, replace the electrode, replace the meter, or request service on pH meter.

10.1.5 Slope

10.1.5.1 After the calibration, the meter will display the slope in percent units. Record the slope in the pH calibration logbook.

10.1.5.2 The slope criteria is 90 – 105%. If not met, recalibrate the meter after performing the steps in Section 10.1.4

10.2 Initial Calibration Verification (ICV)

10.2.1 An analyzed standard with a known pH that is from a source not used for instrument calibration

10.2.2 ICV is a pH 7 buffer solution

10.2.3 Frequency

10.2.3.1 ICV is performed after each Initial Calibration.

10.2.4 Calibration Criteria

10.2.4.1 Each buffer standard reading must be within ± 0.05 from the true value

10.3 Continuing Calibration Verification (CCV)

10.3.1 An analyzed standard with a known pH that is from a source not used for instrument calibration

10.3.1.1 CCV is a pH7 buffer solution

10.3.2 Frequency

10.3.2.1 CCV is performed after every ten samples, including QC.

10.3.3 Calibration Criteria

10.3.3.1 Each buffer standard reading must be within ± 0.05 from the true value.

10.3.3.2 Samples must be bracketed by acceptable CCV performance. If the calibration check standards are not within 0.05 pH units of the true values, the associated samples must be re-analyzed, once. If re-analysis repeats the CCV failure, samples are reported with an NCM noting matrix interference confirmed by re-analysis. See SOP: ST-QA-0036 for further instruction.

11.0 PROCEDURE

11.1 If through evaluation of the sample either visually or by a percent solids analysis, it is determined that the sample is less than 20% aqueous notify the Project Manager so that the request for SW-846 method 9045D (pH in Soil) can be made.

11.2 Aqueous Sample Analysis

11.2.1 Rinse electrode with DI water and dry.

11.2.2 Thoroughly shake sample and pour aliquot into disposable cup, about 10 mL.

11.2.3 Immerse electrode in sample. The pH icon will flash until the reading is stable.

11.2.4 Record pH

11.2.4.1 Record any pH values less than 2 or greater than 12 as < 2 or > 12 , respectively.

11.2.4.2 Record the temperature.

11.2.5 Rinse electrode and dry.

11.2.6 Discard sample aliquot.

11.2.7 Thoroughly shake sample and pour second aliquot into disposable cup, about 10 mL.

11.2.8 Immerse electrode into sample.

11.2.9 Record pH the nearest 0.01 pH units.

11.2.9.1 Record any pH values less than 2 or greater than 12 as < 2 or > 12 , respectively.

11.2.9.2 Record the temperature.

11.2.9.3 The two sample pH readings must agree within ± 0.1 pH units.

11.2.9.4 If pH readings do not agree, repeat analysis with an additional aliquot; if results still do not agree, contact manager/supervisor for further instruction.

11.2.10 The first pH reading is recorded into the LIMS for reporting.

11.2.11 Rinse and dry electrode between sample pH determinations

11.2.11.1 In the event that a sample is analyzed which contains any observable amount of oil, rinse the electrode with a 1:9 Hydrochloric Acid rinse; then rinse the electrode well with DI water and dry before reading the next sample

11.2.12 When the analysis is completed, store electrode in 3M KCL solution.

11.3 Soil Sample Analysis

11.3.1 See SOP, ST-QA-0038 for soil sample aliquots.

11.3.2 Weigh $20 \text{ g} \pm 0.2 \text{ g}$ soil sample for pH analysis into a sample ID labeled centrifuge tube.

- 11.3.3 Record weight in LIMS
- 11.3.4 Add 20 mL of DI water to each sample.
- 11.3.5 Cover and shake the suspension of soil in water for 5 minutes.
 - 11.3.5.1 Additional 20 mL water additions are allowed if working with hygroscopic soils and salts or other problematic matrices, in order to provide sufficient water layer to take pH reading.
- 11.3.6 Allow the beaker to stand without stirring to allow most of the solids to settle, for at least 1 hour. Analyze immediately after the 1 hour settling period
 - 11.3.6.1 If not analyzing leachate immediately after the 1 hour settling period, refrigerate the leachate until ready to analyze.
 - 11.3.6.2 Allow leachate to come up to room temperature before analyzing.
 - 11.3.6.3 Analysis must be done within 48 hours of leaching.
- 11.3.7 Immerse the electrode of the pH meter into the sample to establish a good electrical contact, and read pH of the supernatant water to the nearest 0.01 pH units.
- 11.3.8 Be sure electrode does not come into contact with the soil.
- 11.3.9 Record pH and temperature of the supernatant water to the nearest 0.01 pH units
 - 11.3.9.1 Record any pH values less than 2 or greater than 12 as < 2 or > 12, respectively.
- 11.3.10 Rinse electrode with DI water and dry before reading the next sample.
 - 11.3.10.1 In the event that a sample is analyzed which contains any observable amount of oil, rinse the electrode with a 1:9 Hydrochloric Acid rinse; then rinse the electrode well with DI water and dry before reading the next sample
- 11.3.11 When analysis is complete, store the electrode in 3M KCL solution

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 There are no additional calculations pertaining to this procedure.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.
- 13.2 Method Blank
 - 13.2.1 Not applicable to pH analysis.
- 13.3 Laboratory Control Sample (LCS)
 - 13.3.1 Acceptance Criteria: All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in the LIMS.
 - 13.3.2 Corrective Action for LCS not meeting acceptance criteria:
 - 13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM, (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.
 - 13.3.2.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.

13.3.2.3 RPD excursion for LCS/LCSD – If spike recoveries are acceptable, data is evaluated for reportability and an NCM generated. If spike recoveries and RPD are outside limits, batch is re-prepped/re-analyzed for affected analytes. .

13.4 Matrix Spike/Matrix Spike Duplicate/Sample Duplicate (MS/MSD/SD)

13.4.1 MS/MSD is not applicable to pH analysis.

13.4.2 Sample Duplicate: all analytes should be within established control limits for accuracy (%Recovery) and precision (RPD). Limits can be found in LIMS

13.5 Sample result evaluation

13.5.1 Dilutions

13.5.1.1 Not applicable to pH analysis.

13.5.2 Insufficient Sample

13.5.2.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Demonstration of Capability

14.1.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.2 Training Qualification

14.2.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.2.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.2.3 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type “A” or “B.”

