FINAL
UNIFORM FEDERAL POLICY QUALITY ASSURANCE PROJECT PLAN
FOR
REMEDIAL ACTION

FORMER RAVENNA ARMY AMMUNITION PLANT
RVAAP-063-R-01 GROUP 8
MUNITIONS RESPONSE SITE
PORTAGE AND TRUMBULL COUNTIES, OHIO

Contract No. W912DR-21-D-0005
Delivery Order No. W912DR21F0327

Prepared for

U.S. Army Corps of Engineers, Baltimore District
2 Hopkins Plaza
Baltimore, MD 21201-2930

Prepared by:
HydroGeoLogic, Inc.
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July 2023

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6. AUTHOR(S)
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5d. PROJECT NUMBER
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Planning document for Remedial Action at RVAAP-063-R-01 Group 8 MRS.

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Ohio Environmental Protection Agency

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Mike DeWine, *Governor* Jon Husted, *Lt. Governor* Anne M. Vogel, *Director*

August 31, 2023

TRANSMITTED ELECTRONICALLY

Mr. Kevin M. Sedlak
Restoration Program Manager
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Sent via email to:
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RE: US Army Ravenna Ammunition Plt
RVAAP
Remediation Response
Plans
Federal Facilities
RA
Portage County
267000859272

Subject: **Final Uniform Federal Policy Quality Assurance Project Plan/Remedial Action Work Plan for the RVAAP-063-R-01 Group 8 Munitions Response Site, Version 1.0, Portage and Trumbull Counties, Ohio - Dated July 25, 2023 - Ohio EPA Concurrence**

Dear Mr. Sedlak:

On July 25, 2023, the Ohio Environmental Protection Agency (Ohio EPA), Northeast District Office (NEDO), Division of Environmental Response and Revitalization (DERR) received the "Final Uniform Federal Policy Quality Assurance Project Plan/Remedial Action Work Plan for the RVAAP-063-R-01 Group 8 Munitions Response Site, Version 1.0, Portage and Trumbull Counties, Ohio"[1] (the "Report") dated July 25, 2023. The Report was submitted by Chenega Reliable Services, on behalf of the U.S. Army Corps of Engineers (USACE), Baltimore District, in response to Ohio EPA's June 20, 2023, letter requesting the final document.

Ohio EPA concurs with the Report.

Received 01 SEP 2023

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[1] http://edocpub.epa.ohio.gov/publicportal/ViewDocument.aspx?docid=2480136

If you have any questions concerning this letter or report, please contact me at (330) 963-1235 or nicholas.roope@epa.ohio.gov.

Sincerely,

Nicholas Roope
Environmental Specialist
Division of Environmental Response and Revitalization

NR/cm

ec: Katie Tait, OHARNG RTLS, CJAG
Steve Kvaal, USACE Louisville
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Angela Cobbs, Chenega
Brian Tucker, Ohio EPA, CO, DERR
Megan Oravec, Ohio EPA, NEDO, DERR
Natalie Oryshkewych, Ohio EPA, NEDO, DERR
Thomas Schneider, Ohio EPA, SWDO, DERR

CONTRACTOR'S STATEMENT OF INDEPENDENT TECHNICAL REVIEW

HydroGeoLogic, Inc., has completed the Final Remedial Action Work Plan for the RVAAP-063-R-01 Group 8 Munitions Response Site at the former Ravenna Army Ammunition Plant in Portage and Trumbull counties, Ohio. Notice is hereby given that an independent technical review has been conducted that is appropriate to the level of risk and complexity inherent in the project. During the independent technical review, compliance with established policy, principles, and procedures, and utilizing justified and valid assumptions was verified. This included review of data quality objectives; technical assumptions; methods, procedures, and materials to be used; the appropriateness of data used and level of data obtained; and reasonableness of the results, including whether the product meets customer's needs consistent with law and existing United States Army Corps of Engineers policy.

Reviewed/Approved by: Smith, Alexander Digitally signed by Smith, Alexander Date: 2023.07.10 10:55:13 -04'00' Date: ______________
Alex Smith
Program Manager

Prepared/Approved by: Kimberly Vaughn Date: 7/10/2023
Kimberly Vaughn
Project Manager

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DOCUMENT DISTRIBUTION

Final Uniform Federal Policy Quality Assurance Project Plan for RVAAP-063-R-01 Group 8 Munitions Response Site

Name/Organization	Number of Printed Copies	Number of Electronic Copies
Kevin Sedlak, ARNG Restoration Program Manager, ARNG-IED	0	1
Katie Tait, Environmental Specialist, OHARNG	0	1
Steve Kvaal, USACE Louisville District Project Manager	0	1
Travis McCoun, USACE Baltimore District COR	0	1
Nicole Walworth, USACE Baltimore District TM	0	1
Jennifer Tierney, RVAAP Administrative Record Manager	2	1
Nicholas Roope, Site Coordinator, Ohio Environmental Protection Agency	0	1
Megan Oravec, Environmental Manager, Ohio Environmental Protection Agency	0	Email/Transmittal Letter
Tom Schneider, Ohio Environmental Protection Agency, Federal Facilities	0	Email/Transmittal Letter

ARNG = Army National Guard
COR = Contracting Officer's Representative
IED = Installation and Environment Division
OHARNG = Ohio Army National Guard
RVAAP = Former Ravenna Army Ammunition Plant
TM = Technical Manager
USACE = U.S. Army Corps of Engineers

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TABLE 1 CROSSWALK: OPTIMIZED UFP-QAPP WORKSHEETS TO MR-QAPP MODULE 1

Optimized UFP-QAPP Worksheets		**Munitions Response (MR)-QAPP Module 1:**
1 & 2	Title and Approval Page	Included
3 & 5	Project Organization and QAPP Distribution	Included
4, 7, & 8	Personnel Qualifications and Sign-off Sheet	Included
6	Communication Pathways and Procedures	Included
9	Project Planning Session Summary	Included
10	Conceptual Site Model	Included
11	Project/Data Quality Objectives	Included
12	Measurement Performance Criteria	Included
13	Secondary Data Uses and Limitations	Included
14 & 16	Project Tasks and Schedule	Included
15	Project Action Limits and Laboratory-Specific Detection/Quantitation Limits	Included
17	Sampling Design and Rationale	Included – Title changed to “Survey Design and Project Work Flow”
18	Sampling Locations and Methods	Included
19 & 30	Sample Containers, Preservation, and Hold Times	Included
20	Field Quality Control (QC)	Included
21	Field Standard Operating Procedures (SOPs)	Included
22	Field Equipment Calibration, Maintenance, Testing, and Inspection	Included – Title changed to “Equipment Testing, Inspection, and Quality Control”
23	Analytical SOPs	Included
24	Analytical Instrument Calibration	Included
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection	Included
26 & 27	Sample Handling, Custody, and Disposal	Included

TABLE 1 CROSSWALK: OPTIMIZED UFP-QAPP WORKSHEETS TO MR-QAPP MODULE 1

Optimized UFP-QAPP Worksheets		**Munitions Response (MR)-QAPP Module 1:**
28	Analytical Quality Control and Corrective Action	Included
29	Project Documents and Records	Included – Title changed to "Data Management, Project Documents, and Records"
31, 32, & 33	Assessments and Corrective Action	Included
34	Data Verification and Validation Inputs	Included – Title changed to "Data Verification, Validation, and Usability Inputs"
35	Data Verification Procedures	Included – Title changed to "Data Verification and Validation Procedures"
36	Data Validation Procedures	Included
37	Data Usability Assessment	Included

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LIST OF ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
%	percent
%D	percent difference
%R	percent recovery
%RSD	percent relative standard deviation
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
amsl	above mean sea level
amu	atomic mass unit
AQ	aqueous
ARNG	Army National Guard
ASR	Archives Search Report
B.S.	Bachelor of Science
bgs	below ground surface
CA	Corrective Action
CAR	corrective action request
CASRN	Chemical Abstracts Service Registration Number
CCB	continuing calibration blank
CCV	continuing calibration verification
Cd	cadmium
CENAB	U.S. Army Corps of Engineers, Baltimore District
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFR	Code of Federal Regulations
CHMM	Certified Hazardous Materials Manager
CHSM	Corporate Health and Safety Manager
CIH	Certified Industrial Hygienist
CJAG	Camp James A. Garfield Joint Military Training Center
CoC	chain-of-custody
COC	contaminant of concern
COR	Contracting Officer’s Representative
CPG	Certified Professional Geologist
CQA	Certified Quality Auditor
CQM	Corporate Quality Manager
CQMC	Construction Quality Management for Contractors
CSM	Conceptual Site Model
CSP	Certified Safety Professional
CVAA	cold vapor atomic absorption
cy	cubic yard
DDESB	Department of Defense Explosives Safety Board
DDT	dichlorodiphenyltrichloroethane
DFFO	Director’s Final Findings and Orders
DFW	Definable Feature of Work

LIST OF ACRONYMS AND ABBREVIATIONS (Continued)

DGM	Digital Geophysical Mapping
DL	detection limit
DM	Database Manager
DoD	Department of Defense
DPT	direct-push technology
DQCR	Daily Quality Control Report
DQI	data quality indicator
DQO	data quality objective
EB	equipment blank
EDD	electronic data deliverable
ELAP	Environmental Laboratory Accreditation Program
EPA	U.S. Environmental Protection Agency
FB	field blank
FD	field duplicate
FS	Feasibility Study
ft	foot/feet
FTL	Field Team Lead
FWV	field work variance
GC	gas chromatograph
GC/MS	gas chromatography/mass spectrometry
GIS	Geographic Information System
GPS	Global Positioning System
H&S	Health and Safety
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	high-density polyethylene
HE	high explosive
HGL	HydroGeoLogic, Inc.
HPLC	high performance liquid chromatography
HRR	Historical Records Review
ICAL	initial calibration
ICB	initial calibration blank
ICP-AES	inductively coupled plasma-atomic emission spectroscopy
ICP-MS	inductively coupled plasma-mass spectrometry
ICS	interference check solution
ICV	initial calibration verification
ID	identification
IDW	investigation-derived waste
IED	Installation and Environment Division
ISM	Incremental Sampling Methodology
ITS	instrument test strip

LIST OF ACRONYMS AND ABBREVIATIONS (Continued)

L	liter
lb	pound
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LOD	limit of detection
LOQ	limit of quantitation
M.S.	Master of Science
MB	method blank
MC	munitions constituent
MD	munitions debris
MDAS	material documented as safe
MEC	munitions and explosives of concern
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mL	milliliters
mm	millimeters
MMRP	Military Munitions Response Program
MPC	Measurement Performance Criteria
MPPEH	material potentially presenting an explosive hazard
MPS	milestone payment schedule
MR	Munitions Response
MRS	Munitions Response Site
MRSPP	Munitions Response Site Prioritization Protocol
MS	matrix spike
MSD	matrix spike duplicate
NA	not applicable
$NaHSO_4$	sodium bisulfate
NaOH	sodium hydroxide
NCR	Non-Conformance Report
NE	not established
NGB	National Guard Bureau
OESS	Ordnance Explosives Safety Specialist
ODNR	Ohio Department of Natural Resources
OHARNG	Ohio Army National Guard
Ohio EPA	Ohio Environmental Protection Agency
oz	ounce
P.G.	Professional Geologist
PAL	project action limit
PARCCS	precision, accuracy, representativeness, completeness, and comparability
Pb	lead
PC	Project Chemist
PCB	polychlorinated biphenyls

PDS	post digestion spike
PDT	project delivery team
PgC	Program Chemist
PgM	Program Manager
Ph.D.	Doctor of Philosophy
PM	Project Manager
PMP	Project Management Professional
PP	Proposed Plan
PRG	preliminary remediation goal
PTFE	polytetrafluoroethylene
PWS	Performance Work Statement
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QASP	Quality Assurance Surveillance Plan
QC	Quality Control
QCSM	Quality Control Site Manager
QCSR	Quality Control Summary Report
QSM	Quality System Manual
r^2	correlation coefficient
RA	Remedial Action
RCA	Root Cause Analysis
RCRA	Resource Conservation and Recovery Act
RDX	Research Department Explosive
RI	Remedial Investigation
RME	Risk Management Evaluation
ROD	Record of Decision
RPD	relative percent difference
RRT	relative retention time
RSL	Regional Screening Level
RVAAP	Ravenna Army Ammunition Plant
SDG	sample delivery group
SI	Site Inspection
SO	soil
SOP	standard operating procedure
SSHO	Site Safety and Health Officer
SVOC	semivolatile organic compound
TB	trip blank
TBD	to be determined
TCLP	toxicity characteristic leaching procedure
THQ	target hazard quotient
TM	Technical Manager
TNT	2,4,6-trinitrotoluene

LIST OF ACRONYMS AND ABBREVIATIONS (Continued)

TOI	target of interest
TP	Technical Paper
TPP	Technical Project Planning
UFP	Uniform Federal Policy
URL	Uniform Resource Locator
USACE	U.S. Army Corps of Engineers
UXO	Unexploded Ordnance
UXOQCS	Unexploded Ordnance Quality Control Specialist
UXOSO	Unexploded Ordnance Safety Officer
VOC	volatile organic compound
WAC	waste acceptance criteria
WS	Worksheet

UNIFORM FEDERAL POLICY-QUALITY ASSURANCE PROJECT PLAN
FOR
REMEDIAL ACTION
FORMER RAVENNA ARMY AMMUNITION PLANT
RVAAP-063-R-01 GROUP 8
MUNITIONS RESPONSE SITE
PORTAGE AND TRUMBULL COUNTIES, OHIO

INTRODUCTION

HydroGeoLogic, Inc. (HGL) is conducting a Remedial Action (RA) at the RVAAP-063-R-01 Group 8 Munitions Response Site (MRS) located at the Former Ravenna Army Ammunition Plant (RVAAP) in Portage and Trumbull Counties, Ohio (Figure 10.1). The former RVAAP is now known as Camp James A. Garfield Joint Military Training Center (CJAG). CJAG was previously known as Camp Ravenna Joint Military Training Center (Camp Ravenna), and that name is used in some historical reports. The Group 8 MRS is located just north of the southern facility boundary of CJAG (Figure 10.2).

The federally-owned CJAG facility comprises 21,683 acres and is located in Portage and Trumbull counties, Ohio, approximately 3 miles east-northeast of the City of Ravenna (Figure 10.1). Administrative accountability for CJAG was allocated to the U.S. Property and Fiscal Officer for Ohio in multiple transfers, the last occurring in September 2013. The facility is licensed to the Ohio Army National Guard (OHARNG) for use as a military training facility (Federal Facility Identification (ID) No. OH213820736). To maintain a distinction between historical operations and current activities, the term “RVAAP” will be used for historical discussions, and “CJAG” will be used when referring to the current facility activities.

Army National Guard (ARNG) is the lead federal agency for environmental response actions at CJAG. The *Final Record of Decision for RVAAP-063-R-01 Group 8 Munitions Response Site, Version 1.0* (USACE, 2020) was prepared under the Military Munitions Response Program (MMRP) and presents the selected remedy for the Group 8 MRS. The selected remedy was chosen in accordance with the requirements of the *Comprehensive Environmental Response, Compensation, and Liability Act* (CERCLA) of 1980, as amended by the *Superfund Amendments and Reauthorization Act* of 1986, which requires the issuance of Records of Decision (RODs) for RA taken pursuant to Sections 104, 106, 120, and 122. The *National Oil and Hazardous Substances Pollution Contingency Plan* establishes the regulatory requirements for the ROD in Chapter 40 of the Code of Federal Regulations (CFR), Section 300.430(f)(5).

The Ohio Environmental Protection Agency (Ohio EPA), the supporting state regulatory agency, reviewed and concurred with the *Final Proposed Plan for RVAAP-063-R-01 Group 8 Munitions Response Site, Version 2.0* (HGL, 2020). ARNG’s decision is based on information contained in the Administrative Record file for the Group 8 MRS. The Proposed Plan (PP) presented ARNG’s preferred remedy for addressing the Group 8 MRS and invited public involvement during the comment period (March 4, 2020, through April 4, 2020) and at a public meeting held March 11, 2020. The selected remedy under CERCLA at the Group 8 MRS satisfies the requirements of the *Director’s Final Findings and Orders (DFFO) for RVAAP* (Ohio EPA, 2004), specifically by documenting the Ohio EPA's concurrence with the closeout of the MMRP investigation conducted for this MRS under Section XII, paragraph 26.

The response action selected in the ROD is considered necessary to protect the public health or welfare or environment from actual or threatened releases of hazardous substances to the environment. Based on the information currently available, ARNG believes the selected remedy meets the threshold criteria and provides the best balance of tradeoffs among other alternatives, with respect to the balancing and modifying criteria (detailed in Section J). ARNG expects the selected remedy to satisfy the requirements of CERCLA by protecting human health, complying with applicable or relevant and appropriate requirements, being cost-effective, and utilizing permanent solutions to the maximum extent practical. No unacceptable ecological risk was identified at the Group 8 MRS; therefore, the remedy does not address ecological receptors. However, the Northern Long-Eared Bat, which is a federally threatened species, is present at CJAG and will be considered during removal action activities.

The selected remedy for the Group 8 MRS is *Alternative 3 – Munitions Constituent (MC)-Contaminated Soil Removal*. Mechanical and manual excavation techniques will be used to remove MC-contaminated soil to a depth of 0.5 feet (ft) below ground surface (bgs). As summarized in the Final PP and in Section E.4.2 of the ROD, the only receptor with a complete exposure pathway for risk from MC-contaminated soil is the theoretical Future Resident Receptor. This remedy is protective of the theoretical future Resident Receptor. This alternative is selected over the other alternatives because it provides the greatest protection of human health, results in complete removal of MC-contaminated soil at the MRS and provides the greatest long-term effectiveness. Furthermore, no RA-operation, long-term monitoring costs, or Five-Year Reviews will be required. The selected remedy for the Group 8-MRS includes the following activities:

- Removal of lead- and cadmium-contaminated soil exceeding the Preliminary Remediation Goals (PRGs) to a depth of 0.5 ft bgs;
- Incidental to the surface soil removal, if any munitions debris (MD) is encountered, it will be segregated, inspected, and certified as material documented as safe (MDAS) before disposal;
- Confirmation soil samples will be collected to confirm that the extent of MC-contaminated soil has been removed;
- If required, up to 2.0 ft bgs may be excavated, based on the results of pre-excavation sampling; and
- Implementing this remedy will allow for unlimited use/unrestricted access at the MRS (although future residential use is not anticipated and is theoretical only).

This RA Work Plan is organized to be consistent with the *Uniform Federal Policy for Quality Assurance Project Plans, Optimized Worksheets* (IDQTF, 2020). Project requirements associated with the planning, collection, analysis, and evaluation of material potentially presenting an explosive hazard (MPPEH) and MC remediation activities are structured per the Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP) format. Additional project components, including MMRP and construction activities, are incorporated into the overall document structure where appropriate.

WORKSHEETS #1 AND #2
TITLE AND APPROVAL PAGE

PROJECT IDENTIFYING INFORMATION

Site Name/ Project Name:	RVAAP-063-R-01 Group 8 Munitions Response Site
Site Location/No.:	Former Ravenna Army Ammunition Plant Portage and Trumbull Counties, Ohio
Lead Organization:	U.S. Army Corps of Engineers (USACE), Baltimore District (CENAB)
Contractor	HGL
Contract/Delivery Order No.:	Contract No. W912DR-21-D-0005 Delivery Order No. W912DR21F0327 HGL Project No. BM3001

CONCURRING SIGNATURES

The following signatures indicate the representatives of the subject organizations have reviewed this UFP-QAPP and concur with its implementation as written.

Department of Defense (DoD) Remedial Project Manager (PM)

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Nicole Walworth, CENAB PM — Date

DoD Quality Assurance (QA) Manager

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State Regulatory Agency

Nicholas Roope, Ohio EPA - Northeast District Office Technical Reviewer — Date

PLANS AND REPORTS

- Ohio EPA, 2004. *Director's Final Findings and Orders (DFFO) for RVAAP, Division of Emergency and Remedial Response*. June 10.
- Engineering-environmental Management, Inc. (e2M), 2008. *Final Site Inspection Report, Ravenna Army Ammunition Plant, Ohio, Military Munitions Response Sites, Ravenna Army Ammunition Plant, Ohio*. May.
- Science Applications International Corporation (SAIC), 2010. *Final Facility-Wide Human Health Cleanup Goals for the Ravenna Army Ammunition Plant Ravenna, Ohio*. March.
- ARNG, 2014. *Final Technical Memorandum: Land Uses and Revised Risk Assessment Process for the Ravenna Army Ammunition Plant (RVAAP) Installation Restoration Program, Portage/Trumbull Counties, Ohio*. February.
- CB&I Federal Services LLC (CB&I), 2015. *Remedial Investigation Report for RVAAP-063-R-01 Group 8 MRS*, Version 1.0, Former Ravenna Army Ammunition Plant, Portage and Trumbull Counties, Ohio, Final. May.
- HGL, 2020. *Final Proposed Plan for RVAAP-063-R-01 Group 8 Munitions Response Site*, Version 2.0. January.
- USACE, 2020. *Final Record of Decision for RVAAP-063-R-01 Group 8 Munitions Response Site*, Version 1.0. September.

ANALOG TECHNOLOGY

The undersigned concur that the use of analog technology is justified in the Group 8 MRS RA. Digital geophysical mapping (DGM) was used for the previous remedial investigation and the conclusions reached supporting this RA. The anomaly avoidance required for the current RA will be accomplished with analog technology, to accomplish the remedy.

Digitally signed by
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DoD Remedial PM

Nicole Walworth, CENAB PM — Date

State Regulatory Agency

Nicholas Roope, Ohio EPA - Northeast District Office
Technical Reviewer — Date

WORKSHEETS #3 AND #5
PROJECT ORGANIZATION AND QAPP DISTRIBUTION

(UFP-QAPP Manual Sections 2.3 and 2.4)

This worksheet identifies key project personnel and their points of contact as well as lines of authority and lines of communication among the lead organization, prime contractor, subcontractors, and regulatory agencies. Figure 3.1 provides the project organization for the munitions response team and explosive safety operations activities. Please see the document distribution list placed at the cover pages of this document for the distribution to recipients of the QAPP.

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address
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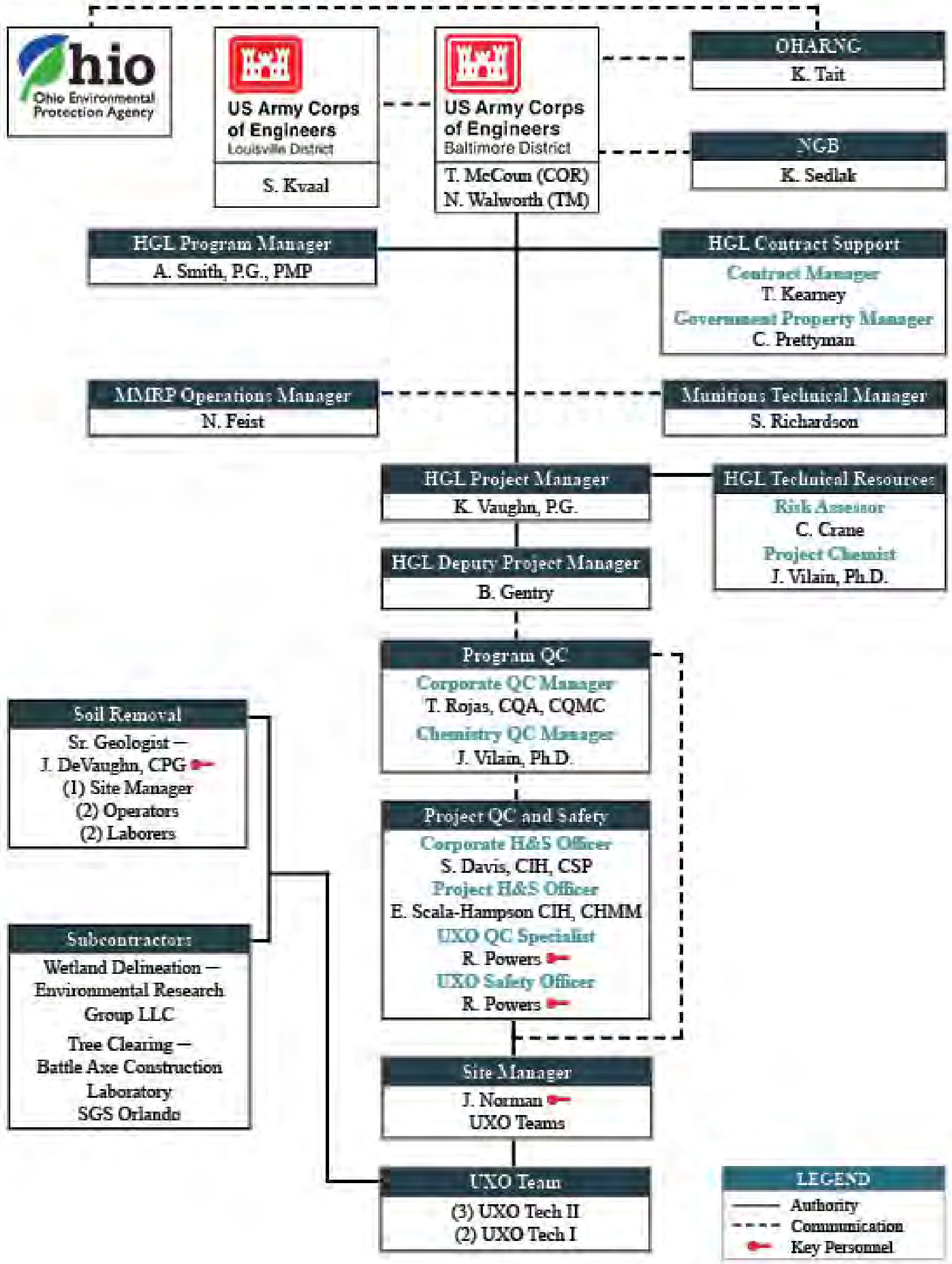
COR = Contracting Officer's Representative
TBD = to be determined
UXOQCS = Unexploded Ordnance Quality Control Specialist
UXOSO = Unexploded Ordnance Safety Officer

WORKSHEETS #3 AND #5 (CONTINUED)
PROJECT ORGANIZATION AND QAPP DISTRIBUTION

Figure 3.1 Project Organizational Structure



CHMM = Certified Hazardous Materials Manager
CIH = Certified Industrial Hygienist
CPG = Certified Professional Geologist
CQA = Certified Quality Auditor
CQMC = Construction Quality Management for Contractors
CSP = Certified Safety Professional
H&S = health and safety
NGB = National Guard Bureau
P.G. = Professional Geologist
Ph.D. = Doctor of Philosophy
PMP = Project Management Professional
TM = Technical Manager
UXO = unexploded ordnance

WORKSHEETS #4, #7, AND #8
PERSONNEL QUALIFICATIONS AND SIGN-OFF SHEET

Organization: HGL

Name/Contact Information	Project Title/Role	Education/Experience	Specialized Training	Required Licenses/Certifications/ Authorizations	Signature/Date
Alex Smith	HGL PgM	B.S., Geology Experience: 25 years	40-Hour HAZWOPER/8-Hour Refresher/8-Hour Supervisor/30-Hour Construction Safety Training USACE CQMC	Professional Geologist Project Management Professional	
Kimberly Vaughn	HGL PM	B.S., Geology Environmental/MMRP Focus Experience: 20 years	P.G.; DoD Quality System Requirements, and Prohibited Practices Training		
Teresa Rojas	Contractor QC Manager	B.S. Chemistry Experience: 36 years	American Society for Quality CQA USACE Construction Quality Manager Six Sigma Green Belt	Contractor QC Manager	
Edie Scala-Hampson	Corporate Health and Safety Manager (CHSM)	M.S., Industrial Engineering		CIH	
Jesse Norman	HGL – Site Manager	Naval School Explosive Ordnance Disposal	2004 HAZWOPER 40-Hour Training, HAZWOPER 8-Hour Refresher Training; HAZWOPER 8-Hour Supervisor Training	Qualified Senior Unexploded Ordnance Supervisor in accordance with DDESB TP-18 USACE CQMC	
Rebecca Powers	HGL – UXOSO/UXOQCS	Basic Explosive Ordnance Disposal School, 2011	HAZWOPER 40-Hour Training, HAZWOPER 8-Hour Refresher Training; 30-Hour Construction Safety	Qualified UXOSO in accordance with DDESB TP-18	
Justin Hersh	HGL Staff Chemist	B.S. Wildlife Biology B.S. Natural Resources and Environmental Science Experience 15 years			

WORKSHEETS #4, #7, AND #8 (CONTINUED)
PERSONNEL QUALIFICATIONS AND SIGN-OFF SHEET

Organization: SGS Orlando (Analytical Laboratory)

Name/Contact Information	Project Title/Role	Education/Experience	Signature/Date
Caitlin Brice	Laboratory Director	M.S., Biology Experience: 10 years	
Norm Farmer	Perfluoroalkyl and Polyfluoroalkyl Substances Technical Director	B.S., Chemistry Experience: 33 years	
Svetlana Izosimova	QA Director	B.S./M.S., Chemical Engineering Ph.D., Colloid Chemistry Experience: 31 years	
Ariel Hartney	Laboratory PM	B.S., Wildlife Ecology and Conservation Experience: 14 years	

Organization: Laboratory Data Consultants (Data Validation)

Name/Contact Information	Project Title/Role	Education/Experience	Signature/Date
Pei Geng	Data Validator PM	M.S., Chemistry Experience: 28 years	

B.S. = Bachelor of Science
DDESB = Department of Defense Explosives Safety Board
HAZWOPER = Hazardous Waste Operations and Emergency Response
M.S. = Master of Science
TP = Technical Paper

WORKSHEET #6
COMMUNICATION PATHWAYS AND PROCEDURES

Communication Driver	Initiator (role) (1)(2)	Recipient(s) (role) (1)	Procedure
General communication between USACE and other project delivery team (PDT) members	CENAB COR or designee	Appropriate PDT member(s)	Communicates directly, as needed, (verbally and/or in writing).
Regulatory interface	CENAB COR	Stakeholders	As required, all materials and information about the project will be forwarded by the CENAB COR, or by the HGL PM with permission from the COR.
Stakeholder agency contacts	Stakeholders	CENAB COR	Communicates directly as needed (verbally and/or in writing).
Project management, Delivery Order administration and logistics	HGL PM	CENAB COR and appropriate PDT member(s)	Communicates directly, as needed, (verbally and/or in writing). The PM will communicate project-related issues, including changes in schedule, changes in scope of fieldwork or delays, and recommendations to stop work, to the CENAB COR by phone, email, or fax by Close of Business, next business day. The PM will also provide project information to the CENAB COR through monthly progress reports, email updates, teleconference calls, and meetings. They will document deviations from the QAPP and corrective actions (CAs) in memoranda to the CENAB COR.
Daily reports	HGL Site Manager	HGL PM and lead technical and site personnel	Documents progress in daily report and submits to HGL PM for onward distribution to PDT. Daily reports will be submitted to PDT by 9 a.m. of the following workday. Field logbooks will be provided upon request. Field progress reports will vary based on the objectives of each definable feature of work (DFW). Examples of these reports are geophysical surveying, intrusive investigation, and daily production reports.

WORKSHEET #6 (CONTINUED)
COMMUNICATION PATHWAYS

Communication Driver	Initiator (role) (1)(2)	Recipient(s) (role) (1)	Procedure
Stop work due to safety issues	Any field or office personnel	HGL Site Manager, SSHO, and other field personnel	Work may be stopped at any time for any safety concern. If unsafe work conditions are noted, the SSHO will stop work immediately. Work will not be allowed to resume until the unsafe condition is corrected. The SSHO will notify the Corporate H&S Officer immediately when a stop work situation is encountered. In some cases, such as inclement weather (for example, lightning or high winds), no CA is required, and work may resume when the SSHO and Corporate H&S Officer determine that conditions allow.
	HGL Site Manager or SSHO	HGL PM	Verbally notify the HGL PM as soon as possible after work stoppage.
	HGL PM	CENAB COR	Notify CENAB verbally or via email as soon as possible after work stoppage.
QAPP changes before fieldwork	HGL PM	CENAB COR	If errors or changed conditions require the modification of the QAPP before fieldwork begins, the HGL PM will prepare revised text. All changes to the QAPP will require final approval from USACE and regulatory agencies.
Minor QAPP changes during project execution	HGL Site Manager, UXOQCS	HGL PM, HGL QC Manager, HGL Project Geophysicist	Minor QAPP changes will be noted on the Daily QC Reports (DQCRs) and forwarded to the Project Geophysicist, HGL PM, and the Project QC Manager, at the end of each day.
Major QAPP changes during project execution	HGL PM	CENAB COR	Within 24 hours, the HGL PM will submit a field change request form to the HGL QA Manager and USACE PM for approval. Following approval, CENAB COR informs stakeholders via email.

WORKSHEET #6 (CONTINUED)
COMMUNICATION PATHWAYS

Communication Driver	Initiator (role) (1)(2)	Recipient(s) (role) (1)	Procedure
Field team finds munitions and explosives of concern (MEC) item(s)	HGL Site Manager	HGL PM, COR, USACE OESS	Verbally notifies HGL PM and USACE OESS immediately
	HGL PM	CENAB COR	Verbally notifies CENAB COR.
	CENAB COR	Stakeholders and other PDT members	Notify other PDT members, as necessary.
Field team ready to conduct MEC disposal operations	HGL Site Manager	HGL PM	Notifies HGL PM and other organizations, as necessary.
	HGL PM	USACE PM, and PDT	HGL PM notifies CENAB COR verbally and other USACE PDT members via email.
Field CAs	HGL Site Manager	HGL PM	CA resulting from either failure to follow QAPP requirements or due to changes in site conditions will be documented by the Site Manager; the Site Manager will communicate the need for CA to the PM on the same business day. Site Manager may initiate interim CA in the field subject to final approval by the PM and Program QA Manager.
Sample receipt discrepancies (e.g., broken or missing samples, improper preservation, missing analysis requests)	HGL Project Chemist (PC)	Laboratory PM	The Laboratory PM will communicate discrepancies in sample receipt to the HGL PM and HGL DM on the same business day the discrepancy is identified. The HGL PM or HGL DM, in consultation with the HGL PC, will instruct the Laboratory PM on the appropriate course of action.
Laboratory QC variances	HGL PC	Laboratory PM	The HGL PC will prepare variance requests in collaboration with the Laboratory PM for transmittal to the USACE PM for approval.
Analytical CAs	HGL PC	Laboratory PM	The need for laboratory CAs will be determined by the HGL PC and/or Laboratory PM/QA Manager. The CA will be documented in memoranda to the HGL PM and USACE PM.
Data verification issues (e.g., incomplete records)	HGL PC	Laboratory PM	The data validators will contact the Laboratory PM directly when a discrepancy is a simple report generation error (such as a skipped page or data missing for a subcontracted analytical method). For systematic problems, such as incorrectly formatted data reports or failure to include required data QC elements, the data validators will contact the HGL PC. The HGL PC will work with the Laboratory PM to ensure that properly formatted data reports are delivered to the data validators on a timely basis.

WORKSHEET #6 (CONTINUED)
COMMUNICATION PATHWAYS

Communication Driver	Initiator (role) (1)(2)	Recipient(s) (role) (1)	Procedure
Data validation issues (e.g., non-compliance with procedures)	HGL PC	Laboratory PM	If it is determined that the laboratory is not in compliance with the requirements of the UFP-QAPP, the HGL PC will coordinate with the SGS PM to bring the laboratory's practices into compliance. In some cases, this will require the preparation of the variance request (see above).
Data review CAs	HGL PC	Laboratory PM	Final analytical data cannot be released until any required validation is complete and the HGL PC has approved the release.
Data tracking and management	HGL DM	HGL PC/PM	The HGL DM or designee will track data from the collection of samples, through login at the laboratory, to delivery of the technical report/sample delivery group of electronic data, and its entry into the project database.

(1) Names and contact information for personnel provided on Worksheets (WSs) #4, 7, & 8.

(2) The initiator may designate another qualified individual to communicate with the recipient(s); however, the initiator shown is responsible for the communication being made.

DM = Database Manager
OESS = Ordnance and Explosives Safety Specialist
SSHO = Site Safety and Health Officer

WORKSHEET #9
PROJECT PLANNING SESSION SUMMARY

Project Kickoff Call

Meeting Date: Tuesday, 05 October 2021
Meeting Time: 1000 Central Time
Meeting Location: Webex Teleconference

Attendees

Name	Org	Role	Number	Email
Travis McCoun	CENAB	COR	443-831-2303	Travis.mccoun@usace.army.mil
Nicole Walworth	CENAB	PM	443-469-1377	Nicole.u.walworth@usace.army.mil
Steven Kvaal	USACE, Louisville District	PM (local)	502-991-1103	Steven.kvaal@usace.army.mil
Kathryn Tait	OHARNG	Environmental Specialist	614-336-6136	Kathryn.s.tait.nfg@army.mil
Kevin Sedlak	ARNG	PM	614-336-6000 ext. 2053	Kevin.m.sedlak.ctr@mail.mil
Mark Leeper	NGB	PM	804-516-3529	Mark.s.leeper.civ@mail.mil
Alex Smith	HGL	PgM	240-586-1341	asmith@hgl.com
Kimberly Vaughn	HGL	PM	512-658-6828	kvaughn@hgl.com
Benjamin (BJ) Shivar	HGL	Deputy PM	704-248-1801	bshivar@hgl.com

The Kickoff Conference Call for the Former RVAAP began at 1000 Central. The meeting was guided by the attached presentation. Meeting topics and discussions are summarized below.

Introductions, Roles, and Responsibilities

Nicole Walworth started the meeting with introductions of the USACE staff. Travis McCoun began discussing background information on the project and adding that CENAB had enough historical information to develop the performance work statement (PWS) with sound assumptions. Mr. McCoun introduced Steven Kvaal as the local PM and Ms. Walworth as the CENAB PM. Mr. McCoun, as the COR, emphasized the Contracting Officer has overall task order authority to direct the contractor, and communications with the Contracting Officer need to be in writing. Mr. McCoun stated the customer for this project is the National Guard. Ms. Walworth stated that CENAB is putting together the PDT to include, but not limited to, an explosives expert and a project geophysicist. Kimberly Vaughn introduced herself as the PM, Alex Smith as the PgM, and Benjamin (BJ) Shivar as the Deputy PM. Ms. Vaughn discussed the lines of communication, as detailed on Slide #4, attached.

Project Management Plan and Quality Assurance Surveillance Plan (QASP)

Ms. Vaughn noted that the Project Management Plan is currently being written and is due to CENAB on or before 27 October 2021. Ms. Vaughn noted that the QASP in the PWS states that it was written in August 2021. Mr. McCoun mentioned it is a Draft QASP and will become Final after the task order award. Mr. McCoun noted the QASP is a living document and can be updated at any time.

Interim Milestone Payment Schedule (MPS)

Ms. Vaughn briefly discussed the MPS, which was submitted during the proposal phase. Mr. McCoun mentioned the less milestones, the better. Mr. Smith added that the MPS set up for this project is typical of other CENAB projects. Mr. McCoun requested a native file of the MPS for a more detailed analysis and review. *Note: Following this teleconference, HGL forwarded the Microsoft Excel file of the proposed milestones.*

Quality Monitoring and Invoicing

Ms. Vaughn summarized the quality monitoring, as described in Slide #8, and briefly mentioned invoicing on Slide #9, attached. Mr. McCoun mentioned that as a rule, no payment will be made without performing quality monitoring. In addition, backup on any invoice should be approved by the COR in advance, before sending the actual invoice. Mr. Smith recommended an 80 percent (%)/20% split on invoicing deliverables (between the Draft and Final versions), and Mr. McCoun concurred.

PWS and Conceptual Site Model (CSM)

Ms. Vaughn discussed the project objectives and how unlimited use/unrestricted exposure could be achieved through the proposed fieldwork plan. Ms. Vaughn further discussed the CSM for both Block D Igloo MRS and the Group 8 MRS. Mr. McCoun noted that the installation wanted to pursue No Further Action and discussed the meaning of Response Complete. He added that the goal was to achieve No Further Action where no land use controls are needed. Ms. Vaughn noted for the Group 8 MRS that based on remedial investigation results, lead and cadmium are the primary contaminants. She added that the initial round of sampling will be performed to define the boundaries for disposal. Mr. McCoun mentioned adding optional funds for various quantities of material to be removed. Ms. Vaughn noted the quantities will be based on soil laboratory analytical data results. Mr. McCoun instructed HGL to monitor soil quantities, and he would add funding, as needed.

Schedule

Ms. Vaughn stated that the current schedule is aggressive. Mr. McCoun was concerned about achievement of the accelerated schedule and added that the contract does have a 5-year period of performance. Mr. McCoun also noted potential delays including limited tree cutting due to nesting season (long-eared bats), long Ohio EPA document reviews, and that wetland delineation needs to occur first. Mr. McCoun wants to make sure HGL's schedule incorporates the necessary time to complete the project without sacrificing quality control. Mr. McCoun recommended staggering tree removal and other vegetation removal to prevent regrowth and erosion issues from developing if there is a delay before MEC removal begins. Lastly, Mr. McCoun noted that November and December are tough months in which to accomplish work due to the holiday season, and contractor and USACE staff availability.

Wetland Delineation

Mr. McCoun noted wetland delineation can occur concurrently with setting up the first Technical Project Planning (TPP) meeting. Ms. Vaughn and Mr. McCoun asked to confirm with the installation if wetland delineation could occur without a formal meeting with Ohio EPA. Ms. Tait agreed that it could be done without a formal meeting and reminded the group there is a potential drainage/wetlands area along the boundary of Group 8 MRS. Mr. McCoun noted that wetland delineation will need to occur at both sites. Ms. Tait confirmed she would accept one report to cover both sites. The scope of work included a report for each MRS, but Ms. Tait said one report is fine.

First TPP Meeting

Ms. Tait recommended a virtual TPP meeting, due to Ohio EPA current telework policies, and the team concurred. Mr. McCoun mentioned the first TPP meeting could occur the first week of December 2021. Mr. McCoun recommended an Army only meeting to go through TPP slides with the technical teams, ahead of the Ohio EPA meeting. Ms. Vaughn stated HGL will put TPP slides together in October or early November 2021 for the Army-only meeting. Mr. McCoun stated the slides will need to be made available to Ohio EPA and the customer 2 weeks prior to the first TPP meeting; additionally, planning for CENAB review time ahead of that deadline is required.

Miscellaneous

Mr. McCoun stated that during project execution, feel free to reach out to any PDT member, as needed, for support. Mr. McCoun emphasized that communication to Ohio EPA goes through the installation. Ms. Tait noted that requests for installation access and work hours need to be coordinated appropriately and expeditiously.

Action Items

OPEN ACTION ITEMS			
No.	**Item/Topic**	**Responsible Party/Expected Date of Resolution**	**Status**
1	HGL to provide native file of the milestone payments proposed.	Kimberly Vaughn – 10/8/2021	Completed.
2	Project Team to review Kickoff Meeting Minutes and provide concurrence.	Project Delivery Team	In Progress.
3	HGL to provide TPP slides for the Army-only meeting.	BJ Shivar – October/early November	In Progress.
4	HGL to provide updated project schedule for review.	PDT	Completed (included with these minutes).

WORKSHEET #10
CONCEPTUAL SITE MODEL

10.0 OVERVIEW

This worksheet is used to present a concise summary of the Group 8 MRS CSM prior to the implementation of the proposed RA: a working, iterative model that depicts the current understanding of sources, pathways, and receptors. The major elements of the CSM include the facility profile, physical profile, release profile, and land use and exposure profile.

10.1 FACILITY PROFILE

10.1.1 Site Location

CJAG is 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 (Figure 10.1). The Group 8 MRS is a 2.65-acre area just north of the southern boundary of CJAG (Figure 10.2). The MRS is located between Buildings 846 and 849 and lies southeast of Load Line #12, just north of the southern facility boundary. Figure 10.2 presents the current boundaries of the MRS.

10.1.2 Site Ownership

The CJAG facility comprises a 21,683-acre federal property that has been transferred to the U.S. Property and Fiscal Officer for Ohio, which subsequently licensed CJAG to OHARNG for use as a military training site.

10.1.3 Site History

RVAAP was constructed in 1940 and 1941 for the assembly/loading and depot storage of ammunition. While serving as an ammunition plant, RVAAP was a U.S. Government-owned and contractor-operated industrial facility. The ammunition plant consisted of 12 munitions assembly facilities, referred to as “load lines.” Load Lines 1 through 4 were used to melt and load 2,4,6-trinitrotoluene (TNT) and Composition B (a mixture of TNT and RDX) into large-caliber shells and bombs. Operations on the load lines produced explosive dust, spills, and vapors that collected on the floors and walls of each building. Periodically, the floors and walls were cleaned with water and steam. After cleaning, the “pink water” wastewater, which contained TNT and Composition B, was collected in concrete holding tanks, filtered, and pumped into unlined ditches for transport to earthen settling ponds. Load Lines 5 through 11 manufactured fuzes, primers, and boosters. From 1946 to 1949, Load Line 12 produced ammonium nitrate for explosives and fertilizers; subsequently, it was used as a weapons demilitarization facility.