16.2.1.2 Sample waste with a Basic pH is generated. All base waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.1.3 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D." This procedure does not generate solvent waste.

16.2.1.4 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid RAD waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 EPA Manual 600/4-79-020, Method for Chemical Analysis of Water and Wastes, Revised March 1983, Method 150.1
- 17.2 SW-846, Method 9040C, pH Electrometric Measurement; Third Edition, Revision 3, Nov. 2004
- 17.3 SW-846, Method 9045D, Soil and Waste pH; Third Edition, Revision 4, Nov. 2004
- 17.4 Standard Methods, Method 4500-H⁺, 2000
- 17.5 TestAmerica Quality Assurance Manual (ST-QAM), current revision
- 17.6 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions
- 17.7 Associated SOPs, current revisions:
 - 17.7.1 ST-QA-0002, Standards Preparation
 - 17.7.2 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.7.3 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the use of Control Charts
 - 17.7.4 ST-QA-0036, Non-conformance Mémorandum (NCM)

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 None

19.0 CHANGES FROM PREVIOUS REVISIONS

- 19.1 Rev 12: Annual Review - No changes to previous revision
- 19.2 Rev 13: Added South Carolina requirement to flag samples run outside 15 minute hold time to Section 8.4.2.
- 19.3 Rev 14:
 - 19.3.1 Removed centrifuge and replaced it with a shaker table in section 6.0.
 - 19.3.2 Updated the definition of a LCS standard in section 9.2.1.
 - 19.3.3 Inserted instructions for the use of an ICV in section 10.0.
 - 19.3.4 Updated soil sample analysis throughout section 11.3.
- 19.4 Rev 15: (06/17/2013)

- 19.4.1 Added HCl to section 5.0
- 19.4.2 Added HCl (1:9) to section 7.0
- 19.4.3 Added second source requirement to 7.4 Calibration Check Standard.
- 19.4.4 Replaced specimen cup and added centrifuge cup in section 6.0 and throughout section 11.0.
- 19.4.5 Removed references to “Clouseau” and “QuantIMS” and replaced with “LIMS”.
- 19.4.6 Updated corrective actions in Section 13.
- 19.4.7 Updated section 15.
- 19.4.8 Updated the use of 7pH buffer solution to clean and protect analytical electrodes throughout sections 9.0, 10.0 and 11.0.
- 19.4.9 Removed non-applicable requirements from section 13.0
- 19.4.10 Updated SM 4500-H⁺ reference in section 17.0
- 19.4.11 Updated attachment 1 and 2
- 19.5 Rev 16: (06/16/2015)
 - 19.5.1 Added pH standard of 2 to Section 7.
 - 19.5.2 Updated holding time requirements in Section 8.
- 19.6 Rev 17 (7/28/16)
 - 19.6.1 Added model number of pH meter to Section 6
 - 19.6.2 Added requirement for slope and slope criteria to Section 10
 - 19.6.3 Updated South Carolina requirement for pH soil sample holding time in Section 8.
 - 19.6.4 Separated information in Section 18 into 2 Sections – 18 to detail method modifications and 19 to document changes from previous SOP revisions. [correcting format error in Revision 16.]
 - 19.6.5 Updated holding time information.
 - 19.6.6 Removed attachments of spreadsheets. Data is now entered into LIMS
 - 19.6.7 Corrected solution probe is to be stored in.

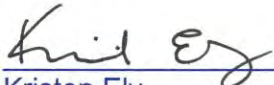
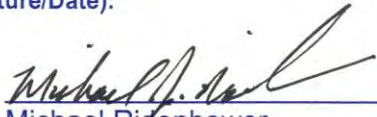
Attachment 1

pH Technique

pH Calibration

1. Prepare the electrode according to the electrode user guide.
2. In the setup mode, select the buffer set (*USA* or *EUR*) that will be used for the automatic buffer recognition feature.
3. In the measurement mode, press  until the arrow icon points to the top line, press  until the **pH** icon is shown and press  to begin the calibration.
4. Rinse the electrode, and ATC probe if being used, with distilled water and place into the buffer.
5. Wait for the **pH** icon to stop flashing.
 - a. Automatic buffer recognition – When the **pH** icon stops flashing the meter will display the temperature-corrected pH value for the buffer.
 - b. Manual calibration – When the **pH** icon stops flashing the meter will display the actual pH value read by the electrode. Press  until the first digit to be changed is flashing, press  /  to change the value of the flashing digit and continue to change the digits until the meter displays the temperature-corrected pH value of the buffer. Once the pH buffer value is set, press  until the decimal point is in the correct location.
6. Press  to proceed to the next calibration point and repeat steps 4 and 5 or press  to save and end the calibration.
7. The actual electrode slope, in percent, will be displayed in the main field and **SLP** will be displayed in the lower field.
 - a. For a one point calibration, press  and  /  to edit the slope and press  to return to the measurement mode.
 - b. For a two or more point calibration, the meter will automatically proceed to the measurement mode after the slope is displayed.

Title: ANALYSIS OF SULFIDE
[SW-846 9034; EPA 376.1; Standard Method 4500 S² C and F]

Approvals (Signature/Date):	
 Kristen Ely Inorganic Department Manager	<u>3/31/16</u> Date
 Michael Ridenhower Health & Safety Manager / Coordinator	<u>3/31/16</u> Date
 Tony L. Byrd Quality Assurance Manager	<u>03-31-2016</u> Date
 for Elaine Wild Laboratory Director	<u>3/31/16</u> Date

This SOP was previously identified as SOP No. ST-WC-0012 Rev. 13

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes.
- 1.2 This method is also applicable to the measurement of water extractable sulfides in soil/sediments and other solids.
- 1.3 This method is suitable for measurement of sulfides in concentration above 1 mg/L for waters.
- 1.4 This method is suitable for measurement of sulfides in concentration above 10 µg/g for soils. The factors which may make this limit higher are the percent moisture for sample and the exact aliquot used in analysis.
- 1.5 This SOP is applicable to EPA Method 376.1, SW-846 Method 9034 and Standard Methods 4500 S²-C and F.
- 1.6 The distillation procedure, SW-846 Method 9030B is described in SOP: ST-IP-0019.
- 1.7 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the LIMS.

2.0 SUMMARY OF METHOD

- 2.1 The sulfide is oxidized to sulfur by adding a known excess amount of iodine. The excess iodine is determined by titration with a standard solution of phenyl arsine oxide (PAO) or sodium thiosulfate until the blue iodine starch complex disappears. As the use of standard sulfide solutions is not possible because of oxidative degradation, quantitation is based on the PAO or sodium thiosulfate.

3.0 DEFINITIONS

- 3.1 See the TestAmerica Quality Assurance Manual (QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

- 4.1 Reduced substances that react with iodine, such as sulfite, thiosulfate and hydrosulfite, will decompose in acid and yield erratic results.
 - 4.1.1 These interferences are eliminated by precipitating Zinc sulfide, removing the supernatant and replacing it with DI water.
- 4.2 Volatile iodine consuming substances will give high results.
- 4.3 Sample distillation interferences are found in SOP: ST-IP-0019.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

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5.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Sodium Sulfide will form Hydrogen Sulfide (HS) gas if combined with water moisture or strong acids. Inhalation of HS gas may be fatal.

5.2 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.**

A complete list of materials used in the method can be found in the reagents and materials section.

Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Sign and Symptom of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Contact can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3 (Ceiling)	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sodium Sulfide	Corrosive	10 ppm (TWA) 15 ppm (STEL)	Will form Hydrogen Sulfide (HS) gas if combined with strong acids. Inhalation of HS gas may be fatal. Symptoms include painful conjunctivitis, headache, nausea, dizziness, coughing and, in extreme cases, pulmonary edema and possible death. Irritant. Contact with skin can produce serious caustic burns with painful inflammation and possible destruction of tissue. Inflammation, tearing and pain may be expected. Severe contact can cause destruction of tissue.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average;			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

6.1 Filtering Flasks.

6.2 Graduated cylinder

6.3 Erlenmeyer Flasks - 250 ml, or equivalent

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- 6.4 Digestion tube, 130mL
- 6.5 Disposable cups, 100mL
- 6.6 Pipettes – adjustable Eppendorfs, or equivalent.
- 6.7 Disposable Pipettes.
- 6.8 Glass Fiber Filter, 0.45 pm.
- 6.9 Volumetric Flasks - 100 ml, 500 ml and 1000 ml.
- 6.10 Balance, analytical.
- 6.11 pH paper, narrow range
- 6.12 Burette, 10-mL

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revisions.
- 7.2 DI water, obtained from the Millipore unit.
- 7.3 Potassium Iodide.
- 7.4 Standard iodine solution, 0.0250 N, commercially available
 - 7.4.1 Dissolve 20 to 25 g Potassium Iodide in DI water in a 1 liter volumetric flask and add 3.2 g iodine (I_2). Allow to dissolve. Dilute to the mark with DI water and standardize against 0.0250 N PAO using a starch indicator.
- 7.5 Sodium Thiosulfate, 0.0250 N or 0.010 N, commercially available
 - 7.5.1 Dissolve 6.205 g $Na_2S_2O_3 \cdot 5H_2O$ in approximately 500 ml DI water in a 1 liter volumetric flask. Add 1.5 ml 6 N NaOH and dilute to the mark with DI water. Use a starch solution as indicator during titration.
- 7.6 Starch indicator, commercially available.
- 7.7 Zinc acetate solution
 - 7.7.1 Dissolve 220g Zinc acetate in 870ml water.
- 7.8 Sodium Hydroxide, NaOH, 6N, commercially available
- 7.9 Hydrochloric acid, HCl, 6N, commercially available.
- 7.10 Sulfide Solution: 1,000 mg/L, commercially available. Dissolve approximately 7.54 g of $Na_2S \cdot 9H_2O$ in 1 liter of deionized water to yield a concentration of approximately 1000 mg/L of S (sulfur conversion factor = 0.133508).
 - 7.10.1 Standards must be prepared at pH > 9 and < 11.
 - 7.10.2 Shelf life: Standard must be made fresh **DAILY**.
 - 7.10.2.1 LCS and Matrix Spike: For waters, spike the LCS and sample into the 500 ml flask with 2 mls of the sulfide solution. Use 200 mls water for the LCS. The true value

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for the LCS and matrix spike (waters) is 10 mg/L. For the LCS and matrix spike (soils), the true value is 200 mg/kg.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 See Sample Preparation SOP, ST-IP-0019 for preservation requirements.
- 8.3 If not immediately analyzed after distillation, store distillate in refrigerator at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a Method Blank, (MB) a Laboratory Control Sample (LCS), Matrix Spike (MS) and a Sample Duplicate (DU).). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
 - 9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request.

9.2 Method Blank (MB)

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.

9.3 Laboratory Control Sample (LCS)

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.

9.4 Matrix Spike (MS)

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.5 Sample Duplicate (DU)

- 9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.6 Procedural Variations/ Nonconformance and Corrective Action

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

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10.0 CALIBRATION AND STANDARDIZATION

- 10.1 The LCS serves as calibration check.
- 10.1.1 Frequency: An LCS or CCV is analyzed at the beginning and end of the analytical sequence.
- 10.2 Standardize the 0.025N I₂ weekly, if not purchase commercially or required by client.
- 10.1.1 Add 10 mls 0.025N Iodine to 200 mls water.
- 10.1.2 Titrate the solution using 0.025N Sodium thiosulfate. Repeat two more times and average volume of titrant.
- 10.1.2.1 Calculate the normality as follows:

$$\text{Normality (I}_2\text{)} = \frac{\text{mL of titrant} \times \text{normality of titrant}}{\text{sample size in mL}}$$
- 10.1.2.2 Standardization of the iodine solution will be performed with each analytical batch or at a maximum frequency of once per week.
- 10.2 No standardization is required for the iodine solution if purchased commercially unless specifically required by the client.
- 10.2.1 If a commercial standardized solution is purchased, this standardization step is not required.
- 10.2.2 Standardize against Potassium bi-iodate after at least two weeks from preparation.
- 10.2.2.1 Dissolve 3.249g of Potassium bi-iodate in DI water. Dilute with DI water to a 1000ml.
- 10.2.2.1.1 This yields a 0.1N KH(IO₃)₂ solution.
- 10.2.2.2 In a 250ml flask, add 1ml Sulfuric acid and 10ml KH(IO₃)₂, and dilute with DI water to 100ml.
- 10.2.2.3 Place flask on stirring plate and stir.
- 10.2.2.4 Add 1g Potassium iodide
- 10.2.2.5 Titrate with 0.1 Sodium thiosulfate until the yellow color is almost discharged.
- 10.2.2.6 Add 1ml of starch indicator and continue titrating until the blue color disappears.
- 10.2.2.7 Normality (Sodium thiosulfate) = $\frac{1}{\text{mL of titrant}}$