In 1950, the facility was placed on standby status, and operations were limited to renovation, demilitarization, normal maintenance of equipment, and munitions storage. Production activities resumed from July 1954 to October 1957 and again from May 1968 to August 1972. Demilitarization and production activities were conducted at Load Lines 1, 2, 3, and 12. Demilitarization activities included disassembling munitions and melting out and recovering explosives using hot water and steam processes. These activities continued through 1992. In addition to production and demilitarization activities at the load lines, other activities conducted at RVAAP included the burning, demolition, and testing of munitions. The locations used as burning and demolition grounds consisted of large, open areas and abandoned quarries. Other areas of concern associated with RVAAP include a landfill, an aircraft fuel tank testing area, and various industrial support and maintenance facilities (CB&I, 2015).

The Group 8 MRS (formerly known as Area Between Building 846 and 849) was used to burn construction debris and rubbish for an unknown period. Before designation as an MRS, the area between Building 846 and 849 was used as a staging area for military vehicles. There are no records available documenting the disposal of munitions at the MRS; however, previous discoveries of MEC and of MD indicated that munitions may have been disposed of at the Group 8 MRS. There have been no CERCLA enforcement actions related to the Group 8 MRS.

10.1.4 Identification of Munitions and Hazardous Substances Known or Suspected to be Present

The Group 8 MRS was reportedly used for the open burning of debris trash for an undetermined amount of time, and as evidenced by the Remedial Investigation (RI) findings, the burning activities may have included munitions demilitarization. The 2007 Site Inspection (SI) recommended the MRS proceed to the RI phase because MEC had been identified historically at the MRS. However, the findings in the RI phase were inconsistent with the historical findings documented in the SI. The items documented in the SI are inconsistent with the types of MPPEH recovered during the RI intrusive investigation. Only MDAS (which does not pose an explosive hazard) was recovered during the RI. Consequently, the post-RI conclusion for the MRS is an incomplete exposure pathway for explosive hazards. Therefore, no further action was required for MEC and as a result, the Feasibility Study (FS) and the PP address the risks present due to MC-related contamination (lead and/or cadmium) in surface soil (0 to 0.5 ft bgs) only.

10.1.5 Summary of Previous Investigations

10.1.5.1 Archives Search Report

In 2004, USACE conducted an archives search under the Defense Environmental Restoration Program that included a historical records search, interview process, and site visit to search for the presence of MEC at the facility. USACE prepared the Final Archives Search Report (ASR) and therein identified 12 areas of concern and 4 additional locations with the potential for containing MEC. Notably, the Group 8 MRS was not identified as one of the original sites that contained MEC as part of the 2004 ASR (USACE, 2004).

10.1.5.2 Historical Records Review

The *Final Military Munitions Response Program Historical Records Review* (HRR) described the Group 8 MRS as the 2.65-acre "Area Between Buildings 846 and 849" and documented the requested name change to the Group 8 MRS. At the time of the HRR records research, OHARNG was using the area as a vehicle staging area. Historical activities at the MRS included burning construction debris and rubbish. The time frame for these activities is not known.

In 1996, MEC was found at the MRS, in the form of a single antipersonnel fragmentation bomb containing high explosives (HEs). MD was found in the form of a demilitarized (i.e., cut in half) 175-millimeter (mm) projectile. The antipersonnel fragmentation bomb with HE was removed and detonated at Open Demolition Area #2. The 175mm projectile (determined to be MD) was also removed from the MRS and was taken to Building 1501 (e2M, 2008).

10.1.5.3 2007 Site Inspection

In 2007, ARNG completed an MMRP SI at CJAG that included the Group 8 MRS. Magnetometer and metal detector-assisted UXO surveys were conducted during the SI field activities over 100% of the MRS. Two unidentifiable T-bar fuzes were found partially buried in the southwest portion of the MRS and were determined at that time to be MPPEH. Because the scope of the SI did not include any intrusive work or handling/disposal of MPPEH, the fuzes were left in place during the inspection; however, the items were not found during the subsequent RI and their final disposition is unknown. Because they were not inspected by UXO qualified personnel, it was conservatively assumed the fuzes contained explosive material and meet the definition of MEC.

MD items identified during the SI field activities included metal fragments from casings and projectiles, burster tubes, and fragments of fuzes. Most of the MD items found had most likely been pressed into the surface soils by the heavy equipment and vehicles that had been stored at the MRS before the SI. Figure 10.3 shows the locations of the SI survey lines and of the T-bar fuzes found during the SI field activities. In addition to the MPPEH and MD, a significant amount of non-munitions related debris consisting of metal trash, fence materials, and wood scraps were found in the general areas where the MPPEH and MD were found. No MPPEH or other debris was identified on the ground surface at the northeast portion of the MRS during the SI. Five surface soil samples were collected at the MRS during the SI field activities using Incremental Sampling Methodology (ISM). The samples were analyzed for explosives, propellants, and target analyte list metals. Lead and thallium were detected in all five samples above the facility screening criteria for background values and one-tenth of the U.S. Environmental Protection Agency (EPA) residential soil PRGs.

Thallium was dismissed as an MC as it was non-munitions related. Antimony, arsenic, aluminum, cadmium, copper, iron, lead, and manganese were detected in at least one sample at concentrations greater than the facility screening criteria and were considered MC. Explosives and propellants were detected at estimated concentrations (i.e., below the method detection limit [DL]); however, no concentrations exceeded one-tenth of the PRGs (e2M, 2008).

10.1.5.4 2011 Remedial Investigation

Between October 31, 2011, and November 14, 2011, CB&I Federal Services, LLC performed a DGM investigation to identify potential subsurface areas of MEC at the Group 8 MRS. Full coverage DGM data was collected on 2.563 acres at the Group 8 MRS. Data were acquired in all accessible areas of the MRS on a line spacing of approximately 2.5 ft. The area surveyed equates to nearly 97% coverage over the 2.65-acre MRS. A small portion could not be investigated because of trees, utility poles, and barbed wire fence.

A total of 264 anomalies were reacquired during the intrusive investigation of 14 trenches within the three areas of high anomaly density. From these intrusive investigations, 359 individual items of MPPEH that weighed approximately 1,418 pounds were recovered from a maximum depth of 4 ft bgs. The maximum depth of MPPEH was established in the RI to be no deeper than 4 ft bgs (CB&I, 2015). Once the item was determined not to be munitions related, it was temporarily removed from the excavation and the Schonstedt magnetometer was used to confirm no additional ferrous items were located beneath the first item. The investigation criteria for trenching were to excavate at a location until the target anomalies were identified; native material was identified and a clear, distinct boundary between the native and fill material was evident; a maximum depth of 10 ft was attained; or the water table was reached. The maximum depth that any of the trenches at the MRS were excavated to was 4 ft bgs, which is the maximum depth that native soils were encountered. Approximately 1,180 pounds (lbs) (277 individual MD items) of MD items were recovered from 9 trenches and 1,281 lbs of "Other Debris" were identified within all 14 trenches (CB&I, 2015). The UXO-qualified personnel documented that all these items were safe and classified them as MDAS (no MEC was identified). The locations of the items and other debris are shown in Figure 10.4. The MDAS was debris that came from the following munitions: M397 series 40mm HE grenades, M49 series 60mm mortars, 20mm projectiles, M72 series 75mm projectiles, M557 series fuzes, 175mm projectiles, HE anti-tank warheads, and assorted fuzes (CB&I, 2015). The MDAS items were solid and/or inert and did not pose an explosive safety hazard.

The MDAS identified during the RI was not consistent with the historically identified MDAS from the HRR and SI. The explosive hazards were re-evaluated during preparation of the FS. Sampling for MC-related contamination was conducted during the RI field work. Four samples were collected using ISM from sampling units of the same size for the entire MRS at depths between 0 and 0.5 ft bgs. Additional samples were proposed in areas with concentrated MD, and three additional ISM soil samples were collected from the bottom of three trenches. All trenches were excavated until native soils were encountered. The three ISM soil samples were collected at depths of 4 to 4.5 ft bgs, below the maximum depth of 4 ft bgs where MD was encountered during the intrusive investigation activities. The trench samples were evaluated/considered as subsurface samples in the human health and ecological risk assessments. Concentrations of cadmium, iron, lead, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, dibenzo(a,h)anthracene, Aroclor-1254, and Aroclor-1260 were detected in the surface soil samples and were carried forward for evaluation in the Human Health Risk Assessment. The following 10 chemicals of potential ecological concern (COPECs) were recommended for evaluation under the Level III Baseline Ecological Risk Assessment evaluation after the Level II Screening in the RI: antimony, cadmium, copper, lead, mercury, zinc, bis(2-ethylhexly)phthalate, di-n-butyl phthalate, Aroclor-1254, and Aroclor-1260. The RI Report concluded that no contaminants in subsurface soil were present at concentrations that pose a risk to either human or ecological receptors. Based on the historical discoveries of MEC, the MRS was assigned an MRS Prioritization Protocol (MRSPP) priority of 4 during the RI. A Priority 1 MRS contains the highest potential hazard, while a Priority 8 MRS contains the lowest potential hazard (HGL, 2020).

10.1.5.5 2019 Feasibility Study

The *Final Feasibility Study for RVAAP-063-R-01 Group 8 Munitions Response Site* was prepared for the Group 8 MRS by ARNG in 2019. The FS evaluated possible alternatives in detail and provided a comparative analysis of those alternatives, based on criteria outlined in the National Oil and Hazardous Substances Pollution Contingency Plan. The FS identified three possible alternatives to address the MC contamination at the Group 8 MRS. The alternatives consisted of (1) No Action, (2) Land Use Controls, and (3) MC-Contaminated Soil Removal (to achieve use of land for UU/UE). The FS also developed the RA objectives based on the potential for MC-related contamination at the Group 8 MRS (HGL, 2019). The FS included a Risk Management Evaluation based on the previously collected RI data. The 2015 RI used the Facility-Wide Human Health Cleanup Goals (SAIC, 2010) in the risk assessment prepared at that time. However, the EPA Regional Screening Levels (RSLs) can be used to reflect current toxicity information and exposure assumptions, as provided in the Technical Memorandum (ARNG, 2014). The Risk Management Evaluation (RME) was therefore performed in the FS to re-assess the contaminants of concern (COCs) using the RSLs, which are protective of the Resident Receptor. The COCs identified for the Resident Receptor in the RI report were re-evaluated with respect to the current Residential Soil RSLs. The RI concluded that no MC-related contamination was present in subsurface soil; therefore, the FS RME evaluated surface soil data collected in the RI. The FS concluded that cadmium in surface soil at the location of sample GR8SS-004M-0001-SO poses a risk to the theoretical future Resident Receptor (Adult and Child). Following the FS, the PP summarizes additional excavation to remove lead in surface soil (see paragraph E.2.6, below). Soil contaminants do not pose a risk to the Industrial Receptor, as the representative receptor under current site use. Additional detail summarizing the MC contamination present at the MRS is shown on Figure 10.5. The RME also re-assessed the ecological risk that was presented in the RI. Because the habitat quality is poor, few birds and mammals forage for food on the MRS. Consequently, the RI overestimated the food and soil ingestion rates and potential exposure to animals. The FS concluded that it is unlikely that site contaminants pose a risk to wildlife communities. The FS established cadmium in surface soil as the MC-related contamination present at the MRS.

The MEC items found in 1996 and 2007 were not consistent with the types of MDAS identified during the RI. No MEC has otherwise been reported since 2007 at the Group 8 MRS and the RI concluded with a 99% confidence level that no MEC are present. Only MDAS (which does not pose an explosive hazard) was recovered during the RI. The MRS has an incomplete exposure pathway for explosive hazards and no further action was recommended for MEC. Therefore, the MEC exposure pathway was re-evaluated during the FS phase and no explosive hazard is anticipated at the Group 8 MRS. The MEC exposure pathway is considered incomplete. The MRSPP priority was also re-evaluated during the FS phase. The project team determined that the MEC items identified historically (during the site inspection) are not representative of the explosive hazards present at the MRS, as demonstrated by the findings during the RI fieldwork and the conclusions of the RI. As a result, the MRSPP was revised, and the Group 8 MRS was assigned a score of 5 during the FS phase.

10.1.5.6 2020 Proposed Plan

In 2020, ARNG completed the PP (HGL, 2020) for the Group 8 MRS. The PP documented the preferred alternative (*Alternative 3 – MC-Contaminated Soil Removal*) to address MC contamination. The PP summarized the determination that no further action was required for MEC, and the FS and the PP both addressed risks present due to MC-related contamination. Following the FS, as documented in the PP, ARNG and Ohio EPA agreed to include additional excavation to remove lead in surface soil at GR8SS-001M and GR8SS-003M to ensure maximum protectiveness is achieved to meet Unrestricted (Residential) Land Use. The concentrations of soil contaminants (COCs) do not pose risks to the Industrial Receptor, which is the Representative Receptor under current site use. Therefore, the PP recommended remediation of the lead and cadmium contamination in GR8SS-004M and the lead contamination in GR8SS-001M and GR8SS-003M (Figure 10.5) to eliminate potential risks to human health under Unrestricted (Residential) Land Use, for a theoretical future Resident Receptor. Remediation of the lead and cadmium contamination in the Group 8 MRS will eliminate potential risks to human health under Unrestricted (Residential) Land Use. The PP established lead and cadmium as the MC-related contamination present in surface soil at the MRS.

10.1.5.7 2020 Record of Decision

In 2020, ARNG finalized the ROD for the selected remedy for the Group 8 MRS, and it was signed on 31 December 2020 (HGL, 2020). The selected remedy will apply throughout the remedial footprint, which is the MRS boundary (Figure 10.5). The theoretical future Resident Receptor (Adult and Child) was evaluated for this MRS. The RA objectives, as summarized from Section H of the ROD, state the following:

- Reduce the unacceptable potential hazard of DoD military munitions on the ground surface and in sediment at the saturated and surface water areas within the MRS to address the likelihood of exposure to the Industrial Receptor via direct contact such that the likelihood of encounter is negligible.
- Reduce the unacceptable potential hazard of DoD military munitions to a depth of 4 ft bgs within the MRS to address the likelihood of exposure to the Industrial Receptor via direct contact such that the likelihood of encounter is negligible.

Information on the conditions under which an acceptable end state will be considered to have been achieved is included in WS 11, Step 2.

10.2 PHYSICAL PROFILE

10.2.1 Topography and Physiography

CJAG is located within the Southern New York section of the Appalachian Plateaus physiographic province, which is characterized by rolling hills, incised streams, and dendritic drainage patterns. Topography at the Group 8 MRS is flat, and the ground surface elevation is approximately 985 ft above mean sea level (amsl) (Figure 10.6). There are no natural streams or ponds located within the MRS, and the MRS is not located within a flood plain. No bogs, kettle lakes, or kames are present at the MRS.

10.2.2 Geology and Soils

The Group 8 MRS is located over the Sharon Member conglomerate unit. The bedrock elevation is approximately 975 ft amsl. The soils identified at the facility are generally derived from the Wisconsin-age silty clay glacial till. The majority of native soil at the facility has been reworked or removed during construction activities. The major soil types found in the Group 8 MRS are silt or clay loams, ranging in permeability from 6.0 × 10-7 to 1.4 × 10-3 centimeters per second (U.S.

Department of Agriculture et al., 1978). The soil type at the Group 8 MRS is the Mahoning-Urban land complex with undulating 2 to 6% slopes (Figure 10.7).

10.2.3 Surface Water

CJAG is located within the Ohio River Basin with a major surface stream running adjacent to the western portion of the facility flowing to the Mahoning River before joining the Michael J. Kirwan Reservoir. After leaving the reservoir, the west branch joins the Mahoning River east of the facility. Surface water drainage for the Group 8 MRS generally flows into drainage ditches along the roadside where it eventually infiltrates the soil. No wetlands are present within the Group 8 MRS. The standing water in Group 8 MRS does not connect to jurisdictional waters. The small ditch (standing water) is present on the southeast corner of the MRS (Figure 10.2).

10.2.4 Hydrogeology

Although groundwater recharge and discharge areas have not been delineated at CJAG, it is assumed that the extensive uplands areas at the facility, primarily located at the western portion of the facility, are regional recharge zones. Sand Creek, Hinkley Creek, and Eagle Creek are presumed to be major groundwater discharge areas.

Based on the data collected at CJAG under the Facility-Wide Groundwater Monitoring Program, the groundwater elevation at the MRS and the immediate vicinity is approximated at a potentiometric high of 960 ft amsl (HGL, 2020). Groundwater flow direction is toward the southeast. The approximate depth to groundwater in the unconsolidated aquifer at the Group 8 MRS is 15 to 20 ft bgs (CB&I, 2015).

10.2.5 Ecology

CJAG is home to a range of vegetation and habitat resources. The habitat at the Group 8 MRS has been influenced and impacted by man-made improvements, including gravel roads. Additionally, historical use of the Group 8 MRS as a burning area has influenced the habitat at the site. The vegetation community present at the Group 8 MRS is categorized as “other land,” in the Anderson Classification, which refers to disturbed areas and/or paved areas lacking identifiable vegetation communities (CB&I, 2015).

The CJAG Integrated Natural Resources Management Plan indicates that one federally listed threatened species, the Northern Long-eared Bat, is known to reside within CJAG (OHARNG, 2014). Additionally, the Ohio Department of Natural Resources (ODNR) has identified several state-listed threatened and endangered plant and animal species. Twelve state listed endangered species (1 mammal, 1 fish, 1 insect, 8 bird, and 1 plant species) and 10 state listed threatened species (5 bird, 1 insect, 4 plants species) are included on the Camp Ravenna Rare Species List. No confirmed sightings of these species within the Group 8 MRS have been reported, and no critical habitats are present within the MRS (CB&I, 2015) (OHARNG, 2014). No unacceptable ecological risk was identified at the Group 8 MRS; therefore, the selected remedy does not address ecological receptors.

10.3 RELEASE PROFILE

The RI Report (CB&I, 2015), as supplemented by the FS and PP evaluations, concluded that MEC was not present at the Group 8 MRS. MC-related contamination only is present at the MRS. Data gathered by ARNG during the SI and subsequent RI for the Group 8 MRS effectively characterized the nature and extent of MC-related contamination at the MRS. Soil samples were collected for the evaluation of MC-related contamination during the RI at areas with concentrated surface and subsurface MD. Soil samples consisted of surface soil ISM samples (collected from 0 to 0.5 ft bgs) and subsurface soil ISM samples (collected from 4 to 4.5 ft bgs). The RI concluded that no MC-related contamination was present in subsurface soil. The additional evaluations provided in the FS and PP concluded that lead and cadmium are the MC-related contamination present in surface soil (0 to 0.5 ft bgs) at the Group 8 MRS.

10.4 LAND USE AND EXPOSURE PROFILE

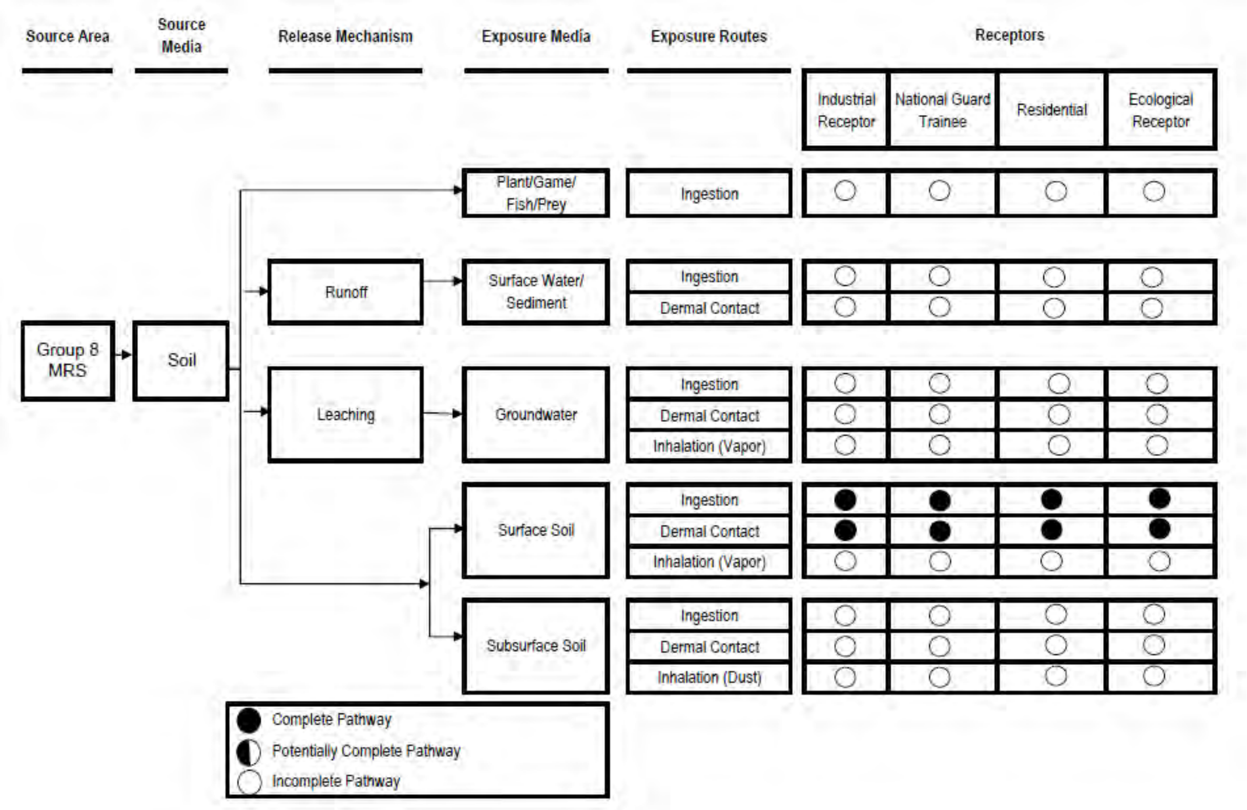
10.4.1 Current and Reasonably Anticipated Future Site Uses

The current land use activities at the MRS are maintenance, natural resource management, sampling, and an access route to adjacent buildings through the existing road network. The MRS is not currently used for military training, but military training is a potential future land use; therefore, the future land use activities at the MRS include maintenance, natural resource management, environmental sampling, and military training.

10.4.2 Current and Reasonably Anticipated Future Receptors and Exposure Pathways

The RI report identified the National Guard Trainee as the Representative Receptor based on the potential future land use of military training. The FS evaluated the Industrial Receptor and the National Guard Trainee as potential receptors because they best reflect current land use and are representative of potential future land use. The primary media of concern for the Industrial Receptor include surface and subsurface soils to a maximum exposure depth of 4 ft bgs. Both the National Guard Trainee and the Industrial Receptor were evaluated as potential receptors for MC risk in the surface soils at the Group 8 MRS, and no risk was identified. However, as summarized in Section E.4.2 of the ROD, an unacceptable risk to a theoretical future Resident Receptor (Adult and Child) was identified from MC-related contamination from 0 to 0.5 ft bgs, when the RME was conducted using the RSLs per the Technical Memorandum (ARNG, 2014). Complete and incomplete exposure pathways for MC are summarized in Figure 10.8. Based on the findings in the RI (CB&I, 2015), a complete exposure pathway does not exist for DoD military munitions verified as MEC in surface and subsurface soils at the Group 8 MRS.

Figure 10.8 Conceptual Site Model



Soil samples were collected during the RI for analysis for MC-related contamination at seven locations. Four surface soil sampling locations were randomly selected in a grid to represent the MRS as part of the ISM. Three subsurface locations were chosen in areas with high-density geomagnetic anomalies. The RI confirmed that unacceptable risk associated with MC-related contamination in surface soil do exist at the Group 8 MRS. In accordance with current guidance, humans are typically considered as the primary, and often the only, receptor to DoD military munitions; therefore, no ecological receptors are identified for the MRS (HGL, 2020).

10.4.3 Access Conditions and Frequency of Use

The MRS is located between Buildings 846 and 849 and lies southeast of Load Line #12, just north of the southern facility boundary. Figure 10.2 presents the current boundaries of the MRS. Buildings in the area are either vacant or used to store military equipment. The MRS is currently restricted access with only vehicles accessing the thru roads on an occasional basis. Once the remedial action is completed demolition of surrounding buildings will occur and the MRS will be used for military training.

WORKSHEET #10 (CONTINUED)
CONCEPTUAL SITE MODEL

Table 10.1 Overview of Conceptual Site Model, Group 8 MRS (RVAAP-063-R-01)

Site Details	Known or Suspected Contamination Source	Potential/Suspected Location and Distribution	Source or Exposure Medium	Current and Future Receptors	Complete Exposure Pathway
Name: Group 8 MRS (RVAAP-063-R-01) **Acreage:** 2.65 **Suspected Past DoD Activities (Release Mechanism):** The Group 8 MRS (formerly known as Area Between Building 846 and 849) was used to burn construction debris and rubbish for an unknown period. Before designation as an MRS, the area between Building 846 and 849 was used as a staging area for military vehicles. There are no records available documenting the disposal of munitions at the MRS; however, previous discoveries of MEC and MD indicated that munitions may have been disposed of at the Group 8 MRS.	The 2015 RI Report, as supplemented by the FS, PP, and ROD, concluded that MEC was not present at the Group 8 MRS. The 2020 ROD established that lead and cadmium are the MC-related contaminants present in surface soil (0 to 0.5 ft bgs).	The horizontal extent of metals contamination associated with unacceptable risk is limited to quadrants GR8SS-001M and GR8SS-003M (lead), and GR8SS-004M (lead and cadmium). The vertical extent of contamination is limited to surface soil (0 to 0.5 ft bgs) in the above quadrants.	Hypothetical Future Resident Receptor (adult and child): lead and cadmium in surface soil (0 to 0.5 ft bgs)	Current: Industrial Receptor Future: Hypothetical Resident (Adult/ Child) Receptor	Exposure by the Hypothetical Resident (Adult/Child) Receptor to cadmium and/or lead contamination in surface soil (0 to 0.5 ft bgs) within the MRS.

WORKSHEET #11
DATA QUALITY OBJECTIVES

11.0 INTRODUCTION

This worksheet documents data quality objectives (DQOs) for MPPEH and MC, which are developed during project planning sessions using a systematic planning process. The process described below is based on EPA's seven-step DQO process as applied to the Group 8 MRS RA.

11.1 MPPEH DQOs

This section presents the DQOs associated with the RA field activities in support of UXO avoidance. UXO avoidance will be performed to ensure the potential for MPPEH contamination in soil is evaluated during the MC contaminated soil excavation activities required to accomplish the remedy for MC contamination.

11.1.1 Step 1: State the Problem

It was determined in the RI that MEC is not present at the Group 8 MRS; however, MPPEH may be present. The maximum depth of MPPEH was established in the RI to be no deeper than 4 ft bgs (CB&I, 2015). The RA was then finalized in the FS, PP and ROD (HGL, 2019; HGL, 2020; and USACE, 2020).

11.1.2 Step 2: Identify the Goals of the Project

The following project objectives apply to the RA data collection for MPPEH avoidance during the Group 8 MRS RA:

1) Provide UXO construction support (UXO avoidance) during the **Pre-Excavation Characterization Soil Sampling** activity and identify and inspect all MPPEH identified on the surface.
2) Perform a visual investigation of all areas where it is determined that MC contaminated soils exist and will be excavated. Remove and inspect all MPPEH identified during the excavation of soils prior to **Pre-Excavation Waste Characterization Soil Sampling**.

11.1.3 Step 3: Identify Information Inputs

This step identifies the types of information inputs associated with each project objective.

Projective Objective 1 – UXO construction support (UXO avoidance) during the ***Pre-Excavation Characterization Soil Sampling.*** Information inputs will include the results of MPPEH discoveries and visual inspections to determine if MEC is present on the surface.

Project Objective 2 – Visual investigation of all areas where it is determined that MC contaminated soils exist during the ***Pre-Excavation Waste Characterization Soil Sampling.*** Information inputs will include results of MPPEH discoveries and visual inspections to determine if MEC is present in the subsurface. UXO construction support (UXO avoidance) will also ensure that no MPPEH is transported offsite while commingled with excavated soils.

11.1.4 Step 4: Define the Boundaries of the Project

This step describes the spatial and temporal boundaries for data collection associated with each of the project objectives.

Projective Objective 1 **–** UXO construction support (UXO avoidance) during the ***Pre-Excavation Characterization Soil Sampling.*** The horizontal spatial boundaries are represented by the overall MRS boundary and specifically the boundaries of quadrants GR8SS-001M, GR8SS-003M, and GR8SS-004M. UXO construction support (UXO avoidance) will be performed within all MC sampling locations collected during sampling of the grid-based locations in each of the quadrants being sampled.

Project Objective 2 **–** Visual inspection of excavations and sampling investigation of the areas where it is determined that MC contaminated soils exist during the ***Pre-Excavation Waste Characterization Soil Sampling.*** Horizontal and vertical spatial boundaries will apply to waste characterization soil sampling and will be based on the final excavation boundaries concurred to by the project team.

11.1.5 Step 5: Develop the Analytic Approach

All MPPEH discovered will be inspected for final determination as MEC or MDAS. If an item is determined to be MEC it will be properly documented and disposed of in accordance with the approved guidance, site plans and applicable Standard Operating Procedures (SOPs). **It should be noted that if MEC is identified the field activities may be halted while the CSM (WS #10) is revised to account for MEC in the Group 8 MRS.** All MDAS will be certified, verified, containerized, and properly disposed/recycled.

11.1.6 Step 6: Specify Performance or Acceptance Criteria

Project-specific measurement performance criteria (MPCs) are presented in WS #12. All identified MPPEH will be properly classified and handled accordingly. HGL UXO technicians will perform the UXO avoidance activities to meet the MPCs and SOPs that are summarized in WS #12.

11.1.7 Step 7: Develop the Detailed Plan for Obtaining Data

This step describes the detailed MPPEH removal, including the decision logic to be applied during results evaluation. Additional details associated with this activity are provided in WSs #17, DFWs.

MPPEH location, ID, and removal will be performed visually and using instrument aided detection during the **Project Objectives 1 and 2** activities as discussed above. DDESB TP-18 qualified UXO personnel will support the ***Pre-Excavation Characterization Soil Sampling***, to ensure that all environmental samples are collected to avoid subsurface metallic responses. DDESB TP-18 qualified UXO personnel will support the ***Pre-Excavation Waste Characterization Soil Sampling*** by operating the earth moving machinery used for the soil excavation so that visual observations of MPPEH can be determined during excavations. Prior to and during soil excavations DDESB TP-18 qualified personnel will perform instrument inspections and removal of MPPEH at the areas where excavations are planned to occur. See additional task-specific details included in WS #17.

11.2 MC DQOs

This section presents the DQOs associated with the characterization and remediation of MC contamination in soil and associated activities.

11.2.1 Step 1: State the Problem

MC-related contamination only is present at the MRS. Data gathered by ARNG during the SI and subsequent RI for the Group 8 MRS effectively characterized the nature and extent of MC-related contamination at the MRS. Soil samples were collected for the evaluation of MC-related contamination during the RI at areas with concentrated surface and subsurface MD. Soil samples consisted of surface soil ISM samples (collected from 0 to 0.5 ft bgs) and subsurface soil ISM samples (collected from 4 to 4.5 ft bgs). The RI concluded that no MC-related contamination was present in subsurface soil. The additional evaluations provided in the FS and PP concluded that lead and cadmium are the MC-related contamination present in surface soil (0 to 0.5 ft bgs) at the Group 8 MRS. The RI Report (CB&I, 2015), as supplemented by the FS and PP evaluations, concluded that MEC was not present at the Group 8 MRS.

The ROD identified the selected remedial alternative to address cadmium and lead soil contamination and consists of the following:

- Removal of lead- and cadmium-contaminated soil to a depth of 0.5 ft bgs;
- Incidental to the surface soil removal, if any MD is encountered, it will be segregated, inspected, and certified as MDAS before disposal;
- Confirmation soil samples will be collected to confirm that the extent of MC-contaminated soil has been removed; and
- Under this alternative, all MC-contaminated soil exceeding the PRGs will be removed, allowing for UU/UE at the MRS (although future residential use is not anticipated and is theoretical only).

Pre-excavation characterization soil sampling is required to further delineate the horizontal and vertical extent of cadmium and lead contamination within three quadrants subject to soil excavation including: GR8SS-001M and GR8SS-003M (lead) and GR8SS-004M (cadmium and lead) (Figure 11.1). The ISM soil sample results from the RI apply to the three overall areas and do not allow for further definition of the horizontal extent of contamination within these areas. Additionally, the ISM sampling was conducted only within the 0 to 0.5 ft bgs and 4 to 4.5 ft bgs depth intervals; therefore, no results are available between these intervals.

Pre-excavation characterization soil sampling will support the development of the necessary excavation boundaries. Pre-excavation waste disposal characterization sampling also will be conducted within the applicable excavation areas to support the profiling, transportation, and off-site disposal of excavated soil.

11.2.2 Step 2: Identify the Goals of the Project

The following project objectives apply to the field data collection program for the Group 8 MRS RA:

1) **Pre-Excavation Characterization Soil Sampling** – Confirm the horizontal and vertical extent of cadmium and lead contamination in surface soil to support the development of excavation boundaries.
2) **Pre-Excavation Waste Characterization Soil Sampling** – Characterize soil to be excavated for management, transportation, and off-site disposal at an appropriately licensed facility.
3) **Confirmation Soil Sampling** – Confirm complete removal of cadmium and lead contamination both vertically and horizontally to support UU/UE at the MRS.
4) **Investigation-Derived Waste (IDW) Water Characterization** – Characterize IDW water generated during equipment decontamination, and possibly excavation dewatering, for management, transportation, and off-site disposal at an appropriately licensed facility.
5) **Backfill Soil Characterization** – Characterize excavation backfill soil to confirm clean and suitable for use.

The remaining steps of the DQO process will address each of the project objectives listed above separately.

11.2.3 Step 3: Identify Information Inputs

This step identifies the types of information inputs associated with each project objective.

Projective Objective 1 – Pre-Excavation Characterization Soil Sampling. Information inputs will include the results of pre-excavation soil sampling to further define the horizontal and vertical extent of cadmium and lead contamination in surface soil within quadrants GR8SS-001M and GR8SS-003M (lead) and GR8SS-004M (cadmium and lead).

Project Objective 2 – Pre-Excavation Waste Characterization Soil Sampling. Information inputs will include results of pre-excavation waste characterization soil sampling to support the management, transportation, and off-site disposal of excavated soil. One composite soil sample will be collected from areas equivalent to five roll-offs. Based on the limited site history and process knowledge available, pre-excavation waste characterization samples will be analyzed for the full list of parameters listed in WS #15 and described in the discussion for Step 5 in Section 11.2.5. Excavation boundaries and the number of waste characterization samples will be developed based on the results of pre-excavation soil characterization sampling associated with Project Objective 1 and submitted for review and stakeholder concurrence in an Excavation Plan.

Project Objective 3 – Confirmation Soil Sampling. Confirmation sampling from sidewalls along the MRS boundary and excavations made to 2.0 ft bgs (representing the maximum depth of pre-excavation characterization sampling) in quadrants GR8SS-001M and GR8SS-003M (lead) and GR8SS-004M (cadmium and lead).

Project Objective 4 – IDW Water Characterization. Information inputs will include results of IDW water characterization sampling to support management, transportation, and off-site disposal. Samples will be collected as needed to characterize equipment decontamination waste water generated during soil sampling and excavation activities. It is also possible that additional IDW water could be generated due to excavation dewatering, if necessary.

Project Objective 5 – Backfill Soil Characterization. Information inputs will include results of backfill soil sampling to support its use as clean excavation backfill. One composite soil sample will be collected from the backfill source per 4,000 cubic yards (cy) of material required based on the planned excavation area boundaries. If existing laboratory analytical results are available for the backfill material, then the results will be reviewed to confirm they are of definitive data quality and meet the requirements for government approval (in lieu of conducting characterization sampling).

11.2.4 Step 4: Define the Boundaries of the Project

This step describes the spatial and temporal boundaries for data collection associated with each of the project objectives.

Projective Objective 1 – Pre-Excavation Characterization Soil Sampling. The horizontal spatial boundaries are represented by the overall MRS boundary and the specific boundaries of quadrants GR8SS-001M, GR8SS-003M, and GR8SS-004M. Pre-excavation soil samples will be collected from grid-based locations in each of the quadrants (Figure 11.2). The vertical spatial boundary for the initial pre-excavation soil sampling for cadmium and lead is 2 ft bgs, based on the planned sample depth intervals at each grid-based location (0.5-1, 1-1.5, and 1.5-2 ft bgs). Since the minimum excavation depth is 0.5 ft bgs, sampling will not be needed from this interval.

Samples from the 0.5-1.0 ft increments will be immediately analyzed for metals of concern. The remainder of the collected characterization samples will be held at the laboratory under preservation conditions. Should results for any grid-based location exceed a project action limit (PAL), the next deepest increment will be analyzed. Samples resulting in concentrations below PALs confirm the vertical limits of contamination.

Project Objective 2 – Pre-Excavation Waste Characterization Soil Sampling. Horizontal and vertical spatial boundaries will apply to waste characterization soil sampling and will be based on the final excavation boundaries. The soil sampling results from Project Objective 1 will be used to finalize the necessary excavation boundaries within quadrants GR8SS-001M, GR8SS-003M, and GR8SS-004M. One composite soil sample will be collected from each distinct excavation area to be representative of the entire excavated soil volume from each area.

The temporal boundary of collecting these samples prior to excavation will eliminate the need for sampling excavated soil stockpiles and the associated time required to complete sample analysis and manage the stockpiles. Pre-excavation waste characterization also will confirm waste management requirements associated with excavated soil prior to mobilization.

Project Objective 3 – Confirmation Soil Sampling. In the event that all pre-excavation samples including the 1.5–2.0 ft bgs increment exceed PALs (Project Action Limits) for a given grid location, that grid area will be excavated to 2.0 ft bgs. A separate confirmation sampling effort, following excavation will be performed for the excavation floor at those sample locations to collect and analyze for metals of concern at the 2.0-2.5-ft bgs increment. Should confirmation samples result in a PAL exceedance, the next six-inch depth increment will also be collected for analysis.

In addition, sidewalls along the outer excavation boundary will be collected at a frequency of one per 20 feet (equivalent to a grid length). Exceedances of PALs from sidewall samples indicate soil contamination beyond the limits of the MRS and will not result in immediate follow-up sampling.

Project Objective 4 – IDW Water Characterization. IDW water generated during soil sampling and excavation activities, and possibly excavation dewatering, will require characterization to support management, transportation, and disposal. The actual number of samples will depend on the volume of water generated, timing of field activities, etc.

A temporal boundary will apply to IDW characterization that will require samples to be collected as soon as practicable at the end of activities that generate waste water. It is also possible that additional IDW water could be generated due to excavation dewatering, if necessary.

Project Objective 5 – Backfill Soil Characterization. Characterization samples will be collected from the backfill and topsoil material at the on-site or off-site source. The actual number of samples will depend on the total volume of backfill soil required for the planned excavation areas.

A temporal boundary will apply to backfill soil sampling, as these samples will be collected far enough in advance of soil excavation activities to allow for laboratory analysis, review of results, and government approval.

11.2.5 Step 5: Develop the Analytic Approach

This step describes the analytic approach to be applied to each type of sampling conducted during field activities. All laboratory analyses will be conducted by SGS North America located in Orlando, Florida. Current SGS Orlando DoD Environmental Laboratory Accreditation Program (ELAP) certifications for planned analytical methods are provided in Appendix F.

Projective Objective 1 – Pre-Excavation Characterization Soil Sampling. Discrete pre-excavation soil samples collected from grid-based locations within each quadrant (Figure 11.2) including GR8SS-001M and GR8SS-003M will be analyzed for lead, and GR8SS-004M will be analyzed for cadmium and lead using method SW-846 6010C. A set of the soil samples will be analyzed by default, with remaining samples held by the laboratory for potential analysis within the applicable hold time (6 months from collection). A detailed description of the decision logic applied to held samples is provided in Step 7 below.

Project Objective 2 – Pre-Excavation Waste Characterization Soil Sampling. Each soil sample collected for pre-excavation waste characterization will be analyzed for the following Resource Conservation and Recovery Act (RCRA) parameters:

- TCLP SVOCs
- TCLP volatile organic compounds (VOCs)
- TCLP herbicides and pesticides
- TCLP metals

- PCBs
- Total sulfides
- Total cyanide
- Nitrate and nitrite
- Paint Filter Test
- Corrosivity (pH)
- Ignitability (flash point)

Project Objective 3 – Confirmation Soil Sampling. Confirmation samples collected from outer sidewalls and from the excavation floor will be analyzed for lead in quadrants GR8SS-001M and GR8SS-003M. Confirmation samples from quadrant GR8SS-004M will be analyzed for lead and cadmium.

Project Objective 4 – IDW Water Characterization. Each water sample collected for waste characterization will be analyzed depending on its provenance. Water extracted from the excavation would be analyzed for the project's COCs plus any other parameter requested by the disposal facility. Water resulting from equipment decontamination would be analyzed for the full suite of waste characterization RCRA parameters as listed for soil waste characterization above, in coordination with the disposal facility requirements.

Project Objective 5 – Backfill Soil Characterization. Soil samples from the backfill and topsoil sources will be analyzed for the following parameters:

- VOCs
- SVOCs
- Pesticides
- Herbicides
- PCBs
- Explosives
- Nitroglycerin
- Nitroguanidine
- Nitrocellulose
- Target analyte list metals
- pH

The backfill characterization parameters are consistent with those provided in the *Final Remedial Design Work Plan for RVAAP Load Lines 1, 2, 3, 4, and 12 (RVAAP-08 to 12)* (SAIC, 2020). Metals results will be compared to facility-specific background values from the *Final Phase II Remedial Investigation Report for Winklepeck Burning Grounds* (SAIC, 2001) and provided in Table 11.1. The project-specific cleanup goals for cadmium and lead supersede the previously established background concentrations. In addition, a geotechnical analysis will be performed on the backfill to evaluate the stability and structural integrity and prevent the risk of subsidence. Grain size, soil type, permeability, and compaction will be tested per the ASTM methods listed in Table 15.11.

11.2.6 Step 6: Specify Performance or Acceptance Criteria

Project-specific MPCs are presented in WS #12, and the PALs and laboratory specific detection/quantitation limits are presented in WS #15. Project-specific MPCs and PALs are the criteria that collected data must meet to satisfy the DQOs. Failure to achieve the MPCs may have an impact on the intended uses of the data, which will be discussed in the Data Usability Assessment Report described on WS #37.

Projective Objective 1 – Pre-Excavation Characterization Soil Sampling. The PALs for cadmium and lead are 71 milligrams per kilogram (mg/kg) and 400 mg/kg, respectively (WS #15.1). Soil sample results will be subject to data verification and validation per the requirements of WS #35 and WS #36.

Project Objective 2 – Pre-Excavation Waste Characterization Soil Sampling. The PALs for waste characterization parameters are based on RCRA regulatory limits (WS #15.11). Waste characterization sampling results are not subject to data validation; however, they will be subject to data verification to confirm completeness.

Projective Objective 3 – Confirmation Soil Sampling. The PALs for cadmium and lead are 71 mg/kg and 400 mg/kg, respectively (WS #15.1). Soil sample results will be subject to data verification and validation per the requirements of WS #35 and WS #36.

Project Objective 4 – IDW Water Characterization. The PALs for waste characterization parameters are based on RCRA regulatory limits (WS #15.11). Waste characterization sampling results are not subject to data validation; however, they will be subject to data verification to confirm completeness.

Project Objective 5 – Backfill Soil Characterization. The PALs for backfill and topsoil characterization are listed in WS 15.2 through 15.11. Backfill soil will be compared to PALs derived from EPA residential soil RSLs, or from site-specific background concentrations (Table 11.1), whichever is greater. A geotechnical analysis will be performed on the backfill (not topsoil) per Table 15.11 to evaluate the expected structural integrity of the final graded site. Soil sample results will be subject to data verification and validation per the requirements of WS #35 and WS #36. If existing laboratory analytical results are available for the backfill material, then the results will be reviewed to confirm they are of definitive data quality and meet the requirements for government approval (in lieu of conducting characterization sampling).

11.2.7 Step 7: Develop the Detailed Plan for Obtaining Data

This step describes the detailed soil sampling and characterization plan, including the decision logic to be applied during results evaluation. Additional details associated with laboratory sample collection and analysis are provided in WSs #17 and #18. Analytical requirements are provided in WSs #19, #20, #24 through #28, and #30.