11.0 PROCEDURE

- 11.1 376.1 Solid and Liquid
- 11.1.1 Unprecipitated Sample
- 11.1.1.1 Place 10ml of standard iodine solution into a 500ml flask. The amount should be estimated to be in excess of the amount of sulfide expected.
- 11.1.1.2 Add distilled water, if necessary, to bring the volume to approximately 20mls.
- 11.1.1.3 Add 2 ml of 6N HCL
- 11.1.1.4
- 11.1.1.4.1 For liquid samples, pipette 200 ml of sample into the flask, keeping the tip of the pipette below the surface of the sample.
- 11.1.1.4.2 For solid samples weigh 20g of sample into a 130mL digestion tube add 100ml of DI and place on shaker for 10min. let particulates settle for approximately 2hrs. pipette 50mls of sample into a 500ml flask, keeping the tip of the pipette below the surface of the sample.
- 11.1.1.5 If the iodine color disappears, add a few drops of 6N HCL. Addition of the sample may cause the pH to rise. If the iodine color is still not present, add more iodine until the color remains. Record the total volume of iodine used.
- 11.1.1.6 Titrate with 0.0250 N sodium thiosulfate using a starch indicator until the blue color disappears. Record the number of milliliters used.

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- 11.1.2 Precipitated samples liquid samples
 - 11.1.2.1 Add the reagent to the sample in the original bottle. Perform steps 1, 3, 5 and 6 from above.
- 11.2 Pretreatment of Water samples (SM 4500-S₂ C and F)
 - 11.2.1 For each sample and QC perform the following:
 - 11.2.2 Place 1.0ml Zinc Acetate solution and 0.50 ml 6N NaOH in a 500ml bottle
 - 11.2.3 Fill to a 100ml with sample and stopper bottle. Be sure there are no air bubbles.
 - 11.2.3.1 For method blanks, fill to 100ml with DI water
 - 11.2.3.2 For LCS, fill to 100ml with prepared LCS solution.
 - 11.2.4 Mix sample vigorously.
 - 11.2.5 Filter sample.
 - 11.2.6 Collect precipitate on a glass fiber filter.
 - 11.2.6.1 Store filter in original 500ml bottle.
 - 11.2.7 Proceed immediately to the titration step 11.1.1.6
- 11.3 Analysis of Sulfide Distillate (SW 9030B and SW 9034), and Pretreated Samples (SM 4500-S₂ C and F)
 - 11.3.1 The sulfide distillation procedure is found in ST-IP-0019.
 - 11.3.2 Clean burette and flask in accordance with cleaning procedure SOP, ST-IP-0004.
 - 11.3.3 Acid Soluble Sulfide sample analysis (SW 9034):
 - 11.3.3.1 For each sample and QC perform the following:
 - 11.3.3.2 Add 65ml iodine solution to titration flask.
 - 11.3.3.3 Add 2ml 6N HCl to titration flask.
 - 11.3.3.4 Pipette the Zinc Acetate scrubbing solutions obtained in Method 9030 to the flask, keeping the end of the pipette below the surface of the iodine solution. The entire scrubber solution is used.
 - 11.3.3.4.1 If in transferring the solution the amber color of the iodine disappears or fades add more acid, sometimes the sample will raise the pH. Extra iodine may not be need but if iodine is needed, record the volume, 0.025N iodine.
 - 11.3.3.5 Record the total volume of standardized 0.025N iodine solution used.
 - 11.3.3.6 Rinse scrubber tube with a known amount of standardized 0.025N iodine solution, 1mL of 6N HCl, and water.
 - 11.3.3.6.1 This rinses the remaining white precipitate (zinc sulfide) from the gas scrubbing bottles into the flask. There should be no visible traces of precipitate after rinsing.
 - 11.3.3.7 Final volume in titration flask should be at least 100ml.
 - 11.3.3.8 If volume is not at least 100ml, add DI water to bring up to 100ml.
 - 11.3.3.9 Proceed to titration step 11.1.1.6.
 - 11.3.4 Acid Insoluble Sulfide sample analysis (SW 9034):
 - 11.3.4.1 For each sample and QC perform the following:
 - 11.3.4.2 Add 65ml iodine solution to titration flask.
 - 11.3.4.3 Add 10ml 6N HCl to titration flask.
 - 11.3.4.4 Pipette the gas sulfide Zinc Acetate scrubbing solutions obtained in Method 9030 to the flask, keeping the end of the pipette below the surface of the iodine solution. The entire scrubber solution is used.
 - 11.3.4.4.1 If in transferring the solution the amber color of the iodine disappears or fades, add more 0.025N iodine.
 - 11.3.4.5 Record the total volume of standardized 0.025N iodine solution used.
 - 11.3.4.6 Rinse scrubber tube with a known amount of standardized 0.025N iodine solution, 1mL of 6N HCl, and water.

- 11.3.4.6.1 This rinses the remaining white precipitate (zinc sulfide) from the gas scrubbing bottles into the flask. There should be no visible traces of precipitate after rinsing.
- 11.3.4.7 Final volume in titration flask should be at least 100ml.
- 11.3.4.8 If volume is not at least 100ml, add DI water to bring up to 100ml.
- 11.3.4.9 Proceed to titration step 11.1.1.6.
- 11.3.5 Sulfide analysis (SM 4500-S₂ F):
- 11.3.5.1 For each sample and QC perform the following
- 11.3.5.2 Add 20ml iodine solution to titration flask.
- 11.3.5.3 Add 2ml 6N HCl to titration flask.
- 11.3.5.4 Pipette 200ml pretreated sample, keeping the end of the pipette below the surface of the iodine solution.
- 11.3.5.4.1 If in transferring the solution the amber color of the iodine disappears or fades, add more 0.025N iodine.
- 11.3.5.5 Record the total volume of standardized 0.025N iodine solution used.
- 11.3.5.6 Proceed to titration step 11.3.8
- 11.3.6 Analysis of Sulfide Distillate obtained by micro distillation
- 11.3.6.1 Add 10mL of deionized water and 2 mL of Iodine solution to the analysis cup.
- 11.3.6.2 Allow tubes to cool for at least 10 minutes after completion of the distillation.
- 11.3.6.3 For each collector tube, hold the tube horizontally and rinse its walls with the distillate in order to homogenize it. Slowly roll the distillate around in the tube to gather all droplets clinging to the tube walls into the bulk of the distillate. Then, slowly return the collector tube to an upright position so that the "D" end is up. Stubborn drops will often fall into the "M" end when the tube is flicked with a finger.
- 11.3.6.4 With the "D" end still up, break the collector tube in half by pulling the "D" end hard towards the user to break it, then twisting and tearing off the "D" end. Discard the "D" end.
- 11.3.6.5 In the remaining "M" end of the collector tube, dilute to the 6 mL mark with DI water. Push the second cap onto the open end of the tube as far as it will go. Shake the tube with a gentle whipping motion to mix. Do not invert the sample.
- 11.3.6.6 Use a transfer pipette to inject the distillate in the "M" end of the collection tube below the surface of the liquid in the analysis cup. Repeat 11.3.6.5 for rinsing any residual distillate from the "M" end of the tube.
- 11.3.6.6.1 If in the transferring of the solution the amber color of the iodine disappears or fades add more acid, sometimes the samples will raise the pH. Extra iodine may not be needed but if iodine is needed record any additional volume of iodine.
- 11.3.6.7 **Note:** when rinsing the collection tubes use minimal amount of DI water necessary. Over dilution record any additional volume of iodine.
- 11.3.6.8 Proceed to 11.3.7 for Titration.
- 11.3.7 Titration for micro distillate
- *use 10 mL burette
- 11.3.7.1 Titrate the solution with standardized 0.025N or 0.010N sodium thiosulfate solution until the amber color fades to yellow.
- 11.3.7.2 Add starch indicator for the solution to turn dark blue, titrate until the blue disappears.
- 11.3.7.3 Record the volume of titrant used.
- 11.3.8 Titration for SM 4500-S₂ F (only):
- 11.3.8.1 Take 500ml bottle containing precipitate and filter.
- 11.3.8.2 Add 20ml iodine solution to titration flask.

- 11.3.8.2.1 If in transferring the solution the amber color of the iodine disappears or fades, add more 0.025N iodine.
- 11.3.8.3 Record the total volume of standardized 0.025N iodine solution used.
- 11.3.8.4 Add 2ml 6N HCl to titration flask.
- 11.3.8.5 Titrate as described in 11.1.1.6.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis QAM.
- 12.2 For water samples use the formula:

$$\text{Sulfide mg/L} = \frac{[(\text{mL} \times \text{N iodine}) - (\text{mL} \times \text{N Na}_2\text{S}_2\text{O}_3)] \times 16000}{\text{mL of sample}}$$

where N = normality of iodine or $\text{Na}_2\text{S}_2\text{O}_3$
- 12.3 For soil samples use the formula:

$$\text{Sulfide } \mu\text{g/g} = \frac{[(\text{mL} \times \text{N iodine}) - \text{mL} \times \text{N Na}_2\text{S}_2\text{O}_3] \times 16000 \times \text{Ext vol original (mL)}}{(\text{Ext vol titrated, mL}) \times (\text{g of sample}) \times (\text{solid fraction})}$$

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the nonconformance memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036. Below is a subset of the data assessment and QC excursion types; for a complete and current listing, please access the LIMS.
- 13.2 Method Blank
 - 13.2.1 Acceptance Criteria:
 - 13.2.1.1 No target analytes may be present in the method blank above the reporting limit.
 - 13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target analyte present above ½ RL), will be noted in LIMS.
 - 13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:
 - 13.2.2.1 Method Blank Contamination – See NCM for corrective action (e.g. reprep/reanalysis, narration).
- 13.3 Laboratory Control Sample (LCS)
 - 13.3.1 Acceptance Criteria:
 - 13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).
 - 13.3.2 Corrective Action for LCS not meeting acceptance criteria:
 - 13.3.2.1 LCS Spike Recovery excursion (high) – See NCM for corrective action (e.g. , reanalysis, narration).
 - 13.3.2.2 LCS Spike Recovery excursion (low) – See NCM for corrective action (e.g. , reanalysis, narration).
 - 13.3.2.3 RPD Duplicate excursion – See NCM for corrective action (e.g. , reanalysis, narration).
- 13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
 - 13.4.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).
 - 13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

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13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. See NCM to determine if re-preparation re-analysis is required.

13.5 Insufficient Sample

13.5.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis and narrative comment stating such is included in the report narrative. The insufficient sample description is included in the NCM within the type defining the excursion.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are recorded in LIMS

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out

16.2.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

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- 17.1 EPA Manual 600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Revised March 1983, Method 376.1.
- 17.2 Standard Methods for the Examination of Water and Waste Water, 19th Edition 1995, Method 4500-S₂-C and Method 4500-S₂-F.
- 17.3 SW-846, Method 9030B Rev 2 1996 and 9034 Rev 0 1996, Sulfides, Third Edition
- 17.4 TestAmerica Quality Assurance Manual (QAM), current revision
- 17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.4 Associated SOPs – current revisions
 - 17.4.1 ST-IP-0004, Glassware Preparation for Inorganic and Trace Metals Analysis
 - 17.4.2 ST-IP-0019, Sulfide Distillation
 - 17.4.3 ST-QA-0002, Standard and Reagent Preparation
 - 17.4.4 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.4.5 ST-QA-0016, IDL/MDL Determination
 - 17.4.6 ST-QA-0036, Non-conformance Memorandum (NCM) Process
 - 17.4.7 ST-PM-0002, Sample Receipt and Chain of Custody

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD.