Projective Objective 1 – Pre-Excavation Characterization Soil Sampling. Pre-excavation soil samples will be collected from each quadrant including GR8SS-001M, GR8SS-003M, and GR8SS-004M. A total of three discrete samples will be collected at each grid location (Figure 11.2) including the 0.5-1, 1-1.5, and 1.5-2 ft bgs depth intervals.

As described in Step 5 above, all 0.5-1.0 ft bgs samples will be analyzed for cadmium and/or lead by default, with the remaining samples held by the laboratory. Additional analysis of the remaining samples will be conducted as needed based on the 0.5-1.0 ft bgs sample results until the horizontal and vertical extent of contamination in each quadrant has been defined.

If the cadmium and/or lead result(s) from a 0.5-1.0 ft bgs sample exceed their respective cleanup goals (71 mg/kg and 400 mg/kg, respectively), then the applicable adjacent held samples will be analyzed for the applicable metals. This iterative process will continue until the horizontal and vertical extent of contamination in each quadrant has been defined or until all samples for a specific grid location have been analyzed. If the results of a sample from the 1.5-2.0 ft increment exceed cleanup goals, confirmation sampling will be performed following excavation (see Project Objective 3, below).

Project Objective 2 – Pre-Excavation Waste Characterization Soil Sampling. Composite samples will be collected from distinct excavation areas equivalent to five roll-offs to allow for the pre-excavation characterization of soil for waste management, transportation, and disposal purposes. Waste characterization soil samples will be analyzed for the RCRA parameters listed in Step 5 above. Each excavation area will be assigned a unique identifier to ensure the correct associated waste characterization sample results are applied to the soil excavated from each area. More detail is provided in Section 17.2.5.

Project Objective 3 – Confirmation Soil Sampling. Discrete samples from each grid-based location will serve as confirmation of removal of contaminated soils from the MRS. Samples collected during pre-excavation characterization that do not exceed cleanup goals may serve as confirmation that the undisturbed, unexcavated soil meets cleanup goals. Confirmation samples will be collected from the excavation floor at grid locations that exceeded cleanup goals in the 1.5-2.0 ft bgs increment. A discrete sample will be collected in the center of the 400 square-foot grid location from the upper six inches of the excavation floor and sent for lead and/or cadmium analysis. If the cadmium and/or lead result(s) from an excavation floor confirmation sample exceed their respective cleanup goals, then an additional six-inch increment will be sampled and analyzed for the applicable metals. This iterative process will continue until vertical extent of contamination in each quadrant has been defined.

Following excavation, the outer sidewalls of the excavation will be sampled at a frequency of one per 20 feet (equivalent to a grid length). Sidewall samples will not be collected where excavations extend to building foundations. Soil will be collected from the midpoint of the grid edge and along the complete vertical height of the sidewall. If the horizontal extent of contamination has not been defined at grid locations along the outer boundaries of the quadrants via sidewall sampling, then HGL will coordinate with USACE to determine if additional soil sampling is required.

Project Objective 4 – IDW Water Characterization. IDW water generated during soil sampling and excavation activities, and possibly excavation dewatering, will be containerized in 55-gallon drums and/or larger poly tanks, as necessary per the actual volume of water. If drums are used, a single composite sample will include aliquots from up to 10 drums. If one or more poly tanks are used, a single composite sample will be collected from a total volume of no more than 500 gallons. Waste characterization water samples will be analyzed for the full suite of RCRA parameters listed in Step 5 above.

Project Objective 5 – Backfill Soil Characterization. Characterization samples will be collected from the backfill and topsoil material at the on-site or off-site source at a frequency of 1 sample per 4,000 cy of backfill material required to complete excavation backfill. Backfill material subject to sampling at the source area may include in situ material (i.e., not yet excavated) or previously excavated and stockpiled material. Backfill soil samples will be collected using a stainless-steel hand auger, soil probe, etc. as applicable to actual conditions. Backfill characterization soil samples will be analyzed for the parameters listed in Step 5 above. If existing laboratory analytical results are available for the backfill material, then the results will be reviewed to confirm they are of definitive data quality and meet the requirements for government approval (in lieu of conducting characterization sampling).

Table 11.1 RVAAP Background Metals Concentrations

Parameter	Background Concentration (mg/kg)	Parameter	Background Concentration (mg/kg)
Aluminum	17,700	Lead	400*
Antimony	0.96	Magnesium	3,030
Arsenic	15.4	Manganese	1,450
Barium	88.4	Mercury	0.036
Beryllium	0.88	Nickel	21.1
Cadmium	71*	Potassium	927
Calcium	15,800	Selenium	104
Chromium	17.4	Silver	0
Cobalt	10.4	Sodium	123
Copper	17.7	Thallium	0
Cyanide	0	Vanadium	31.1
Iron	23,100	Zinc	61.8

Source: *Phase II Remedial Investigation Report for Winklepeck Burning Grounds* (SAIC, 2001) unless noted otherwise

* Project-specific cleanup goal supersedes background concentration

WORKSHEET #12
MEASUREMENT PERFORMANCE CRITERIA

MPPEH Locate, Inspection, and Removal

Measurement	Data Quality Indicator (DQI)	Specification	Activity Used to Assess Performance
Site Preparation (Personnel)	Completeness	All staff and equipment mobilize to the site. Field personnel review documents and the Site Manager and UXOSO/UXOQCS provide site-specific training to all field personnel.	Site Manager submits a daily report documenting that mobilization is complete and site-specific training is accomplished.
Site Preparation (Tree and Vegetation Clearance)	Completeness	Entire MRS is cleared of trees smaller than 4 inches in Diameter measured at the chest and vegetation.	UXOQCS submits a daily report documenting areas of the MRS cleared.
Instrument Test Strip (ITS) Construction (Analog)	Accuracy/ Precision/ Completeness	ITS is developed, installed, and surveyed with analog equipment twice per day. ITS construction details are documented on the DQCR, Access database, or in Preparatory or Initial Checklist. Daily ITS survey performance and QC are documented. Analog instruments are expected to be used it assist with the visual inspections occurring prior to and during soil excavation activities.	DQCR for activity.
Visual Anomaly Reduction	Completeness/ Comparability	100% of the planned areas requiring soil removal will have visual surveys to ensure no MPPEH is being transported off site.	Results are documented in grid sheets and daily reports.
Visual Anomaly Resolution	Accuracy/Completeness	Excavation continues vertically to a depth of the expected soil removal. Excavated soils will screened using metallic screens 1-inch x 1-inch in diameter and all metallic debris will be removed and inspected as MPPEH until a determination of MEC or MDAS is determined.	Weekly Analog data deliverable

WORKSHEET #12.1
MEASUREMENT PERFORMANCE CRITERIA: VOLATILE ORGANIC COMPOUNDS

Laboratory: SGS North America
Matrix: Water/Soil
Analytical Group/Method: Volatile Organics/SW846 8260D
Concentration Level: Low
Data Type: Definitive

DQI	QC Sample or Measurement Performance Activity	MPC[1]
Precision, Representativeness	Field Duplicate	≤ 30% RPD Water, ≤ 50% RPD Soil
Accuracy	LCS, LCSD, MS, MSD	DoD QSM 5.3 as noted in WS #28
Precision	LCSD, MSD RPD	DoD QSM 5.3 as noted in WS #28
Accuracy	Surrogate Spike	DoD QSM 5.3 as noted in WS #28
Bias	Equipment Rinse Blank	Not detected > LOQ
	Trip Blank (TB)	Not detected > LOQ
Bias, Representativeness	Temperature Blank[2]	0ºC - 6ºC
Bias	Laboratory Method Blank (MB)	No analytes detected > ½ the LOQ
Sensitivity	Laboratory limit of detection (LOD) determination and verification	≤ LOQ
Completeness	Not applicable	< 10% data rejected

[1] QC acceptance criteria from DoD Quality System Manual (QSM) 5.3 (DoD QSM for Environmental Laboratories Version 5.3, May 2019 http://www.denix.osd.mil/edqw/) as noted in WSs where applicable.
[2] If a temperature blank is not provided; other temperature measurement procedures may be used.

SW846 = Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, November 1986, and its updates. https://www.epa.gov/hw-sw846
°C = degrees Celsius
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
LOQ = limit of quantitation
MS = matrix spike
MSD = matrix spike duplicate
RPD = relative percent difference

WORKSHEET #12.2
MEASUREMENT PERFORMANCE CRITERIA: SEMI VOLATILE ORGANIC COMPOUNDS

Laboratory: SGS North America
Matrix: Water
Analytical Group/Method: Semi Volatile Organics/SW846 8270E
Concentration Level: Low
Data Type: Definitive

DQI	QC Sample or Measurement Performance Activity	MPC[1]
Precision, Representativeness	Field Duplicate	≤ 30% RPD
Accuracy	LCS, LCSD, MS, MSD	DoD QSM 5.3 as noted in WS #28
Precision	LCSD, MSD RPD	DoD QSM 5.3 as noted in WS #28
Accuracy	Surrogate Spike	DoD QSM 5.3 as noted in WS #28
Bias	Equipment Rinse Blank	Not detected > LOQ
Bias, Representativeness	Temperature Blank[2]	0ºC - 6ºC
Bias	Laboratory MB	No analytes detected > ½ the LOQ
Sensitivity	Laboratory LOD determination and verification	≤ LOQ
Completeness	Not applicable	< 10% data rejected

[1] QC acceptance criteria from DoD QSM 5.3 (DoD QSM for Environmental Laboratories Version 5.3, May 2019 http://www.denix.osd.mil/edqw/) as noted in WSs where applicable.

[2] If a temperature blank is not provided; other temperature measurement procedures may be used.

SW846 = Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, November 1986, and its updates. https://www.epa.gov/hw-sw846

WORKSHEET #12.3
MEASUREMENT PERFORMANCE CRITERIA: PESTICIDES

Laboratory: SGS North America
Matrix: Water
Analytical Group/Method: Gas Chromatograph (GC) Organics/SW846 8081B
Concentration Level: Low
Data Type: Definitive

DQI	QC Sample or Measurement Performance Activity	MPC[1]
Precision, Representativeness	Field Duplicate	≤ 30% RPD
Accuracy	LCS, LCSD, MS, MSD	DoD QSM 5.3 as noted in WS #28
Precision	LCSD, MSD RPD	DoD QSM 5.3 as noted in WS #28
Accuracy	Surrogate Spike	DoD QSM 5.3 as noted in WS #28
Bias	Equipment Rinse Blank	Not detected > LOQ
Bias, Representativeness	Temperature Blank[2]	0ºC - 6ºC
Bias	Laboratory MB	No analytes detected > ½ the LOQ
Sensitivity	Laboratory LOD determination and verification	≤ LOQ
Completeness	Not applicable	< 10% data rejected

[1] QC acceptance criteria from DoD QSM 5.3 (DoD QSM for Environmental Laboratories Version 5.3, May 2019 http://www.denix.osd.mil/edqw/) as noted in WSs where applicable.
[2] If a temperature blank is not provided; other temperature measurement procedures may be used.
SW846 = Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, November 1986, and its updates. https://www.epa.gov/hw-sw846

WORKSHEET #12.4
MEASUREMENT PERFORMANCE CRITERIA: PCBS

Laboratory: SGS North America
Matrix: Water/Soil
Analytical Group/Method: GC Organics/SW846 8082A
Concentration Level: Low
Data Type: Definitive

DQI	QC Sample or Measurement Performance Activity	MPC[1]
Precision, Representativeness	Field Duplicate	≤ 30% RPD Water, ≤ 50% RPD Soil
Accuracy	LCS, LCSD, MS, MSD	DoD QSM 5.3 as noted in WS #28
Precision	LCSD, MSD RPD	DoD QSM 5.3 as noted in WS #28
Accuracy	Surrogate Spike	DoD QSM 5.3 as noted in WS #28
Bias	Equipment Rinse Blank	Not detected > LOQ
Bias, Representativeness	Temperature Blank[2]	0ºC - 6ºC
Bias	Laboratory MB	No analytes detected > ½ the LOQ
Sensitivity	Laboratory LOD determination and verification	≤ LOQ
Completeness	Not applicable	< 10% data rejected

[1] QC acceptance criteria from DoD QSM 5.3 (DoD QSM for Environmental Laboratories Version 5.3, May 2019 http://www.denix.osd.mil/edqw/) as noted in WSs where applicable.
[2] If a temperature blank is not provided; other temperature measurement procedures may be used.
SW846 = Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, November 1986, and its updates. https://www.epa.gov/hw-sw846

WORKSHEET #12.5
MEASUREMENT PERFORMANCE CRITERIA: HERBICIDES

Laboratory: SGS North America
Matrix: Water/Soil
Analytical Group/Method: GC Organics/SW846 8151A
Concentration Level: Low
Data Type: Definitive

DQI	QC Sample or Measurement Performance Activity	MPC[1]
Precision, Representativeness	Field Duplicate	≤ 30% RPD Water, ≤ 50% RPD Soil
Accuracy	LCS, LCSD, MS, MSD	DoD QSM 5.3 as noted in WS #28
Precision	LCSD, MSD RPD	DoD QSM 5.3 as noted in WS #28
Accuracy	Surrogate Spike	DoD QSM 5.3 as noted in WS #28
Bias	Equipment Rinse Blank	Not detected > LOQ
Bias, Representativeness	Temperature Blank[2]	0ºC - 6ºC
Bias	Laboratory MB	No analytes detected > ½ the LOQ
Sensitivity	Laboratory LOD determination and verification	≤ LOQ
Completeness	Not applicable	< 10% data rejected

[1] QC acceptance criteria from DoD QSM 5.3 (DoD QSM for Environmental Laboratories Version 5.3, May 2019 http://www.denix.osd.mil/edqw/) as noted in WSs where applicable.
[2] If a temperature blank is not provided; other temperature measurement procedures may be used.
SW846 = Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, November 1986, and its updates. https://www.epa.gov/hw-sw846

WORKSHEET #12.6
MEASUREMENT PERFORMANCE CRITERIA: EXPLOSIVES

Laboratory: SGS North America
Matrix: Water/Soil
Analytical Group/Method: High Performance Liquid Chromatography (HPLC) Organics/SW846 8330
Concentration Level: Low
Data Type: Definitive

DQI	QC Sample or Measurement Performance Activity	MPC[1]
Precision, Representativeness	Field Duplicate	≤ 30% RPD Water, ≤ 50% RPD Soil
Accuracy	LCS, LCSD, MS, MSD	DoD QSM 5.3 as noted in WS #28
Precision	LCSD, MSD RPD	DoD QSM 5.3 as noted in WS #28
Accuracy	Surrogate Spike	DoD QSM 5.3 as noted in WS #28
Bias	Equipment Rinse Blank	Not detected > LOQ
Bias, Representativeness	Temperature Blank[2]	0ºC - 6ºC
Bias	Laboratory MB	No analytes detected > ½ the LOQ
Sensitivity	Laboratory LOD determination and verification	≤ LOQ
Completeness	Not applicable	< 10% data rejected

[1] QC acceptance criteria from DoD QSM 5.3 (DoD QSM for Environmental Laboratories Version 5.3, May 2019 http://www.denix.osd.mil/edqw/) as noted in WSs where applicable.

[2] If a temperature blank is not provided; other temperature measurement procedures may be used.

SW846 = Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, November 1986, and its updates. https://www.epa.gov/hw-sw846

WORKSHEET #12.7
MEASUREMENT PERFORMANCE CRITERIA TABLE – METALS

Laboratory: SGS North America
Matrix: Water and Soil
Analytical Group/Method: Metals/SW846 6010D/6020A
Concentration Level: Low
Data Type: Definitive

DQI	QC Sample or Measurement Performance Activity	MPC[1,2]
Precision, Representativeness	Field Duplicate	≤ 30% RPD Water, ≤ 50% RPD Soil
Accuracy	LCS, LCSD, MS, MSD	WS #15, DoD QSM 5.3 as noted in WS #28
Precision	LCSD, MSD RPD	DoD QSM 5.3 as noted in WS #28
Bias, Representativeness	Temperature Blank[3]	0ºC - 6ºC
Bias	Laboratory MB	No analytes detected > ½ the LOQ
Bias	Dilution Test (only required if MS or MSD percent recovery (%R) fails and native concentration is ≥50x LOQ)	Five-fold dilution ±10% of the original measurement
Bias	Post-digestion Spike (only required if MS or MSD %R fails and native concentration is <50x LOQ)	Recovery within 80% - 120%
Sensitivity	Laboratory LOD determination and verification	≤ LOQ
Completeness	Not applicable	< 10% data rejected

[1] QC acceptance criteria from DoD QSM 5.3 (DoD QSM for Environmental Laboratories Version 5.3, May 2019 http://www.denix.osd.mil/edqw/) as noted in WSs where applicable.
[2] QC acceptance criteria from EPA Method (https://www.epa.gov/esam/epa-method-2008-determination-trace-elements-waters-and-wastes-inductively-coupled-plasma-mass) as noted in WSs where applicable.
[3] If a temperature blank is not provided; other temperature measurement procedures may be used.
SW846 = Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, November 1986, and its updates. https://www.epa.gov/hw-sw846

WORKSHEET #12.8
MEASUREMENT PERFORMANCE CRITERIA TABLE – QC TESTS FOR WASTE CHARACTERIZATION ANALYSES

The following analytical methods will be used to characterize aqueous and solid IDW to ensure proper storage and disposal of this material. These analyses will be required to meet the requirements presented in the analytical methods and the laboratory SOPs and no project-specific MPC are presented. The analyses that will be performed to characterize project IDW are presented in the following tables.

Analyses required to comply with 40 CFR 261.21 through 24:

Parameter	Matrix	Analytical Method
TCLP[1]	Soil	SW1311
VOCs[2]	Water and soil extract	SW8260D
SVOCs[2]	Water and soil extract	SW8270E
Metals[2]	Water and soil extract	SW6020A
Mercury	Water and soil extract	SW7470A
PCBs	Water and soil extract	SW8082
Percent moisture[3]	Soil	SM540G-1997
Paint filter test	Soil	SW9095B
Pesticides	Soil	SW8081B
Herbicides	Soil	SW8151A
Total Cyanide	Water and Soil	SW9012B
Sulfide	Water and Soil	SM4500S F-2000
Nitrate/Nitrite	Water and Soil	SW9056A
pH	Water	SW9040C
pH[3]	Soil	SW9045D
Flash point	Water	SW1010A
Ignitability	Soil	Per SOP

[1] Prior to sampling soil IDW, it must be determined whether TCLP is appropriate. Consult the requirements of the waste disposal facility and any applicable federal, state, or local discharge permits or other requirements.

[2] Analyte lists as presented in 40 CFR 261.24, see WS #15.12.

[3] Percent moisture and pH are also measured for solid matrix environmental samples; this method is listed with the other IDW parameters because the test will be performed in accordance with the laboratory SOP and the results from this test will be used without review or validation.

SM = analytical method source is Standard Methods for the Analysis of Water and Wastewater (1997)

SW = analytical method source is the EPA's Test Methods for Analysis of Water and Waste, Physical/Chemical Methods (SW-846), through Update V (EPA, 2015)

WORKSHEET #13
SECONDARY DATA USES AND LIMITATIONS

Data type	Source	Data uses relative to current project	Factors affecting the relevance, strength, and reliability of secondary data
Wetlands Survey	Final Wetland Delineation Report (ERG, 2022)	Project planning and work scheduling	None
GIS data	Environmental System Research Institute, ARNG	Data layers to support work plan and project report ArcGIS figure development and field mapping.	Data are assumed to be accurate when obtained from known sources with internal QA/QC programs. The relative accuracy of GIS data will be confirmed in the field with GPS instrumentation or comparison with new civil survey data collected during field activities.
Site History	• ASR (2004) • HRR (2007) • SI (2007) • RI (2011) • FS (2019) • PP (2020) • ROD (2020)	• Identified MRS location and history • Identified potential MEC and MC presence • Evaluated potential risk from MC presence in surface soil • Established selected remedy for the MRS	None
CSM, Land Use	• RI (2011)	• Facility profile • Physical profile • Release profile • Land use and exposure profile	None

WORKSHEETS #14 AND #16
PROJECT TASKS AND SCHEDULE

DFW	Activity	Responsible party	Planned start date	Planned completion date	Deliverable(s)	Deliverable due date
MPPEH/MC #1	Mobilization and Vegetation Clearance	HGL	3/5/23	3/8/23	Daily/Weekly QC Reports, Vegetation Clearance Data Submittal	Daily/Weekly; Following vegetation clearance
MPPEH/MC #2	Soil Sample Grid Layout	HGL	3/9/23	3/13/23	Daily/Weekly QC Reports, Mag and Dig MPPEH Removal Data Submittal	Daily/Weekly; Following Mag and Dig
MPPEH/MC #3	Pre-Excavation Soil Sample Collection	HGL	3/14/23	3/16/23	Daily/Weekly QC Reports	Daily/Weekly
MC #3	Analysis and Pre-Excavation Sampling Report	HGL	3/17/23	10/24/23	Daily/Weekly QC Reports; Pre-Excavation Sampling Report (including lab data and validation reports)	Daily/Weekly; Draft Sampling Report due 6/30/22
MPPEH/MC #4	Excavation and Backfill	HGL	10/25/23	TBD based on excavation volume	Daily/Weekly QC Reports	Daily/Weekly
MPPEH #5	Final Disposition of MPPEH	HGL	TBD	TBD	Daily/Weekly QC Reports	Daily/Weekly
MC #5	Waste Characterization and Disposal	HGL	10/25/23	TBD based on excavation volume	Daily/Weekly QC Reports; MPPEH Processing, Excavation, Transport and Delivery, and Backfill Submittal	Daily/Weekly; Following excavation activities
MC #6	Site Restoration and Demobilization	HGL	TBD	TBD	Daily/Weekly QC Reports; Final Site Restoration Data Submittal	Daily/Weekly; Following backfill

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

In all cases, the laboratory is required to report concentrations at or greater than the DL as detected results. Results with quantitation at or greater than the DL but below the corresponding LOQ will be reported as detections by the laboratory with the qualification of J. Non-detected results and results below the corresponding DL will be reported by the laboratory as the DL and qualified U. Laboratory-assigned qualifiers may be subsequently modified during the data validation process (see WS #36). The PALs listed in WS #15.1 for cadmium (71 mg/kg) and lead (400 mg/kg) represent the cleanup goals defined in the ROD (USACE, 2020) and apply to the pre-excavation characterization sampling within the MRS. WSs 15.2 to 15.10 present the PALs associated with backfill material to be used to restore the site following excavation. Waste characterization parameters are provided in WS #15.11.

WORKSHEET #15.1
REFERENCE LIMITS AND EVALUATION TABLE FOR PRE-EXCAVATION CHARACTERIZATION– METALS BY SW846 METHOD SW6010D

Analyte	CASRN	SGS Orlando			PAL[1] (mg/kg)
		DL (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	
Cadmium	7440-43-9	0.025	0.05	0.20	71
Lead	7439-92-1	0.05	0.20	1.0	400

[1] The PAL value is from the EPA RSLs for residential soil, target hazard quotient (THQ) 0.1, May 2021

CASRN = Chemical Abstracts Service Registration Number

WORKSHEET #15.2
REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – VOCS BY METHOD 8260D

Analyte	CASRN	SGS Orlando			PAL[1] (µg/kg)
		DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)	
1,1,1,2-Tetrachloroethane	630-20-6	1.0	2.0	5.0	2000
1,1,1-Trichloroethane	71-55-6	1.0	2.0	5.0	810,000
1,1,2,2-Tetrachloroethane	79-34-5	1.0	2.0	5.0	600
1,1,2-Trichloroethane	79-00-5	1.0	2.0	5.0	150
1,1-Dichloroethane	75-34-3	1.8	2.0	5.0	3,600
1,1-Dichloroethene	75-35-4	1.0	2.0	5.0	2,300
1,1-Dichloropropene	563-58-6	1.0	2.0	5.0	NE
1,2,3-Trichlorobenzene	87-61-6	1.4	3.5	5.0	6,300
1,2,3-Trichloropropane	96-18-4	1.3	3.5	5.0	51
1,2,4-Trichlorobenzene	120-82-1	1.0	3.5	5.0	5,800
1,2,4-Trimethylbenzene	95-63-6	1.0	2.0	5.0	30,000
1,2-Dibromo-3-chloropropane	96-12-8	1.9	3.5	5.0	53
1,2-Dibromoethane	106-93-4	1.0	2.0	5.0	3.6*
1,2-Dichlorobenzene	95-50-1	1.0	2.0	5.0	180,000
1,2-Dichloroethane	107-06-2	1.0	2.0	5.0	460
1,2-Dichloropropane	78-87-5	1.0	2.0	5.0	1,600
1,3,5-Trimethylbenzene	108-67-8	1.0	2.0	5.0	27,000
1,3-Dichlorobenzene	541-73-1	1.0	2.0	5.0	NE
1,3-Dichloropropane	142-28-9	1.0	2.0	5.0	160,000
1,4-Dichlorobenzene	106-46-7	1.2	2.0	5.0	2,600
2,2-Dichloropropane	594-20-7	1.0	2.0	5.0	NE
2-Butanone (MEK)	78-93-3	7.3	15	25	2,700,000
2-Chlorotoluene (o-Chlorotoluene)	95-49-8	1.0	2.0	5.0	160,000
2-Hexanone	591-78-6	7.5	15	25	20,000
4-Chlorotoluene (p-Chlorotoluene)	106-43-4	1.0	2.0	5.0	160,000
4-Isopropyltoluene (p-Isopropyltoluene)	99-87-6	1.0	2.0	5.0	NE
4-Methyl-2-pentanone (MIBK)	108-10-1	7.5	15	25	3,300,000
Acetone	67-64-1	100	150	200	7,000,000
Benzene	71-43-2	1.2	2.0	5.0	1200
Bromobenzene	108-86-1	1.0	2.0	5.0	29,000
Bromochloromethane	74-97-5	1.5	2.0	5.0	15,000
Bromodichloromethane	75-27-4	1.0	2.0	5.0	290
Bromoform	75-25-2	1.0	2.0	5.0	19,000
Bromomethane (Methyl bromide)	74-83-9	2.0	3.5	5.0	680
Carbon disulfide	75-15-0	1.0	2.0	5.0	77,000
Carbon tetrachloride	56-23-5	1.0	2.0	5.0	650
Chlorobenzene	108-90-7	1.0	2.0	5.0	28,000
Chlorodibromomethane	124-48-1	1.0	2.0	5.0	8,300
Chloroethane	75-00-3	2.0	3.5	5.0	540,000
Chloroform	67-66-3	1.3	2.0	5.0	320
Chloromethane (Methyl chloride)	74-87-3	2.0	3.5	5.0	11,000

WORKSHEET #15.2 (CONTINUED)
REFERENCE LIMITS AND EVALUATION TABLE – VOCS BY METHOD 8260D

Analyte	CASRN	SGS Orlando DL (µg/kg)	SGS Orlando LOD (µg/kg)	SGS Orlando LOQ (µg/kg)	PAL[1] (µg/kg)
cis-1,2-Dichloroethene	156-59-2	1.4	2.0	5.0	16,000
cis-1,3-Dichloropropene	10061-01-5	1.0	2.0	5.0	NE
Dibromomethane (Methylene bromide)	74-95-3	1.0	2.0	5.0	2,400
Dichlorodifluoromethane	75-71-8	2.0	3.5	5.0	8,700
Ethylbenzene	100-41-4	1.0	2.0	5.0	5,800
Hexachlorobutadiene	87-68-3	1.3	2.0	5.0	1,200
Isopropylbenzene	98-82-8	1.0	2.0	5.0	190,000
Methyl tert-butyl ether (MTBE)	1634-04-4	1.0	2.0	5.0	47,000
Methylene chloride	75-09-2	11	15	20	35,000
m,p-Xylene	179601-23-1	1.1	4.0	10	NE
Naphthalene	91-20-3	2.0	3.5	5.0	2,000
n-Butylbenzene	104-51-8	1.0	2.0	5.0	390,000
n-Propylbenzene	103-65-1	1.0	2.0	5.0	380,000
o-Xylene	95-47-6	1.0	2.0	5.0	64,000
sec-Butylbenzene	135-98-8	1.0	2.0	5.0	780,000
Styrene	100-42-5	1.0	2.0	5.0	600,000
tert-Butylbenzene	98-06-6	1.0	2.0	5.0	780,000
Tetrachloroethene	127-18-4	1.3	2.0	5.0	8,100
Toluene	108-88-3	10	15	20	490,000
trans-1,2-Dichloroethene	156-60-5	1.0	2.0	5.0	7,000
trans-1,3-Dichloropropene	10061-02-6	1.0	2.0	5.0	NE
Trichloroethene	79-01-6	1.0	2.0	5.0	410
Trichlorofluoromethane	75-69-4	2.0	3.5	5.0	2,300,000
Vinyl acetate	108-05-4	16	20	25	91,000
Vinyl chloride	75-01-4	1.0	2.0	5.0	59
Xylenes (total)	1330-20-7	1.1	4.0	10	58,000

[1] The PAL value is from the EPA RSLs for residential soil, THQ 0.1, May 2021

µg/kg = micrograms per kilogram

NE = not established under EPA Maximum Contaminant Level or ODNR groundwater regulations

WORKSHEET #15.3
REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – SVOCS BY METHOD 8270E

Analyte	CASRN	SGS Orlando			PAL[1] (µg/L)
		DL (µg/L)	LOD (µg/L)	LOQ (µg/L)	
1,2,4-Trichlorobenzene	120-82-1	20	33	170	5,800
1,2-Dichlorobenzene	95-50-1	17	67	170	180,000
1,2-Diphenylhydrazine [Azobenzene]	122-66-7	17	33	170	680
1,3-Dichlorobenzene	541-73-1	18	67	170	NE
1,4-Dichlorobenzene	106-46-7	22	67	170	2,600
1-Methylnaphthalene	90-12-0	17	33	170	18,000
2,4,5-Trichlorophenol	95-95-4	27	33	170	630,000
2,4,6-Trichlorophenol	88-06-2	19	33	170	6,300
2,4-Dichlorophenol	120-83-2	19	33	170	19,000
2,4-Dimethylphenol	105-67-9	44	67	170	130,000
2,4-Dinitrophenol	51-28-5	170	500	830	13,000
2,4-Dinitrotoluene	121-14-2	17	33	170	1,700
2,6-Dinitrotoluene	606-20-2	21	33	170	360
2-Chloronaphthalene	91-58-7	17	33	170	480,000
2-Chlorophenol	95-57-8	20	33	170	39,000
2-Methylnaphthalene	91-57-6	17	33	170	24,000
2-Methylphenol (o-Cresol)	95-48-7	20	33	170	320,000
2-Nitroaniline	88-74-4	39	67	170	63,000
2-Nitrophenol	88-75-5	18	33	170	NE
2,2'-Oxybis(1-chloropropane)	108-60-1	21	33	170	310,000
3,3'-Dichlorobenzidine	91-94-1	40	67	170	1,200
3-Nitroaniline	99-09-2	19	67	170	NE
3&4-Methylphenol [m/p-Cresol][2]	65794-96-9	27	67	170	320,000
4,6-Dinitro-o-cresol [4,6-Dinitro-2-methylphenol]	534-52-1	67	130	330	510
4-Bromophenyl phenyl ether	101-55-3	17	33	170	NE
4-Chloro-3-methylphenol	59-50-7	19	33	170	630,000
4-Chloroaniline [p-Chloroanline]	106-47-8	42	67	170	2,700
4-Chlorophenyl phenyl ether	7005-72-3	17	33	170	NE
4-Nitroaniline	100-01-6	48	67	170	25,000
4-Nitrophenol	100-02-7	170	330	830	NE
Acenaphthene	82-32-9	18	33	170	NE
Acenaphthylene	208-96-8	17	33	170	NE
Aniline	62-53-3	36	67	170	44,000
Anthracene	120-12-7	19	33	170	1,800,000
Benzidine	92-87-5	330	830	1700	0.5*
Benzoic acid	65-85-0	170	330	830	25,000,000
Benzo(a)anthracene	56-55-3	17	33	170	1,100
Benzo(a)pyrene	50-32-8	20	33	170	110*
Benzo(b)fluoranthene	205-99-2	18	33	170	1,100
Benzo(g,h,i)perylene	191-24-2	17	33	170	NE
Benzo(k)fluoranthene	207-08-9	22	33	170	11,000
Benzyl alcohol	100-51-6	17	33	170	630,000

WORKSHEET #15.3 (CONTINUED) REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – SVOCS BY METHOD 8270E

Analyte	CASRN	SGS Orlando DL (µg/L)	SGS Orlando LOD (µg/L)	SGS Orlando LOQ (µg/L)	PAL[1] (µg/L)
bis(2-Chloroethoxy)methane	111-91-1	17	33	170	19,000
bis(2-Chloroethyl)ether	111-44-4	19	33	170	230
bis(2-Ethylhexyl)phthalate	117-81-7	33	120	330	39,000
Butyl benzyl phthalate	85-68-7	33	67	170	290,000
Carbazole	86-74-8	23	33	170	NE
Chrysene	218-01-9	17	33	170	110,000
Di-n-butyl phthalate	84-74-2	67	120	330	630,000
Di-n-octyl phthalate	117-84-0	33	67	170	63,000
Dibenzo(a,h)anthracene	53-70-3	21	33	170	110*
Dibenzofuran	132-64-9	17	33	170	7,800
Diethyl phthalate	84-66-2	33	120	330	5,100,000
Dimethyl phthalate	131-11-3	33	67	170	NE
Fluoranthene	206-44-0	17	33	170	240,000
Fluorene	86-73-7	18	33	170	240,000
Hexachlorobenzene	118-74-1	17	33	170	78*
Hexachlorobutadiene	87-68-3	17	67	170	1,200
Hexachlorocyclopentadiene	77-47-4	33	67	170	180
Hexachloroethane	67-72-1	20	67	170	1,800
Indeno(1,2,3-cd)pyrene	193-39-5	20	33	170	1,100
Isophorone	78-59-1	17	33	170	570,000
N-Nitrosodi-n-propylamine	621-64-7	17	33	170	78*
N-Nitrosodimethylamine	62-75-9	28	67	170	2.0*
N-Nitrosodiphenylamine	86-30-6	18	67	170	110,000
Naphthalene	91-20-3	17	33	170	2,000
Nitrobenzene	98-95-3	17	33	170	5,100
Pentachlorophenol	87-86-5	170	330	830	1,000
Phenanthrene	85-01-8	17	33	170	NE
Phenol	108-95-2	17	33	170	1,900,000
Pyrene	129-00-0	19	33	170	180,000
Pyridine	110-86-1	67	120	330	7,800

*This action limit is below the LOQ. The laboratory methods and analytical limits provided are appropriate and are routinely used for environmental analyses. The laboratory limits are comparable to those obtainable through other commercial laboratories. The impact of the action level being below the LOQ for an analyte will be discussed during risk assessment and reporting summaries.

[1] The PAL value is from the EPA RSLs for residential soil, THQ 0.1, May 2021

[2] The RSL for 3- & 4- methylphenol is not established, so the more conservative RSL for 3-methylphneol was used.

µg/L = micrograms per liter

NE = not established under EPA Maximum Contaminant Level or ODNR groundwater regulations

WORKSHEET #15.4
REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – PESTICIDES BY METHOD 8081B

Analyte	CASRN	SGS Orlando DL (µg/kg)	SGS Orlando LOD (µg/kg)	SGS Orlando LOQ (µg/kg)	PAL[1] (µg/kg)
4,4'-DDD	72-54-8	0.46	0.83	3.3	190
4,4'-DDE	72-55-9	0.60	0.83	3.3	2,000
4,4'-DDT	50-29-3	0.50	0.83	3.3	1,900
Aldrin	309-00-2	0.52	0.83	1.7	NE
alpha-BHC	319-84-6	0.52	0.83	1.7	86
alpha-Chlordane	5103-71-9	0.51	0.83	1.7	3,600
beta-BHC	319-85-7	0.49	0.83	1.7	300
delta-BHC	319-86-8	0.47	0.83	1.7	NE
Dieldrin	60-57-1	0.46	0.83	1.7	34
Endosulfan-I	959-98-8	0.38	0.83	1.7	NE
Endosulfan-II	33213-65-9	0.39	0.83	1.7	NE
Endosulfan sulfate	1031-07-8	0.44	0.83	3.3	38,000
Endrin	72-20-8	0.83	1.7	3.3	1,900
Endrin aldehyde	7421-93-4	0.38	0.83	3.3	NE
Endrin ketone	53494-70-5	0.52	0.83	3.3	NE
gamma-BHC (Lindane)	58-89-9	0.50	0.83	1.7	71
gamma-Chlordane	5103-74-2	0.48	0.83	1.7	3,600
Heptachlor	76-44-8	0.49	0.83	1.7	130
Heptachlor epoxide	1024-57-3	0.49	0.83	1.7	70
Methoxychlor	72-43-5	0.66	1.7	3.3	32,000
Toxaphene	8001-35-2	25	41	83	490

*This action limit is below the LOQ. The laboratory methods and analytical limits provided are appropriate and are routinely used for environmental analyses. The laboratory limits are comparable to those obtainable through other commercial laboratories. The impact of the action level being below the LOQ for an analyte will be discussed during risk assessment and reporting summaries.

[1] The PAL value is from the EPA RSLs for residential soil, THQ 0.1, May 2021

NA = not applicable., PAL values are not applicable to surrogate compounds.

NE = not established

WORKSHEET #15.5
REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – PCBS BY METHOD 8082A

Analyte	**CASRN**	**SGS Orlando**			**PAL[1] (µg/kg)**
		DL (µg/kg)	**LOD (µg/kg)**	**LOQ (µg/kg)**	
Aroclor 1016	12674-11-2	6.6	12	17	410
Aroclor 1221	11104-28-2	8.3	12	17	200
Aroclor 1232	11141-16-5	8.3	12	17	170
Aroclor 1242	53469-21-9	6.6	12	17	230
Aroclor 1248	12672-29-6	6.6	12	17	230
Aroclor 1254	11097-69-1	6.6	12	17	120
Aroclor 1260	11096-82-5	6.6	12	17	240

[1] The PAL value is from the EPA RSLs for residential soil, THQ 0.1, May 2021

WORKSHEET #15.6
REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – HERBICIDES BY METHOD 8151A

Analyte	Acronym	CASRN	SGS Orlando DL (µg/kg)	SGS Orlando LOD (µg/kg)	SGS Orlando LOQ (µg/kg)	PAL[1] (µg/kg)
2,4-Dichlorophenoxy acetic acid	2,4-D	94-75-7	8.4	17	33	70,000
2(2,4,5-Trichlorophenoxy) propionic acid	2,4,5-TP (Silvex)	93-72-1	0.93	1.7	3.3	51,000
2,4,5-Trichlorophenoxy acetic acid	2,4,5-T	93-76-5	0.85	1.7	3.3	63,000

[1] The PAL value is from the EPA RSLs for residential soil, THQ 0.1, May 2021

WORKSHEET #15.7
REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – EXPLOSIVES BY METHOD 8330B

Analyte	CASRN	SGS Orlando			PAL[1] (µg/kg)
		DL (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)	
1,3,5-Trinitrobenzene	99-35-4	50	75	100	220,000
1,3-Dinitrobenzene	99-65-0	50	75	100	630
2,4,6-Trinitrotoluene	118-96-7	50	75	100	3,600
2,4-Dinitrotoluene	121-14-2	50	75	100	1,700
2,6-Dinitrotoluene	606-20-2	50	75	100	360
2-amino-4,6-Dinitrotoluene	35572-78-2	50	75	100	770
2-Nitrotoluene (o-Nitrotoluene)	88-72-2	50	75	100	3,200
3,5-Dinitroaniline	618-87-1	50	75	100	2,500
3-Nitrotoluene (m-Nitrotoluene)	99-08-1	50	75	100	630
4-amino-2,6-Dinitrotoluene	19406-51-0	51	75	100	770
4-Nitrotoluene (p-Nitrotoluene)	99-99-0	50	75	100	25,000
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	50	75	100	8,300
Nitrobenzene	98-95-3	50	75	100	5,100
Nitroglycerine	55-63-0	250	500	1,000	630
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	51	75	100	390,000
PETN	78-11-5	250	500	1,000	57,000
Tetryl	479-45-8	50	75	100	16,000

[1] The PAL value is from the EPA RSLs for residential soil, THQ 0.1, May 2021

WORKSHEET #15.8
REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – METALS BY METHOD 6010D

Analyte	CASRN	SGS Orlando DL (mg/kg)	SGS Orlando LOD (mg/kg)	SGS Orlando LOQ (mg/kg)	PAL[1] (mg/kg)
Aluminum	7429-90-5	1.8	2.5	10	17,700
Antimony	7440-36-0	0.065	0.25	1.0	3.1
Arsenic	7440-38-2	0.10	0.25	0.5	15.4
Barium	7440-39-3	0.05	0.10	10	1,500
Beryllium	7440-41-7	0.025	0.05	0.25	16
Cadmium	7440-43-9	0.025	0.05	0.20	71
Calcium	7440-70-2	2.5	5.0	250	15,800
Chromium, total	7440-47-3	0.05	0.10	0.5	300[2]
Cobalt	7440-48-4	0.025	0.05	2.5	10.4
Copper	7440-50-8	0.05	0.10	1.25	310
Iron	7439-89-6	0.85	2.5	15	23,100
Lead	7439-92-1	0.05	0.20	1.0	400
Magnesium	7439-95-4	1.8	5.0	250	3,030
Manganese	7439-96-5	0.025	0.05	0.75	1,450
Nickel	7440-02-0	0.025	0.05	2.0	150
Potassium	7440-09-7	10	25	500	927
Selenium	7782-49-2	0.12	0.25	1.0	104
Silver	7440-22-4	0.045	0.10	0.5	39
Sodium	7440-23-5	25	100	500	123
Thallium	7440-28-0	0.025	0.25	0.5	0.078*
Vanadium	7440-62-2	0.025	0.05	2.5	39
Zinc	7440-66-6	0.15	0.25	1.0	2,300

* This action limit is below the LOQ. The laboratory methods and analytical limits provided are appropriate and are routinely used for environmental analyses. The laboratory limits are comparable to those obtainable through other commercial laboratories. The impact of the action level being below the LOQ for an analyte will be discussed during risk assessment and reporting summaries.

[1] The PAL value is from the greater of EPA RSLs for residential soil, THQ 0.1, May 2021, or the site-specific background values (SAIC, 2001).

[2] The PAL for total chromium is not established, the value for hexavalent chromium is listed since it is the more conservative of the chromium species listed in the RSL tables.

WORKSHEET #15.9
REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – METALS BY METHOD 6020A

Analyte	CASRN	SGS Orlando			PAL[1] (mg/kg)
		DL (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	
Aluminum	7429-90-5	1.0875	6.25	25	17,700
Antimony	7440-36-0	0.025	0.125	0.25	3.1
Arsenic	7440-38-2	0.025	0.125	0.25	15.4
Barium	7440-39-3	0.025	0.125	0.25	1,500
Beryllium	7440-41-7	0.027	0.125	0.25	16
Cadmium	7440-43-9	0.025	0.125	0.25	71
Calcium	7440-70-2	1.8	12.5	25	15,800
Chromium, total	7440-47-3	0.025	0.125	0.25	300[2]
Cobalt	7440-48-4	0.025	0.125	0.25	10.4
Copper	7440-50-8	0.025	0.125	0.25	310
Iron	7439-89-6	1.975	6.25	25	23,100
Lead	7439-92-1	0.025	0.125	0.25	400
Magnesium	7439-95-4	1.30	12.5	25	3,030
Manganese	7439-96-5	0.025	0.125	0.25	1,450
Nickel	7440-02-0	0.025	0.125	0.25	150
Potassium	7440-09-7	1.6425	12.5	25	927
Selenium	7782-49-2	0.045	0.125	0.25	104
Silver	7440-22-4	0.025	0.125	0.25	39
Sodium	7440-23-5	1.20	12.5	25	123
Thallium	7440-28-0	0.025	0.125	0.25	0.078*
Vanadium	7440-62-2	0.025	0.125	0.25	39
Zinc	7440-66-6	0.0725	0.125	0.25	2,300

* This action limit is below the LOQ. The laboratory methods and analytical limits provided are appropriate and are routinely used for environmental analyses. The laboratory limits are comparable to those obtainable through other commercial laboratories. The impact of the action level being below the LOQ for an analyte will be discussed during risk assessment and reporting summaries.

[1] The PAL value is from the EPA RSLs for residential soil, THQ 0.1, May 2021, or the site-specific background values (SAIC, 2001).

[2] The PAL for total chromium is not established, the value for hexavalent chromium is listed since it is the more conservative of the chromium species listed in the RSL tables.

WORKSHEET #15.10
REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – MERCURY BY METHOD 7471B

Analyte	CASRN	SGS Orlando			PAL[1] (mg/kg)
		DL (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	
Mercury	7439-97-6	0.0042	0.017	0.042	1.1

[1] The PAL value is from the EPA RSLs for residential soil, THQ 0.1, May 2021

WORKSHEET #15.11
REFERENCE LIMITS AND EVALUATION TABLE FOR BACKFILL CHARACTERIZATION – GEOTECHNICAL ANALYSIS

Parameter	Test Method	Criteria
Grain Size	ASTM D 422	No criteria, results used as a basis for classification
Soil Type (Classification)	ASTM D 2487	SC, SC-SM, CL, or CH
Permeability	ASTM D 5084	≤ 10^{-4} cm/sec
Compaction	ASTM D 698	90% of ASTM maximum dry density

WORKSHEET #15.12
REFERENCE LIMITS AND EVALUATION TABLE FOR WASTE CHARACTERIZATION

Method	Analyte	RCRA Limit[1] (mg/L)	SGS Orlando LOQ (mg/L)
Analysis of Aqueous Waste and Solid Waste Extracts for Toxicity Characteristic			
VOCs SW8260D	1,1-Dichloroethene	0.7	0.002
	1,2-Dichloroethane	0.5	0.002
	2-Butanone (methyl ethyl ketone)	200	0.015
	Benzene	0.5	0.002
	Carbon tetrachloride	0.5	0.002
	Chlorobenzene	100	0.002
	Chloroform	6.0	0.002
	Tetrachloroethene	0.7	0.002
	Trichloroethene	0.5	0.002
	Vinyl chloride	0.2	0.002
SVOCs SW8270E	1,4-Dichlorobenzene	7.5	0.05
	2,4,5-Trichlorophenol	400	0.05
	2,4,6-Trichlorophenol	2.0	0.05
	2,4-Dinitrotoluene	0.13	0.05
	Cresols, total (2-,3-, & 4-methylphenol)	200	0.05
	Hexachlorobenzene	0.13	0.05
	Hexachlorobutadiene	0.5	0.05
	Hexachloroethane	3.0	0.05
	Nitrobenzene	2.0	0.05
	Pentachlorophenol	100	0.25
	Pyridine	5.0	0.1
Metals SW6020A	Arsenic	5.0	0.1
	Barium	100	2
	Cadmium	1.0	0.05
	Chromium	5.0	0.1
	Lead	5.0	0.05
	Selenium	1.0	0.1
	Silver	5.0	0.1
SW7470A	Mercury	0.2	0.005
SW8081B	Chlordane	0.03	0.00083
	Endrin	0.02	0.0017
	gamma-BHC (lindane)	0.4	0.00083
	Heptachlor	0.008(3)	0.00083
	Heptachlor epoxide	0.008(3)	0.00083
	Methoxychlor	10	0.0017
	Toxaphene	0.5	0.041
SW8151A	2,4-Dichlorophenoxyacetic acid (2,4-D)	10	0.002
	2,4,5-TP (silvex)	1.0	0.0002
PCBs			
SW8082A	PCBs, total (aqueous)	50[2]	NA[3]
	PCBs, total (soil)	50 mg/kg[2]	NA[3]
Reactivity			
9012E	Cyanide	NA	0.01
SM4500S F-2000	Sulfide	NA	1.0
9056A	Nitrate	NA	0.1
9056A	Nitrite	NA	0.1

Method	Analyte	RCRA Limit[1] (mg/L)	SGS Orlando LOQ (mg/L)
Corrosivity			
SM4500H+ B-2000	pH (aqueous)	pH <2 (acidic) or pH >12.5 (basic)	0.01 Standard Unit
SW9045D	pH (soil)		
Ignitability			
1010A	Flash point (aqueous)	<60 °C	NA
1010A MOD	Ignitability (solid)	<60 °C	NA

[1] The RCRA limits were taken from the November 2021 EPA Title 40 CFR, Subchapter 1 Solid Waste, Part 261 Identification and listing of Hazardous Waste, Sections 21 through 24.
[2] Value is from the Toxic Substances Control Act (40 CFR 761.61).
[3] The values of each detected Aroclor will be summed; non-detected results will be assigned a value of zero.
mg/L = milligrams per liter

WORKSHEET #17
SURVEY DESIGN AND PROJECT WORK FLOW

17.0 INTRODUCTION

This WS describes the sampling design and associated rationale for each DFW for MPPEH and MC that will be required to successfully complete field operations during this project and achieve the DQOs described in WS #11. A DFW consists of an activity or task that is separate and distinct from other activities, requires separate control, and can be roughly correlated to the DQOs summarized in WS #11. The following subsections specify the technical approach, broken down into a series of DFWs, for completion of the field activities planned, which include:

MPPEH DFWs:

- MPPEH #1: UXO Avoidance/Construction Support during Site Mobilization and Vegetation Clearing
- MPPEH #2: UXO Avoidance/Construction Support during Soil Sample Grid Layout
- MPPEH #3: UXO Avoidance/Construction Support during Soil Sample Collection
- MPPEH #4: Visual inspection, instrument aided MPPEH, locate, inspect, and removal prior to and during Soil Excavation
- MPPEH #5: MPPEH final disposition

MC DFWs:

- MC #1: Site Mobilization and Vegetation Clearing
- MC #2: Soil Sample Grid Layout
- MC #3: Soil Sample Collection
- MC #4: Soil Excavation and Backfill
- MC #5: Waste Characterization and Disposal
- MC #6: Site Restoration and Demobilization

Specific SOPs associated with the DFWs described above are listed in WS #21 and provided in Appendix D. All listed MPPEH DFWs listed above will be applicable during the RA MC DFWs listed (1 through 6) which will accomplish the remedy.

17.1 MPPEH DFWs

This section describes the DFWs associated with the location, ID, and classification of MPPEH items from within the identified MC contaminated soil excavation areas at the Group 8 MRS. MPPEH ID and removal will be completed prior to and during to the completion of all soil excavation activities covered in the MC DFWs, Section 17.2.

17.1.1 MPPEH DFW #1: UXO Avoidance/Construction Support during Site Mobilization and Vegetation Clearing

HGL will provide a qualified DDESB TP-18 UXO personnel during all activities discussed below in Section 17.2.1 using the procedures described in HGL SOP 501.08 MEC Construction Support and 408.507 MEC Anomaly Avoidance Support (Appendix D) to provide UXO avoidance as needed while the site is being mobilized and vegetation clearing is occurring.

The UXOQCS will be responsible for establishing an ITS for the UXO personnel to test their analog instrument daily. It will be the responsibility of the UXO Team Leader to document that each operator and their instrument perform their equipment checks daily for all MPPEH activities discussed below.

All MPPEH identified during this activity will be inspected and determined to be either MEC or MDAS and will be handled as discussed in Section 17.1.5 below.

17.1.2 MPPEH DFW #2: UXO Avoidance/Construction Support during Soil Sample Grid Layout

HGL will provide a qualified DDESB TP-18 UXO personnel during all activities discussed below in section 17.2.2 using the procedures described in HGL SOP 501.08 MEC Construction Support and 408.507 MEC Anomaly Avoidance Support (Appendix D) to provide UXO avoidance as needed for emplacement of the 225 soil sampling grids located within the three quadrants. The UXO technicians will employ the use of a Schonstedt Ga 52Cx or similar detector for determining the safe location of each grid corner location.

All MPPEH identified during this activity will be inspected and determined to be either MEC or MDAS and will be handled as discussed in Section 17.1.5 below.

17.1.3 MPPEH DFW #3: UXO Avoidance/Construction Support during Soil Sample Collection

HGL will provide a qualified DDESB TP-18 UXO personnel during all activities discussed below in section 17.2.3 using the procedures described in HGL SOP 501.08 MEC Construction Support and 408.507 MEC Anomaly Avoidance Support (Appendix D) to provide UXO avoidance as needed for pre-excavation characterization soil samples to be collected from each grid location. Samples will be collected using a direct-push technology (DPT) drill rig equipped with a Macro Core sampler and single-use disposable liners. The UXO team will check each sampling location with their analog instrument ensuring that there is no response indicative of a metallic object in the sub-surface where the sample location is planned. The UXO team will be responsible for locating an area within the sampling grid safe for the environmental sample to be collected.

All MPPEH identified during this activity will be inspected and determined to be either MEC or MDAS and will be handled as discussed in Section 17.1.5 below.

17.1.4 MPPEH DFW #4: Visual Inspection, instrument aided MPPEH, locate, inspect, and removal prior to and during Soil Excavation

HGL will provide a qualified DDESB TP-18 UXO personnel during all activities discussed below in section 17.2.4 using the procedures described in HGL SOP 501.05 Analog and Digital MEC Operations (Appendix D) to provide UXO instrument assisted MPPEH removal as needed before and during the removal of soils with cadmium and lead at concentrations greater than their respective cleanup goals.

Once it has been determined which grids will require soil excavations (due to the detections of cadmium and lead at concentrations greater than their respective cleanup goals) the UXO team will perform a visual inspection and remove all individual MPPEH anomalies from the surface prior to each excavation phase. These excavations will continue until the respective depth of the planned excavations is reached (based on sampling results). HGL will build a portable metallic screening table that can be placed on the top of a roll-off container. The excavator operator will feather the soils over the screen to separate any residual MPPEH that may be present in excavated soils. In the event the area to be excavated is saturated with non-munition related debris the UXO team will be allowed to use the earth moving machinery to scrape the area(s) in six-inch lifts, exposing the metallic debris to be visually inspected for MPPEH.

All MPPEH identified during this activity will be inspected and determined to be either MEC or MDAS and will be handled as discussed in Section 17.1.5 below.

17.1.5 MPPEH DFW #5: MPPEH Final Disposition

A detailed account of all MPPEH encountered during the investigation will be maintained.

MPPEH that is inspected, verified, and certified to be free of explosive hazards will be classified as MDAS. MDAS generated during the project will be stored in a secure area inside locked and sealed containers. Once the field investigation is complete, the sealed containers will be shipped to an Army-approved facility for proper disposal in accordance with the Waste Management Plan (Appendix C) and HGL SOP 408.504 MPPEH Inspection and Management (Appendix D). Certification and disposal of MPPEH and MDAS will be monitored by the UXOQCS using the SOPs described on WS #21.

In the event that MPPEH determined to be MEC is identified, all site activities will cease and a determination on how to proceed will discussed with the onsite USACE OESS, CJAG, HGL PM, and the USACE PM for approval prior to MEC disposal operations occurring or RA activities continuing.

17.2 MC DFWs

This section describes the DFWs associated with the characterization and removal of soil contaminated with cadmium and lead at concentrations greater than their respective cleanup goals at the Group 8 MRS.

Several activities described in the DFWs below will result in the generation of field GIS/survey or laboratory analytical data. HGL will coordinate with the contractor (Leidos) that manages the CJAG RVAAP Environmental Information Management System database to ensure the unique sample nomenclature are appropriate for inclusion in the database and that the necessary data are transferred upon project completion.

17.2.1 MC DFW #1: Site Mobilization and Vegetation Clearing

Mobilization includes the movement of equipment, supplies, and personnel required for activities described in this RA Work Plan. The field management team consisting of an HGL Site Manager and UXOSO/UXOQCS will mobilize at the beginning of fieldwork and oversee all work described in this UFP-QAPP. The field management team will have already reviewed the available documentation relating to the site, the RA Work Plan, Accident Prevention Plan/Site Safety and Health Plan, and additional data obtained during previous site visits. Existing site conditions will be captured with photography and positions of features to be restored (roads, driveways) will be recorded with GPS for reference during the site restoration and demobilization phase.

Site facilities will include the excavated soil loadout area, IDW staging area, temporary storage, portable toilet, and field personnel vehicle parking area (Figure 11.1). It is anticipated that quadrant 2 (where no excavations will occur), will serve as the equipment staging area. However, if unanticipated delays occur that require roll-off containers to be staged for longer that will not fit within quadrant 2, an area north of Building 845 may be used. This option will be discussed with CJAG in advance and concurred to by CJAG, at that time. If necessary, HGL will install gravel or protection to harden the ground in areas to be used for staging. The CJAG Building 1036 Contractor General Use Building, or other suitable location as approved by OHARNG/CJAG Department of Public Works, will be used as the field office for the field mobilization.

Vegetation clearing will be conducted as needed based on actual site conditions and will include the excavated soil loadout area, equipment staging area, and the area in and around the Group 8 MRS. Historically the area around Buildings 846 and 849 has been mowed occasionally, so significant vegetation clearing during mobilization is not anticipated.

Erosion control measures will be installed prior to intrusive activities per the Environmental Protection Plan (Appendix B). A Stormwater Pollution Prevention Plan has been prepared and is included as an attachment to the Environmental Protection Plan (Appendix B). Silt fencing and/or straw wattles will be installed into portions of the drainage ditches located outside of the planned excavation area, along with other measures described in the Stormwater Pollution Prevention Plan, to prevent surface water and sediment migration beyond the MRS boundary.

On-site personnel will contact Range Control (614-336-6041) to confirm their arrival/departure from the MRS and planned activities for the day. Normal work hours will be from 0700 hours to 1600 hours on weekdays. Work on weekends or holidays must be coordinated ahead of time with the OHARNG (Katie Tait, 614-336-6136). Warehouse buildings in the Group 8 MRS area are not to be accessed by project personnel.

Utility clearance activities will follow the procedures outlined in Appendix D SOP 411.03. Prior to any excavation and/or intrusive soil sampling, CJAG's Department of Public Works will be contacted to ensure no utilities are present in the area. Additionally, HGL will obtain a private utility location service during site preparation. Due to the extensive nature of excavation activities (based on MC sampling data), broad areas of utilities clearance will be conducted before beginning any intrusive activities. In addition, onsite detectors (e.g., handheld Schonstedt GA-52Cx) may be used to identify underground utilities as work progresses. However, excavation along or near the existing overhead power lines may be required, which will require close coordination with CJAG personnel and the owner/operator of the power line. Due to the close proximity of the overhead line to excavation activities, close coordination will be required and may require de-energizing the line and securing using lockout/tagout procedures.

17.2.2 MC DFW #2: Soil Sample Grid Layout

The Group 8 MRS boundary and the 75 sample grids for each quadrant including GR8SS-001M, GR8SS-003M, and GR8SS-004M will be established after mobilization and vegetation clearing are complete (Figure 11.2). The soil sample grid layout will be setup using a centimeter-accurate GPS unit verified with a known control point. Grid locations will be marked using pin flags and/or spray marking paint. Within each quadrant, grid locations will be assigned unique identifiers in the format G8SL-001M-001 through G8SL-001M-075 (for quadrant 001M) as shown in WS #18.

17.2.3 MC DFW #3: Soil Sample Collection

Pre-excavation characterization soil samples will be collected from each grid location using a DPT drill rig equipped with a Macro Core sampler and single-use disposable liners. Samples from each soil boring will be collected from 0.5-1, 1-1.5, and 1.5-2 ft bgs depth intervals and will be analyzed for lead. Samples collected from quadrant G8SS-004M will be analyzed for cadmium and lead (WS #15.1). Multiple pushes to 2 ft bgs may be required to collect the necessary soil sample volume if sample recovery is limited due to gravel/debris or for field duplicate (FD) and MS/MSD samples. Soil sample numbers will be assigned as described in WS #18 to distinguish between each quadrant, grid location, required depth intervals, and field QC sample type.

Only samples from the 0.5-1.0 ft bgs interval will initially be analyzed and evaluated for exceedances above the PALs for cadmium and lead. An exceedance will trigger the analysis of the next deepest depth interval for that grid point. All pre-excavation samples will be held in cold storage at SGS Orlando until the lab is directed to analyze the sample or dispose of it.

Once the delineation of PAL exceedances for lead and cadmium is complete and an excavation footprint defined, waste characterization samples will be collected using a stainless-steel hand auger. The number of samples will depend on the excavation volume. Composite samples will be collected from areas equivalent to five roll-offs and analyzed for parameters listed in WS #15.12. Waste characterization sampling will be detailed further in the pre-excavation sampling report.

Soil descriptions and notes regarding field observations will be documented on a soil boring log form (ENG Form 1836 or equivalent). Soil sampling will be documented on a Field Sampling Report. Excess soil cuttings generated during soil sampling will be containerized and managed as IDW. The Macro Core sampler will be decontaminated prior to use at each distinct grid location using a detergent/potable water wash and potable water rinse. A new disposable liner also will be used at each grid location. Waste decontamination water will be containerized and managed as IDW.

A Pre-Excavation Report will be developed in the form of a Data Usability Assessment following the analysis of pre-excavation and waste characterization sampling. The report will be submitted to all members of the PDT (ARNG, USACE, OHARNG, and Ohio EPA) for review, comment, and acceptance. The report will include a description of the vertical and horizontal limits of excavation. Excavation limits will be designed to remove all contaminated soils as defined by the PALs. In addition, the results of the backfill and topsoil sampling will be presented for review and approval. Any additional logistical considerations needed to perform the work (phasing, erosion control, compaction methods, etc.) will be included in the Pre-Excavation Report. Following the Pre-Excavation Report, subsequent analytical results of post-excavation confirmation sampling and/or backfill concurrence requests will be submitted in Field Change Requests which will be reviewed and approved by all members of the PDT (ARNG, USACE, OHARNG, and Ohio EPA).

Although the presence of MEC is not anticipated, UXO-qualified personnel will conduct an anomaly avoidance survey at each soil sampling location to identify potential MEC or MPPEH items on the surface or in the subsurface prior to starting intrusive work. The search will be conducted visually and using a handheld analog magnetometer or electromagnetic detector (such as the Schonstedt GA52-CX, White's All Metals Detector, Garrett AT Max, or similar instruments). An analog handheld magnetometer is capable of identifying potential MEC or MPPEH at a depth of up to 4 ft bgs, although the detection capability decreases with increasing depth and decreasing object size. If a metallic anomaly is identified, then the soil sample location will be moved as needed within the associated grid cell to a cleared location.

17.2.4 MC DFW #4: Soil Excavation, Confirmation Sampling, and Backfill

After the initial surface MPPEH removal is complete for the identified excavation areas a construction crew will be mobilized to conduct excavation to remove soil with cadmium and lead at concentrations greater than their respective cleanup goals. Excavation will be conducted using a D6 dozer, excavator, and front-end loader to remove contaminated soil. Results of the pre-excavation waste characterization soil sampling will be used to identify distinct excavation areas with RCRA hazardous and/or non-hazardous soil (as applicable) to allow for segregation of the material during excavation and loadout. Should dewatering be necessary due to precipitation, water will be collected from a temporary pit dug to collect water within the excavation via a sump pump to a drum or larger polyurethane tank. Due to the shallow nature of the excavation, groundwater infiltration is not anticipated. Excavated soil will be loaded directly into roll-off boxes at the excavation site to minimize the need for double-handling and staging in stockpiles. Additionally metallic screens will be placed on top of the roll-off containers. These screens will be 1 inch by 1 inch in diameter and will be used to capture any metallic debris remaining in the subsurface soils. All metallic debris will be inspected as MPPEH until it is determined to be MEC or MDAS in accordance with Section 17.1.5 above.

Once the contaminated soil has been removed from an excavation area, discrete confirmation samples will be collected from the sidewalls of the outer boundary of the excavation. Sidewall confirmation samples will be collected at a frequency of one per 20 ft (equivalent to a grid length). Sidewall samples will not be collected where excavations extend to building foundations. Soil will be collected from the midpoint of the grid edge and along the complete vertical height of the sidewall. If the horizontal extent of contamination has not been defined at grid locations along the outer boundaries of the quadrants via sidewall sampling, then HGL will coordinate with USACE to determine if additional soil sampling is required.

Confirmation samples will be collected from the excavation floor at grid locations that exceeded cleanup goals in the 1.5-2.0 ft bgs increment. A discrete sample will be collected in the center of the 400 square-foot grid location from the upper six inches of the excavation floor and sent for lead and/or cadmium analysis. If the cadmium and/or lead result(s) from an excavation floor confirmation sample exceed their respective cleanup goals, then an additional six-inch increment will be sampled and analyzed for the applicable metals. This iterative process will continue until vertical extent of contamination in each quadrant has been defined.

Confirmation samples will be analyzed under short turnaround to minimize the duration of an open excavation. Preliminary results will be used to determine whether an area can be backfilled. Phasing of excavation areas and confirmation sampling will be detailed in the Pre-Excavation Report. Confirmation results will be presented in Ravenna Field Change Request Form (Appendix E) and submitted for review by USACE, OHARNG, and Ohio EPA for authorization to backfill. Clean backfill soil will be placed in the excavation and compacted using the excavator bucket.

After soil excavation and backfill activities are completed, six inches of topsoil will be placed across the excavated areas to prevent surface water ponding and support vegetation growth. The final grade elevation will match the original grade and include existing drainage features. The topsoil area will be seeded and stabilized and impacted portions of the pre-existing gravel roads will be replaced to match existing grade. Erosion control measures identified in the Environmental Protection Plan (Appendix B) will be installed and maintained until sufficient vegetation growth is achieved.

Characterization samples will be collected from the backfill material at the source at a frequency of 1 sample per 4,000 cy of material. The actual quantity of backfill material will not be known until the excavation dimensions are confirmed after the pre-excavation soil characterization described in DFW #3. Backfill material subject to sampling at the source area may include in situ material (i.e., not yet excavated) or previously excavated and stockpiled material. Backfill soil samples will be collected using a stainless-steel hand auger, soil probe, etc. as applicable to actual conditions. Backfill characterization soil samples will be analyzed for the parameters listed in WS 15.2 through 15.10. If existing laboratory analytical results are available for the backfill material, then the results will be reviewed to confirm they are of definitive data quality and meet the requirements for government approval (in lieu of conducting characterization sampling).

17.2.5 MC DFW #5: Waste Characterization and Disposal

The following types of waste are anticipated during on-site activities:

- Waste soil (excavated and soil sampling cuttings)
- Waste water (decontamination and excavation dewatering)
- Municipal trash (expended personal protective equipment, general trash)

Pre-characterized waste soil generated during excavation, and excess soil cuttings generated during pre-excavation soil sampling activities, will be transported for off-site disposal at one or more appropriately licensed facilities. A truck scale will be utilized to maximize the volume of soil in the roll-offs relative to the road weight limit.

If disposal of soil cuttings is required prior to the start of excavation activities, then one composite soil sample will be collected and analyzed for the full suite of RCRA parameters listed in WS #15.11.

Waste water generated during soil sampling and excavation activities, and possibly excavation dewatering, will be containerized in 55-gallon drums and/or larger poly tanks, as necessary per the actual volume of water. If drums are used, a single composite sample will include aliquots from up to 10 drums. If one or more poly tanks are used, a single composite sample will be collected from a total volume of no more than 500 gallons. Waste characterization water samples will be analyzed for the full suite of RCRA parameters listed in WS #15.11.

Each disposal facility receiving waste soil or water will provide HGL with return manifests, and weight tickets for each load received and processed. Municipal trash will include expended personal protective equipment and general waste not impacted by site contaminants, and will disposed daily at a dumpster procured by HGL. Original manifests, return manifests, and weight tickets will be turned in to OHARNG at the end of the project. Wastes will be tracked using the form included in the Waste Management Plan (Appendix C). OHARNG will sign all profiles and manifests. Attachment C1 of the Waste Management Plan includes all manifest preparation instructions including point-of-contact, required signatures, and approval authorities.

17.2.6 MC DFW #6: Site Restoration and Demobilization

All equipment utilized during soil sampling, soil excavation, and site restoration activities will be demobilized from the Group 8 MRS as soon as practicable. Before any equipment is removed from the MRS, it will be cleaned/decontaminated to ensure that no contaminants will be accidentally transported to areas outside the MRS. Roads and driveways will be restored to original condition per photos and GPS collected during the site preparation phase. Railroad base does not need to be restored using ballast, but as former railroad beds are currently used for driving access, they will be restored, if impacted, to pre-RA conditions as described above for roads and driveways. Waste located in the applicable staging area will be transported for off-site disposal as soon as possible after characterization and transportation documentation is complete.

Following soil removal, the area previously cleared of vegetation will be seeded with native seed mixes approved by the OHARNG. Per CJAG preferred seed types in regularly mowed areas, the final restoration will utilize a seed mixture that contains 40% Kentucky bluegrass (*Poa pratensis*), 30% Perennial ryegrass (*Lolium perenne*), 20% Hard fescue (*Festuca brevipila*), and 10% Creeping red fescue (*Festuca rubra*). The mixture will be broadcast at a ratio of 18 lbs per acre.

Prior to seeding, the topsoil will be tilled to provide a loose and suitable seed bed for germination. Two inches of straw mulch will be added as cover following the seeding. Seeding will occur following backfill, regardless of the time of year; however, it is recognized that grass seeding is most effective in the late-summer months. Therefore, revegetation will be monitored and supplemented as needed until coverage reaches 70%. Once revegetation coverage criteria are met, erosion controls will be removed from the site.

WORKSHEET #18
SAMPLING DESIGN AND RATIONALE

Sample Location	Sample Designation	Sample Depth (ft bgs)	QA/QC Sample Designation		Analytical Parameters
			FD	MS/MSD	
Pre-Excavation Soil - Excavation Area 001M					
G8SL-001M-A1	G8SL-001M-A1-1.0-REG	0.5 – 1	G8SL-001M-A1-1.0-FD	G8SL-001M-A1-1.0-MS/MSD	Pb
	G8SL-001M-A1-1.5-REG	1.0 – 1.5	G8SL-001M-A1-1.5-FD	G8SL-001M-A1-1.5-MS/MSD	
	G8SL-001M-A1-2.0-REG	1.5 – 2.0	G8SL-001M-A1-2.0-FD	G8SL-001M-A1-2.0-MS/MSD	
G8SL-001M-A2	G8SL-001M-A2-1.0-REG	0.5 – 1			
	G8SL-001M-A2-1.5-REG	1.0 – 1.5			
	G8SL-001M-A2-2.0-REG	1.5 – 2.0			
G8SL-001M-A3 thru G8SL-001M-O5	Same format and depths as above		Collected as needed to maintain frequency requirements per regular samples for FD (1 per 10) and MS/MSD (1 per 20)		Pb
Pre-Excavation Soil - Excavation Area 003M					
G8SL-003M-A6	G8SL-003M-001-1.0-REG	0.5 – 1.0	G8SL-003M-A6-1.0-FD	G8SL-003M-A6-1.0-MS/MSD	Pb
	G8SL-003M-001-1.5-REG	1.0 – 1.5	G8SL-003M-A6-1.5-FD	G8SL-003M-A6-1.5-MS/MSD	
	G8SL-003M-001-2.0-REG	1.5 – 2.0	G8SL-003M-A6-2.0-FD	G8SL-003M-A6-2.0-MS/MSD	
G8SL-003M-A7	G8SL-003M-002-1.0-REG	0.5 – 1.0			
	G8SL-003M-002-1.5-REG	1.0 – 1.5			
	G8SL-003M-002-2.0-REG	1.5 – 2.0			
G8SL-003M-A8 thru G8SL-003M-O10	Same format and depths as above		Collected as needed to maintain frequency requirements per regular samples for FD (1 per 10) and MS/MSD (1 per 20)		Pb
Pre-Excavation Soil - Excavation Area 004M					
G8SL-004M-P6	G8SL-004M-P6-1.0-REG	0.5 – 1.0	G8SL-004M-P6-1.0-FD	G8SL-004M-P6-1.0-MS/MSD	Cd/Pb
	G8SL-004M-P6-1.5-REG	1.0 – 1.5	G8SL-004M-P6-1.5-FD	G8SL-004M-P6-1.5-MS/MSD	
	G8SL-004M-P6-2.0-REG	1.5 – 2.0	G8SL-004M-P6-2.0-FD	G8SL-004M-P6-2.0-MS/MSD	
G8SL-004-P7	G8SL-004M-P7-1.0-REG	0.5 – 1.0			
	G8SL-004M-P7-1.5-REG	1.0 – 1.5			
	G8SL-004M-P7-2.0-REG	1.5 – 2.0			
G8SL-004M-P8 thru G8SL-004M-AD10	Same format and depths as above		Collected as needed to maintain frequency requirements per regular samples for FD (1 per 10) and MS/MSD (1 per 20)		Cd/Pb

WORKSHEET #18 (CONTINUED)
SAMPLING DESIGN AND RATIONALE

Sample Location	Sample Designation	Sample Depth (ft bgs)	QA/QC Sample Designation		Analytical Parameters
			FD	MS/MSD	
Confirmation Samples (as needed from excavation floor)					
G8SL-001M-A1	G8SL-001M-A1-2.5-REG	2.0-2.5	G8SL-001M-A1-2.5-FD	G8SL-001M-A1-2.5-MS/MSD	Cd/Pb
G8SL-001M-A2 thru G8SL-004M-AD10	Same format and depths as above		Collected as needed to maintain frequency requirements per regular samples for FD (1 per 10) and MS/MSD (1 per 20)		Cd/Pb
Confirmation Samples from Sidewalls					
G8SL-001M-A1	G8SL-001M-A1-SWN-REG	Entire height of sidewall	G8SL-001M-A1-SWN-FD	G8SL-001M-A1-SWN-MS/MSD	Cd/Pb
G8SL-001M-A1	G8SL-001M-A1-SWE-REG	Entire height of sidewall	G8SL-001M-A1-SWN-FD	G8SL-001M-A1-SWN-MS/MSD	Cd/Pb
G8SL-001M-A2 thru G8SL-004M-AD10	Same format and depths as above		Collected as needed to maintain frequency requirements per regular samples for FD (1 per 10) and MS/MSD (1 per 20)		Cd/Pb
Backfill Soil Characterization					
G8SL-BKFL-001	G8SL-BKFL-001-REG	Not Applicable	Not Applicable		See WS 15
G8SL-BKFL-002	G8SL-BKFL-002-REG				
G8SL-BKFL-003	G8SL-BKFL-003-REG				
Waste Soil Disposal Characterization					
G8WS-WAC-001	G8SL-WAC-001-REG	Varies per Excavation Lift	Not Applicable		Full Suite WAC[1]
G8WS-WAC-002	G8SL-WAC-002-REG				
G8WS-WAC-003	G8SL-WAC-003-REG				
G8WS-WAC-004	G8SL-WAC-004-REG				
G8WS-WAC-005	G8SL-WAC-005-REG				
G8WS-WAC-006	G8SL-WAC-006-REG				
Waste Water Disposal Characterization					
G8WW-WAC-001	G8WA-WAC-001-REG	Not Applicable	Not Applicable		Full Suite WAC[1]
G8WW-WAC-002	G8WA-WAC-002-REG				
G8WW-WAC-003	G8WA-WAC-003-REG				

* The actual number of pre-excavation soil, confirmation, waste disposal, and backfill soil characterization samples may vary depending on field conditions

[1] The list of full suite waste acceptance criteria (WAC) analytical parameters is provided in WS #12.8

Cd = cadmium

Pb = lead

WORKSHEETS #19 AND #30
SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

The following table includes all analytical methods that are presented in WS #23 and discussed in more detail in WS #12 and WS #15.

Matrix	Parameter	Analytical/Preparation Method (SOP Reference)	Containers	Preservation Requirements	Maximum Holding Time
Soil	VOCs	SW8260D / MS020.2 and OP041.10	a) 3 x 40-mL glass vials, Teflon septum b) 3 x 40-mL glass vials, Teflon septum c) 3 x 40-mL glass vials, Teflon septum d) Sealed syringe-type corer (Encore® or Terracore® equivalent)	a) Cool ≤6°C; $NaHSO_4$ to pH ≤2 b) Cool ≤6°C, methanol c) Cool ≤6°C, deionized water d) Cool ≤6°C	a) 14 days b) 14 days c) 48 hours; must be frozen within 48 hours d) 48 hours; must be frozen within 48 hours
	SVOCs	SW8270E and SW1311 / MS021.2 and OP040.10	1 x 8-oz amber glass jar with HDPE-lined lid	Cool ≤6°C protected from light	14 days to extraction / 40 days from extraction to analysis
	Herbicides	SW8151A and SW1311 / GC031.11 and OP040.10	1 x 4-oz amber glass jar with HDPE-lined lid	Cool ≤6°C protected from light	14 days to extraction / 40 days from extraction to analysis
	Pesticides	W8081B and SW1311 / GC015.16 and OP040.10	1 x 4-oz amber glass jar with HDPE-lined lid	Cool ≤6°C protected from light	14 days to extraction / 40 days from extraction to analysis
	PCB	SW8082A / GC014.16	1 x 4-oz amber glass jar with HDPE-lined lid	Cool ≤6°C protected from light	14 days to extraction / 40 days from extraction to analysis
	Explosives	SW8330B / GC034.10	1 x 8-oz amber glass jar with HDPE-lined lid	Cool ≤6°C protected from light	14 days to extraction / 40 days from extraction to analysis
	Metals	MET107.04/MET108.05	4oz or 8oz glass jar with PTFE Lid	Cool ≤6°C	6 months
	Mercury	MET105.13	4oz or 8oz glass jar with PTFE Lid	Cool ≤6°C	28 days

WORKSHEETS #19 AND #30 (CONTINUED)
SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

Matrix	Parameter	Analytical/Preparation Method (SOP Reference)	Containers	Preservation Requirements	Maximum Holding Time
Waste Soil[1]	VOCs	SW8260D and SW1311 / MS020.2 and OP041.10	1 x 300 mL glass jar; Teflon-lined lid	Cool ≤6°C; zero headspace; protected from light	14 days to leach / 14 days from leaching to analysis
	SVOCs	SW8270E and SW1311 / MS021.2 and OP040.10	1 x 300 mL glass jar; Teflon-lined lid	Cool ≤6°C; protected from light	14 days to leach / 7 days to extraction/ 40 days from extraction to analysis
	Pesticides	SW8081B and SW1311 / GC015.16 and OP040.10	1 x 300 mL glass jar; Teflon-lined lid	Cool ≤6°C; protected from light	14 days to leach / 7 days to extraction/ 40 days from extraction to analysis
	Herbicides	SW8151A and SW1311 / GC031.11 and OP040.10	1 x 300 mL glass jar; Teflon-lined lid	Cool ≤6°C; protected from light	14 days to leach / 7 days to extraction/ 40 days from extraction to analysis
	PCBs	SW8082A / GC014.16	1 x 4-oz amber glass jar with HDPE-lined lid	Cool ≤6°C protected from light	14 days to extraction / 40 days from extraction to analysis
	Metals	SW6020A, SW7470A, and SW1311 / MET107.04, MET 106.13, and OP040.10	1 x 300 mL glass jar; Teflon-lined lid	Cool ≤6°C; protected from light	6 months (Metals) 28 days (Mercury)
	Reactivity	SW846 Chapter 7.3 / GN113.12	1 x 4-oz glass jar with HDPE-lined lid	Cool ≤6°C; protected from light	14 days
	Sulfide	SM4500S F-2000 / GN140.16	3 x 250-mL HDPE	Cool ≤6°C; NaOH and zinc acetate to pH>9	7 days
	Ignitability	SW1010A MOD / GN121.13	1 x 4-oz glass jar with HDPE-lined lid	Cool ≤6°C; protected from light	14 days
	Corrosivity (pH)	SW9045D / GN137.16	1 x 4-oz glass jar with HDPE-lined lid	Cool ≤6°C	7 days
	Paint Filter	SW9095B / GN127.08	1 x 4-oz glass jar with HDPE-lined lid	NA	NA

WORKSHEETS #19 AND #30 (CONTINUED)
SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

Matrix	Parameter	Analytical/Preparation Method (SOP Reference)	Containers	Preservation Requirements	Maximum Holding Time
Waste Water	VOCs	SW8260D and SW1311 / MS020.2 and OP041.10	1 x 1-L amber glass bottles, Teflon septum	Cool ≤6°C; zero headspace; protected from light	14 days to leach / 14 days from leaching to analysis
	SVOCs	SW8270E and SW1311 / MS021.2 and OP040.10	1 x 1-L amber glass	Cool ≤6°C; protected from light	14 days to leach / 7 days to extraction / 40 days from extraction to analysis
	Pesticides	SW8081B and SW1311 / GC015.15 and OP040.10	1 x 1-L amber glass	Cool ≤6°C; protected from light	14 days to leach / 7 days to extraction / 40 days from extraction to analysis
	Herbicides	SW8151A and SW1311 / GC031.11 and OP040.10	1 x 1-L amber glass	Cool ≤6°C; protected from light	14 days to leach / 7 days to extraction / 40 days from extraction to analysis
	RCRA Metals	SW6020A, SW7470A, and SW1311 / MET107.04, MET 106.13, and OP040.10	1 x 1-L HDPE bottles	Cool ≤6°C; protected from light	6 months (Metals) 28 days (Mercury)
	PCBs	SW8082A / GC014.16	2 x 1-L or 2 x 250 mL amber glass, Teflon septum	Cool ≤6°C protect from light	7 days to extraction / 40 days from extraction to analysis
	Sulfide	SM4500S F-2000 / GN140.16	3 x 250-mL HDPE	Cool ≤6°C; NaOH and zinc acetate to pH>9	7 days
	Flash Point	SW1010A / GN121.13	1 x 250-mL glass bottle	Cool ≤6°C	24 hours
	Corrosivity (pH)	SM4500H+ B-2000 / GN137.16	1 x 250-mL HDPE	Cool ≤6°C	24 hours

[1] TCLP extracts will be containerized, preserved, and stored in accordance with the laboratory's quality manual and the associated preparation SOPs.

HDPE = high-density polyethylene
L = liter
mL = milliliter
$NaHSO_4$ = sodium bisulfate
NaOH = sodium hydroxide
oz = ounce
PTFE = polytetrafluoroethylene

WORKSHEET #20
FIELD QUALITY CONTROL SUMMARY

This worksheet summarizes the types and estimated quantities of field QC samples required for the project including the following:

- Field duplicate – “FD” appended to the associated regular Sample ID
- Matrix spike – “MS” appended to the associated regular Sample ID
- Matrix spike duplicate – “MSD” appended to the associated regular Sample ID

During pre-excavation soil and backfill soil characterization sampling, field duplicate samples will be collected at a rate of approximately 1 per 10 regular field samples and MS/MSD pairs will be collected at a rate of approximately 1 per 20 regular field samples. Field duplicate samples will be collected from a separate advancement of the split spoon or auger, immediately adjacent to the parent sample to preserve the independence of the duplicate sample. Field duplicates will receive a dedicated sample time. MS/MSD samples can be collected from homogenized increments within the same split spoon as the parent sample. MS/MSD samples will share the sample time with the parent sample.

WORKSHEET #20 (CONTINUED)
FIELD QUALITY CONTROL SUMMARY

Activity	Matrix	Analytical Parameter	Regular Samples	FD	MS	MSD	TB	EB	FB	Total Analyses
Pre-Excavation Soil (0.5-1.0 ft Interval)[1]	Soil	Metals (Cd/Pb)	225	23	12	12	NA	NA	NA	272
Pre-Excavation Soil (1.0-1.5 ft Interval)[2]	Soil	Metals (Cd/Pb)	225	23	12	12	NA	NA	NA	272
Pre-Excavation Soil (1.5-2.0 ft Interval)[2]	Soil	Metals (Cd/Pb)	225	23	12	12	NA	NA	NA	272
Confirmation Soil[3]	Soil	Metals (Cd/Pb)	TBD	TBD	TBD	TBD	NA	NA	NA	TBD
Field Blank (Potable Water Source)	Water	Full Suite (See WS 15)	NA	NA	NA	NA	TBD	NA	1	1
Backfill Soil[4]	Soil	Full Suite (See WS 15)	TBD	NA	NA	NA	TBD	NA	NA	TBD
Waste Soil[5]	Soil	Metals, SVOCs, PCBs (see WS 15)	6	NA	NA	NA	NA	NA	NA	6
Waste Water[5]	Water	Metals, SVOCs, PCBs (see WS 15)	3	NA	NA	NA	NA	NA	NA	3

[1] Indicates initial default sampling of 0.5-1.0 ft depth interval from the three excavation areas.

[2] Samples from the 1.0-1.5, and 1.5-2.0 ft depth intervals will be held at the laboratory and may be analyzed pending evaluation of initial samples results in order to horizontally and vertically bound the extent of cadmium and lead contamination.

[3] Total number of confirmation samples will be dependent on number of grids excavated to 2.0 ft bgs.

[4] Total number of backfill soil characterization samples may vary based on volume of backfill soil needed (1 sample per 4,000 cy).

[5] Total number of waste characterization samples estimated, actual number may vary based on field conditions.

Field QC Sample Collection Frequencies:

FD = 1 per 10 regular samples
MS/MSD = 1 per 20 regular samples
TB = one per cooler containing VOC samples.
EB = equipment blank
FB = field blank

WORKSHEET #21
FIELD SOP REFERENCES

SOP Reference	Title and Revision Date	Originating Organization	Equipment Type	Modified for Project Work?	Comments
General Site Activities					
H&S 12.1	Control of Hazardous Energy Program – Lockout/Tagout Procedure (Revision 6, June 2020)	HGL	Lockout tags and locks	No	Applicable when overhead lines need to de-energized during heavy equipment operation
201.505	Hazard Communication (Revision 9, 9/26/22)	HGL	Container labels	No	Applicable to managing soils, chemicals, and waste.
201.512	Control of Hazardous Energy (Revision 7, 9/27/21)	HGL	Lockout tags and locks	No	Applicable when overhead lines need to de-energized during heavy equipment operation
401.501	Field Logbook Use and Maintenance (Revision 4, 3/21/22)	HGL	Field logbook	No	Applicable to all field activities
300.07	Environmental Database Quality Control (Revision 3, 12/21/20)	HGL	Electronic field and laboratory data	No	Applicable to field GIS and laboratory data
411.03	Subsurface Utility Avoidance (Revision 3, 9/29/20)	HGL	TBD	No	CJAG requirements for intrusive sampling up to 2 ft depth and potentially deeper excavations
501.06	Vegetation Removal on MMRP Sites (Revision 2, 12/29/21)	HGL	TBD	No	Pre-sampling and excavation.
MPPEH Locate, Inspection, and Removal					
501.08	MEC Construction Support (Revision 1, 12/16/20)	HGL	Analog Detector	No	Applicable to field activities requiring UXO avoidance.
408.507	MEC Anomaly Avoidance Support (Revision 3, 9/13/22)	HGL	Analog detector	No	Applicable to field activities requiring UXO avoidance.
501.05	Analog and Digital MEC Operations (Revision 2, 10/16/19)	HGL	Analog Detector	No	Applicable to field activities requiring MPPEH subsurface removal.
408.504	MPPEH Inspection and Management (Revision 6, 5/29/22)	HGL	TBD	No	Applicable to field activities requiring MPPEH handing and inspections.