- 18.1 376.1 :
 - 18.1.1 For soil samples, a DI leach is performed. The leachate is then treated as an aqueous sample.
 - 18.1.2 If the iodine color disappears with addition of sample, a few drops of 6N HCL are added before addition of more iodine. This is to ensure the sample did not increase the pH.

19.0 CHANGES TO PREVIOUS

- 19.1 Added method 376.1 to the section 11.0.
- 19.2 Updated section 7.0 by changing the amount sulfide used in a LCS for waters.
- 19.3 Removed Low-levels and High-Levels LCS from section 10.0.
- 19.4 Rev, 10. Added digestion tubes and disposable cups to section 6.0.
- 19.5 Rev, 10. Added 1,000 mg/L of commercial grade sulfide solution to section 7.0.
- 19.6 Rev, 10. Increased the amount of sulfide solution standard from 250 to 500mls and 100 to 200 mls in section 7.10.2.
- 19.7 Rev, 10. Increased the flask size from 250mls to 500mls in section 11.1.1.4.
- 19.8 Revision 11:
 - 19.8.1 Updated section 11.0 by adding instructions for Analysis of Sulfide Distillate obtained by micro distillation.
- 19.9 Revision 12:
 - 19.9.1 Updated titration reference step in sections 11.4 and 11.5.
- 19.10 Revision 13: (10/31/2014)
 - 19.10.1 Updated reagent Sodium Thiosulfate in section 7.5
 - 19.10.2 Updated micro distillation analysis procedure in section 11.4.6.
 - 19.10.3 Updated titration procedure in section 11.5.
- 19.11 Revision 14: (03/31/2016)
 - 19.11.1 Removed references to Quantims, Clouseau, and Client Requirement Sheets throughout
 - 19.11.2 Updated acronym for Sample Duplicate to DU, to reflect LIMS
 - 19.11.3 Made grammatical corrections throughout

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- 19.11.4 Updated procedure in section 11 to make it easier to follow, and to more accurately reflect actual procedure performed.
- 19.11.5 Added modifications to section 18 for 376.1
- 19.12 Revision 14: (03/29/2017) Annual review, no changes

**Title: FLASH POINT BY PENSKEY-MARTENS CLOSED CUP
TESTER/CLEVELAND OPEN CUP
[SW-846 1010A; ASTM D92-02a; ASTM D93-02;]**

Approvals (Signature/Date):	
 _____ Jacob Boyd Wet Chemistry Group Lead	11/16/16 _____ Date
 _____ Michael Ridenhower Health & Safety Manager / Coordinator	11/16/16 _____ Date
 _____ Kristen Ely Quality Assurance Manager	11/17/16 _____ Date
 _____ Elaine Wild Laboratory Director	11/16/16 _____ Date

This SOP was previously identified as SOP No. ST-WC-0026 Rev. 17

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1.0 SCOPE AND APPLICATION

- 1.1 This Standard Operating Procedure is appropriate for the determination of the flash point by The Cleveland Open Cup and Pensky-Martens closed cup tester of fuel oils, lube oils, suspension of solids, liquids that tend to form a surface film under test conditions, other liquids of similar viscosity and soils.
- 1.2 This method is based on ignitability characteristic which in turn can be used to identify waste materials or products that either present fire hazards under routine storage, disposal, and transportation or are capable of severely exacerbating a fire once started.
- 1.3 This method measures the properties of a sample in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard/risk of materials or products under actual fire conditions.
- 1.4 This method is applicable to samples having a flash point range from -5 to 370°C. Because this SOP specifically addresses the RCRA Ignitability requirement, directions are given only for determining flashpoints below 60 °C. All other samples are reported as having a flashpoint above 60 °C. The actual flashpoint above 60 °C (up to 110°C) can be determined by extension of the procedure given here, at client request.
- 1.5 This SOP is applicable to SW-846 Method 1010A , ASTM Methods D93-02 and D92-02a.
- 1.6 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

- 2.1 The sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapor above the sample to ignite.

3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Flash Point: The lowest temperature at which application of a test flame causes the vapor of a specimen to momentary flash or ignite under specified conditions of the test.
- 3.3 P-Xylene: An extremely flammable, colorless liquid in its pure (95%) form. Used as a flash point check standard in the test.

4.0 INTERFERENCES

- 4.1 Ambient pressure, sample homogeneity, drafts, and operator bias can affect flash point values.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, the facility addendum to the CSM, and this document.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 In the event a sample ignites in the test apparatus do not attempt to remove the sample. Turn off the apparatus and flame. The flame should go out when the cup is closed. If this does not happen the flame may be extinguished by covering the sample with a non-flammable material. After the apparatus has cooled the sample may be removed.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

5.4 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Xylene	Flammable Irritant	100 ppm (TWA)	Inhalation of vapors may be irritating to the nose and throat. Inhalation of high concentrations may result in nausea, vomiting, headache, ringing in the ears, and severe breathing difficulties, which may be delayed in onset. High vapor concentrations are anesthetic and central nervous system depressants. Skin contact results in loss of natural oils and often results in a characteristic dermatitis. May be absorbed through the skin. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 Pensky-Martens Closed Flash Point Tester with cover assembly, or equivalent.

6.2 Flashpoint Cup with heat resistant handle.

6.3 Thermometer with a range from -5 to 110 °C.

- 6.4 Propane gas cylinder.
- 6.5 Connecting lines and ferrules.
- 6.6 Rubber tubing 1/8"(o.d.).
- 6.7 Cleveland Open Cup Flashpoint tester or equivalent.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 P-Xylene (99% pure), reagent grade.
- 7.3 Calcium chloride, reagent grade.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Store sample at $\geq 0^{\circ}\text{C}$ to 6°C in tight glass containers to avoid any loss of volatile material.
- 8.3 Holding time, from date of collection, is 180 days or contract specific requirements.
- 8.4 Samples of very viscous materials that do not contain volatile contaminants may be warmed until they are tested. However, no sample should be heated more than is absolutely necessary. It shall never be heated above a temperature of 17°C (30°F) below its expected flash point.
- 8.5 Samples containing dissolved or free water that also do not contain volatile contaminants may be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Warming the sample is permitted, but it should not be heated for prolonged periods or above a temperature of 17°C (30°F) below its expected flash point.