WORKSHEET #21 (CONTINUED)
FIELD SOP REFERENCES

SOP Reference	Title and Revision Date	Originating Organization	Equipment Type	Modified for Project Work?	Comments
MC Contaminated Soil Characterization and Removal					
201.522	Excavation and Trenching (Revision 9, 4/6/22)	HGL	Augers, shovels, excavation equipment	No	Applicable to sampling and excavation.
201.528	Drilling Safety (Revision 6, 4/29/22)	HGL	DPT drill rig	No	Applicable to sampling
201.531	Forklifts and Earthmoving Equipment (Revision 6, 5/3/22)	HGL	Excavators and similar earthmoving equipment	No	Applicable to soil removal and restoration.
403.02	Hand-Operated Auger Soil Sampling (Revision 2, 8/1/19)	HGL	Hand auger soil sampling	No	Applicable to pre-excavation waste characterization soil sampling
403.04	Direct-Push Technology Soil Sampling (Revision 3, 6/18/20)	HGL	DPT Macro Core soil sampler with disposable liner	No	Applicable to pre-excavation soil sampling
411.02	Sampling Equipment Cleaning and Decontamination (Revision 5, 6/18/20)	HGL	Decontamination liquids, potable water, deionized water, Alconox, brushes, tubs, etc.	No	Applicable to non-dedicated stainless-steel sampling equipment
411.03	Subsurface Utility Avoidance (Revision 3, 9/29/20)	HGL	Checklists and records	No	Applicable to excavation

WORKSHEET #22
EQUIPMENT TESTING, INSPECTION, AND QUALITY CONTROL
(UFP-QAPP MANUAL SECTION 3.1.2.4)

Table 22.1 MPPEH Locate, Inspection, and Removal

Measurement Quality Objective	SOP Reference	Frequency	Responsible Person/ Report Method/ Verified by	Acceptance Criteria	CA
Construct ITS: Verify as-built ITS against design plan (Analog sensors)	502.2	Once following ITS construction	UXOQCS/ DQCR and Access Database/ Lead Organization	Small industry standard object seed items for analog methods buried at 30 centimeters; All seeds buried horizontally in the cross-track orientation	RCA/CA: Make necessary changes to seeded items and re-verify
Ongoing Instrument Function Test (Analog)	501.05	Beginning and end of each day and each time instrument is turned on	Field Team Leader/ Running QC Summary/ Project/QC Geophysicist or designee	Audible response consistent with expected change in tone in presence of standard object	RCA/CA: Make necessary repairs and re-verify
Documenting recovered sources (All sensors)	501.05	Daily	UXOQCS/ GIS data recorded/ QC Geophysicist	All metallic debris collected is documented for the following attributes: Designation as UXO, MD, range-related debris, or other debris; UXO and MD described by type, weight, depth, and as TOI or non-TOI. Photos displaying all MD recovered (individual MD photos not necessary) and photos showing all surfaces of each MEC are recorded.	RCA/CA; document questionable information in database

RCA = Root Cause Analysis
TOI = target of interest

WORKSHEET #23
ANALYTICAL SOP REFERENCES

Lab SOP Number	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix and Analytical Group	SOP Options or Equipment Type	Modified for Project Work? (Y/N)
MS 020.2	Analysis of volatile organics by GC/MS Rev. Date: 12/2020	Definitive	VOCs (SO and AQ)	GC	No
MS 021.2	Analysis of semi-volatile organics by GC/MS 8270E Rev. Date: 10/2020	Definitive	SVOCs (SO and AQ)	GC/MS	No
GC.015.16	Analysis of Organophosphorus Pesticides by Gas Chromatography Electron Capture Detector, Rev Date: 12/2020	Definitive	Organophosphorus Pesticides (SO and AQ)	GC	No
GC 014.16	Analysis of polychlorinated biphenyls by Gas Chromatography, electron capture detector Rev. Date: 12/2020	Definitive	Pesticide and PCBs (SO and AQ)	GC	No
GC 031.11	Analysis of chlorinated herbicides by Gas chromatography, electron capture detector Rev Date: 12/2020	Definitive	Herbicides (SO and AQ)	GC	No
GC034.10	Analysis of Nitroaromatics, Nitramines, and Nitrate Esters by HPLC Method SW-846 8330B; 01/2021	Definitive	Water and Solids / Explosives	HPLC	No
MET 108.05	Metals by Inductively Coupled Plasma Atomic Emission Spectrometry 6010D, Rev. Date: 12/2020	Definitive	Metals (SO and AQ)	ICP-AES	No
MET 107.04	Metals by Inductively Coupled Plasma – Mass Spectrometry Rev. Date: 12/2020	Definitive	Metals (SO and AQ)	ICP-MS	No
MET 105.13	Cold Vapor Analysis of Mercury for Soils, Rev. Date: 12/2020	Definitive	Mercury (SO)	CVAA	No
MET 106.13	Cold Vapor Analysis of Mercury for Water Samples, Rev. Date: 12/2020	Definitive	Mercury (AQ)	CVAA	No
OP040.10	Standard Operating Procedure for The Toxicity Characteristic Leaching of Semi-volatile Organics and Metals (TCLP), Rev. Date: 09/2020	Preparation	Preparation (SO)	NA	No
OP041.10	Standard Operating Procedure for The Toxicity Characteristic Leaching of Volatile Organics (TCLP), Rev. Date: 09/2020	Preparation	Preparation (SO)	NA	No

WORKSHEET #23 (CONTINUED)
ANALYTICAL SOP REFERENCES

Lab SOP Number	Title, Date, and URL (if available)	Definitive or Screening Data	Matrix and Analytical Group	SOP Options or Equipment Type	Modified for Project Work? (Y/N)
GN 113.12	Micro Distillation of Cyanide in Aqueous and Solid Matrices, Rev. Date: 08/2016	Definitive	Cyanide (SO and AQ)	Lachat	No
GN 140.17	Standard operating procedure for Sulfide Rev. Date: 09/2021	Screening	Sulfide (SO and AQ)	Titrator	No
GN 137.16	pH by Electrode Rev. Date: 05/2020	Definitive	pH (SO and AQ)	pH meter	No
GN 127.08	Standard Operating Procedure for Paint filter Rev. Date: 08/2016	Screening	Liquids and water waste	NA	No
GN 121.13	Standard Operating Procedure for Ignitability (Pensky Martens) Rev. Date 10/2018	Screening	Flash point (SO and AQ)	Pensky-Martens	No
GN 161.13	Percent Solids Rev. Date 09/2015	Screening	Percent Solids (SO)	balances	No

AQ = aqueous
CVAA = cold vapor atomic absorption
GC/MS = gas chromatography/mass spectrometry
ICP-AES = inductively coupled plasma-atomic emission spectroscopy
ICP-MS = inductively coupled plasma-mass spectrometry
SO = soil
URL = Uniform Resource Locator

WORKSHEET #24.1
ANALYTICAL INSTRUMENT CALIBRATION - ORGANIC METHODS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Method Reference
GC/MS	Five-point initial calibration for all target analytes (six points required for curve)	Initially; thereafter, as the continuing calibration fails	Target analyte evaluation: r^2 ≥0.990 or %RSD ≤15% for each analyte	1) Evaluate system 2) Recalibrate as necessary	8260D, 8270E
			Target analyte minimum mean relative response factor requirement met.	1) Evaluate system 2) Recalibrate as necessary	8270E
	ICV (must be from a second source)	Following initial calibration	%R = 80 to 120%	1) Evaluate system 2) Recalibrate as necessary	8260D, 8270E
	Instrument tuning	Every 12 hours; marks the beginning of an analytical sequence	Ion peaks meet method requirements	1) Halt analytical sequence 2) Evaluate system 3) Retune and recalibrate as necessary	8260D, 8270E
	CCV	Every 12 hours, after instrument tune	RRT within ±0.06 RRT units for each analyte and surrogate (it is acceptable to update RRT windows using the CCV to account for minor fluctuations or after routine instrument maintenance) Each target compound %D ≤20% Internal standard retention time within ±30 seconds and peak area within 50-200% of retention time and peak area in the midpoint standard of the corresponding initial calibration	1) Evaluate system 2) Clean system 3) Recalibrate if necessary 4) Reanalyze affected samples since the last in-control CCV	8260D, 8270E
			Target analyte minimum relative response factor requirement met.	1) Evaluate system 2) Recalibrate as necessary	8270E

WORKSHEET #24.1 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION -ORGANIC METHODS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Method Reference
GC and HPLC	Five-point initial calibration for all target analytes (six points required for curve)	Initial calibration prior to sample analysis. Perform instrument re-calibration once per year minimum.	%RSD of calibration factor for each analyte ≤method-specific maxima linear – $r^2 > 0.99$	1) Evaluate system 2) Recalibrate as necessary	8081B, 8082A, 8151A, 8330B
	ICV (must be from a second source)	Immediately following initial calibration	Each target compound %D ≤method-specific maxima	1) Evaluate system 2) Recalibrate as necessary	8081B, 8082A, 8151A, 8330B
	Endrin/DDT breakdown check (Method 8081B only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤ 15% for both 4,4′-DDT and endrin	1) Evaluate system 2) Clean system 3) Reanalyze breakdown check.	8081B
	Retention time verification	Update at start of run or daily	All standards within retention time window	1) Correct problem 2) Re-analyze all samples analyzed since the last retention time check	8081B, 8082A, 8151A, 8330B
	CCV	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	Each target compound %D ≤method-specific maxima	1) Evaluate system 2) Clean system 3) Reanalyze affected samples since the last in-control CCV	8081B, 8082A, 8151A, 8330B

%D = percent difference
%RSD = percent relative standard deviation
CCV = continuing calibration verification
ICV = initial calibration verification
r^2 = correlation coefficient
RRT = relative retention time

WORKSHEET #24.2
ANALYTICAL INSTRUMENT CALIBRATION - INORGANIC METHODS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Method Reference[2]
ICP-AES/ ICP-MS	Instrument tuning (ICP-MS only)	Prior to initial calibration	Mass calibration ≤0.1 amu from the true value; resolution <0.9 amu full width at 10% peak height; %RSD ≤5% for at least four replicate analyses.	1) Retune instrument 2) Reanalyze tuning solution	6020A
	Initial Calibration	Daily, minimum of one blank and one standard	None	NA	6020A, 6010D
	Initial Calibration	Daily, optional multipoint calibration	$r^2 \geq 0.995$	1) Evaluate system 2) Recalibrate	6020A, 6010D
	ICV; must be from a second source	Following initial calibration	%R = 90% to 110%	1) Evaluate system 2) Recalibrate as necessary	6020A, 6010D
	Low-level calibration check (at LOQ)	Daily, if multipoint calibration not performed	%R = 80% to 120%	1) Evaluate system 2) Recalibrate as necessary	6020A, 6010D
	CCV and low-level CCV	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	CCV: All analytes within 10% of expected value and %RSD of replicate integrations <5% Low-level CCV: All analytes within 30% of expected value and %RSD of replicate integrations <5%	1) Repeat calibration 2) Reanalyze all samples since last successful CCV	6020A, 6010D
	ICB/CCB	After every ICV/CCV	No analytes detected ≥1/2 LOQ or 1/10 the amount measured in any sample; no analytes with negative drift with absolute value >1/2 LOQ	1) Correct problem 2) Analyze calibration blank and previous 10 samples	6020A, 6010D
	ICS A	At the beginning of an analytical run	Spiked analytes: Within 20% of expected value Non-spiked analytes absolute value <1/2 LOQ (unless they are a verified trace impurity from one of the spiking solutions)	1) Terminate analysis and correct problem 2) Reanalyze ICS 3) Reanalyze all affected samples	6020A, 6010D
	ICS AB	At the beginning of an analytical run	Within 20% of expected value	1) Terminate analysis and correct problem 2) Reanalyze ICS 3) Reanalyze all affected samples	6020A, 6010D

WORKSHEET #24.2 (CONTINUED)
ANALYTICAL INSTRUMENT CALIBRATION - INORGANIC METHODS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Method Reference[2]
CVAA	Initial Calibration	Daily	$r^2 \geq 0.995$	1) Evaluate system 2) Recalibrate	7470A, 7471B
	ICV; must be from a second source	Following initial calibration	%R = 90% to 110%	1) Evaluate system 2) Recalibrate as necessary	7470A, 7471B
	CCV	Before sample analysis, after every 10 samples, and at the end of the analysis sequence	%R = 80% to 120%	1) Repeat calibration 2) Reanalyze all samples since last successful CCV	7470A, 7471B
	ICB/CCB	After every ICV/CCV	No analytes detected ≥1/2 LOQ or 1/10 the amount detected in any sample; no analytes with negative drift with absolute value >1/2 LOQ	1) Correct problem 2) Analyze calibration blank and previous 10 samples	7470A, 7471B
Ultraviolet-visible spectroscopy Automatic Colorimetry, Cyanide	ICAL - the instrument is calibrated by a minimum 5-point curve and a blank	At beginning of each day, or if QC is out of criteria	If multiple calibration standards are used, r^2 must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance; check calibration standards; reanalyze affected data	9012B
	ICV – Second Source	Following ICAL, prior to samples analysis	%R must be within 90–110% of the true value.	Investigate reasons for failure, reanalyze once; if still unacceptable, repeat ICAL	9012B
	ICB	Before beginning a sample sequence.	No analytes detected > ½ LOD.	Correct the problem, reprepare, and reanalyze	9012B
	CCV	At beginning and end of sequence and after every 10 samples	%R must be within 90–110% of true value.	Recalibrate and/or perform necessary equipment maintenance; check calibration standards; reanalyze affected data.	9012B
	CCB	After the initial CCV, after every 10 field samples; and at end of sequence	No analytes detected > ½ LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and previous 10 samples.	9012B

amu = atomic mass unit
CCB = continuing calibration blank
ICAL = initial calibration
ICB = initial calibration blank
ICS = interference check solution

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

The project laboratory is SGS Orlando and is operating under a quality system that conforms to International Organization for Standardization 17025:2005, and all appropriate activities are documented in the laboratory's QA manuals. The laboratory QA manual is considered business confidential but can be made available upon request.

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

Sample Collection (Personnel/Organization): HGL Field Staff
Sample Packaging (Personnel/Organization): HGL Sample Manager
Coordination of Shipment (Personnel/Organization): HGL Sample Manager; SGS Orlando Sample Receipt Manager
Type of Shipment/Carrier: FedEx
Field Sample Storage (number of days from sample collection): Samples will be stored on site no longer than approximately 3 days prior to shipping (unless sample method hold times require expedited shipping)
Sample Receipt and Analysis:
Sample Receipt (Personnel/Organization): SGS Orlando Sample Receipt Manager/Staff
Sample Custody and Storage (Personnel/Organization): SGS Orlando Sample Management Staff
Sample Preparation (Personnel/Organization): SGS Orlando Organic Preparation Staff, Inorganic Preparation Staff, and Bench Chemists
Sample Determinative Analysis (Personnel/Organization): SGS Orlando Bench Chemists
Sample Archiving:
Sample Extract/Digestate Storage (number of days from extraction/digestion): 60 days from data report release or as required per the observational metals soil sample approach (i.e., sample hold and analyze upon notification from HGL)
Sample Disposal:
Personnel/Organization: SGS Orlando Sample Management Staff
Number of Days from Analysis: 60 days from data report release or as required per the observational metals soil sample approach (i.e., sample hold and analyze upon notification from HGL)

WORKSHEETS #26 AND #27 (CONTINUED)
SAMPLE HANDLING, CUSTODY, AND DISPOSAL

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to the laboratory):

HGL will maintain chain-of-custody (CoC) records for all field and field QC samples. A sample is defined as being under a person's custody if any of the following conditions exist: (1) it is in his or her possession; (2) it is in his or her view after being in the individual's possession; (3) it was in his or her possession and is locked up; or (4) it is in a designated secure area after being in his or her possession.

Procedures to ensure the custody and integrity of the samples begin at the time of sampling and continue through transport, sample receipt, preparation, analyses, storage, data generation, reporting, and sample disposal. Records concerning the custody and condition of the samples are maintained in the field and laboratory records. All sample containers will be sealed in a manner that will prevent tampering or indicate tampering, should it occur. In no instance will sample containers be sealed with tape.

Sample Labeling: Each sample will have a unique sample ID number assigned in accordance with the site-specific sample IDs presented in WS #18. Field QC samples will be identified in accordance with the ID protocols presented in WS #20. The following information will be included on the label:

- Project ID;
- Sample ID;
- Type of sample matrix;
- Preservative added;
- Date and time of collection;
- Required analytical methods; and
- Sampler's initials.

The samples labels will be placed on the sample containers so as not to obscure any QA/QC data on the bottles. Sample information will be printed in a legible manner using a permanent (indelible) ink marker or will be preprinted. Field ID must be sufficient to enable cross referencing with the appropriate sample documentation forms. CoC forms will be completed at the time of collection, including all required information, and ensuring that the CoC information matches the information on the sample labels.

Sample Packaging: Preservation reagents will be added to sample containers before or immediately after collection of the sample, as indicated in WSs #19 and #30. Samples requiring cool temperature preservation will immediately be placed on ice and will be kept chilled during the work day until packaged for shipment to the laboratory.

WORKSHEETS #26 AND #27 (CONTINUED)
SAMPLE HANDLING, CUSTODY, AND DISPOSAL

Sample coolers will be supplied by the laboratory. When packaging samples for shipment, the cooler drainage plug will be closed and the cap will be sealed in place with duct tape. Sample containers will be placed inside sealed plastic bags as a precaution against cross-contamination caused by leakage or breakage. Bagged sample containers will be placed in the coolers in such a manner as to eliminate the chance of breakage during shipment. Ice in plastic bags will be placed in the coolers to keep the samples at 6 ºC or less throughout shipment. Prior to sealing the cooler, the sampler's copy of the CoC forms will be detached and preserved for the project file. The remaining portion of the completed CoC forms will be attached to the underside of the cooler lid in a sealed plastic bag. The cooler will then be taped shut and at least two completed custody seals will be affixed across the gap between the lid and body of the cooler.

Sample Shipment: Samples collected in the field will be transported by a laboratory courier (if available) or shipped to the laboratory as expeditiously as possible. Sample shipment will be performed in accordance with all applicable U.S. Department of Transportation regulations. Samples will be shipped to the laboratory by the procedures identified in this worksheet. Arrangements will be made between HGL and the laboratory point-of-contact for samples that are to be delivered to a laboratory on a weekend so that sample condition and holding times are not compromised.

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal):

The designated sample custodian(s) and staff are responsible for samples received at the laboratory. In addition to receiving samples, the sample receipt staff is also responsible for documentation of sample receipt and storage before and after sample analysis. Summaries of the minimal laboratory receipt procedures are as follows:

- Upon receipt, sign, date, and document the time of sample receipt on the airbills or other shipping manifests received from the couriers.
- Sign the CoC form assuming custody of the samples. If a CoC form is not received with a set of samples, the laboratory will immediately notify the HGL PC.
- Inspect the sample cooler for integrity and then document the following information:
    - Type of courier and whether the samples were shipped or hand delivered (copies of the airbills are maintained).
    - Availability and condition of custody information.
    - Sample temperature.
    - If the temperature of the samples upon receipt at the laboratory exceeds the temperature requirements, individual sample containers will be measured. All exceedances will be documented in laboratory records, and the laboratory must contact the HGL PC immediately and document any decision regarding the potentially affected samples.
    - Presence of leaking or broken containers and indication of sample preservation.
- Match the sample container information (e.g., sample tag/label), CoC records, and all pertinent information associated with the sample. The sample custodian will then verify sample identity to ensure that all information is correct. Any inconsistencies are resolved with the HGL PC through the Laboratory PM. CA measures are documented before sample analysis proceeds.
- Verify the holding time has not been exceeded. If a sample has exceeded holding time, the HGL PC must be notified.

Samples and extracts will be archived at the laboratory in accordance with this worksheet. The laboratory is also responsible for the proper management and disposal of all sample residuals and extracts, following all applicable federal, state, and local laws, rules, and regulations.

WORKSHEETS #26 AND #27 (CONTINUED)
SAMPLE HANDLING, CUSTODY, AND DISPOSAL

Sample ID Procedures:

All field samples will receive a unique sample ID designation as detailed in WS #18 (field samples) and WS #20 (field QC samples). Sample IDs will clearly differentiate field QC samples (including duplicates and MS/MSDs) and IDW samples from environmental samples.

CoC Procedures:

Documentation of the CoC of the samples is necessary to demonstrate that the integrity of the samples has not been compromised between collection and delivery to the laboratory. A CoC record to document the transfer of custody from the field to the laboratory will accompany each sample cooler. All information requested in the CoC record will be completed. In addition, the airbill number assigned by the overnight courier will be listed on the CoC record or the general logbook. One copy of the CoC form will be retained by the samplers and placed in the project records file. The remaining pages will be sealed in a plastic bag and placed inside of the cooler. Upon receipt at the laboratory, the COC forms will be completed and a cooler receipt form will be completed. It is the responsibility of the laboratory to document the condition of custody seals and sample integrity upon receipt.

The following sample-specific information concerning the sample will be documented on each CoC form:

- Unique sample ID number;
- Date and time of sample collection;
- Designation of MS/MSD;
- Preservative used;
- Analyses required;
- Name of collector(s);
- Serial numbers of custody seals and transportation cases (if used); and
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories.

Including a bill of lading or airbill tracking number on the CoC form is not possible if the airbill is generated at the transporter drop-off location (FedEx), as the shipping cooler(s) will already be prepared for shipment with the CoC form sealed inside. In this case the airbill number(s) will be provided to the laboratory PM via email as soon as possible after shipment drop-off is completed.

In addition to the information above, the field team will record the source of sample (including name, location, and sample type) and any location-specific QC (such as field duplicates and ambient blanks) in the field logbook at the time of collection. Sample-specific information also will be recorded on sample-specific sample collection sheets and retained in the project file. Other pertinent field data, such as field screening measurements or visual observations, will be recorded in the field logbook or on task-specific pre-printed log forms and retained in the project file.

WORKSHEET #28.1
METHOD QC TABLE – GC/MS, GC, AND HPLC METHODS

QC Element	Frequency	Method/SOP QC Acceptance Limits[1]	CA	Person(s) Responsible for CA	DQI
Screening Level QC Elements					
MB	Every analytical batch (maximum of 20 samples)	Target analytes not detected >½ LOQ and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater)	1) Rerun 2) Evaluate batch 3) Reanalyze or qualify results as necessary	Section Manager/ Laboratory Analyst	Accuracy/Bias and Representativeness
LCS (and LCSD, if performed)	Every analytical batch (maximum of 20 samples)	Analyte-specific %R and RPD acceptance criteria	1) Rerun 2) Evaluate batch 3) Reanalyze or qualify results as necessary	Section Manager/ Laboratory Analyst	Accuracy/Bias (and Precision)
MS/MSD	As indicated on CoC forms, and as required for batch control	Analyte-specific %R and RPD acceptance criteria (NA to air methods or if parent sample concentration ≥4x the spike level)	1) Evaluate MS/MSD to assess matrix interference 2) Evaluate batch and qualify results as necessary	Section Manager/ Laboratory Analyst	Accuracy/Bias and Precision
Surrogate Recovery	Every sample	Surrogate-specific %R acceptance criteria	1) Rerun 2) Reanalyze or qualify results as necessary	Section Manager/ Laboratory Analyst	Accuracy/Bias
Definitive Level QC Elements – GC/MS Methods					
Internal Standard Performance	Every sample	Peak area within 50-200% of the peak area in the initial calibration midpoint standard or most recent CCV standard on days initial calibration is not performed Retention time within ±10 seconds of the initial calibration midpoint standard or most recent CCV standard on days initial calibration is not performed	1) Rerun 2) Reanalyze or qualify results as necessary	Section Manager/ Laboratory Analyst	Accuracy/Bias
RRT Position	Once per initial calibration and at the beginning of the analytical shift	RRT within ±0.06 RRT units for each analyte and surrogate	1) Correct problem 2) Recalibrate instrument 3) Reanalyze results as necessary	Section Manager/ Laboratory Analyst	Analyte ID

WORKSHEET #28.1 (CONTINUED)
METHOD QC TABLE – GC/MS, GC, AND HPLC METHODS

QC Element	Frequency	Method/SOP QC Acceptance Limits[1]	CA	Person(s) Responsible for CA	DQI
Mass spectrometer results	All positive results must be confirmed	Spectral match to reference spectrum	1) Analyst must evaluate results to confirm ID if spectral match does not meet criteria 2) Section manager must review analyst's determination	Section Manager/ Laboratory Analyst	Analyte ID
Definitive Level QC Elements – GC and HPLC Methods					
Retention time window position	Once per initial calibration and at the beginning of the analytical shift	All peaks associated with positive results must elute within the established retention time window; for total petroleum hydrocarbons, the window is determined for carbon ranges.	1) Correct problem 2) Recalibrate instrument 3) Reanalyze results as necessary	Section Manager/ Laboratory Analyst	Analyte ID
Internal Standard Performance (as applicable)	Every field sample, standard, blank, and QC sample	Retention time within ± 0.06 RRT units from the retention time of the midpoint standard in the ICAL. Peak area within 50-200% of the peak area of the ICAL midpoint standard. On days when an ICAL is not performed, the daily initial CCV can be used.	1) Evaluate potential for matrix effects. 2) Re-extract or dilute and reanalyze; or qualify results, as necessary.	Analyst	Accuracy Bias
Confirmation column	All positive results must be confirmed	Result not confirmed using second column or detector	1) Analyst must evaluate data to determine if unconfirmed result is a detection 2) Section manager must review analyst's determination	Section Manager/ Laboratory Analyst	Analyte ID
		Results between primary and second column RPD ≤40%; not required for multicomponent analytes	1) Analyst must select result to report in accordance with method requirements and laboratory SOP 2) Section manager must review analyst's determination	Section Manager/ Laboratory Analyst	Accuracy/Bias

[1] Method-specific acceptance criteria are presented in the corresponding tables of WSs #12 and 15.

WORKSHEET #28.2
METHOD QC TABLE – METALS AND WET CHEMISTRY METHODS

QC Element	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI
Screening Level QC Elements					
MB	Every preparation batch (maximum of 20 samples)	Analytes not detected >½ LOQ and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); no negative values with absolute value >1/2 LOQ	1) Rerun 2) Evaluate batch 3) Redigest affected samples or qualify results as appropriate	Laboratory Manager/ Laboratory Analyst	Accuracy/Bias and Representativeness
LCS (and LCSD, if performed)	Every preparation batch (maximum of 20 samples)	Analyte-specific %R and RPD acceptance criteria	1) Rerun 2) Evaluate batch 3) Reanalyze or qualify results as necessary	Laboratory Manager/ Laboratory Analyst	Accuracy/Bias and Precision
MS (and MSD, if performed)	Every preparation batch (maximum of 20 samples)	Analyte-specific %R and RPD acceptance criteria %R discrepancies NA if parent sample concentration ≥ 4x the spike level	1) Rerun 2) Evaluate batch 3) Qualify sample results as appropriate	Laboratory Manager/ Laboratory Analyst	Accuracy/Bias and Precision
Laboratory Duplicate (if performed)	Every preparation batch (maximum of 20 samples)	RPD ≤ method criteria if both results >5x the LOQ; absolute difference <LOQ for evaluation of low-level results (<5x LOQ)	1) Rerun 2) Evaluate batch 3) Qualify sample results as appropriate	Laboratory Manager/ Laboratory Analyst	Precision
Definitive Level QC Elements (Methods 6010D, 6020A, 7470A, 7471B)					
Internal Standards (SW6020A only)	Every field sample, standard, and QC sample	Internal standard intensity in the samples within 30-120% of intensity of the internal standard in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze the sample at 5-fold dilutions until the criteria is met. For failed QC samples, correct problem and rerun all associated failed samples.	Analyst	Accuracy Bias

WORKSHEET #28.2 (CONTINUED)
METHOD QC TABLE – METALS AND WET CHEMISTRY METHODS

QC Element	Frequency/ Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI
Serial Dilution	If MS %R discrepancy affects a target analyte present at ≥50x LOQ	%D ≤10% for all analytes present in the parent sample at concentrations ≥50x LOQ	1) Evaluate PDS results; if PDS results are in control for all analytes with a serial dilution discrepancy, report data; otherwise: 2) Rerun 3) Evaluate batch 4) Qualify sample results as appropriate	Laboratory Manager/ Laboratory Analyst	Accuracy/Bias
PDS	If MS %R discrepancy affects a target analyte present at <50x LOQ	%R = 80%-120%	1) Rerun 2) Evaluate batch 3) Qualify sample results as appropriate	Laboratory Manager/ Laboratory Analyst	Accuracy/Bias
Definitive Level QC Elements (Method 9012B)					
High and low distilled standards	Every preparation batch (maximum of 20 samples)	%R = 85%-115%	1) Rerun 2) Evaluate batch 3) Qualify sample results as appropriate	Laboratory Manager/ Laboratory Analyst	Accuracy/Bias

PDS = post digestion spike

WORKSHEET #29
DATA MANAGEMENT, PROJECT DOCUMENTS, AND RECORDS

This worksheet summarizes the documents and records that will be generated during on-site data collection activities. All documents and records will be maintained in the project file (hard copy) or on electronic media/HGL SharePoint website subject to daily backup. Hard copy forms will be scanned for electronic storage as PDF files. Electronic log forms populated in Microsoft Word/Excel or other software packages will be printed/exported to PDF format for storage and inclusion in daily reports, as applicable.

Document/Record	Generation Frequency	Person Responsible for Generation	Person Responsible for Verification
General Field Operations and QC			
Three-Phase Checklist (Preparatory, Initial, Follow-Up)	Prior to (Preparatory), at task startup (Initial), and throughout task execution (Follow-Up)	HGL Field Team Lead (FTL)	HGL PM
DQCR	Daily during on-site activities	HGL FTL	HGL PM
Daily H&S Tailgate Meeting Log	Daily during on-site activities	HGL SSHO	HGL PM
ITS Testing	Daily before MPPEH on-site activities	HGL FTL	HGL UXOQCS
Field Activity Daily Log	Daily during on-site activities	HGL FTL or SSHO	HGL PM
GIS Data	Daily after data collection	HGL field staff	HGL FTL
Digital Photograph	Daily after data collection	HGL field staff	HGL FTL
Equipment Calibration Log	Daily prior to use	HGL field staff	HGL FTL
Field Logbook/Notes	Daily during field activities	HGL field staff	HGL FTL
Telephone logs, emails, faxes, and correspondence	Continuously during field activities	HGL field staff	HGL PM
DD Form 1348-1A	As required for batches of MPPEH	HGL field staff	HGL Site Manager
ENG Form 1836	Per boring	HGL field staff	HGL PM
Deliverables			
Project Work Plans (Field Sampling Plan, Sampling and Analysis Plan, UFP-QAPP, Accident Prevention Plan/Site Safety and Health Plan)	Upon completion of Preliminary Draft, Draft, and Final versions	HGL Staff	HGL PM
Project Management Deliverables (Monthly Project Status Reports, Accruals)	Monthly during project execution	HGL PM	HGL PgM
Telephone logs, emails, faxes, and correspondence	Continuously during project execution	HGL Staff	HGL PM

WORKSHEET #29 (CONTINUED)
DATA MANAGEMENT, PROJECT DOCUMENTS, AND RECORDS

Document/Record	Generation Frequency	Person Responsible for Generation	Person Responsible for Verification
Laboratory Sample Collection and QC Documentation			
CoC Form	Continuously during sampling	HGL Sampler	HGL FTL
Sample Collection Form	Continuously during sampling	HGL Sampler	HGL FTL
Courier/FedEx Airbill	Each sample shipment	HGL Sample Manager	HGL FTL
Laboratory Analysis and Reporting			
Sample Receipt, Custody, and Tracking Records	Continuously from sample receipt	Laboratory Sample Receipt Staff	Laboratory PM
Standard Traceability Log	Continuously during sample analysis	Laboratory Analytical Staff	Laboratory Section Manager/QA Manager
Equipment Calibration Log	Continuously during sample analysis	Laboratory Analytical Staff	Laboratory Section Manager/QA Manager
Sample Preparation Log	Continuously during sample analysis	Laboratory Analytical Staff	Laboratory Section Manager/QA Manager
Analytical Run Log	Continuously during sample analysis	Laboratory Analytical Staff	Laboratory Section Manager/QA Manager
Equipment Maintenance, Testing, and Inspection Log	Continuously during sample analysis	Laboratory Analytical Staff	Laboratory Section Manager/QA Manager
Analytical Discrepancy Form	Continuously during sample analysis	Laboratory Analytical Staff	Laboratory Section Manager/QA Manager
Reported Analytical Results	Continuously during data reporting	Laboratory Analytical Staff	Laboratory Section Manager/QA Manager
Reported Results for Standards, QC Checks, and QC Samples	Continuously during sample analysis	Laboratory Analytical Staff	Laboratory Section Manager/QA Manager
Data Package Completeness Checklist	Continuously during data reporting	Laboratory Analytical Staff/Section Manager	Laboratory PM/QA Manager
Sample Disposal Record	Continuously during sample disposal	Assigned Laboratory Staff	Laboratory OM/QA Manager
Extraction and Cleanup Record	Continuously during sample analysis	Laboratory Analytical Staff	Laboratory Section Manager/QA Manager
Raw Data (Electronic)	Continuously during data reporting	Laboratory Analytical Staff	Laboratory Database Manager/QA Manager
Electronic Data Package	One per Sample Delivery Group (SDG) during data reporting	Laboratory Database Manager	HGL Database Manager
Full Data Package (PDF)	One per SDG during data reporting	Laboratory PM	HGL PC
QC Summary Report (QCSR)	One per field sampling event	HGL PC/Database Manager	HGL Program Chemist (PgC)
Telephone logs, emails, faxes, and correspondence	Continuously during project execution	Laboratory Staff	Laboratory PM

WORKSHEET #29 (CONTINUED)
DATA MANAGEMENT, PROJECT DOCUMENTS, AND RECORDS

Document/Record	Generation Frequency	Person Responsible for Generation	Person Responsible for Verification
Laboratory Data Assessment Documents and Records			
Data Validation Report	One per SDG during data validation	Data Validation Staff	Data Validation PM
Database QC Spreadsheet	One per SDG during data validation	HGL Database Manager	HGL PM
Data Usability Assessment	One per SDG during data validation	HGL PC	HGL PgC
Telephone logs, emails, faxes, and correspondence	Continuously during project execution	Data Validation PM/HGL Staff	HGL PM
Waste Management Documentation			
MDAS Disposal Documentation	After each shipment of MDAS off site	HGL field staff	HGL Site Manager
Waste Tracking Log	Daily when waste is generated and stored on site	HGL field staff	HGL FTL
Waste Disposal Profile/ Manifest/Bill of Lading/ Letter of Acceptance	Upon completion of waste shipment from site and acceptance at disposal facility	HGL Hazardous Waste Material Manager	HGL PM

WORKSHEETS #31, #32, AND #33
ASSESSMENTS AND CORRECTIVE ACTION

For this project, related activities are grouped as follows:

1. Site preparation (DFW 1)
2. Pre-Excavation Sampling (DFW 2-3)
3. Excavation and Backfill (DFW 4-6)

For each group of related activities, assessment activities will occur during the following phases:

- **Preparatory Phase:** Comprises the planning and design process leading up to field activities. The UXOQCS will perform a Preparatory Phase assessment before beginning each group of activities. The purpose of this assessment is to review applicable specifications and plans to verify that the necessary resources, conditions, and controls are in place and comply with specifications before field work begins.
- **Initial Phase:** Occurs at the startup of field activities. The purpose of this phase is to check preliminary work for compliance with specifications, check for omissions, and resolve differences of interpretation.
- **Follow-up Phase:** Covers the routine, day-to-day activities at the site. One or more follow-up assessments will be conducted during each related group of activities, depending on the duration of field activities, and the nature of any assessment findings.

WORKSHEETS #31, #32 AND #33 (CONTINUED)
ASSESSMENTS AND CORRECTIVE ACTION

Assessments

Assessment Type	Assessment Frequency	Responsible Party (Title, Organization)			
		Performing Assessment	**Responding to Assessment Findings**	**Identifying/ Implementing CA**	**Monitoring Effectiveness of CA**
Management Assessment	Based on internal management request	HGL PgM	HGL PgM	HGL PgM	HGL PgM
Independent Assessment	Based on internal management request	Independent Organization Point of Contact	HGL PgM	HGL PgM	HGL PgM
Laboratory Audit/ Inspection	Biennial or as needed (DoD ELAP)	Laboratory Contractor	Laboratory PgM	Laboratory PgM	Laboratory QA/QC Manager
QA/QC Audit	Once per project (if scheduled)	HGL Lead Auditor	HGL PM/QCSM/FTL (minor deficiencies) HGL CQM/CHSM/PgM (major deficiencies)	HGL PM/QCSM/FTL (minor deficiencies) HGL CQM/CHSM/PgM (major deficiencies)	HGL PM/QCSM/FTL (minor deficiencies) HGL CQM/CHSM/PgM (major deficiencies)
QCSR (Analytical Data)	Project, task, field event specific	HGL PgC	HGL PgC/PC/DM	HGL PgC/PC/DM	HGL PgC
Readiness Review	Prior to field mobilization	HGL QCSM	HGL PM/QCSM	HGL QCSM	HGL PgM
Field Inspection	Task-specific	HGL QCSM	HGL PgM	HGL QCSM	HGL PgM
Preparatory Phase Inspection/Meeting	Prior to DFW startup	HGL QCSM	HGL PgM	HGL QCSM	HGL PgM
Initial Phase Inspection	Upon or immediately after DFW startup	HGL QCSM	HGL PgM	HGL QCSM	HGL PgM
Follow-Up Phase Inspection	During and/or at completion of DFW	HGL QCSM	HGL PgM	HGL QCSM	HGL PgM
Final Inspection		HGL QCSM	HGL PgM	HGL QCSM	HGL PgM

CQM = Corporate Quality Manager
QCSM = Quality Control Site Manager

Guidance on Audits and Inspections

Assessment and Audit Frequency:
Technical inspections and assessments will be conducted during initial stages of fieldwork to identify and correct problems as quickly as possible. Independent assessments will be performed in response to project management requests. Laboratory audits may be conducted by contractor personnel every two years in accordance with the frequency required by the DoD ELAP if they are deemed necessary to confirm DoD ELAP accreditation audits. More frequent audits may be deemed necessary based on laboratory data quality performance, reporting, or other related issues that could arise over the course of the contract. The laboratory audit will include all sample analysis procedures that will be performed by the laboratory being audited. The QA Manager or PM may conduct audits at a greater frequency than indicated in WS #31. Successful DoD ELAP audits are needed to demonstrate environmental testing laboratories are compliant with the DoD QSM, Version 5.1 (DoD, 2017).

Management Assessments and Independent Assessments:
Management assessments and independent assessments may be used to review sample collection, handling, analysis, and documentation procedures. Assessment results are used to evaluate a system’s ability to produce data that fulfill program objectives and to identify any areas requiring CAs. Inspections are routine qualitative reviews of the overall sampling or measurement system, or they may have a narrow focus, such as a follow-up inspection. Assessments provide an overall examination of the measurement system. Assessment and inspection records are reviewed by the QA Manager or designated staff to determine whether data will fulfill the program objectives. Additional inspections or reviews for designated methods may be conducted, or additional information may be requested if data quality problems are indicated.

Management Assessments
Management assessments may be conducted at the request of the Site Operations Manager, PM, or other employees in management authority. Management assessments are informal reviews of work progress, functionality, adherence to policies and procedures, compliance with requirements, or effectiveness of implementation. They provide the basis for follow-up inspections or independent assessments whenever deficiencies are indicated. All observations are documented, and any recommendations or CAs are submitted to the QA Manager for tracking, implementation, additional review (if required) and completion.

Independent Assessments
Independent assessments may be conducted at the request of the PM or by personnel who have the authority and organizational independence to provide an unbiased review of the system or procedure. When performed, a detailed checklist will be used for each procedure or system reviewed and will contain items that delineate the critical aspects of the procedure under review. All observations are documented, and the checklist is submitted with a written assessment and recommendations to the QA Manager, PM, USACE COR, representatives of the audited organization, and others as appropriate. The information and any CA documentation also will be summarized and included in program reports.

Guidance on Audits and Inspections (Continued)

Field Inspections:
Field inspections are on-site, qualitative reviews of a sampling or analysis system. Inspections are conducted, preferably at the beginning of the sampling task, by the PgM or designee, field lead, or a designated qualified technical staff member who has the authority to act independently of the project staff. Critical items for field inspections include:

- Calibration procedures and documentation for field instruments
- Documentation in field logbooks and on sampling data sheets
- Document control
- Equipment decontamination procedures
- Sample collection, storage, and transportation procedures
- CoC procedures for sample documentation and for transfer to a laboratory
- Work instructions

The checklist for each inspection will contain detailed questions regarding the critical items requiring yes/no answers and comments. A debriefing session will be held for all participants to discuss any inspection results and to discuss any required CA. The reviewer then completes the inspection and submits a report, including observations of strengths and deficiencies and any recommendations for improvements. Detailed checklists will be provided for each final version of the site-specific plans. Examples of these checklists are provided in Attachment 4 and are for informational purposes only.

Inspections for Field Activities:
Inspections will be performed on materials or services to determine compliance with contractual, planning, and other requirements. Criteria will be established prior to the inspection and will be based on project specifications, requirements, code specifications, and product acceptability. Acceptance criteria will be adequate for the activity and will be verified during inspection activities. Inspection may be performed and verified through visual observation, measurement of materials or equipment, examination of documentation/certification, evaluation of performance, or testing. Inspection forms must be developed based on the definable features of work described in site-specific work plans.

Inspections may be performed using the three-phase inspection method. The preparatory inspections will be performed prior to start-up and will examine training, procedures, equipment and materials, work plans and documents, and overall readiness to perform work. Initial inspections will be performed when work begins on a particular feature of work and will include an examination of the quality of workmanship and a review of control testing for compliance with contract and work plan requirements. Follow-up inspections will be performed to verify compliance with procedures and will ensure the continuation of quality and safety standards established during preparatory and initial inspections until completion of the definable work feature. Final follow-up inspections will be conducted at the completion of each task. Participants in this inspection may include QA (USACE) and QC (contractor). The final follow-up inspection will be performed to ensure that the completed feature of work meets contract requirements. Any deficiencies noted during this inspection will be documented, and a determination will be made as to the CAs that may be necessary to mitigate the deficiency. All significant deficiencies must be corrected prior to turnover.

Records of inspections will be maintained in the project files. At a minimum, inspection files will include inspection reports/checklists, inspection responses, any supporting documents, and applicable client comments.

Guidance on Audits and Inspections (Continued)

Receipt Inspections:
Standard contractor procurement procedures will be used to obtain supplies and consumables. These procedures are initiated by the task manager, field coordinator, or other technical lead personnel and then forwarded to project management and procurement personnel for approval and supplier contact. In the request for supplies, the requestor must provide specifications of the material, including any required certifications of purity or QC level. Sample supplies and consumables must be inspected upon receipt to verify that they meet these specifications and that any required manufacturer’s documentation is present and retained for the contractor’s project files. Any damaged, unsealed, or used equipment (unless adequately cleaned and returned to service) will not be accepted. For items that may come in contact with the sampled matrix or sampling device, assurances should be made, through adequate receipt inspection, that such materials are not contaminated. They must remain sealed or be adequately decontaminated before field use. Examples include decontamination water, chemical reagents, in-line water filters, sample bottles and jars, sampling probes/instruments, bailers, soil spoons, or augers.

Performance and System Audits:
Scheduled project/laboratory audits will be performed, as project activities allow, to review and evaluate the adequacy of field activities and laboratory performance and to ascertain if the UFP-QAPP is being completely and uniformly implemented. The PgM or designee is responsible for requesting and establishing an audit team. Biennial audits may be supplemented by additional audits for one or more of the following reasons:

- Significant changes are made in field or laboratory protocols
- It is necessary to verify that a CA has been taken on a nonconformance reported in a previous audit
- Audit is requested by the PM

The objectives of performance and systems audits are (1) to verify that the UFP-QAPP developed for this project is being implemented according to the specified requirements, (2) to assess the effectiveness of the plan, (3) to identify nonconformances, and (4) to verify that identified deficiencies are corrected. Upon discovery of any significant deviation from the UFP-QAPP, the PC and PgM or designee shall be informed of the nature and extent of the deviation. A nonconformance will be documented and a CA will be taken to remedy the deviation.

Assessment Findings and CA:
All observations and assessment findings will be documented, and the checklist will be submitted with a written assessment and recommendations, including any required or recommended CAs to the PgM or designee, PM, USACE COR, representatives of the audited organization, and others as appropriate. The information and any CA documentation also will be summarized and included in program reports.

Guidance on Audits and Inspections (Continued)

Nonconformance Documentation:
Complex field investigation, remediation, sampling, and analysis tasks, such as those performed routinely as part of USACE environmental projects, are sometimes subject to nonconformances. A nonconformance is defined as an unplanned deviation that occurs during the implementation of a task that cannot usually be corrected until after it has occurred. Nonconformance activities may include using unapproved methods, not following procedures specified in the UFP-QAPP or task work plan, or substituting unapproved materials or equipment to perform an activity. Nonconforming supplies may also include suspect and counterfeit items. All nonconformance activities and/or material must go through a cycle of being identified, documented, assessed, corrected, and reported.

The ID of a nonconformance is the responsibility of every person assigned to support the USACE project. This responsibility is incorporated into each person's understanding of his or her tasks, as assigned by the supervisor or task leader, and each person's function on the project. As individuals perform their duties on the project, they must constantly be aware of the scope of the activity and recognize when a deviation from the planned activity has occurred or is occurring. After recognizing the deviation, they must take action by informing the HGL PM and documenting in writing (using a Non-Conformance Report [NCR] form) the specifics of what occurred. The HGL QCSM will maintain a status log of open and closed nonconformances. The log will also serve as the basis for numbering each discrepancy and tracking it through closure. If the nonconforming activity, service, or material is severe, it may require cessation of all activities.

Satisfactory resolution of nonconformances must be verified by the HGL QCSM. Nonconformances are not to be closed until the required corrective and preventative actions have been completed to the satisfaction of the HGL QCSM or until long-term CAs have been established and implemented. Nonconformances will be monitored until the action is verified as complete and closed as documented on the NCR. Nonconformances and associated documentation will be documented in the project file and referenced and discussed in the final task report.

Field Work Variance Documentation:
Field Work Variances (FWVs) are similar to nonconformances with respect to how they are defined, resolved, and documented. The primary difference is the timing of the occurrence of the deviation. A FWV can be identified prior to implementation of a task, while a nonconformance is generally not identified until the task is in progress or complete. Therefore, with a FWV, alternative techniques, modified methods, or a change in task and DQOs can be considered. Substitute data, alternate success criteria, or even the deletion of data points may be contemplated after gathering information on the reason for the deviation and examining the intended use of the data as planned. Project FWV will be subject to the same stepwise process of ID, documentation, assessment, correction, and reporting as nonconformances. This process has been designed to ensure key information is recorded by the personnel who identify FWVs, review the documentation, assess the impact on task objectives, and consider alternative strategies for CA. FWVs will be documented in the project file and will be referenced and discussed in the final task report.