9.0 QUALITY CONTROL

- 9.1 Batch
 - 9.1.1 A sample batch is a maximum of twenty environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a

group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

9.1.2 Conditions must be the same for all samples and QC samples.

9.1.3 For this analysis the batch may contain up to 20 environmental samples a Method Blank (MB), a Laboratory Control Sample (LCS) and a Sample Duplicate (DU). In the event that there is insufficient sample to analyze a Sample Duplicate, an LCS Duplicate (LCSD) is analyzed.

9.2 Method Blank

9.2.1 A blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure. For ignitability, a blank matrix is considered to be a material that flashes above the ignitability range of the method.

9.2.2 A method blank must be prepared with every preparation batch (20 or fewer samples of the same matrix).

9.3 Laboratory Control Sample

9.3.1 A blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be performed with every preparation batch.

9.3.3 An LCS is not required for Cleveland Open Cup analysis.

9.4 Sample Duplicates

9.4.1 Definition: Sample Duplicate – a separate aliquot of a field sample taken through the entire analytical process.

9.4.2 Sample Duplicates do not count towards the 20 samples in an analytical batch.

9.5 Procedural Variations/ Nonconformance and Corrective Action

9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.5.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Thermometer calibration

10.1.1 Thermometers are verified annually. See SOP: ST-QA-0005.

10.2 Rotator verification

10.2.1 Rotation is not a variable setting (on/off switch). Apparatus must be verified annually. (for Pensky-Martens only)

10.3 The flash point of a check standard (P-Xylene) must be within a value obtained from the vendor's MSDS and within $\pm 10\%$.

10.4 Initial Calibration Verification

10.4.1 To annually verify the performance of the Flashpoint apparatus, an ICV will be analyzed at least once per year. Once this demonstration is verified, a secondary working standard will be used for routine analysis of the LCS in each sample batch.

10.4.2 A p-Xylene source different than the one used for the LCS is used as the initial calibration verification standard (for Pensky-Martens only).

10.4.3 ICV acceptance criteria: $\pm 10\%$ °C.

11.0 PROCEDURE

11.1 Flashpoint Correction

11.1.1 For both procedures, the flashpoint must be corrected for barometric pressure.

11.1.2 Use the INORG-0168_Flashpoint_Correction spreadsheet

11.2 Pensky-Martens Closed Cup

11.2.1 Thoroughly clean and dry all parts of the cup and its accessories before starting the test, being sure to remove any solvent which had been used to clean the apparatus.

11.2.2 Invert sample three times and carefully pour into flashpoint cup filling up to mark of grooved ring.

11.2.3 Place cup into tester and wipe away excess sample from cup.

11.2.4 Insert thermometer into aperture on cover until it is just above the blades.

11.2.5 Place cover on cup making sure that the locking devices are properly engaged.

11.2.6 Rotate arm of lower line on cover in upward direction.

11.2.7 Open main valve on propane tank.

11.2.8 Open secondary valve slightly and light the flame of lower line.

11.2.9 Rotate arm of lower line downward igniting the upper ignition line.

11.2.10 Adjust the test flame of upper line by means of the valve screw on the burner block to approximately 1/2" in length.

11.2.11 Turn blade under cover with spring handle to stir either manually or by connecting to a stirring motor. Recommended stirring speed is between 90 - 120 rpm.

11.2.12 Read initial temperature which should be 25°C or less.

11.2.13 To lower test flame into cup, turn operating knob of black handle on cover clockwise, observe reaction for any flash. This whole step should be done quickly, < 3 seconds, and also do not stir the sample while applying the test flame.

11.2.14 Turn on power of the tester. Rotate temperature controlling dial to 100 % output. Adjust the supply of heat at such a rate that the temperature as indicated by thermometer increases 5° to 6°C (9° to 11° F) / minute.

11.2.15 For samples for which the flashpoint is suspected to be below 60 °C, also cool sample to -5° C. Repeat step 11.1.13 as the temperature increases every 1°C (2° F) i.e. 25° C, 26° C, 27° C.. etc. until a flash is observed or temperature exceeds 60°C (140° F).

11.2.16 If the sample is known to have a flash point above 60°C (140°F), apply the test flame in the manner just described at each temperature that is a multiple of 2°C (5°F) i.e. 25°C, 27°C, 29°C,...etc., until a flash is observed or temperature exceeds 60°C (140°F).

11.2.17 Record as the flash point the temperature read on the thermometer at the time of the flame application that causes a distinct flash in the interior of the cup.

11.2.18 Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash. The actual flash will have occurred when a large flame propagates itself over the surface of the sample.