Assessment Findings and CA Responses

Assessment Type	Deficiency Documentation	Person(s) Notified of Findings	Notification Timeframe	CAR Documentation	Person(s) Notified of CAR	CAR Response Timeframe
Management Assessment	NCR	HGL PgM/PM	5 days	CAR	HGL PgM	30 days
Independent Assessment	NCR	HGL PgM/PM/ CQM/FTL	5 days	CAR	HGL PM	30 days
Receipt Inspection	NCR	HGL CQM/ QCSM/FTL	NA	Item rejected or replaced by vendor	HGL CQM/ QCSM/PM	NA
Laboratory Audit/ Inspection	NCR	HGL PgM/PM	NA	CAR	HGL CQM/PgC	NA
QCSR	NCR	USACE Chemist/ Data End Users	NA	NA	NA	NA
Field Inspection	NCR	HGL CQM/ QCSM/PM	NA	CAR	HGL CQM/QCSM	NA
Preparatory Phase Inspection	NA	HGL QCSM/ PM/FTL	NA	NA	NA	NA
Initial Phase Inspection	NCR	HGL QCSM/ PM/FTL	5 days	CAR (Based on severity)	HGL PgM/ CQM/PM	30 days
Follow-Up Phase Inspection	NCR	HGL QCSM/ PM/FTL	5 days	CAR (Based on severity)	HGL PgM/ CQM/PM	30 days
Final Inspection	NCR	HGL QCSM/ PM/FTL	5 days	CAR (Based on severity)	HGL PgM/ CQM/PM	30 days

CAR = corrective action request

WORKSHEET #34
DATA VERIFICATION AND VALIDATION INPUTS

This worksheet lists the inputs that will be used during data verification and validation. Inputs include planning documents, field records, and laboratory records. 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 UFP-QAPP.

Item	Description	Verification (Completeness)	Validation (Conformance to specifications)
Planning Documents/Records			
1	Approved UFP-QAPP	X	
2	Contract	X	
3	Field SOPs	X	
4	Laboratory SOPs	X	
Field Records			
5	Field logbooks	X	
6	Equipment calibration logs	X	
7	CoC forms	X	
8	Sample location coordinates/maps/surveys	X	
9	Relevant correspondence	X	
10	FWV	X	
11	Inspection reports/checklists	X	
12	Correction action requests/reports	X	
13	Underground utility clearance tickets/forms	X	
14	Facility badging/access requests	X	
15	Field GIS data	X	
16	Digital photographs	X	
17	Intrusive investigation results	X	
18	DD Form 1348-1A	X	X
19	MDAS disposition documentation	X	X
Laboratory Analytical Data Package/Deliverables			
17	Cover sheet (laboratory identifying information)	X	X
18	Case narrative	X	X
19	Internal laboratory QC	X	X
20	Sample receipt records	X	X
21	Sample chronology (dates and times of receipt, preparation, and analysis)	X	X
22	Communication records	X	X
23	Project-specific performance testing sample results	X	X
24	LOD/LOQ establishment and verification	X	X
25	Standards traceability	X	X
26	Instrument calibration records	X	X
27	Definition of laboratory data qualifiers	X	X
28	Results reporting forms	X	X
29	QC sample results	X	X
30	CA reports	X	X
31	Raw data	X	X
32	Electronic data deliverables (EDDs)	X	X

WORKSHEET #35
DATA VERIFICATION PROCEDURES

This worksheet documents procedures that will be used to verify project data. Data verification is a completeness check to confirm that all required activities were conducted, all specified records are present, and the contents of the records are complete. The data inputs required for data verification and the associated processes are identified below.

Verification Input	Description	Person(s) Responsible
CoC forms (site shipping)	CoC forms will be reviewed upon completion and verified against the packed sample coolers and site sampling requirements. This QC check will be verified by initialing the CoC form next to the shipper's signature. A copy of the CoC form will be retained in the project file and the original and one copy will be taped inside the cooler in a waterproof bag.	HGL FTL
Field logs/forms	Field logs/forms will be reviewed on a daily basis. This review will be performed to verify that all field monitoring equipment was maintained, calibrated, and operated properly. In addition, the review denotes all required information has been correctly documented in the field logbooks and sample documentation sheets.	HGL FTL
CoC forms (laboratory receipt)	CoC forms will be reviewed and compared to cooler contents. Any discrepancies (sample bottles, sample IDs, requested methods) will be communicated to the Laboratory PM for resolution.	HGL PC/ Laboratory PM
Analytical data package	Ensure that the appropriate analytical samples have been collected, appropriate site IDs have been used, and the correct analytical methods have been applied.	HGL PC
Analytical data package (data validation)	Review the analytical reports to establish that all required forms, case narratives, samples, CoC forms, logbooks, and raw data have been included. This verification is conducted as part of data validation.	HGL PC
EDD (laboratory export)	All EDDs will be verified against the requirements of the project format requirements prior to transmittal to HGL.	Laboratory PM/DM
EDD (HGL import)	Any EDD nonconformances from the laboratory are reviewed and addressed before the data is processed further. This check is performed on the EDD to ensure that it is in the correct format and that it contains the correct standard values. Any errors or warnings are addressed before processing the data further.	HGL DM
Project database	All data qualifiers applied to the project database by manual entry will receive a 100% QC check for accuracy and completeness. Prior to final approval, each EDD output will receive a 10% QC check of electronically reported results against the hardcopy laboratory reports.	HGL DM

WORKSHEET #36
DATA VALIDATION PROCEDURES

On a site-specific basis, identify all analytical methods that will be used and the level of data quality (screening or definitive) that the data review process will be required to support. All categories of field samples collected at a site should be accounted for in this table.

Validation Stage	Matrix	Analytical SOP[1]	Validation Criteria	Data Validator
Data Review Step IIa				
Data Verification	All	All	Package Completeness Holding Times: WS #19 Narrative: Additional items noted for resolution or clarification	Staff Validator, or Subcontractor
Data Validation – Screening (Stage 2A)	Site-specific[2]	Site-specific[2]	DQIs: Method-specific criteria presented in WSs #12, #15, and #28 Qualification: Appendix E, Table E.1, screening level items. All data collected will undergo at least Stage 2A validation.	Staff Validator, or Subcontractor
Data Validation – Definitive (Stage 2B)	Site-specific[2]	Site-specific[2]	DQIs: Method-specific criteria presented in WSs #12, #15, #24, and #28 Qualification: *DoD Data Validation Guidelines* (Nov 2019) and associated Data Validation Modules 1, 2, 3, and 4 (Feb 2022), screening and definitive level items. All data collected will undergo Stage 2a validation and Stage 2B validation of methods that support Stage 2B validation.	Staff Validator, or Subcontractor
Data Validation – Full Review (Stage 4)	Site-specific[2]	Site-specific[2]	DQIs and Qualification: As for definitive data validation. This level of review incorporates evaluation of raw data (e.g., quantitation reports, chromatograms, and mass spectra), recalculation of results, and data reduction. At the discretion of the PC, a sufficient number of laboratory reports corresponding to no less than 10% of the data collected will undergo Stage 4 validation. The data selected will be representative of 10% of samples collected.	Staff Validator, or Subcontractor
Data Review Step IIb				
Senior Review	All	All	See WS #37 and *DoD Data Validation Guidelines* (Nov 2019) and associated Data Validation Modules 1, 2, 3, and 4 (Feb 2022)	PC
Overall Assessment	All	All	See WS #37 and *DoD Data Validation Guidelines* (Nov 2019) and associated Data Validation Modules 1, 2, 3, and 4 (Feb 2022)	Project Manager

[1] Refer to WS #23.

[2] The matrices sampled and the associated level of validation required for each method used to support site characterization will be based on site specific DQOs.

Data Validation Process Overview

An overview of the data validation process is presented in the following table. This process is described in full in the DoD Data Validation Guidelines (Nov 2019) and associated Data Validation Modules 1, 2, 3, and 4 (Feb 2022)

Validation Stage	Validation Input	Description	Person Responsible for Validation
Data Review Step IIa			
Data Verification	Laboratory data reports (see WS #35)	The data validator will verify data package completeness, review case narratives, evaluate sample delivery and condition, and evaluate preparation and analysis holding times (WS #19).	Staff Validator or Subcontractor
Data Validation	Laboratory data reports	The data validator will perform an evaluation of sample- and batch-related QC results for screening or screening and definitive QC elements, as required for each method on a site-specific basis.	Staff Validator or Subcontractor
Data Review Step IIb			
Senior Review	Data validation reports	The HGL PC will approve of all validation results and final qualifiers and perform an overall evaluation of analytical performance against QAPP requirements.	PC
Overall Assessment	Project documentation (WS #33)	The HGL PM will perform a complete review of the project dataset and documentation and determine whether the sampling plan was executed as specified (that is, the number, location, and type of field samples were collected and analyzed as specified in the Work Plan). The HGL PM all will evaluate whether sampling procedures were followed with respect to equipment and proper sampling support (for example, techniques, equipment, decontamination, volume, temperature, and preservatives).	PM

The following provides a brief explanation of the data validation qualifiers assigned to results during the data review process by a data validator.

Qualifier	Definition
U	The analyte was not detected and was reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
J	The reported result was an estimated value with an unknown bias.
J+	The result was an estimated quantity, but the result may be biased high.
J-	The result was an estimated quantity, but the result may be biased low.
N	The analysis indicates the presence of an analyte for which there was presumptive evidence to make a "tentative identification."
NJ	The analyte has been “tentatively identified” or “presumptively” as present and the associated numerical value was the estimated concentration in the sample.
UJ	The analyte was not detected and was reported as less than the LOD or as defined by the customer. However, the associated numerical value is approximate.
X	The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project QC criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Acceptance or rejection of the data should be decided by the project team (which should include a PC), but exclusion of the data is recommended.

WORKSHEET #37
DATA USABILITY ASSESSMENT

Personnel who will be responsible for participating in the data usability assessment include:

- PM – Kimberly Vaughn
- PC – Joseph Vilain, Ph.D.
- Staff Chemist – Justin Hersh
- QC Manager – Theresa Rojas

The key component of the assessment will be the determination of the pre-excavation characterization soil sampling results being confirmed as definitive for cadmium and lead with LODs/LOQs sufficiently lower than their respective cleanup goals. The assessment will include an evaluation of the QC elements relating to precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) (both sample collection and analytical) described in this worksheet. The impact of any data gaps resulting from sampling incompleteness or rejected data will be evaluated in a data quality evaluation included as an appendix to the project report.

Evaluation activities will be documented in the QA reports listed in WS #29. An overall assessment of the impact of data usability issues will be presented in the project report. The usability assessment will evaluate the overall dataset generated during pre-excavation characterization soil sampling (including the observational technical approach consisting of laboratory sample hold and optional analysis to full define the horizontal and vertical extent of cadmium and lead contamination).

Waste characterization sample results will support off-site transportation and disposal determinations and are not subject to data validation or statistical evaluation. The results will be subject to data verification review for completeness; however, they are used as-is to prepare the necessary waste profiles, manifests, or bills-of-lading. The waste characterization sample results also will support the on-site management of the waste materials based on their classification as RCRA/Toxic Substances Control Act hazardous or non-hazardous.

It is the responsibility of the PC and the laboratory to ensure that the data meets the method DLs, reporting limits/minimum detected activities, and laboratory QC limits listed in this UFP-QAPP. During the data validation assessment, non-conformances are documented, and data is qualified for use in making decisions. Data gaps will be present if a sample is not collected, a sample is not analyzed for the requested parameters, or the data is determined to be unusable. The need for further investigation will be determined on a case-by-case basis, depending on whether data can be extrapolated from adjacent sampling locations or whether the results are unnecessary based on the results from adjacent locations. All data is usable as qualified by the data validator, except for data qualified for possible exclusion. Estimated and/or biased results are usable. Outliers, if present, can be addressed on a case-by-case basis.

Usability assessment process and procedures:

Compliance with measurement quality objectives for the DQIs (e.g., PARCCS) will be evaluated to support assessment of data usability. Data qualified with the "X" qualifier as excluded is evaluated by the project team, which includes the Project Chemist, to consider rejection. Data qualified with the "J" qualifier is considered estimated. The project team will determine if any bias that might be present in the qualified results affects the usability of the data for the intended purpose. Several different types of laboratory QC information (field and lab duplicates, MS/MSD, LCS, and etc.) will be used as multiple lines of evidence to understand the possible bias before concluding that data is usable for decision-making purposes.

Evaluative procedures used to assess overall measurement error associated with the project:

After all data evaluations are completed, any limitations on the use of data will be known and the limitations will be considered when making decisions. After data validation and an overall review of DQIs, the data will be reconciled with the DQOs to determine whether sufficient data of acceptable quality are available to make a decision.

The following is a summary of the usability assessment process and all procedures including interim steps and any statistics, equations, and computer algorithms that will be used:

The QC program to be used is designed to obtain DQIs for each field procedure and analytical method. The PARCCS criteria are the qualitative and quantitative indicators of data quality. An objective of this UFP-QAPP is to ensure that collected data are precise, accurate, representative, complete, and comparable to actual site conditions. QC results will be used to assess all project data. All affected project samples will be qualified if low/high-biased QC results are consistently reported. PARCCS criteria are defined as follows:

Precision

Precision measures the reproducibility of measurements. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Precision is evaluated by comparing the RPDs of field versus field duplicate, MS versus MSD, LCS versus LCSD – also known as blank spike – and blank spike duplicate against the limits established by the laboratory. The formula for the calculation of precision is:

$$\text{RPD}\,\% = \left(\frac{(X_1 - X_2)}{(X_1 + X_2)/2}\right) \times 100$$

Where:

X_1 = Concentration of analyte in sample, and
X_2 = Concentration of analyte in corresponding replicate/duplicate sample.

Precision is determined for analytical results using field and laboratory duplicates or duplicate MS samples. Precision measures the reproducibility of measurements under a given set of conditions.

Accuracy

Accuracy is the statistical measurement of correctness and includes components of random error (variability due to imprecision) and systematic error. A measurement is accurate when the reported value does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is measured by comparing the %R (organic and inorganic) of analytes spiked into LCSs and MS and/or MSD samples to the DoD QSM control limits. Recoveries outside the control limits indicate a cause other than normal measurement error. CA may include instrument recalibration, reanalysis of the QC sample, or reanalysis of the samples in the batch. For organic analyses, surrogate compound recoveries and tracer yields, respectively, are also used to assess accuracy and method performance for each sample analyzed. The calculation used for %R/yield is expressed as:

$$\text{Percent Recovery} = \frac{X - D}{D} \times 100$$

Where:

X represents the value/activity of the spike sample, and
D represents the spike concentration added.

Accuracy of analytical results reported in environmental samples also is measured against any contamination present in laboratory MBs and instrument blanks, as well as field blanks, such as trip and equipment rinsate blank samples. Frequency of sampling and analysis of laboratory and field blanks is specified on Worksheet #20.

The temperature receipt of the cooler is measured from a representative sample in the cooler (the temperature of the outside of a container measured with an infrared gun). The cooler temperature is recorded and reported by the laboratory for evaluation during data validation. CA for coolers received at temperatures outside the acceptance limits (< 6°C) may result in the qualification of results or resampling of affected samples.

Sensitivity

Sensitivity is the ability of the method or instrument to detect the contaminant of concern and other target compounds at the level of interest. Sensitivity is achieved for a majority of the methods by the use of a low-level calibration standard. For those methods that require a multi-level ICAL, the low-level calibration standard is spiked at or below the quantitation level specified on Worksheet #15. The criterion used to measure the performance of this QC sample is the ICAL acceptance criteria specified in the DoD QSM and summarized on Worksheet #24. The CA performed if the acceptance criterion is not achieved is summarized on Worksheet #24.

Representativeness

Representativeness is the degree to which data accurately and precisely expresses a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Although representativeness is a qualitative measurement, it is evaluated through a multistep process beginning with evaluation of precision and accuracy data. Project design (see Worksheets #14 and

#16) is one of the critical inputs that determine if the data collected is representative of the population sampled.

Representativeness of individual samples will be controlled by sample collection and handling in accordance with the requirements of Worksheets #14 and #16 and the HGL SOPs presented Appendix C. The sample containers and preservation methods presented in Worksheets #19 and #30 will be used to ensure that samples arriving at the laboratory retain the appropriate degree of representativeness. The temperature of each cooler delivered to the laboratory is measured on receipt using a representative sample in the cooler (the temperature of the outside of a container measured with an infrared gun). The cooler temperature is recorded and reported by the laboratory for evaluation during data validation. CA for coolers received at temperatures outside the acceptance limits (< 6°C) may result in the qualification of results or resampling of affected samples. The holding times presented in Worksheets #19 and #30 have been established to ensure that samples retain representativeness at the time of extraction and analysis.

Representativeness will also be assessed using field and laboratory blank samples. Field blank samples (TBs and EBs) will be collected at the frequency presented in Worksheet #20. An MB will be analyzed with each analytical or preparation batch (as appropriate to the analytical method) to determine potential contamination introduced during routine laboratory procedures. ICBs and CCBs will be analyzed as required by analytical methods. Acceptable performance of blank samples will provide evidence that compounds detected in the environmental samples are site-related and are not artifacts of shipping, storage, field procedures, or laboratory procedures.

Comparability

Comparability is a qualitative parameter that expresses the confidence with which one data set may be compared to another. This is prime concern when current data is being integrated with historical data. Comparability of data is maximized by SOPs in the field and laboratory, standardized analytical methods, and consistent units of measure. The laboratory shall make the necessary provisions to ensure the comparability of all data. These procedures include, but are not limited to, the use of standard units and reporting formats, the use of calculations as referenced in the methodology for quantitation, and the use of standard measures of accuracy and precision for QC samples.

Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount planned to be obtained under correct, normal conditions. In order to meet the needs of the data users, project data must meet the MPC for data completeness specified in UFP-QAPP Worksheets #12.

Completeness will be evaluated qualitatively and quantitatively. The qualitative evaluation of completeness will be determined as a function of all events contributing to the sampling event. This includes items such as samples arriving at the laboratory intact, properly preserved, and in sufficient quantity to perform the requested analyses.

The quantitative description of completeness will be defined as the percentage of QC parameters that are acceptable. The completeness criterion for this project is 100% for each analyte/matrix, particularly for perfluorooctanoic acid/perfluorooctanesulfonic acid/perfluorobutanesulfonic acid. Completeness will be calculated for each analyte in each matrix (UFP-QAPP Manual Section 2.6.2.6). Data validation and data quality assessment will determine the data that are valid (usable) and the data that are rejected or missing. The quantitative assessment of completeness will be calculated for each analytical method as the number of valid (usable) data points for the target analyte in samples of the subject matrix, divided by the total number of samples of the subject matrix analyzed for the target analyte. Following the completion of the analytical testing, the percent completeness for each target analyte in each matrix will be calculated by the following equation:

$$\text{Completeness (\%)} = (S/R) \times 100$$

Where:

S = Number of usable sample results
R = Number of samples collected for each parameter analyzed

The completeness acceptance criterion for samples collected in the field will be 100% of the quantity of samples planned for collection in the UFP-QAPP. CA may be implemented to re-collect samples where necessary and possible (e.g., modifying a planned sample location, sample jars broken during shipment). Laboratory notification of sample receipt and conditions will be used to evaluate, as soon as possible, whether any problems during sample shipment would necessitate recollection of samples.

REFERENCES

Army National Guard (ARNG), 2014. *Final Technical Memorandum: Land Uses and Revised Risk Assessment Process for the Ravenna Army Ammunition Plant (RVAAP) Installation Restoration Program,* Portage/Trumbull Counties, Ohio. February.

CB&I Federal Services LLC (CB&I), 2015. *Remedial Investigation Report for RVAAP-063-R-01 Group 8 MRS, Version 1.0,* Former Ravenna Army Ammunition Plant, Portage and Trumbull Counties, Ohio, Final. May.

Department of Defense (DoD), 2017. Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.1.

Department of Defense (DoD), 2019. General Data Validation Guidelines. Environmental Data Quality Workgroup. November 4.

Department of Defense (DoD), 2022. Data Validation Guidelines Modules 1, 2, 3, and 4. Environmental Data Quality Workgroup. Updated February 9.

Engineering-environmental Management, Inc. (e2M), 2008. *Final Site Inspection Report, Ravenna Army Ammunition Plant,* Ohio, Military Munitions Response Sites, Ravenna Army Ammunition Plant, Ohio. May.

Environmental Research Group (ERG), 2022. *Final Wetland Delineation Report,* Former Ravenna Army Ammunition Plant, Portage and Trumbull Counties, Ohio. November.

HydroGeoLogic, Inc. (HGL), 2019. *Final Feasibility Study for RVAAP-063-R-01 Group 8 Munitions Response Site, Version 1.0*. July.

HGL, 2020. *Final Proposed Plan for RVAAP-063-R-01 Group 8 Munitions Response Site, Version 2.0*. January.

Intergovernmental Data Quality Task Force (IDQTF), 2020. *Uniform Federal Policy (UFP) for Quality Assurance Project Plans, Munitions Response QAPP Toolkit*. Update 1. April.

Ohio Army National Guard (OHARNG), 2014. *Integrated Natural Resources Management Plan (INRMP) at the Camp Ravenna Joint Military Training Center,* Portage and Trumbull Counties, Ohio. December.

Ohio Army National Guard (OHARNG), 2018. *Environmental Procedures*, OHARNG Energy & Environmental Branch. January 25.

Ohio Environmental Protection Agency (Ohio EPA), 2004. *Director's Final Findings and Orders (DFFO) for RVAAP*, Division of Emergency and Remedial Response. June.

Science Applications International Corporation (SAIC), 2001. *Final Phase II Remedial Investigation Report for Winklepeck Burning Grounds*. April.

SAIC, 2010. Final Facility-Wide Human Health Cleanup Goals for the Ravenna Army Ammunition Plant Ravenna, Ohio. March.

SAIC, 2020. *Final Remedial Design Work Plan for RVAAP Load Lines 1, 2, 3, 4, and 12 (RVAAP-08 to 12).* October.

U.S. Department of Agriculture, Soil Conservation Service, in cooperation with Ohio Department of Natural Resources, Division of Land and Soils, and Ohio Agriculture Research and Development Center, 1978. *Soil Survey of Portage County*.

U.S. Army Corps of Engineers (USACE), 2004. *Archive Search Report for the Ravenna Army Ammunition Plant*, Ravenna, Ohio. June.

USACE, 2020. *Final Record of Decision for RVAAP-063-R-01 Group 8 Munitions Response Site, Version 1.0*. September.

U.S. Environmental Protection Agency (EPA), 2015. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Update V*.

APPENDIX A

FIGURES

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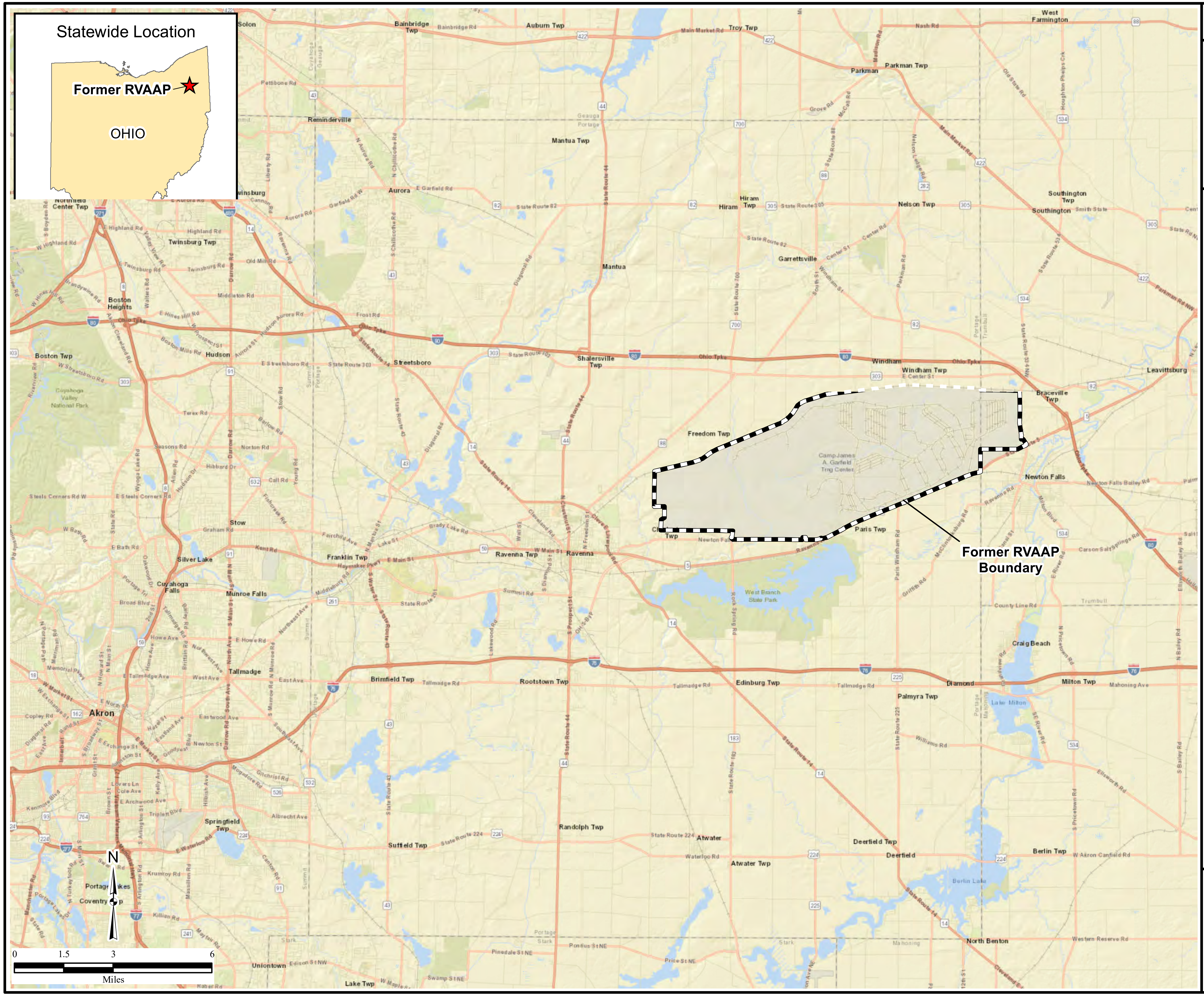
Figure 10.1
Former Ravenna Army Ammunition Plant Location



Legend

▭ Installation Boundary

Note:
RVAAP=Ravenna Army Ammunition Plant
UFP-QAPP=Uniform Federal Policy-Quality-Assurance Project Plan

\\srv-gst-01\HGLGIS\Ravenna_AAP_BM3001\Group_8\QAPP_Prelim\(10-01)Ravenna_Location_Map.mxd
3/3/2022 TA
Source: HGL,CB&I, USACE, e²M
ArcGIS Online World Street Map

Figure 10.2
Group 8 MRS
Site Location

Legend

● MPPEH (T-Bar Fuze)

Single Anomaly Results (2015 Remedial Investigation)

- ● MDAS
- □ Surface Metal
- ■ Other Debris
- ▽ Control Point (QC)

— Road

-+-+- Former Railroad

🟦 Standing Water

⬚ MRS

⬚ Installation Boundary

Notes:
MDAS=material documented as safe
MPPEH=material potentially presenting an explosive hazard
MRS=munitions response site
QC=quality control
RVAAP=Ravenna Army Ammunition Plant
UFP-QAPP=Uniform Federal Policy-Quality-Assurance Project Plan

*\\srv-gst-01\HGLGIS\Ravenna_AAP_BM3001\Group_8\QAPP_Prelim\
(10-02)Group8_MRS_Site_Location.mxd
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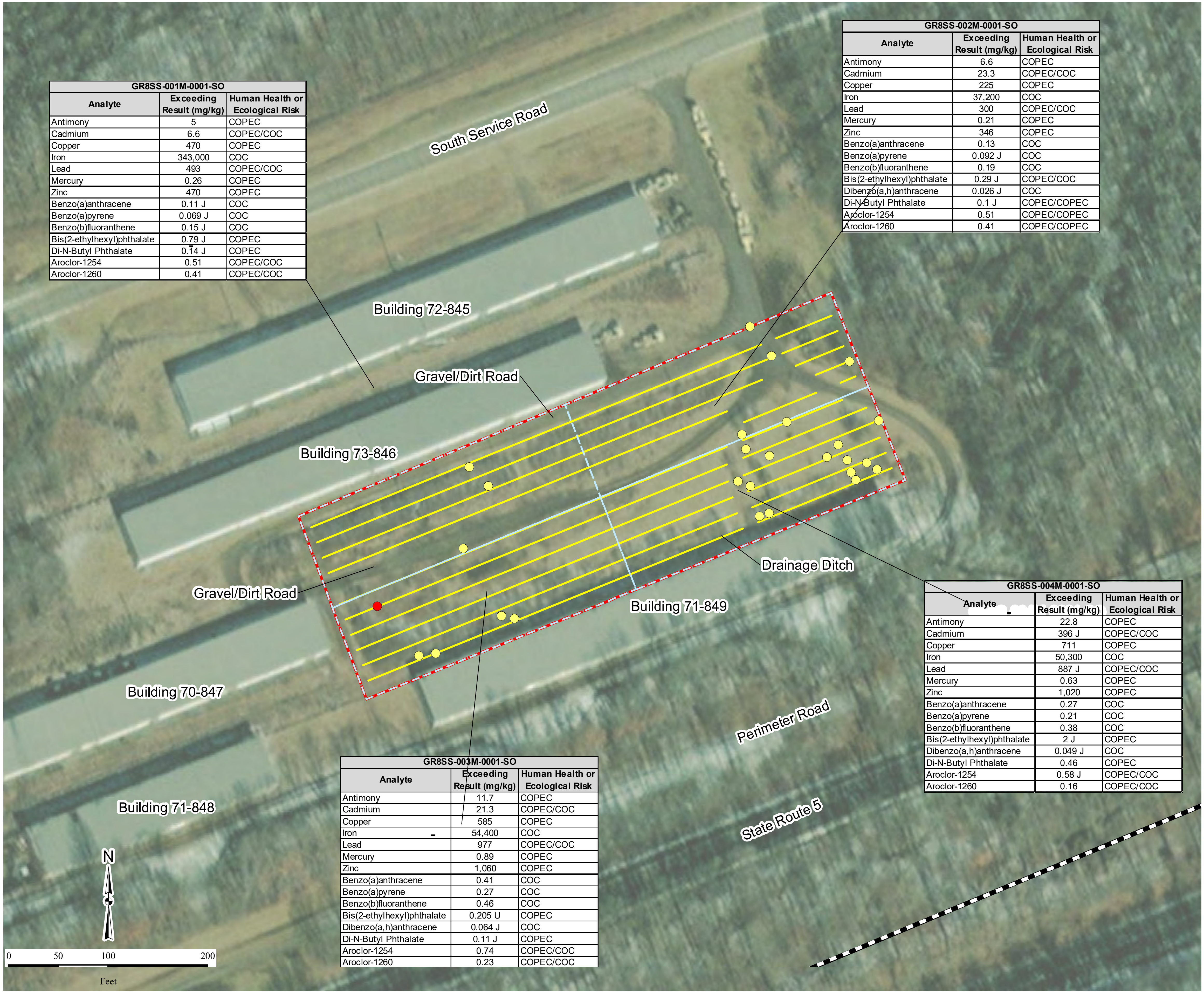
Source: HGL,CB&I, USACE, e²M
ArcGIS Online Imagery*

Figure 10.3
Group 8 MRS
Site Inspection Results



GR8SS-001M-0001-SO		
Analyte	**Exceeding Result (mg/kg)**	**Human Health or Ecological Risk**
Antimony	5	COPEC
Cadmium	6.6	COPEC/COC
Copper	470	COPEC
Iron	343,000	COC
Lead	493	COPEC/COC
Mercury	0.26	COPEC
Zinc	470	COPEC
Benzo(a)anthracene	0.11 J	COC
Benzo(a)pyrene	0.069 J	COC
Benzo(b)fluoranthene	0.15 J	COC
Bis(2-ethylhexyl)phthalate	0.79 J	COPEC
Di-N-Butyl Phthalate	0.14 J	COPEC
Aroclor-1254	0.51	COPEC/COC
Aroclor-1260	0.41	COPEC/COC

GR8SS-002M-0001-SO		
Analyte	**Exceeding Result (mg/kg)**	**Human Health or Ecological Risk**
Antimony	6.6	COPEC
Cadmium	23.3	COPEC/COC
Copper	225	COPEC
Iron	37,200	COC
Lead	300	COPEC/COC
Mercury	0.21	COPEC
Zinc	346	COPEC
Benzo(a)anthracene	0.13	COC
Benzo(a)pyrene	0.092 J	COC
Benzo(b)fluoranthene	0.19	COC
Bis(2-ethylhexyl)phthalate	0.29 J	COPEC/COC
Dibenzo(a,h)anthracene	0.026 J	COC
Di-N-Butyl Phthalate	0.1 J	COPEC/COPEC
Aroclor-1254	0.51	COPEC/COPEC
Aroclor-1260	0.41	COPEC/COPEC

GR8SS-004M-0001-SO		
Analyte	**Exceeding Result (mg/kg)**	**Human Health or Ecological Risk**
Antimony	22.8	COPEC
Cadmium	396 J	COPEC/COC
Copper	711	COPEC
Iron	50,300	COC
Lead	887 J	COPEC/COC
Mercury	0.63	COPEC
Zinc	1,020	COPEC
Benzo(a)anthracene	0.27	COC
Benzo(a)pyrene	0.21	COC
Benzo(b)fluoranthene	0.38	COC
Bis(2-ethylhexyl)phthalate	2 J	COPEC
Dibenzo(a,h)anthracene	0.049 J	COC
Di-N-Butyl Phthalate	0.46	COPEC
Aroclor-1254	0.58 J	COPEC/COC
Aroclor-1260	0.16	COPEC/COC

GR8SS-003M-0001-SO		
Analyte	**Exceeding Result (mg/kg)**	**Human Health or Ecological Risk**
Antimony	11.7	COPEC
Cadmium	21.3	COPEC/COC
Copper	585	COPEC
Iron	54,400	COC
Lead	977	COPEC/COC
Mercury	0.89	COPEC
Zinc	1,060	COPEC
Benzo(a)anthracene	0.41	COC
Benzo(a)pyrene	0.27	COC
Benzo(b)fluoranthene	0.46	COC
Bis(2-ethylhexyl)phthalate	0.205 U	COPEC
Dibenzo(a,h)anthracene	0.064 J	COC
Di-N-Butyl Phthalate	0.11 J	COPEC
Aroclor-1254	0.74	COPEC/COC
Aroclor-1260	0.23	COPEC/COC

Legend

●	MPPEH (T-Bar Fuze)
●	MDAS
—	Line Abreast Survey Area
⬚	Surface ISM Soil Sample Area (2015 RI Delineated MC Contamination)
⬚	MRS
⬚	Installation Boundary

HHRA COCs - Surface Soil Only
Resident Receptor
Aroclor-1254
Aroclor-1260
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Cadmium
Dibenzo(a,h)anthracene
Iron
Lead
National Guard Trainee
Cadmium
Lead

ERA COPECs - Surface Soil Only
Antimony
Aroclor-1254
Aroclor-1260
Bis(2-ethylhexyl)phthalate
Cadmium
Copper
Di-N-Butyl Phthalate
Lead
Mercury
Zinc
No habitat present onsite

Notes:
Surface soil defined as 0 ft bgs to 0.5 ft bgs.

COC=Chemical of Concern
COPEC=Chemical of Potential Ecological Concern
ERA=Ecological Risk Assessment
ft bgs=feet below ground surface
HHRA=Human Health Risk Assessment
ISM=incremental sampling method
J=estimated value
MC=munitions constituent
MDAS=material documented as safe
mg/kg=milligrams per kilogram
MPPEH=material potentially presenting an explosive hazard
MRS=munitions response site
RI=remedial investigation
RVAAP=Ravenna Army Ammuntion Plant
SI=site investigation
U=undetected
UFP-QAPP=Uniform Federal Policy-Quality-Assurance Project Plan

\\srv-gst-01\HGLGIS\Ravenna_AAP_BM3001\Group_8\QAPP_Prelim
(10-03)Group8_SI_MRS_Boundaries.mxd
12/8/2022 TH
Source: HGL,CB&I, USACE, e²M
ArcGIS Online Imagery

Figure 10.4
Group 8 MRS
2015 Remedial Investigation Results

Legend

Single Anomaly Results

- ● MDAS
- □ Surface Metal
- ■ Other Debris
- ▽ Control Point (QC)

Trench Results

- ▬ MDAS Identified
- ▬ MDAS and Other Debris Identified
- ▬ Other Debris Identified

- ▭ MRS
- ▭ Installation Boundary

Notes:
MDAS=material documented as safe
MRS=munitions response site
QC=quality control
RVAAP=Ravenna Army Ammuntion Plant
UFP-QAPP=Uniform Federal Policy-Quality-Assurance Project Plan

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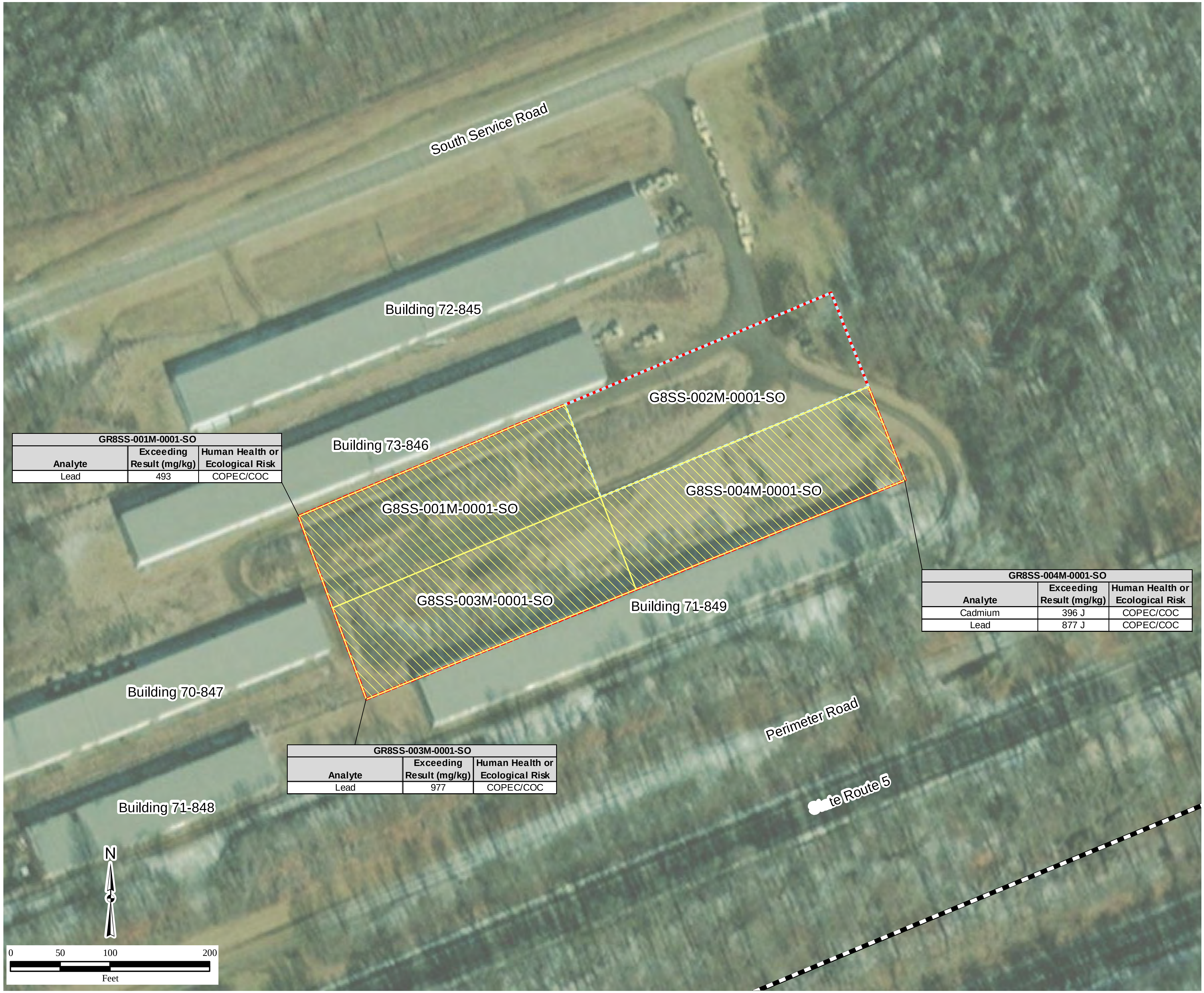
(10-04)Group8_2015_RI_Results.mxd
12/8/2022 TH
Source: HGL,CB&I, USACE, e²M
ArcGIS Online Imagery

Figure 10.5
2019 Feasibility Study
Risk Management Evaluation
Delineated MC Contamination



GR8SS-001M-0001-SO		
Analyte	Exceeding Result (mg/kg)	Human Health or Ecological Risk
Lead	493	COPEC/COC

GR8SS-004M-0001-SO		
Analyte	Exceeding Result (mg/kg)	Human Health or Ecological Risk
Cadmium	396 J	COPEC/COC
Lead	877 J	COPEC/COC

GR8SS-003M-0001-SO		
Analyte	Exceeding Result (mg/kg)	Human Health or Ecological Risk
Lead	977	COPEC/COC

Legend

▨	Excavation Area
⬚	Surface ISM Soil Sample Area
⬚	MRS
⬚	Installation Boundary

HHRA COCs - Surface Soil Only
Resident Receptor
Cadmium
Lead
National Guard Trainee (Commercial/Industrial Worker)
Cadmium
Lead
ERA COPECs - Surface Soil Only
Cadmium
Lead
No habitat present onsite

Notes:
Surface soil defined as 0 ft bgs to 0.5 ft bgs.

COC=Chemical of Concern
COPEC=Chemical of Potential Ecological Concern
ERA=Ecological Risk Assessment
ft bgs=feet below ground surface
HHRA=Human Health Risk Assessment
ISM=incremental sampling method
J=estimated value
MC=munitions constituent
mg/kg=milligrams per kilogram
MRS=munitions response site
RVAAP=Ravenna Army Ammuntion Plant
UFP-QAPP=Uniform Federal Policy-Quality-Assurance Project Plan

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(10-05)Group8_Delineated_MC.mxd
12/8/2022 TH
Source: HGL,CB&I, USACE, e²M
ArcGIS Online Imagery

Figure 10.6
Group 8 MRS
Site Topography

Legend

- — Topographic Contour (2ft Interval)*
- ▭ MRS
- ▭ Installation Boundary

Notes:
* Contours are dated 2016 and from Portage County GIS.
Values are in feet only those within the MRS are labeled.
MRS=munitions response site
RVAAP=Ravenna Army Ammuntion Plant
UFP-QAPP=Uniform Federal Policy-Quality Assurance Project Plan

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12/8/2022 TH
Source: HGL,CB&I, USACE, e^{2}M
ArcGIS Online USA Topo Maps

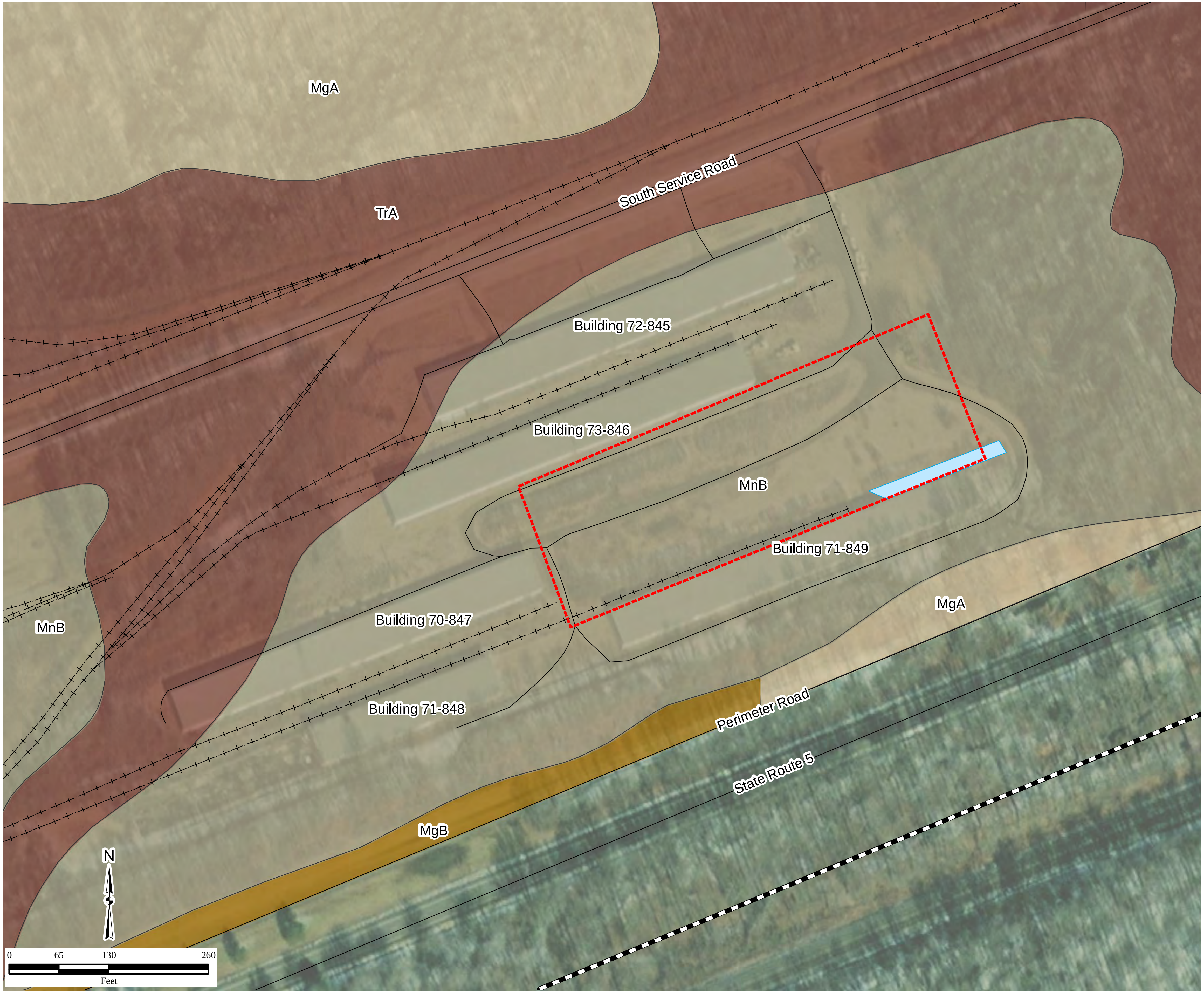

Figure 10.7
Group 8 MRS
Soil Types

Legend

—— Road

-+-+- Former Railroad

▭ Standing Water

▭ MRS

▭ Installation Boundary

<u>Soil Types</u>:

▨ MgA (Mahoning silt loam)

▨ MgB (Mahoning silt loam)

▨ MnB (Mahoning-Urban land complex)

▨ TrA (Trumbull silt loam)

Notes:
MRS=munitions response site
RVAAP=Ravenna Army Ammuntion Plant
UFP-QAPP=Uniform Federal Policy-Quality Assurance Project Plan

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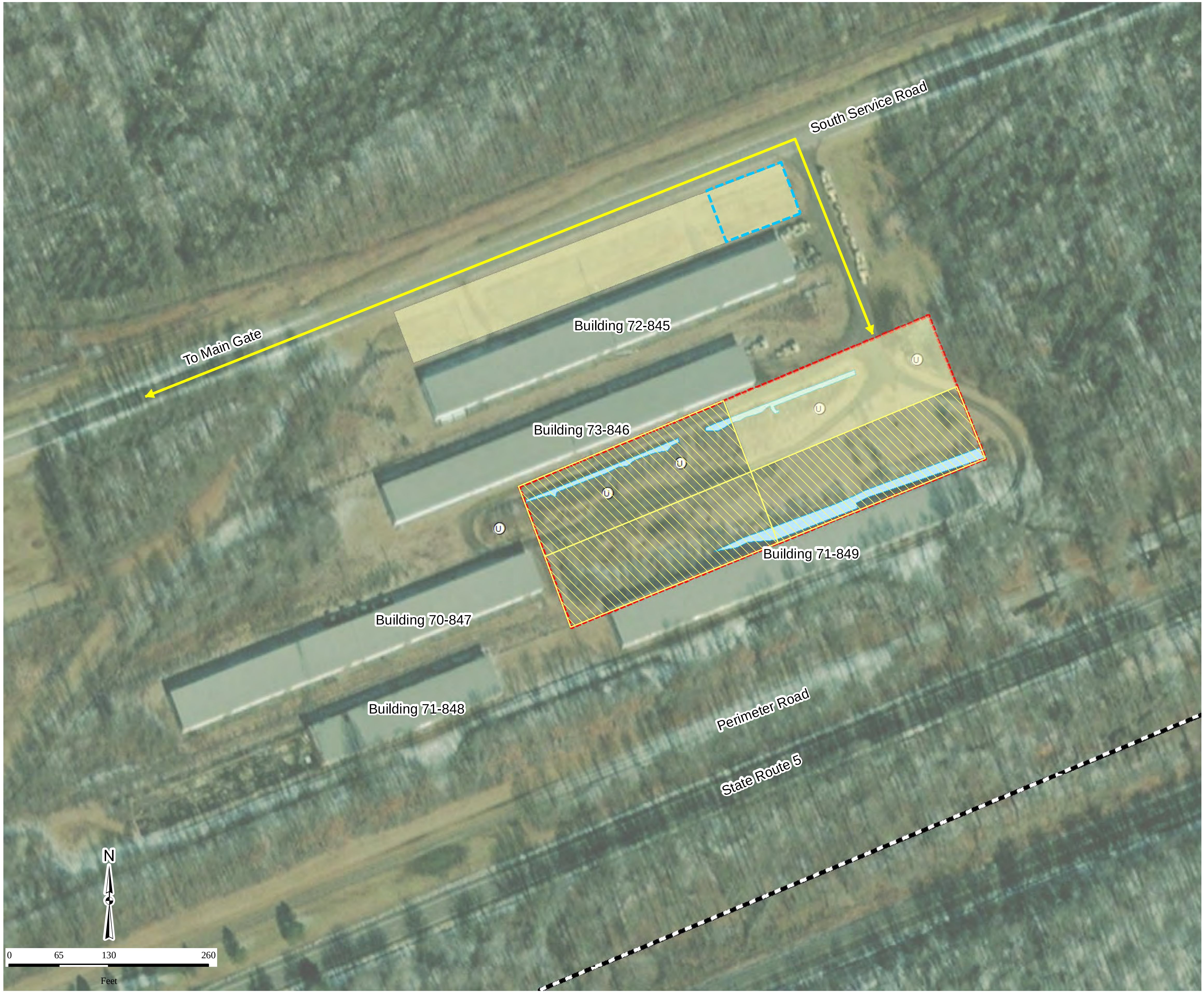
(10-07)Group8_Soils.mxd
12/8/2022 TH
Source: HGL, CB&I, Shaw, USACE, e^2M
ArcGIS Online Imagery

Figure 11.1
Group 8 MRS
Remedial Action Plan and Site Features



Legend

Ⓤ	Power Pole
↔	Construction/Haul Route
⬚	Temporary Storage and Portable Bathroom
▨	Excavation Area
🟨	Staging/Load-out Area
🟦	Ditch
⬚	MRS
⬚	Installation Boundary

Notes:
MRS=munitions response site
RVAAP=Ravenna Army Ammuntion Plant
UFP-QAPP=Uniform Federal Policy-Quality-Assurance Project Plan

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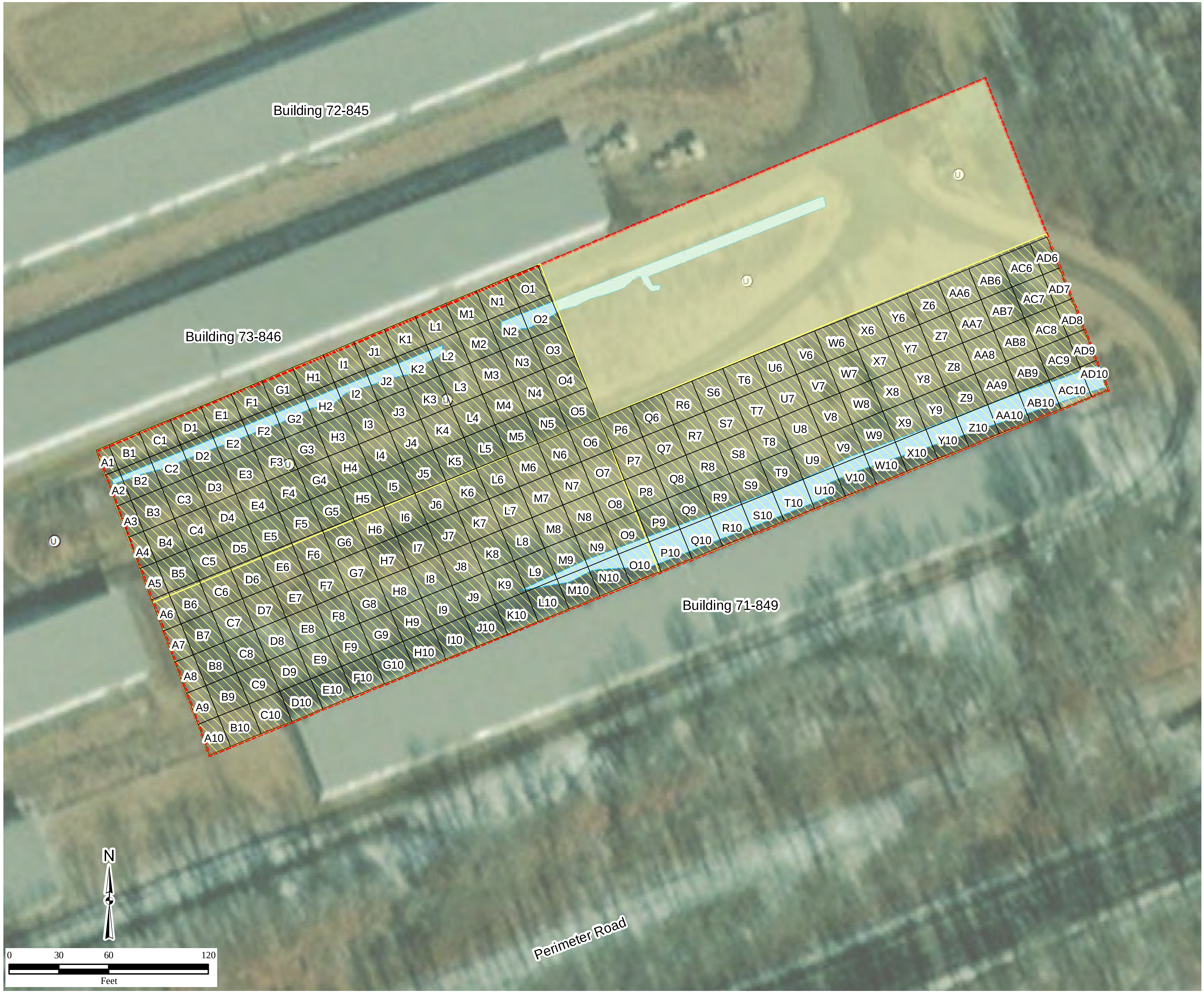
(11-01)Group8_MRS_Site_Features.mxd
12/22/2022 RR
Source: HGL,CB&I, USACE, e^2M
ArcGIS Online Imagery*

Figure 11.2
Group 8 MRS
Munitions Constituents Sampling Plan



Legend

- Ⓤ Power Pole
- ☐ Sampling Grid (20x20ft)
- ▨ Excavation Area
- 🟨 Staging/Load-out Area
- 🟦 Ditch
- ⬚ MRS

Notes:
MRS=munitions response site
RVAAP=Ravenna Army Ammuntion Plant
UFP-QAPP=Uniform Federal Policy-Quality-Assurance Project Plan

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12/9/2022 RR
Source: HGL,CB&I, USACE, e²M
ArcGIS Online Imagery

APPENDIX B

ENVIRONMENTAL PROTECTION PLAN

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APPENDIX B
ENVIRONMENTAL PROTECTION PLAN

REMEDIAL ACTION
FORMER RAVENNA ARMY AMMUNITION PLANT
RVAAP-063-R-01 GROUP 8 MRS
MUNITIONS RESPONSE SITE
PORTAGE AND TRUMBULL COUNTIES, OHIO

Contract No. W912DR-21-D-0005
Delivery Order No. W912DR21F0327

Prepared for

U.S. Army Corps of Engineers, Baltimore District
2 Hopkins Plaza
Baltimore, MD 21201-2930

Prepared by:
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Reston, Virginia 20190

July 2023

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LIST OF TABLES

LIST OF ATTACHMENTS

LIST OF ACRONYMS AND ABBREVIATIONS

APP	Accident Prevention Plan
BMP	Best Management Practice
CJAG	Camp James A. Garfield Joint Military Training Center
EPA	U.S. Environmental Protection Agency
EPP	Environmental Protection Plan
ESA	Endangered Species Act
HGL	HydroGeoLogic, Inc.
IAW	in accordance with
IDW	investigation-derived waste
INRMP	Integrated Natural Resources Management Plan
MDAS	material documented as safe
mg/L	milligrams per liter
MRS	Munitions Response Site
OHARNG	Ohio Army National Guard
PM	Project Manager
POL	petroleum, oil, and lubricant
PPE	personal protective equipment
RAWP	Remedial Action Work Plan
RCRA	Resource Conservation and Recovery Act
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SUXOS	Senior Unexploded Ordnance Supervisor
SWPPP	Stormwater Pollution Prevention Plan
TCLP	toxicity characteristic leaching procedure
USACE	U.S. Army Corps of Engineers
USC	U.S. Code
USDOT	U.S. Department of Transportation
USFWS	United States Fish and Wildlife Service
UXO	unexploded ordnance

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1.0 ENVIRONMENTAL PROTECTION PLAN

This Environmental Protection Plan (EPP) describes the approach, methods, and operational procedures that will be employed to protect the natural environment during performance of all remedial action field tasks associated with the Remedial Action at Camp James A. Garfield Joint Military Training Center (CJAG) Group 8 Munitions Response Site (MRS). The EPP addresses the potential impacts that the proposed actions may have on the surrounding environment and suggests measures to be implemented during the proposed actions to protect identified environmentally sensitive areas.

1.1 PLAN OBJECTIVES

The plan also identifies environmental management controls that will prevent and/or decrease the environmental impact in and around the project location. The objectives of this plan are to:

- Define methods and procedures to minimize the polluting of air, water, and land resources;
- Protect identified, environmentally sensitive cultural and/or historical resources; and
- Execute the field activities in the project work area in accordance with (IAW) all applicable federal, state, and local regulations.

1.2 COMPLIANCE AND REQUIREMENTS

The Contractor, HydroGeoLogic, Inc., (HGL) will follow all applicable regulations and obtain all necessary permits concerning environmental protection, pollution control, and abatement necessary for the proposed field operations. Applicable statutes may include, but are not limited to, the following:

- Clean Water Act, Title 33 U.S. Code (USC) §1344
- Wetlands Mitigation-Compensation Policy and Supplemental Guidelines
- Resource Conservation and Recovery Act, Title 42 USC §6969 et seq.
- Clean Air Act National Ambient Air Quality Standards Particulates, Title 42 USC §7401
- Fish and Wildlife Coordination Act (16 USC 661)
- Migratory Bird Treaty Act (16 USC 703)
- Endangered Species Act (ESA) (16 USC 1531, 50 Code of Federal Regulations 402)
- ESA of 1973 (16 USC 1536)
- Sikes Act Improvement Act (16 USC 670a et seq.)
- AR 200-1, Environmental Protection and Enhancement
- Bald and Golden Eagle Protection Act of 1940 (16 USC 668-668d, 54 Stat. 250)
- U.S. Department of Defense Instruction 4715.03, Natural Resources Conservation Program

2.0 POLLUTION MINIMIZATION METHODS

2.0.1 Based on the nature of the vegetation clearing and intrusive site work to be conducted the environmental impacts to land, air, or water will be minimized using the methods described in this EPP. The vegetation clearance will include mowing (if needed) and the removal of small trees/bushes from the drainage ditch and will be minimized to extent possible. The *Wetland Delineation Report* (ERG, 2022) indicates that the drainage ditches located along the southern MRS boundary do not discharge to any surface water feature, and any standing water infiltrates into the underlying soil. Based on publicly available aerial photography from November 2021, the area subject to clearing of small trees and brush along the drainage ditch will cover approximately 0.5 acres north of Building 71-849. No trees greater than 4 inches in diameter will be removed unless specifically approved by CJAG Environmental Office. The remainder of the MRS appears to be mowed intermittently by CJAG and should only require additional mowing (if needed) which will not result in significant ground disturbance.

2.0.2 As the expected disturbance area dimensions total more than 1 acre in size, a Stormwater Pollution Prevention Plan (SWPPP) is required to address stormwater and erosion controls. The SWPPP has been developed and is included as an attachment to this EPP. After completion of the pre-excavation soil characterization sampling the SWPPP will be revised, if necessary, to include the final planned excavation dimensions and specific locations of the applicable stormwater and erosion controls.

2.0.3 Additionally, stormwater and erosion Best Management Practices (BMPs) will be utilized around the general work area and MRS. The BMPs will include silt fence and straw wattles to limit potential sediment migration into portions of the drainage ditch located outside of the final planned excavation areas. The drainage ditch also may be blocked using clean soil to prevent surface water and sediment migration to the southwest beyond the MRS boundary (if actual field conditions warrant).

2.0.4 Other than during the possible disposal of an unexploded ordnance (UXO) item by detonation, noise is not anticipated to be a concern. Dust levels will be monitored visually by the Site Safety and Health Officer (SSHO). If project personnel recognize an increase in pollution potential, the work will be stopped temporarily, and the U.S. Army Corps of Engineers (USACE) and CJAG will evaluate the situation to take the appropriate steps to mitigate environmental impacts.

3.0 INITIAL ENVIRONMENTAL SURVEY

3.0.1 Prior to beginning site activities, the Senior UXO Supervisor (SUXOS) will coordinate with the CJAG Environmental Department Biologist, the Contractor's Project Manager (PM), the SUXOS and the SSHO to assess the site and develop a layout plan of the operating area to document conditions of areas in and adjacent to the site of the work, vegetation and brush cutting, equipment storage areas, and access routes. The following items will be identified on the layout plan: vegetation approved for cutting, endangered and protected species or habitats, and cultural or historical resource areas.

3.0.2 During the initial site assessment, photographs of the site and the surrounding area will be taken to document conditions prior to work activities. This includes taking generally representative photographs of the site and photographs of areas that will be used for administrative support, storage, and/or stockpiles. The photographs will aid in restoring the area to its original conditions after work activities are completed. Where environmentally sensitive areas are known to be present, the project team will assess the environmental impact and discuss any procedures, equipment, or training which may be required to mitigate/eliminate any adverse impact. This initial site assessment will be conducted at the beginning of field work activities.

3.0.3 Memoranda for Record, reviewing the possible effects on cultural/historical resources and on threatened/endangered species and habitats resulting from the proposed activities for this project, have been issued by the Ohio Army National Guard (OHARNG). These memoranda concluded that no cultural/historical resources are present and therefore affected by project activities; because habitat for the Northern Long-eared Bat, a federally listed threatened species, is present within the Group 8 MRS, vegetation clearing activities will be conducted as described in Section 3.2.4.2 of this EPP, to minimize adverse effects. The Memoranda for Record are included as Attachment B3.

3.1 IDENTIFICATION AND LOCATION OF KNOWN NATURAL RESOURCES

A discussion of the existing environmental conditions and natural resources known to be present at the Group 8 MRS is presented in the following sections.

3.1.1 Federally or State Threatened or Endangered Plant and Animal Species

According to the CJAG Integrated Natural Resources Management Plan (INRMP) and U.S. Fish and Wildlife Service (USFWS) Threatened and Endangered Species List, there is one federally listed threatened species (Northern Long-eared Bat) that is known to reside within CJAG. Additionally, 12 state-listed endangered species (1 mammal, 1 fish, 1 insect, 8 bird, and 1 plant species) and 10 state-listed threatened species (5 bird, 1 insect, 4 plants species) are included on the Camp Ravenna Rare Species List. Six federally listed species are known to occur within in Portage and Trumbull Counties, Ohio to include the threatened northern monkshood, the endangered Mitchell's satyr butterfly, the endangered clubshell, the endangered Indiana bat, threatened northern long-eared bat, and threatened eastern massasauga. Table B.1-1 identifies Federally listed Plant and Animal Species within Portage and Trumbull Counties, Ohio. No confirmed sightings of these species within the Group 8 MRS have been reported and no critical habitats are present within the MRS. No federally listed endangered species have been identified within CJAG.

3.1.2 Migratory Birds

3.1.2.1 As summarized in the CJAG INRMP dated December 2014, CJAG is in compliance with the Migratory Bird Treaty Act. The Department of Defense has a memorandum of understanding with USFWS for protection of migratory birds. Over 214 species of birds are known to migrate through, stop or call home within CJAG. The Forest Management Program has been able to perpetuate large, quality forested habitat by controlling harvesting/logging to small selective areas. No impact has been observed; therefore, no firm restrictions have been placed on harvesting during the breeding season. However, because of the listed bat species, harvesting will be avoided between April and September.

3.1.2.2 The potential impacts to migratory birds were evaluated as part of this EPP. The field activities are anticipated to be completed in 2023. No potential impact to migratory birds is anticipated.

Table B-1.1
Federal Threatened and Endangered Plant and Animal Species

Common Name	Scientific Name	Federal Status	Image
Northern monkshood	*Aconitum noveboracense*	Threatened	
Mitchell's satyr butterfly	*Neonympha mitchellii*	Endangered	
Clubshell	*Pleurobema clava*	Endangered	

Table B-1.1 (Continued)
Federal Threatened and Endangered Plant and Animal Species

Indiana bat	*Myotis sodalis*	Endangered	
Northern long-eared bat	*Myotis septentrionalis*	Threatened	
Eastern massasauga	*Sistrurus catenatus*	Threatened	

3.2 PROTECTION OF NATURAL RESOURCES

Field activities outlined in this Uniform Federal Policy-Quality Assurance Project Plan (see Worksheet #17) will be conducted in a manner to minimize impacts to the natural resources present at the MRS. Additional information for specific natural resources and plans for their protection are detailed below.

3.2.1 Project Site Monitoring

3.2.1.1 For evaluation of potential habitats, the CJAG Environmental Office will evaluate the project site along with the SUXOS ahead of the field activities. Site-specific input will be provided to onsite field teams prior to beginning field activities regarding the importance of endangered species, identification and avoidance of endangered species and general site-specific concurrence on planned vegetation and brush cutting. Any identified threatened and endangered species present within the project area are not expected to be impacted.

3.2.1.2 In the event that unanticipated threatened or endangered species are encountered, work will halt within 150 yards of the sighting or habitat identified and the field team will notify the SUXOS. The SUXOS will immediately report the find to the HGL PM, and the CJAG and USACE team will be notified.

3.2.2 Wetlands

Wetlands are saturated areas where water is present all the time or for varying periods of time during the year. Wetlands provide habitat for waterfowl, fish, other terrestrial and aquatic animals, and a wide variety of plant life. Wetlands also provide resting and feeding places on migration routes, as well as food, shelter, breeding areas, and nurseries for many species. Wetlands protection is mandated by Executive Order 11990. A jurisdictional wetlands delineation was performed at the Group 8 MRS in April 2022 (ERG, 2022). While the two ditches on the southern boundary of the MRS exhibit wetland characteristics, they were determined to not be jurisdictional due to the lack of connectivity to traditional navigable waters, their artificial nature, and ephemerality. No jurisdictional wetlands were identified in the project area.

3.2.3 Water Resources

Water resources and floodplain protection is mandated by Executive Order 11988. Based on available aerial photography, no water resources, defined as sources of water useful or potentially useful to humans for agricultural, industrial, household, or recreational use, are expected to be impacted by the project. There are no major surface water bodies or navigable waters in the area of the MRS and the surface water protection measures described in Section 4.2.9 will be used.

3.2.4 Trees and Shrubs

3.2.4.1 The Group 8 MRS is situated in a flat, relatively open grassy area. Small trees and brush are present along the drainage ditch located along the southern MRS boundary north of Building 71-849. Based on publicly available aerial photography, the grassy portions of the MRS are mowed occasionally by CJAG. The habitat at the Group 8 MRS has been influenced and impacted by man-made improvements, including gravel roads. Additionally, historical use of the Group 8 MRS as a burning area has also influenced the habitat at the site. The vegetation community present at the Group 8 MRS is categorized as "other land".

3.2.4.2 Brush clearing will be minimized to the extent possible and will only include excavation areas and adjacent areas needed for equipment access. No trees greater than 4 inches in diameter will be removed unless specifically approved by Camp Ravenna Environmental Office. Trees will only be cut between October 1st and March 31st, as required to avoid impacting the nesting season of the Northern Long-eared Bat. Woody debris generated during vegetation clearing will be chipped on-site, with the chips transported to Dan Gallagher Farms Mulch & Sawdust for recycling as mulch.

3.2.5 Cultural or Historical Resources

3.2.5.1 There are no known cultural or archaeological resources that could be impacted by the planned work. There are no historical buildings or other structures within the MRS. Cultural or archaeological surveys have not been completed for the Group 8 MRS; however, due to the historic nature of former activities, cultural/archaeological resources are unlikely to exist.

3.2.5.2 If cultural or archaeological resources are identified prior or during on-site activities, they will be marked and avoided, with coordination with CJAG Environmental Department, during completion of the project. If any cultural or archaeological materials or resources are discovered within the project area, the SUXOS/SSHO will immediately report the find to the PM so that USACE and CJAG can be notified. Site work will be suspended and will resume only after obtaining approval from USACE. The geographic information system data files or imagery depicting locations of cultural or archaeological resources will not be released to the public without permission from CJAG. Attachment B1 to this EPP presents the Inadvertent Discovery Standard Operating Procedures provided by CJAG, which details the necessary steps to be followed in such an event.

4.0 SITE-SPECIFIC MITIGATION MEASURES

4.1 WASTE DISPOSAL

All waste generated will be properly characterized and disposed of IAW all applicable regulations and through approved channels. It is expected that only uncontaminated trash will be generated as a result of this project. Uncontaminated trash will be stored in plastic trash bags for disposal as municipal solid waste through a local trash disposal facility.

4.1.1 Non-Hazardous Waste

4.1.1.1 The anticipated non-hazardous waste streams resulting from on-site activities include:

- Soil cuttings from pre-excavation sampling;
- Excavated soil;
- Metal debris classified as material documented as safe (MDAS);
- Wastewater from soil sampling and excavation equipment decontamination;

- Wastewater from excavation dewatering; and
- Municipal trash.

4.1.1.2 Soil cuttings generated during pre-excavation soil characterization sampling activities will be containerized in 55-gallon drums and managed as investigation-derived waste (IDW). One composite waste characterization sample will be collected from the soil cuttings drums and submitted for off-site laboratory analysis of the required Waste Acceptance Criteria parameters. If the soil cuttings are classified as Resource Conservation and Recovery Act (RCRA) non-hazardous waste they, will be stored at a location approved by CJAG until they can be added to and disposed along with similarly-classified excavated soil. Non-hazardous waste soil will be disposed at U.S. Ecology in Detroit, Michigan. Metal debris classified as MDAS will be collected and disposed off-site for recycling at Demil Metals located in Northbrook, Illinois.

4.1.1.3 Wastewater will be generated during soil sampling and excavation equipment decontamination activities. Decontamination wastewater will be containerized in 55-gallon drums and characterized in the same manner described previously for soil cuttings. Although not anticipated, wastewater generated due to excavation dewatering will be handled in the same manner as decontamination wastewater. Non-hazardous wastewater will be disposed at Clean Water Environmental in Dayton, Ohio.

4.1.1.4 Municipal trash will include expended personal protective equipment (PPE) and general trash generated by field personnel during on-site activities. Municipal trash will be containerized and transferred to a dumpster procured by HGL for disposal at a local municipal landfill.

4.1.1.5 Any IDW may include PPE and sampling equipment consumables to conduct field activities. Scrap metal may be generated as a result of the investigation of metallic geophysical anomalies. Nonhazardous IDW such as non-munitions related metal scrap will be collected and properly disposed of. It is expected that solid IDW (e.g., rubber gloves or other protective PPE, acetate split spoon sleeves) will be collected separately in trash bags and disposed of as municipal solid waste. Chipped trees and brush generated during vegetation clearing will be transported off-site at the Dan Gallagher Farms Mulch and Sawdust facility, in Manuta, Ohio for recycling as mulch.

4.1.2 Hazardous Waste

Although the generation of hazardous waste is not anticipated, elevated concentrations of cadmium and/or lead in soil could result in a portion of the soil cuttings and/or excavated soil being classified as RCRA hazardous waste per their associated toxicity characteristic leaching procedure (TCLP) metals regulatory limits of 1 milligram per liter (mg/L) (cadmium) and 5 mg/L (lead). The results of the pre-excavation soil characterization sampling will provide total concentrations and TCLP results for the final planned excavation areas. The availability of the pre-excavation data will assist in the appropriate management of excavated soil during on-site activities. Hazardous waste soil cuttings and/or excavated soil will be disposed at U.S. Ecology in Detroit, Michigan. Appropriate on-site housekeeping practices will be maintained during the course of the project. All wastes generated during on-site activities will be managed and transported in accordance with applicable U.S. Department of Transportation (USDOT) regulations and disposed at appropriately-licensed facilities. Any transporter of hazardous waste will need to be approved by the Defense Logistics

Agency Disposition Services. Should any waste be characterized as hazardous, the following steps will have to be followed:

- 30-day notification prior to waste generation;
- 60-day period to remove all hazardous waste from the site; and
- Contractor hazardous waste training is required.

4.1.3 Packaging, Labeling, Storage, and Disposal

4.1.3.1 As mentioned previously, non-hazardous soil cuttings generated during pre-excavation characterization soil sampling will be labeled IAW applicable regulations and stored at a location approved by CJAG. For disposal the soil cuttings will be added to the excavated soil. If the soil cuttings cannot be stored at the facility, then they will be transported and disposed at an appropriately-licensed off-site facility. Although not anticipated, hazardous soil cuttings will be disposed at an appropriately-licensed off-site facility as soon as possible after generation.

4.1.3.2 As described previously, chips generated during vegetation clearing will be transported to an off-site facility (Dan Gallagher Farms Mulch and Sawdust, or similar facility) for recycling as mulch. Municipal trash will be containerized in the MRS and the project staging area as appropriate and will be transported to an off-site municipal landfill for disposal. These materials will be non-hazardous debris and will not require specialized containerization, labelling, or transport.

4.1.3.3 Any waste generated by the field teams will be collected, stored, and labeled IAW applicable regulations. In general, all project waste (excluding vegetation chips and municipal trash) will be containerized and labelled as “Analysis Pending” waste until characterization laboratory results are available and reviewed. Excavated soil assumed to be hazardous based on pre-excavation characterization soil sampling will be labelled and managed per RCRA requirements immediately upon generation. The transportation and disposal of all project wastes will be conducted in accordance with USDOT and Ohio regulations by the HGL subcontractor.

4.1.4 Manifesting and Transporting Wastes

If generated, the transportation of hazardous waste will require the completion of a disposal facility waste profile and for each waste stream and a transportation manifest for each shipment. Additional Ohio regulations may necessitate additional sampling of hazardous waste by the disposal facility. Non-hazardous waste transportation will require a waste profile for each waste stream (excluding vegetation chips and municipal trash) and a non-hazardous waste manifest for each shipment. Regardless of waste type or classification, transportation will be conducted by a licensed subcontractor in accordance with applicable USDOT regulations. Documentation of receipt/acceptance will be provided by each disposal facility (excluding municipal trash).

4.1.5 Compliance with USDOT Shipping Regulations

Transportation of all wastes and materials will be conducted IAW applicable USDOT regulations, including use of labels and placards, and documentation of transportation. A OHARNG representative will review and certify any requested waste profiles and manifests. An HGL representative will sign the necessary manifests related to the disposal of any MDAS.

4.2 OTHER MATERIALS AND ACTIVITIES

4.2.1 Security of Hazardous Materials

4.2.1.1 Field team personnel will provide security during working hours for the work area. Small quantity hazardous materials may include, but not be limited to:

- Lubricants
- Hydraulic fluid
- Marking paint
- Insect repellant
- Gasoline/diesel fuel

4.2.1.2 These items will be securely stored during working hours, either inside field vehicles or within the field office. HGL will maintain an inventory list of these materials and Safety Data Sheets will be accessible to field team personnel. Any hazardous materials related to equipment used for vegetation and tree cutting (e.g., fuel, motor oil, etc.) will be kept in USDOT-approved containers and stored in the field vehicles (i.e., in a truck bed-mounted fuel tank, or appropriate portable flammables containers compliant with Occupational Safety and Health Administration and National Fire Protection Association requirements).

4.2.2 Burning Activities

No burning is allowed, nor will be conducted, during remedial activities.

4.2.3 Dust and Emission Control

U.S. Environmental Protection Agency (EPA) has established National Ambient Air Quality Standards pursuant to Sections 109 and 301(a) of the Clean Air Act. These standards, expressed in micrograms per cubic meter, establish safe concentration levels for each criteria pollutant. Standards have been set for six pollutants: particulate matter, sulfur dioxide, carbon monoxide, nitrogen dioxide, ozone, and lead. The proposed project would result in a minimal amount of air pollution in the form of smoke and fugitive dust emissions. Smoke and fugitive dust emissions may result from support vehicles; however, impact from these operations should not be significant and should not have any long-term environmental impact on air quality. Excavation and fill activities may result in increased concentrations of airborne dust. The SSHO will monitor dust levels visually and initiate mitigation measures (e.g., water spraying), as necessary.

4.2.4 Noise Control and Prevention

It is expected that mechanical equipment (e.g., excavator, trucks, skid steer, vegetation clearing equipment) will be the primary source of noise on this project. The field team will minimize the noise emissions from this equipment to the extent possible by ensuring that trucks and equipment are properly functioning. The lack of any residential areas within close proximity of the MRS will eliminate any nuisance noise impacts. Noise monitoring, action levels, and hearing protection requirements for site workers are addressed in the Accident Prevention Plan (APP)/Site Safety and

Health Plan (SSHP). Brush clearing will not be conducted from April thru September to avoid negative impacts to nesting birds and roosting bats.

4.2.5 Spill Control and Prevention

4.2.5.1 Spill Potential

The most likely sources of spills during planned activities include equipment fueling, hydraulic or fuel line failure/leaks, and liquid IDW container failure/leaks. Equipment refueling and addition of lubricants, oil, hydraulic fluids will be conducted using portable containers or dedicated fuel trucks. An inventory of hazardous materials and chemicals present on site will be maintained and associated Safety Data Sheets will be available on site as described in the APP/SSHP.

4.2.5.2 Spill Control Measures

4.2.5.2.1 Equipment refueling will be conducted using ground containment (plastic trays, plastic sheeting) and/or absorbent pads to capture small quantities of spilled liquids. Spilled liquids on equipment surfaces will be cleaned with absorbent pads. Care will be taken by site workers to avoid overfilling liquid reservoirs and tanks.

4.2.5.2.2 A spill kit will be present in the MRS, project staging area, or at other locations where the storage or handling of hazardous/flammable liquids is conducted. Spill kits will be commercially manufactured and suitable for the specific materials used (e.g., universal, oil, fuel). If any significant leak (e.g., leaking or ruptured lines or tanks) is observed, operation will cease immediately to reduce flow rate and allow for workers to initiate containment and cleanup. CJAG Range Control and the OHARNG Environmental Office will contacted as soon as possible after a spill and reporting will include the use of the First Responder Reporting Form provided in Attachment B2 within 24 hours. This form will be present in each work area and in each support vehicle while on site.

4.2.5.3 Spill Prevention

4.2.5.3.1 Vehicles will be maintained in good operating condition and left running only when necessary. Project support vehicles will be fueled, maintained, and serviced at an off-site location. Routine cleaning or washing of support vehicles or equipment (excluding decontamination) will not be permitted on site.

4.2.5.3.2 Power equipment and vehicles will be inspected a minimum of once daily, and continually during use, to identify petroleum, oil, and lubricant (POL) leaks. Liquid IDW containers will be inspected at least once daily, and upon each use, to identify potential leaks. Any soil visibly impacted by a POL or IDW fluid leak will be removed, containerized, and disposed as IDW.

4.2.5.3.3 When refueling vehicles or equipment, the following measures will be taken to minimize the potential for and impact from any spills:

- A spill pan will be placed beneath the vehicle/equipment fuel inlet during fueling or refueling.
- The operator will not leave the fueling operation, will be present, and will continually monitor the activity throughout the time it takes to complete the operation.
- When the vehicle/equipment is full, the fuel flow will be suspended and the nozzle will be carefully removed from the equipment to ensure no fuel drips from the nozzle.

4.2.5.3.4 Safety cans or other approved portable service containers of flammable liquids having a flash point at or below 73 degrees Fahrenheit will be clearly labelled. Drums, barrels, and flammable-liquid containers will be tightly capped. Project IDW and chemicals will be stored in a manner to prevent contact with stormwater, including the use of tarpaulins and/or storage under a roofed structure.

4.2.5.3.5 All equipment/waste transport drivers will be trained in USDOT and EPA spill prevention measures. The transport driver will be required to remain on duty and with the truck during loading/filling operations to protect against spills. Site workers will be trained on spill prevention, containment, and retrieval methods at the start of work. In the event of a spill of any size, CJAG spill response guidelines and reporting procedures must be followed. Attachment B2 includes Form 200-1-24 – First Responder Reporting, which will be kept in each field vehicle. All spills must be reported to Range Control and to the OHARNG Environmental Office.

4.2.5.4 <u>Large Spill Response</u>

4.2.5.4.1 A large spill that will require immediate reporting to CJAG will be defined as one that meets one or more of the following criteria:

- Spills of 5 gallons or more of fuel or oil;
- Spills covering an area of 5 feet or larger;
- Spills entering a waterway, storm drain, sewer system, or surface water; or
- Spills posing a threat to public health or welfare.

4.2.5.4.2 CJAG Range Control and the OHARNG Environmental Office will contacted as soon as possible after a spill and reporting will include the use of the CJAG First Responder Reporting Form provided in Attachment B2 within 24 hours. This form will be present in each work area and in each support vehicle while on site.

4.2.5.5 <u>Other Spill Response</u>

4.2.5.5.1 For a spill that does not qualify as a large spill, response actions will include the following:

- Contain or confine the spill by placing adsorbent materials around the site;
- Immediately clean up spills including any fuel leakage, oil drips, or hydraulic line rupture that may occur during the operation of trucks, heavy equipment, etc.; and
- Collect and containerize the spilled medium and materials.

4.2.5.5.2 CJAG Range Control and the OHARNG Environmental Office will contacted as soon as possible after a spill and reporting will include the use of the First Responder Reporting Form provided in Attachment B2 within 24 hours. This form will be present in each work area and in each support vehicle while on site.

4.2.5.6 Spill Mitigation Equipment

The following spill mitigation equipment will be available on site for use during the removal actions:

- Drip pans;
- Shovels;
- 55-gallon drums (for containerization); and
- Spill kit consisting of sorbent pads and personal protective equipment.

4.2.6 Storage Areas and Temporary Facilities

The Contractor anticipates establishing a temporary field office and storage area to support operations required during this project. Upon project completion, all temporary facilities and associated debris will be removed from the site.

4.2.7 Access Routes

4.2.7.1 Crews entering and exiting the work sites will use the existing gravel roads in the vicinity of the MRS. Off-road vehicle and equipment travel outside the MRS boundary is not anticipated.

4.2.7.2 If the need for significant off-road travel or operations is identified, CJAG and the USACE will be notified and approval obtained. In general, the following measures will be taken to minimize the environmental effects from vehicle and equipment travel within and around the MRS:

- Any ruts or tracks created by field activities will be restored. The ruts will be filled in and leveled to maintain positive drainage off the roads.
- Site workers will avoid driving equipment and vehicles into wet, unstable, or contaminated areas.
- Vehicles and equipment will remain on existing roads and driveways to a point as close to the site or work area as possible, and site workers will walk the remaining distance to the work area.

4.2.8 Trees and Shrubs Protection and Restoration

There are no known sensitive trees or shrubs in the MRS. No trees larger than 4 inches in diameter will be removed without the approval of the CJAG Environmental Office. As described previously, vegetation clearance will include mowing the grassy areas and the removal of small trees and bushes within the drainage ditch along the southern MRS boundary north of Building 71-849. Vegetation clearing will be minimized to the extent possible to provide access the required portions of the MRS. Requirements specific to excavation backfill and general site restoration are provided in Sections 17.2.4 and 17.2.6, respectively, in the Remedial Action Work Plan (RAWP).

4.2.9 Control of Water Run-on and Runoff (Erosion Control)

4.2.9.1 Although the anticipated maximum excavation area may be up to approximately 2 acres (i.e., all of MRS subareas 001M, 003M, and 004M), the actual excavation dimensions will not be known until the pre-excavation soil characterization sampling is completed and the results evaluated. If the final expected disturbance area dimensions total more than 1 acre in size, then a SWPPP will be required to address stormwater and erosion controls. If needed, the SWPPP will be developed and submitted prior to the field mobilization and will include the final planned excavation dimensions and specific locations of the applicable stormwater and erosion controls.

4.2.9.2 Even if a SWPPP is not required, stormwater and erosion BMPs will be utilized around the general work area and MRS. The BMPs may include silt fence and straw wattles to limit potential sediment migration into portions of the drainage ditch located outside of the final planned excavation areas. The drainage ditch also may be blocked using clean soil to prevent surface water and sediment migration to the southwest beyond the MRS boundary (if actual field conditions warrant).

4.2.10 Decontamination and Disposal of Equipment

Small tools decontamination will apply to stainless steel sampling equipment (direct-push technology Macro Core barrels, hand augers, bowls, spoons, etc.) used during pre-excavation characterization soil sampling and IDW characterization sampling. Small tools decontamination may be conducted in the project staging area or in the MRS, as needed to facilitate site operations. Excavator decontamination will include dry decontamination of the excavator bucket using a broom and shovel if site conditions warrant. Excavator bucket decontamination will be conducted within the MRS excavation areas to avoid potential cross-contamination beyond the MRS. Dry decontamination debris will be added to the excavated soil for transportation and off-site disposal. If site conditions do not allow for dry decontamination, then the excavator bucket will be decontaminated using a steam cleaner within secondary containment. Wastewater from all decontamination activities will be containerized, managed, and disposed as IDW.

4.2.11 Minimizing Areas of Disturbance

All activities associated with this project will be conducted to minimize impacts to land resources within and outside of the MRS boundary. Areas affected by the project will be restored, as practical, to their original condition. As described previously, requirements specific to general site restoration are provided in Section 17.2.6 in the RAWP.

4.3 PROCEDURES FOR POST-ACTIVITY CLEANUP

All wastes will be removed from each site immediately upon completion of each day's field activities. Therefore, no post-activity cleanup will be required.

4.4 AIR MONITORING PLAN

No contact or potential contact with hazardous materials is expected within the investigation areas; therefore, no routine air monitoring will be undertaken. Should potential chemical hazards be discovered, an appropriate monitoring plan shall be developed and implemented prior to continuation of the investigation to verify compliance with applicable laws and regulations.

4.5 GREEN AND SUSTAINABLE EFFORTS

The initiatives below will be implemented by the project team to support the Army’s goal of preserving natural resources, minimize energy use, minimize carbon dioxide emissions, maximize recycling and reuse of materials, and minimize the Army’s environmental footprint.

- Use equipment, supplies, and materials that are locally available and are reusable and/or recyclable;
- Select local providers for field operations wherever practical;
- Complete site restoration for excavated areas as quickly as possible;
- Identify sensitive areas prior to field work (e.g., wetlands, sensitive habitats and species, historic properties, cultural resources, etc.) and plan field work to avoid or mitigate impacts;
- Plan travel to and from the site to minimize the number of trips and vehicles used;
- Inspect and maintain vehicles and equipment to ensure optimal fuel efficiency and to minimize emissions;
- Use phosphate-free detergents instead of organic solvents or acids to decontaminate sampling equipment; and
- Make use of reusable PPE wherever practical.

5.0 REFERENCES

Camp James A. Garfield Joint Military Training Center (CJAG), 2014. *Final Integrated Natural Resources Management Plan.* December.

Environment Research Group (ERG), 2022. Wetland Delineation Report, Former Ravenna Army Ammunition Plant, Portage and Trumbull Counties, Ohio. September.

ATTACHMENT B1

INADVERTENT DISCOVERY STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE NO. 5A
OHARNG Procedures for Inadvertent Discovery of Cultural Materials at Camp Ravenna Joint Military Training Center
(taken from OHARNG ICRMP and modified for CRJMTC)

Contact(s): Kim Ludt, OHARNG CRM, 614-336-6569
(Alternate contact, CRJMTC Environmental Office, 614-336-6568/6136)
CRJMTC Range Control 614-336-6041 or MARCS radio Channel #1

Scope: This Standard Operating Procedure (SOP) outlines the steps to be taken upon inadvertent discovery of human remains or artifacts at Camp Ravenna Joint Military Training Center (CRJMTC) during construction, demolition, training events, or other ground disturbing activities. If archaeological surveys or excavations become necessary as a result of the inadvertent discovery, they must be conducted by a person meeting the Secretary of Interior's professional qualification standards for archaeology. Anyone who does not meet these standards and engages in any excavations, including probing during metal detecting, shall be considered to be looting the cultural resources of CRJMTC and subject to prosecution under Archaeological Resources Protection Act (ARPA). This SOP is intended for all Ohio Army National Guard (OHARNG) personnel, contractors, and users of CRJMTC.