11.2.19 Turn off tester and rotate temperature controlling dial back to 0% output.

11.2.20 Close secondary valve on propane tank between samples.

11.2.21 Pour used sample into appropriate disposal container.

11.2.22 Rinse cup thoroughly with tap water, soap, DI water, and acetone.

11.2.23 Repeat analysis if required.

11.3 Cleveland Open Cup Method

- 11.3.1 Thoroughly clean and dry all parts of the cup and its accessories before starting the test, being sure to remove any solvent which had been used to clean the apparatus.
- 11.3.2 Invert sample three times and carefully pour into flashpoint cup filling up to mark of grooved ring.
- 11.3.3 Place cup into tester and wipe away excess sample from cup.
- 11.3.4 Insert thermometer into aperture on cover until it is just above the blades.
- 11.3.5 Rotate arm of lower line on cover in upward direction.
- 11.3.6 Open main valve on propane tank.
- 11.3.7 Open secondary valve slightly and light the flame of lower line.
- 11.3.8 Adjust the test flame line by means of the valve screw.
- 11.3.9 Slide flame arm over sample for less than three seconds
- 11.3.10 Turn on power of the tester. Rotate temperature controlling dial to 100 % output. Adjust the supply of heat at such a rate that the temperature as indicated by thermometer increases 5° to 6°C (9° to 11° F) / minute.
- 11.3.11 For samples for which the flashpoint is suspected to be below 60 °C, repeat step 11.1.13 as the temperature increases every 1°C (2° F) i.e. 25° C, 26° C, 27° C. etc. until a flash is observed or temperature exceeds 60°C (140° F).
- 11.3.12 If the sample is known to have a flash point above 60°C (140°F), apply the test flame in the manner just described at each temperature that is a multiple of 2°C (5°F) i.e. 25°C, 27°C, 29°C,...etc., until a flash is observed or temperature exceeds 60°C (140°F).
- 11.3.13 Record, as the observed flash point, the reading on the thermometer at the time the flame application causes a distinct flash in the interior of the cup.
- 11.3.14 Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash. The actual flash will have occurred when a large flame propagates itself over the surface of the sample.
- 11.3.15 Turn off tester and rotate temperature controlling dial back to 0% output.
- 11.3.16 Close secondary valve on propane tank between samples.
- 11.3.17 Pour used sample into appropriate disposal container.
- 11.3.18 Rinse cup thoroughly with tap water, soap, DI water, and acetone.
- 11.3.19 Repeat analysis if required.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 Observe and record the flash point of sample tested to the nearest 0.5° C (1° F).
- 12.3 Equation to correct flashpoint for barometric pressure

$$\text{Corrected Flashpoint} = C + 0.033 * (760 - P)$$

C = Observed Flashpoint, °C

P = Ambient Barometric Pressure, mmHg

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

- 13.2 Method Blank
 - 13.2.1 If the method blank flashes within the temperature range of this test, do not proceed with analysis.
- 13.3 Laboratory Control Sample (LCS)
 - 13.3.1 Acceptance Criteria:
 - 13.3.1.1 All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD).
 - 13.3.2 Corrective Action for LCS not meeting acceptance criteria:
 - 13.1.1.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM, (unless prohibited by client requirements). Samples with detects for the analyte with a high bias in the LCS are re-prepped and re-analyzed.
 - 13.1.1.2 LCS Spike Recovery excursion (low) – the batch is re-prepped and re-analyzed for the affected analytes.
 - 13.1.1.3 RPD excursion for LCS/LCSD – If spike recoveries are acceptable, data is evaluated for reportability and an NCM generated. If spike recoveries and RPD are outside limits, batch is re-prepped/re-analyzed for affected analytes.
- 13.4 RPD (%D)
 - 13.4.1 % D excursion for sample/sample duplicate – If there is physical evidence of non-homogeneity or matrix interference, the data is reported with an NCM describing the physical evidence; otherwise the sample should be re-prepped and re-analyzed.
- 13.5 Insufficient Sample
 - 13.5.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of this SOP.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
 - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for

performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
 - 16.2.1 The following waste streams are produced when this method is carried out.
 - 16.2.1.1 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.
 - 16.2.1.2 Solvent waste generated. Solvent waste must be accumulated in the appropriate waste accumulation container, labeled as Drum Type "D".

17.0 REFERENCES

- 17.1 EPA Method 1010A, SW-846, "Test Methods for Evaluating Solid Waste", Revised September 1986.
- 17.2 ASTM Methods D93-02 and D92-02a ASTM 2003 Volume 05.01
- 17.3 Instruction Manual for the Koehler K 16200 Electric Pensky-Martens Closed Cup Flash Tester.
- 17.4 TestAmerica St. Louis Quality Assurance Manual (ST-QAM)
- 17.5 Corporate Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.6 Associated SOP:
 - 17.6.1 ST-PM-0002, Sample Receipt and Chain of Custody
 - 17.6.2 ST-QA-0002, Standard and Reagent Preparation
 - 17.6.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.
 - 17.6.4 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.6.5 ST-QA-0036, Non-conformance Memorandum (NCM) Process
 - 17.6.6 ST-IP-0004, Labware Preparation for Inorganic and Trace Metals Analysis

18.0 CLARIFICATIONS, MODIFICATIONS TO REFERENCE METHOD

- 18.1 This method is applicable to samples having a flash point range from -5 to 370°C. Because this SOP specifically addresses the RCRA Ignitability requirement, directions are given only for determining flashpoints below 60 °C. All other samples are reported as having a flashpoint above 60 °C. The

actual flashpoint above 60 °C can be determined by extension of the procedure given here, at client request.

19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Updated method reference from 1010 to 1010A throughout document.
- 19.2 Rev. 12
 - 19.2.1 In section 10.0, updated the ICV and CCV control limits to ± 1.1 .
- 19.3 Rev. 13:
 - 19.3.1 Updated the ASTM method.
- 19.4 Rev. 14:
 - 19.4.1 Removed references to QuantIMS and Clouseau
 - 19.4.2 Updated section 9.1.3 to note that an LCSD must be performed in the event that no volume exists to run a Sample Duplicate.
 - 19.4.3 Updated section 9.3.2 to correct the frequency that an LCS must be analyzed.
 - 19.4.4 Added header for section 10.4.
 - 19.4.5 Added header for section 10.5 and clarified in 10.5.1 that a CCV must be run at the end of the analytical sequence.
 - 19.4.6 Section 13 updated with corrective actions
 - 19.4.7 Section 15 updated
- 19.5 Rev. 15:
 - 19.5.1 Added section 10.4.1 to mirror method requirement of an annual apparatus check with an independent certified reference material (ICV). This clarifies that the ICV is now an annual check, not a daily check.
 - 19.5.2 Removed reference to CCV from section 10. A CCV is not required by method ASTM D93-11.
 - 19.5.3 Section 10.5 removed. A sample duplicate is not required for every sample that flashes, according to ASTM D93-11.
- 19.6 Rev. 16: (09/30/2015)
 - 19.6.1 Updated LCS and ICV acceptance criteria in section 10
- 19.7 Rev. 17: (10/6/16)
 - 19.7.1 Corrected ASTM methods in title, section 1 and section 17
 - 19.7.2 Updated required purity of *m*-xylene in Section 7 from 95% to 99%
 - 19.7.3 Updated Storage temperature in Section 8.
- 19.8 Rev. 18: (11/16/2016)
 - 19.8.1 Added barometric pressure correction (Section 11.1 and 12.3)