Statutory Reference(s):

- Native American Graves Protection and Repatriation Act and its implementing regulation (43 Code of Federal Regulations [CFR] 10)
- ARPA
- National Historic Preservation Act and its implementing regulation (36 CFR 800)

Procedures: In the event that artifacts or human remains are encountered, the ground disturbing activity should stop immediately, and the following steps should be followed.

- Report any observations or discoveries of artifacts or human remains immediately to CRJMTC Range Control (614-336-6041 or Multi-Agency Radio Communication System radio Channel #1). Range Control will immediately notify the OHARNG Cultural Resources Manager (CRM)/CRJMTC Environmental Office.
- The Range Control or the CRM will secure any artifacts or human remains, as appropriate. If human remains are suspected, they are not to be disturbed and Range Control will promptly notify Ohio State Highway Patrol or Federal Bureau of Investigation, as appropriate.
- The CRM and Range Control will take measures to protect the location from further disturbance until appropriate parties are notified.
- If a concentration of artifacts or a burial site is identified as the source of materials discovered, the CRM will make arrangements for site recordation and stabilization, in

consultation with the Ohio Historic Preservation Office and any interested Native American tribes.

- Once the site has been cleared by the CRM and CRJMTC Range Control, the activity may resume. Depending on the findings, activities may be cleared to resume in 48 hours or up to 6 months.

ATTACHMENT B2

FORM 200-1-24 – FIRST RESPONDER REPORTING

FIRST RESPONDER REPORTING FORM

(Print all information)

Collect as much of the information on the top half of this form as possible before making initial notification. Complete the top and bottom of the form before turning in to Camp Ravenna.

Name of individual reporting spill:______________________________________

When did the spill occur (Date and Time)?__________________________________

Spill Location (Building or area name / number, indoors or out; if vehicle involved, type and bumper number):____

What was spilled? __________________ **How much was spilled?**__________________

Rate at which material is currently spilling:__________________________________

Extent of spill travel?____

Did the spill reach water (ditch, creek, stream, pond, well head)?______________________

Number of injured personnel and type injuries, if applicable:________________________

Do you need the Fire Department to respond to protect life, property, and environment?__________

Unit: ______________________ **State:** _____ **Report Date & Time:** __________

On Scene Coordinator Name and Grade: __________________ **Phone:**__________

How did the spill occur (be specific)?____________________________________

____

What remedial action was taken?____

____

Was soil and absorbent material generated? __________ **How much?**________________

What is the location of the soil and absorbents?________________________________

Was the Environmental Office contacted (yes or No, date and time)?____________________

Who did you talk to in the Environmental Office?_______________________________

Was the site cleared by the Env. Office (Yes or No, date and time)?____________________

Who cleared the site (name and grade, date and time)?___________________________

Initial information is critical. Get as much information as you can, but don't hesitate to make the initial notification if a spill is moving or worsening rapidly!

This form must be completed for all releases and turned-in to Camp Ravenna Range Control within 24 hours.

AGOH Form 200-1-24 (25 Jul 19)

FIRST RESPONDER SPILL/RELEASE RESPONSE ACTIONS

Units or contractors performing training or other operations at Camp Ravenna shall be responsible for adhering to the provisions identified in the Camp Ravenna Integrated Contingency Plans (ICP). A copy of the ICP may be obtained from the Camp Ravenna Environmental Supervisor. Following discovery of a spill (any size), the procedures outlined below shall be executed where applicable:

1. **If necessary, initiate evacuation of the immediate area.**
2. **Notify Camp Ravenna Range Control via two-way radio or by calling (614) 336-6041, and report information contained on the "First Responder Reporting Form" if it is known or can reasonably be determined. This form has been copied on the opposite side of this page. If Range Control cannot be reached, contact a Camp Ravenna OSC (listed below).**
3. **Stop spill flow when possible without undue risk of personal injury.**
4. **If trained, contain the spill using available spill response equipment or techniques.**
5. **Make spill scene OFF LIMITS to unauthorized personnel.**
6. **Restrict all sources of ignition when flammable substances are involved.**
7. **Report to the OSC upon his/her arrival to the scene.**
8. **Turn in a completed copy of the Camp Ravenna First Responder Form to Camp Ravenna Range Control for ALL releases, even ones cleaned up by the reporter.**

TELEPHONE NUMBERS

When **Camp Ravenna Range Control** is ***not available***, **the Camp Ravenna OSC *must be contacted* by the discoverer/first responder following a release if it is in water, at or above a reportable quantity (25 gallons or more of POL), a hazardous or extremely hazardous substance, a hazardous waste, or involves fire, explosion, or is otherwise a major incident.**

NAME			
Camp Ravenna Range Control	Operations and Training	(614)336-6041	(614) 202-5783
Tim Morgan (Primary OSC)	Environmental Supervisor	(614)336-6568	(330)322-7098
Brad Kline (Alternate OSC)	Environmental Specialist	(614)336-4918	Contact Alternate
Katie Tait (Alternate OSC)	Environmental Specialist	(614)336-6136	Contact Alternate
Joint Forces Command (Alternate POC)	OHARNG Emergency Center	(888)637-9053	(888)637-9053

Off-site (from Camp Ravenna area code 614 phones)
Ravenna Dispatch ...…………………………………..…. 9-1-330 296-6486

SEE REVERSE FOR FIRST RESPONDER REPORTING FORM

ATTACHMENT B3

MEMORANDA FOR RECORD

OHIO ARMY NATIONAL GUARD
CAMP JAMES A. GARFIELD JOINT MILITARY TRAINING CENTER
ENVIRONMENTAL OFFICE BUILDING
1438 STATE ROUTE 534 SW
NEWTON FALLS, OH 44444-8503

NGOH-IMR-ENV 17 June 2022

MEMORANDUM FOR RECORD

SUBJECT: ESA Review for the Group 8 Munitions Response Site (MRS) Removal Action as part of the Ravenna Army Ammunitions Plant (RVAAP) Restoration Program at the Camp James A. Garfield Joint Military Training Center (C-JAG), Portage County, Ohio

1. The Army National Guard (ARNG)/Ohio Army National Guard (OHARNG) has contracted HGL to complete a removal action at the Group 8 MRS as part of the RVAAP Restoration Program. The Group 8 MRS is located south of South Service Road within the eastern end of the Group 8 buildings in the southern central portion of C-JAG in Portage County, Ohio.

2. The purpose of the removal action is to remediate contaminated surface soil (0 – 1 foot bgs) with a maximum removal depth of 4 feet bgs (if needed due to identified additional contamination based on confirmation samples) to achieve unrestricted use. The MRS is approximately 2.65 acres of which approximately 2 acres will be disturbed as part of the removal action. The MRS was previously used to burn construction debris and trash as part of the former RVAAP operations and therefore is previously disturbed. A small wetland along a ditch line is located on the southeastern corner of the MRS. Vegetation consists of grass-covered areas with limited shrubs and trees. Tree and brush cutting will occur from 1 October to 31 March to avoid the roosting season for the Northern Long Eared Bat. A wetlands delineation will be performed within the MRS prior to ground disturbance. Based on the amount of ground disturbance, a stormwater pollution prevention plan (SWPPP) will be developed and erosion controls will be installed and managed. Grading and seeding with an OHARNG approved seed mix will occur at the end of the removal action. See Attachment 1 for a map of the MRS.

3. The following listed species provided by the US Fish and Wildlife Service are known to occur in Portage County, Ohio. This list is based on the latest T&E species accessed via US Fish & Wildlife Service IPaC website https://ipac.ecosphere.fws.gov, accessed 27 APR 2022 (see Attachment 2). The presence of general habitat within the project area, federally designated critical habitat within the project area, and federally designated critical habitat at C-JAG has also been included.

Common Name	**Scientific Name**	**Federal Status**	**Habitat within Action Area**	**Critical Habitat at C-JAG**	**Determination**
Indiana Bat	*Myotis sodalis*	Endangered	Yes	No	No Effect (Absent at C-JAG)
Mitchell's Satyr Butterfly	*Neonymapha mitchellii*	Endangered	No	No	No Effect

Northern Long-Eared Bat	*Myotis septentrionalis*	Threatened	Yes	No	*May Affect, not likely to adversely affect
Eastern Massasauga	*Sistrurus catenatus*	Threatened	No	No	No Effect
Northern Monkshood	*Aconitum noveboracense*	Threatened	No	No	No Effect

* = Determination made by USFWS according to 2015 C-JAG Biological Evaluation (BE) adopted as informal consultation on 12 MAY 2015.

4. Intensive biological surveys have been conducted at C-JAG for all of the above listed species. In accordance with the C-JAG Integrated Natural Resources Management Plan (INRMP), bat surveys are conducted on five-year cycles in coordination with the USFWS. To date, six base-wide bat surveys have been conducted at C-JAG (Tawse, 1999; Davey Resource Group, 2002; Duffey & Brack, 2005; Johnson, 2010; EnviroScience, 2016). The latest basewide bat survey commenced in 2021 (delayed one year due to survey prohibition related to COVID19) and will conclude in 2022. Habitat suitable for the Threatened Northern Long-eared Bat (NLEB) does exist at C-JAG as does habitat for the Endangered Indiana bat which, to date, has not been found on site and is considered absent at C-JAG according to the USFWS. In addition, this project will not harm or destroy habitat suitable for the Endangered Mitchell's Satyr Butterfly, the Threatened Eastern Massasauga, or the Threatened Northern Wild Monkshood. To date, these three species have not been found on C-JAG though five and ten year basewide surveys continue to occur.

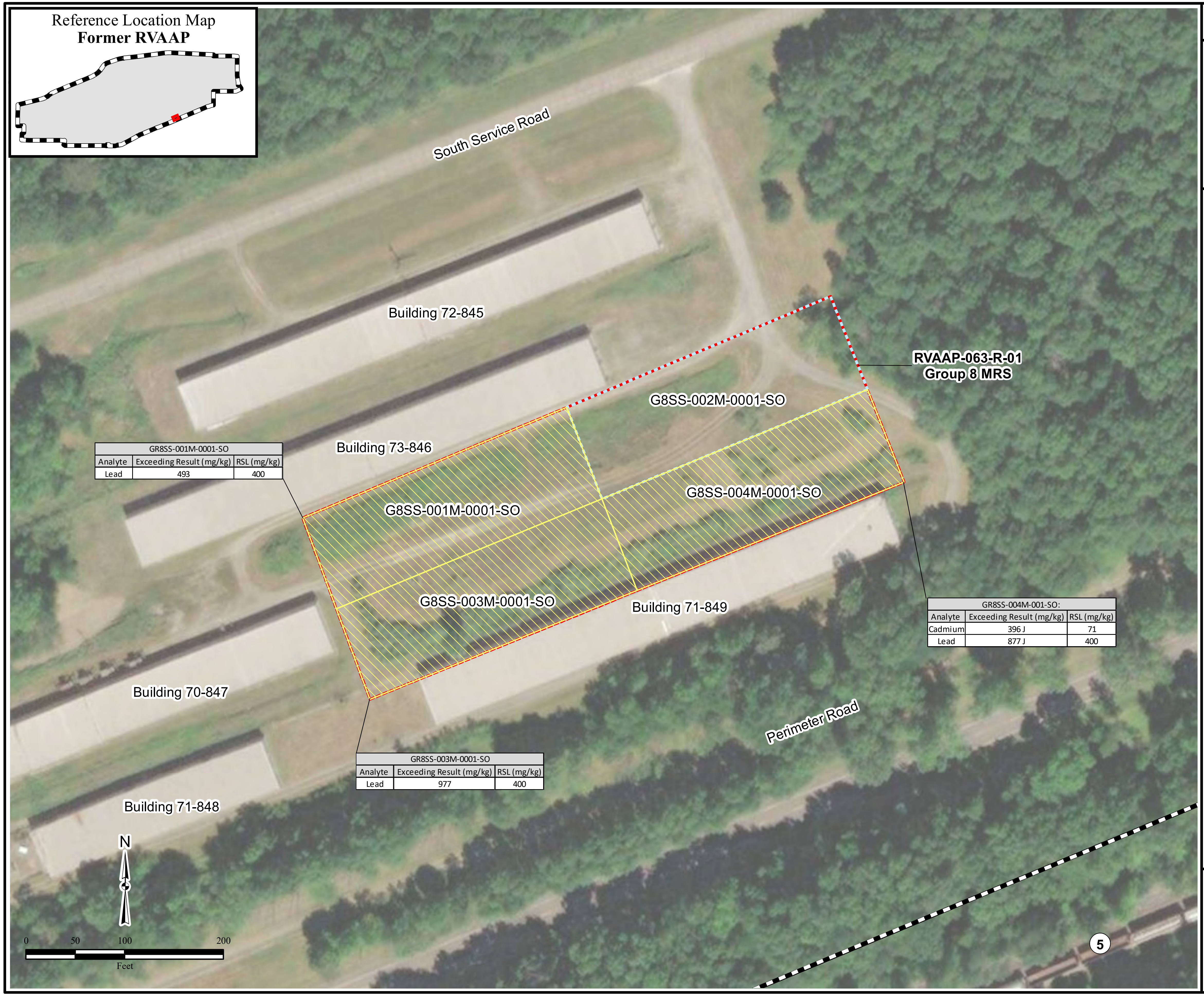
5. Based on the above review, the absence of federally designated critical habitat at C-JAG, the fact that tree felling will occur between OCT-MAR in accordance with NLEB BE, and because habitat for any federally-listed species aside from bats does not exist at or within the proposed action area, the OHARNG has concluded **No Effect** will occur to any known listed species as a result of this action with the exception of the federally threatened NLEB in which this action **May affect, not likely to adversely affect** this species present at C-JAG. This "May affect" determination has already been acknowledged and accepted by the USFWS in accordance with the 2015 C-JAG (then CRJMTC) BE, adopted as informal consultation on 12 MAY 2015.

6. The POC for this action is Mr. Brian Riley, Natural Resources Manager, at 614-336-4564 or by email, brian.p.riley17.nfg@army.mil.

Attachments
1. Group 8 MRS Map
2. USFWS Official Species List, 27 APR 2022

////////////BPR///////////
BRIAN P. RILEY
CIV, ESII, OHARNG
Natural Resources Manager

Attachment 1: Group 8 MRS Map



GR8SS-001M-0001-SO		
Analyte	Exceeding Result (mg/kg)	RSL (mg/kg)
Lead	493	400

GR8SS-004M-001-SO:		
Analyte	Exceeding Result (mg/kg)	RSL (mg/kg)
Cadmium	396 J	71
Lead	877 J	400

GR8SS-003M-0001-SO		
Analyte	Exceeding Result (mg/kg)	RSL (mg/kg)
Lead	977	400

HGL—Record of Decision
Former RVAAP, Ohio

Figure 6
2019 Feasibility Study Risk Management Evaluation Delineated MC Contamination Former RVAAP Portage and Trumbull Counties, Ohio

Legend

▨	Excavation Area
⬚	Surface ISM Soil Sample Area
⬚	MRS
⬚	Installation Boundary

HHRA COCs - Surface Soil Only
Resident Receptor
Cadmium
Lead

Notes:
Surface soil defined as 0 ft bgs to 0.5 ft bgs.

COC=Chemical of Concern
ft bgs=feet below ground surface
HHRA=Human Health Risk Assessment
ISM=incremental sampling method
J=estimated value
MC=munitions constituent
mg/kg=milligrams per kilogram
MRS=munitions response site
RVAAP=Ravenna Army Ammunition Plant

\\srv-gst-01\HGLGIS\Ravenna_AAP\Group8\ROD\(06)Group8_Delineated_MC.mxd
6/23/2020 TH
Source: HGL,CB&I, USACE, e²M
ArcGIS Online Imagery

HGL HydroGeoLogic, Inc

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Attachment 2: USFWS Official Species List, 27 APR 2022

United States Department of the Interior

FISH AND WILDLIFE SERVICE
Ohio Ecological Services Field Office
4625 Morse Road, Suite 104
Columbus, OH 43230-8355
Phone: (614) 416-8993 Fax: (614) 416-8994

In Reply Refer To:
Project Code: 2022-0036474
Project Name: General Project - Military Mission Support

April 27, 2022

Subject: List of threatened and endangered species that may occur in your proposed project location or may be affected by your proposed project

To Whom It May Concern:

The enclosed species list identifies threatened, endangered, proposed and candidate species, as well as proposed and final designated critical habitat, that may occur within the boundary of your proposed project and/or may be affected by your proposed project. The species list fulfills the requirements of the U.S. Fish and Wildlife Service (Service) under section 7(c) of the Endangered Species Act (Act) of 1973, as amended (16 U.S.C. 1531 *et seq.*).

New information based on updated surveys, changes in the abundance and distribution of species, changed habitat conditions, or other factors could change this list. Please feel free to contact us if you need more current information or assistance regarding the potential impacts to federally proposed, listed, and candidate species and federally designated and proposed critical habitat. Please note that under 50 CFR 402.12(e) of the regulations implementing section 7 of the Act, the accuracy of this species list should be verified after 90 days. This verification can be completed formally or informally as desired. The Service recommends that verification be completed by visiting the ECOS-IPaC website at regular intervals during project planning and implementation for updates to species lists and information. An updated list may be requested through the ECOS-IPaC system by completing the same process used to receive the enclosed list.

The purpose of the Act is to provide a means whereby threatened and endangered species and the ecosystems upon which they depend may be conserved. Under sections 7(a)(1) and 7(a)(2) of the Act and its implementing regulations (50 CFR 402 *et seq.*), Federal agencies are required to utilize their authorities to carry out programs for the conservation of threatened and endangered species and to determine whether projects may affect threatened and endangered species and/or designated critical habitat.

A Biological Assessment is required for construction projects (or other undertakings having similar physical impacts) that are major Federal actions significantly affecting the quality of the human environment as defined in the National Environmental Policy Act (42 U.S.C. 4332(2)(c)). For projects other than major construction activities, the Service suggests that a biological evaluation similar to a Biological Assessment be prepared to determine whether the project may affect listed or proposed species and/or designated or proposed critical habitat. Recommended contents of a Biological Assessment are described at 50 CFR 402.12.

If a Federal agency determines, based on the Biological Assessment or biological evaluation, that listed species and/or designated critical habitat may be affected by the proposed project, the agency is required to consult with the Service pursuant to 50 CFR 402. In addition, the Service recommends that candidate species, proposed species and proposed critical habitat be addressed within the consultation. More information on the regulations and procedures for section 7 consultation, including the role of permit or license applicants, can be found in the "Endangered Species Consultation Handbook" at:

http://www.fws.gov/endangered/esa-library/pdf/TOC-GLOS.PDF

Migratory Birds: In addition to responsibilities to protect threatened and endangered species under the Endangered Species Act (ESA), there are additional responsibilities under the Migratory Bird Treaty Act (MBTA) and the Bald and Golden Eagle Protection Act (BGEPA) to protect native birds from project-related impacts. Any activity, intentional or unintentional, resulting in take of migratory birds, including eagles, is prohibited unless otherwise permitted by the U.S. Fish and Wildlife Service (50 C.F.R. Sec. 10.12 and 16 U.S.C. Sec. 668(a)). For more information regarding these Acts see https://www.fws.gov/birds/policies-and-regulations.php.

The MBTA has no provision for allowing take of migratory birds that may be unintentionally killed or injured by otherwise lawful activities. It is the responsibility of the project proponent to comply with these Acts by identifying potential impacts to migratory birds and eagles within applicable NEPA documents (when there is a federal nexus) or a Bird/Eagle Conservation Plan (when there is no federal nexus). Proponents should implement conservation measures to avoid or minimize the production of project-related stressors or minimize the exposure of birds and their resources to the project-related stressors. For more information on avian stressors and recommended conservation measures see https://www.fws.gov/birds/bird-enthusiasts/threats-to-birds.php.

In addition to MBTA and BGEPA, Executive Order 13186: *Responsibilities of Federal Agencies to Protect Migratory Birds*, obligates all Federal agencies that engage in or authorize activities that might affect migratory birds, to minimize those effects and encourage conservation measures that will improve bird populations. Executive Order 13186 provides for the protection of both migratory birds and migratory bird habitat. For information regarding the implementation of Executive Order 13186, please visit https://www.fws.gov/birds/policies-and-regulations/executive-orders/e0-13186.php.

We appreciate your concern for threatened and endangered species. The Service encourages Federal agencies to include conservation of threatened and endangered species into their project planning to further the purposes of the Act. Please include the Consultation Code in the header of this letter with any request for consultation or correspondence about your project that you submit to our office.

Attachment(s):

- Official Species List

Official Species List

This list is provided pursuant to Section 7 of the Endangered Species Act, and fulfills the requirement for Federal agencies to "request of the Secretary of the Interior information whether any species which is listed or proposed to be listed may be present in the area of a proposed action".

This species list is provided by:

Ohio Ecological Services Field Office
4625 Morse Road, Suite 104
Columbus, OH 43230-8355
(614) 416-8993

Project Summary

Project Code: 2022-0036474

Event Code: None

Project Name: General Project - Military Mission Support

Project Type: Military Operations

Project Description: General operation, maintenance, training project for Portage County, OH, portion of Camp James A. Garfield Joint Military Training Center (C-JAG).

Project Location:

Approximate location of the project can be viewed in Google Maps: https://www.google.com/maps/@41.19679795,-81.08693398110162,14z

Counties: Portage County, Ohio

Endangered Species Act Species

There is a total of 6 threatened, endangered, or candidate species on this species list.

Species on this list should be considered in an effects analysis for your project and could include species that exist in another geographic area. For example, certain fish may appear on the species list because a project could affect downstream species.

IPaC does not display listed species or critical habitats under the sole jurisdiction of NOAA Fisheries[1], as USFWS does not have the authority to speak on behalf of NOAA and the Department of Commerce.

See the "Critical habitats" section below for those critical habitats that lie wholly or partially within your project area under this office's jurisdiction. Please contact the designated FWS office if you have questions.

---

[1] NOAA Fisheries, also known as the National Marine Fisheries Service (NMFS), is an office of the National Oceanic and Atmospheric Administration within the Department of Commerce.

Mammals

NAME	STATUS
Indiana Bat *Myotis sodalis* There is **final** critical habitat for this species. The location of the critical habitat is not available. Species profile: https://ecos.fws.gov/ecp/species/5949	Endangered
Northern Long-eared Bat *Myotis septentrionalis* No critical habitat has been designated for this species. Species profile: https://ecos.fws.gov/ecp/species/9045	Threatened

Reptiles

NAME	STATUS
Eastern Massasauga (=rattlesnake) *Sistrurus catenatus* No critical habitat has been designated for this species. Species profile: https://ecos.fws.gov/ecp/species/2202	Threatened

Insects

NAME	STATUS
Mitchell's Satyr Butterfly *Neonympha mitchellii mitchellii* No critical habitat has been designated for this species. Species profile: https://ecos.fws.gov/ecp/species/8062	Endangered
Monarch Butterfly *Danaus plexippus* No critical habitat has been designated for this species. Species profile: https://ecos.fws.gov/ecp/species/9743	Candidate

Flowering Plants

NAME	STATUS
Northern Wild Monkshood *Aconitum noveboracense* No critical habitat has been designated for this species. Species profile: https://ecos.fws.gov/ecp/species/1450	Threatened

Critical habitats

THERE ARE NO CRITICAL HABITATS WITHIN YOUR PROJECT AREA UNDER THIS OFFICE'S JURISDICTION.

IPaC User Contact Information

Agency:	State of Ohio
Name:	Brian Riley
Address:	1438 State Route 534 SW
Address Line 2:	-
City:	Newton Falls
State:	OH
Zip:	44444
Email	brian.p.riley17.nfg@army.mil
Phone:	6143364564

STATE OF OHIO
ADJUTANT GENERAL'S DEPARTMENT
2825 West Dublin Granville Road
Columbus, Ohio 43235-2789

NGOH-IMR-ENV 26 May 2022

MEMORANDUM FOR RECORD

SUBJECT: National Historic Preservation Act Compliance for the Removal Action at the Group 8 Munitions Response Site (MRS) as part of the Ravenna Army Ammunition Plant (RVAAP) Restoration Program at the Camp James A. Garfield Joint Military Training Center (CJAG)

1. The Army National Guard (ARNG)/Ohio Army National Guard (OHARNG) has contracted HGL to complete a soil removal action at the Group 8 MRS as part of the RVAAP Restoration Program. The Group 8 MRS is located south of South Service Road within the eastern end of the Group 8 buildings in the southern central portion of CJAG in Portage County, Ohio.

2. The purpose of the removal action is to remediate contaminated surface soil (0 – 1 foot bgs) with a maximum removal depth of 4 feet bgs (if needed due to identified additional contamination based on confirmation samples) to achieve unrestricted use. The MRS is approximately 2.65 acres of which approximately 2 acres will be disturbed as part of the removal action. The MRS was previously used to burn construction debris and trash as part of the former RVAAP operations and therefore is previously disturbed. A small wetland along a ditch line is located on the southeastern corner of the MRS. Vegetation consists of grass-covered areas with limited shrubs and trees. Tree and brush cutting will occur from 1 October to 31 March to avoid the roosting season for the Northern Long Eared Bat. A wetlands delineation will be performed within the MRS prior to ground disturbance. Based on the amount of ground disturbance, a stormwater pollution prevention plan (SWPPP) will be developed and erosion controls will be installed and managed. Grading and seeding with an OHARNG approved seed mix will occur at the end of the removal action. See Attachment 1 for a map of the MRS.

3. There will be no historic archaeological sites affected by the proposed undertaking. In 2015, a Phase I archaeological survey was completed within and around the site identified in this MFR. The survey did not identify any archaeological sites within the proposed work area. There are no sites that meet the eligibility criteria for listing in the National Register of Historic Places (NRHP) within or near the proposed work area.

4. There will be no historic architectural properties affected by the proposed undertaking. The Ravenna Army Ammunition Plant (RVAAP) was placed into modified caretaker status in October 1993 and maintenance stopped on all but a few buildings. In 1993, the United States Army Materiel Command Programmatic Agreement to Cease Maintenance, Excess and Dispose of Select Buildings (PA CMED) was developed. This PA established procedures for maintenance and disposal of buildings located within the confines of the former RVAAP in Ohio and installations in six other states. Any nearby buildings are included in the PA. As such, the Section 106 compliance responsibilities to manage these architectural resources have been met, and all impacts to those architectural resources have been mitigated through implementation of the PA CMED. In 2002, the OHARNG consulted with the OHPO again regarding Section 106 responsibilities for buildings covered by the PA CMED. In a letter dated February 11, 2003, the OHPO concurred that Section 106 requirements were met. In addition, these buildings are covered under the Program Comments for WWII and Cold War Era (1939-1974) Army Ammunition Storage Facilities and Army Ammunition Production Facilities and Plants

5. The OHARNG regularly consults with 14 federally recognized Tribes regarding archaeological surveys and projects at CJAG. Draft archaeological surveys are sent to these tribes for comment and review. No concerns were expressed regarding the above-mentioned surveys. The OHARNG has established a good working relationship with tribes that have ancestral ties to OHARNG owned properties. Consultation with tribes has shown that the greatest interest is the inadvertent discovery of human remains or NAGPRA related items and results of archaeological surveys. Since there are no known historic properties that tribes attach religious or cultural significance to within the project areas, additional consultation is not necessary for the proposed undertakings. Although it is unlikely archaeological resources will be encountered, in the event of an inadvertent discovery of human remains or funerary items, the OHARNG will follow *Procedures for Inadvertent Discovery of Cultural Materials at Camp Ravenna Joint Military Training Center*. These procedures were taken from Standard Operating Procedure #5 of the OHARNG Integrated Cultural Resources Management Plan and modified specifically for the facility.

6. There are no known historic properties within the proposed work area; therefore the OHARNG makes the determination that no historic properties will be affected by the proposed undertaking. Copies of pertinent correspondence and survey reports are available upon request from the OHARNG Cultural Resources Manager. Any questions or concerns regarding Cultural Resources for the OHARNG should be directed to the undersigned at (614) 336-6569 or via e- mail at kimberly.s.ludt.nfg@army.mil.

//////S//////

KIMBERLY S. LUDT
OHARNG Cultural Resources Manager

Attachment 1 – Map of the Group 8 MRS

STANDARD OPERATING PROCEDURES NO. 5A
OHARNG Procedures for Inadvertent Discovery of Cultural Materials at Camp Ravenna Joint Military Training Center
(taken from OHARNG ICRMP and modified for CRJMTC)

Contact(s): Kim Ludt, OHARNG Cultural Resources Manager, 614-336-6569
(Alternate contact, CRJMTC Environmental Office, 614-336-6568/6136)
CRJMTC Range Control 614-336-6041 or MARCS radio Channel #1

Scope: This Standard Operating Procedure (SOP) outlines the steps to be taken upon inadvertent discovery of human remains or artifacts at Camp Ravenna Joint Military Training Center (CRJMTC) during construction, demolition, training events, or other ground disturbing activities. If archaeological surveys or excavations become necessary as a result of the inadvertent discovery, they must be conducted by a person meeting the Secretary of Interior's professional qualification standards for archaeology. Anyone who does not meet these standards and engages in any excavations, including probing during metal detecting, shall be considered to be looting the cultural resources of CRJMTC and subject to prosecution under ARPA. This SOP is intended for all OHARNG personnel, contractors and users of CRJMTC.

Statutory Reference(s):

- Native American Graves Protection and Repatriation Act (NAGPRA) and its implementing regulation (43 CFR 10)
- Archaeological Resources Protection Act (ARPA)
- National Historic Preservation Act (NHPA) and its implementing regulation (36 CFR 800).

Procedures: In the event that artifacts or human remains are encountered, the ground disturbing activity should stop immediately and the following steps should be followed.

- Report any observations or discoveries of artifacts or human remains immediately to CRJMTC Range Control (614-336-6041 or MARCS radio Channel #1). Range Control will immediately notify the OHARNG Cultural Resources Manager (CRM)/CRJMTC Environmental Office.
- The Range Control or the CRM will secure any artifacts or human remains, as appropriate. If human remains are suspected, they are not to be disturbed and Range Control will promptly notify Ohio State Highway Patrol or Federal Bureau of Investigation, as appropriate.
- The CRM and Range Control will take measures to protect the location from further disturbance until appropriate parties are notified.
- If a concentration of artifacts or a burial site is identified as the source of materials discovered, the CRM will make arrangements for site recordation and stabilization, in consultation with the OHPO and any interested Native American tribes.
- Once the site has been cleared by the CRM and CRJMTC Range Control, the activity may resume. Depending on the findings, activities may be cleared to resume in 48 hours or up to 6 months.

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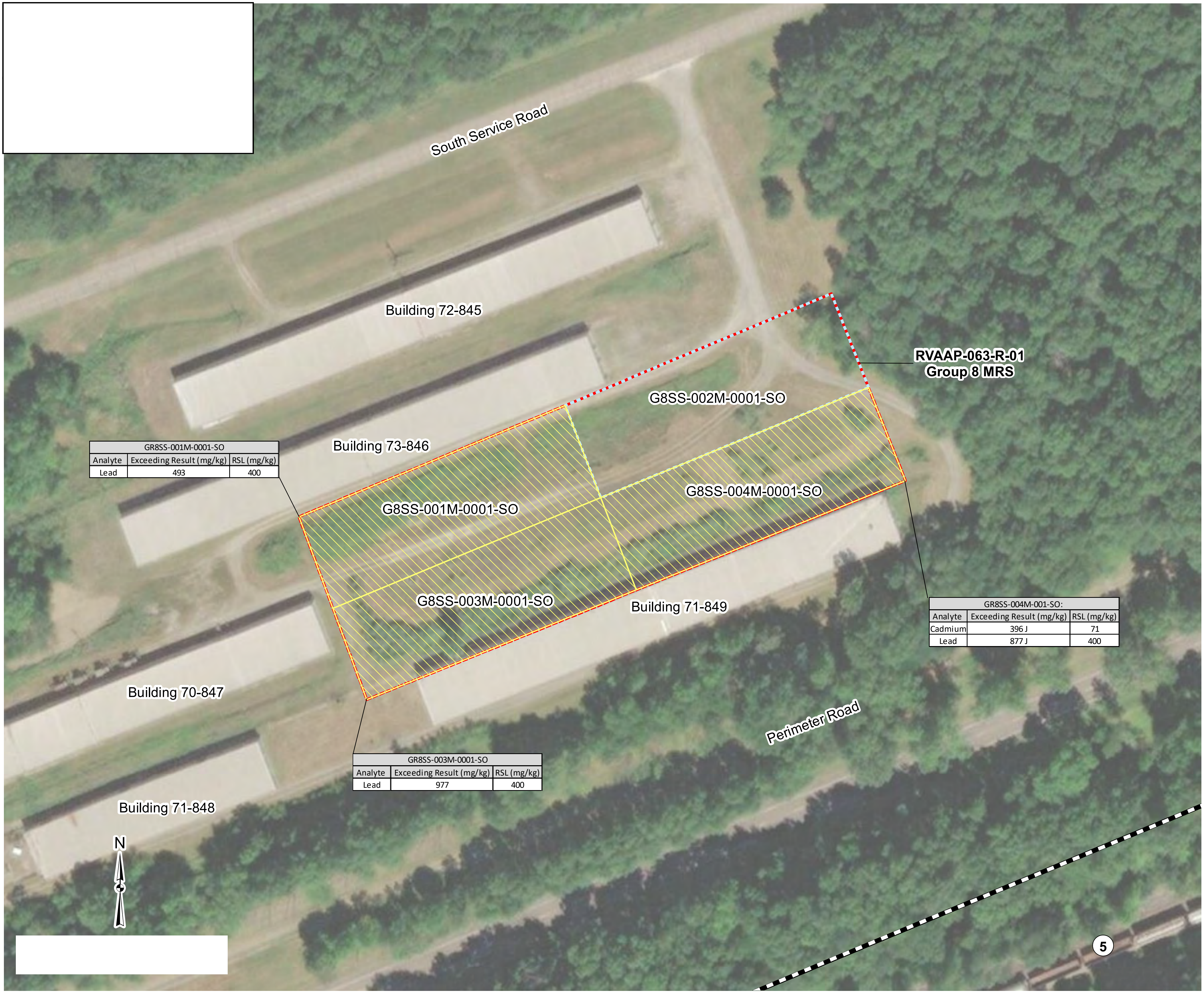
Figure 6
2019 Feasibility Study Risk Management Evaluation Delineated MC Contamination Former RVAAP Portage and Trumbull Counties, Ohio



GR8SS-001M-0001-SO		
Analyte	Exceeding Result (mg/kg)	RSL (mg/kg)
Lead	493	400

GR8SS-004M-001-SO:		
Analyte	Exceeding Result (mg/kg)	RSL (mg/kg)
Cadmium	396 J	71
Lead	877 J	400

GR8SS-003M-0001-SO		
Analyte	Exceeding Result (mg/kg)	RSL (mg/kg)
Lead	977	400

Legend

▨	Excavation Area
⬚	Surface ISM Soil Sample Area
⬚	MRS
⬚	Installation Boundary

HHRA COCs - Surface Soil Only
Resident Receptor
Cadmium
Lead

Notes:
Surface soil defined as 0 ft bgs to 0.5 ft bgs.

COC=Chemical of Concern
ft bgs=feet below ground surface
HHRA=Human Health Risk Assessment
ISM=incremental sampling method
J=estimated value
MC=munitions constituent
mg/kg=milligrams per kilogram
MRS=munitions response site
RVAAP=Ravenna Army Ammunition Plant

\\srv-gst-01\HGLGIS\Ravenna_AAP\Group8\ROD\(06)Group8_Delineated_MC.mxd
6/23/2020 TH
Source: HGL,CB&I, USACE, e²M
ArcGIS Online Imagery

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ATTACHMENT B4

STORMWATER POLLUTION PREVENTION PLAN

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STORMWATER POLLUTION PREVENTION PLAN

REMEDIAL ACTION
FORMER RAVENNA ARMY AMMUNITION PLANT
RVAAP-063-R-01 GROUP 8
MUNITIONS RESPONSE SITE
PORTAGE AND TRUMBULL COUNTIES, OHIO

Contract No. W912DR-21-D-0005
Delivery Order No. W912DR21F0327

Prepared for:

U.S. Army Corps of Engineers, Baltimore District
2 Hopkins Plaza
Baltimore, Maryland 21201

Prepared by:

HydroGeoLogic, Inc.
11107 Sunset Hills Road, Suite 400
Reston, Virginia 20190

July 2023

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LIST OF FIGURES

ACRONYMS AND ABBREVIATIONS

ARNG	Army National Guard
bgs	below ground surface
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CJAG	Camp James A. Garfield
FS	Feasibility Study
HGL	HydroGeoLogic, Inc.
MC	munitions constituents
MD	munitions debris
MEC	munitions and explosives of concern
MRS	Munitions Response Site
NPDES	National Pollutant Discharge Elimination System
Ohio EPA	Ohio Environmental Protection Agency
QAPP	Quality Assurance Protection Plan
QCS	Quality Control Specialist
RA	Remedial Action
RI	Remedial Investigation
SWPPP	Stormwater Pollution Prevention Plan
TBD	to be determined

STORMWATER POLLUTION PREVENTION PLAN
REMEDIAL ACTION
BLOCK D IGLOO MUNITIONS RESPONSE SITE
FORMER RAVENNA ARMY AMMUNITION PLANT, OHIO

1.0 GENERAL INFORMATION

Hydrogeologic (HGL) has prepared this Stormwater Pollution Prevention Plan (SWPPP) as part of a Remedial Action Uniform Federal Policy Quality Assurance Project Plan (QAPP) to address removal of soil contaminated with munitions constituents (MC) at the Group 8 Munitions Response Site (MRS). This SWPPP builds on the project Environmental Protection Plan (EPP, Appendix B of the QAPP) and includes:

- Site Features Map (included as Figure B.4.1)
- Erosion and Sedimentation Control Plan (included as Figure B.4.2)

HGL will be implementing these design plans as part of field activities conducted at Camp James A. Garfield (CJAG) (formerly known as the Ravenna Army Ammunition Plant) for the U.S. Army Corps of Engineers, Baltimore District, under Contract No. W912DR-21-D-0005, Delivery Order No. W912DR21F0327.

Section 402 of the Clean Water Act established the National Pollutant Discharge Elimination System (NPDES) program to limit discharges of pollutants. In Ohio, the Ohio Environmental Protection Agency (Ohio EPA) reviews and approves coverage under a NPDES general permit to dischargers of stormwater from construction activities.

No formal permit is required for this project because it is being conducted in accordance with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) process. CERCLA response actions are exempted by law from the requirement to obtain Federal, State, or local permits related to any activities conducted completely on site. However, this SWPPP is intended to ensure that the substantive portions of the NPDES requirements are met.

2.0 BACKGROUND

Evidence from the 2015 Remedial Investigation (RI) suggests that MC, in the form of lead and cadmium, are present in surface soils at the Group 8 MRS. The Group 8 MRS (formerly known as Area Between Building 846 and 849) was used to burn construction debris and rubbish for an unknown period. Before designation as an MRS, the area between Building 846 and 849 was used as a staging area for military vehicles. There are no records available documenting the disposal of munitions at the MRS; however, previous discoveries of Munitions of Explosive Concern (MEC) and of munitions debris (MD) indicated that munitions may have been disposed of at the Group 8 MRS. Sampling for MC-related contamination was conducted during the RI field work. The RI Report concluded that no contaminants in subsurface soil were present at concentrations that pose a risk to either human or ecological receptors. The 2019 Feasibility Study (FS) concluded that cadmium in surface soil at the location of quadrant GR8SS-004M poses a risk to the theoretical future Resident Receptor (Adult and Child). Following the FS, the Army National Guard (ARNG) and Ohio EPA agreed to include additional excavation to remove lead in surface soil at quadrants GR8SS-001M and GR8SS-003M to ensure maximum protectiveness is achieved to meet Unrestricted (Residential) Land Use. The 2020 Proposed Plan recommended remediation of the lead and cadmium contamination in GR8SS-004M and the lead contamination in GR8SS-001M and GR8SS-003M to eliminate potential risks to a theoretical future Resident Receptor.

Remedial action is required to mitigate risks to current and reasonably anticipated future receptors. The Decision Document signed August 2020 identified the following remedial action for the MRS:

- Removal of lead- and cadmium-contaminated soil exceeding the Preliminary Remediation Goals to a depth of 0.5 feet below ground surface (bgs);
- Incidental to the surface soil removal, if any MD is encountered, it will be segregated, inspected, and certified as material documented as safe before disposal;
- Confirmation soil samples will be collected to confirm that the extent of MC-contaminated soil has been removed;
- If required, up to 2.0 feet bgs may be excavated, based on the results of confirmation sampling; and
- Implementing this remedy will allow for unlimited use/unrestricted access at the MRS (although future residential use is not anticipated and is theoretical only).

3.0 NATURE OF CONSTRUCTION ACTIVITY

The function of the construction activity is the removal of contaminated soils to reduce the exposure risk to potential future receptors. Minimal woody vegetation is present in the Group 8 MRS. Vegetation clearance will include mowing (if needed). The Wetland Delineation Report (Environmental Research Group [ERG], 2022) indicates that the drainage ditches located along the southern MRS boundary do not discharge to any surface water feature, and any standing water infiltrates into the underlying soil. Based on publicly available aerial photography from November 2021, the area subject to clearing of small trees and brush along the drainage ditch will cover approximately 0.5 acres north of Building 71-849. No trees greater than 4 inches in diameter will be removed unless specifically approved by CJAG Environmental Office. All vegetation removed from the MRS will be recycled for firewood or mulch. Tree and vegetation removal will follow Best Management Practices including erosion control devices to protect wetlands and streams at the site. Trees will be cut to within 4 to 6 inches of the ground surface to allow for proper subsurface surveys. All stumps will be left in place unless it is determined that they need to be removed to investigate an anomaly.

Vertical limits of excavation will be determined through pre-characterization sampling using a Direct Push Technology drill rig. Once samples have been analyzed and an excavation plan is developed, soils will be excavated using heavy equipment and put directly into lined roll-offs staged in quadrant GR8SS-002. Waste characterization will be performed prior to excavation minimizing the anticipated duration of staging full roll-offs.

Backfilling will occur concurrent with excavation activities to the degree feasible. Backfill source material will be analyzed for potential contaminants prior to acquisition and be preapproved by CJAG. Filled areas will be compacted and graded to the original pre-excavation elevations. Site restoration includes seeding with a site-approved grass seed mix, application of mulch, and 12 months of monitoring and supplemental seeding.

The planned start date of site preparation is March 2023. The estimated finish date of removal and restoration activities is October 2023.

4.0 SITE PLAN

Figure B.1.1 is a Site Features Map that includes the MRS boundary (which serves as the limits of construction activities), topography, and surface water features. No jurisdictional wetlands were identified in the Wetland Delineation Report (Appendix I of the Block D Igloo QAPP). However, isolated drainage ditches are present within the MRS. The ditches are within the excavation footprint and do not seem to discharge outside of the MRS. The proposed staging area is within quadrant GR8SS-02. Equipment, vehicle staging, and rolloff staging is expected to be wholly contained within the MRS.

HGL will implement the following erosion and sedimentation control measures prior to excavation, as shown in Figure B.1.2.

1. A silt fence or straw wattle will be installed at the ends of the existing drainage ditches outside of the planned excavation area. In addition, silt fence or straw wattle will be installed on the north, west, and south boundaries of the MRS, where runoff would be expected. Controls will be installed perpendicular to runoff across the slope at relative low points into the drainage basin. The following are guidelines for installing these erosion controls.
    a. Straw wattles will be placed in a row along the contour with adjacent bales securely tied with either wire or nylon string. Each bale will be anchored with two metal or wooden stakes driven into the ground at least 1.5 feet.
    b. Wattles will be placed in the ground at least four inches.
    c. Silt fences will be placed on the contour with posts spaced no more than 10 feet apart. A trench 8 to 12 inches deep will be excavated along the silt fence line. Filter fabric will extend into the trench. Soil excavated from the trench will be used to backfill the trench and cover the bottom of the filter fabric.
2. A temporary vehicle tracking pad will be installed in the driveway exiting the MRS to the north prior to the South Service Road. Vehicles exiting the Group 8 MRS will drive over the tracking pad before turning on to South Service Road. The tracking pad will be a modular fabricated pad designed to remove excess soil from vehicle tires prior to exiting the area. A sediment trap will be placed on the downgradient edge of the tracking pad to capture sediment during rain events.
3. Following removal actions, the area previously cleared of vegetation will be seeded with native seed mixes approved by the Ohio Army National Guard. Per the directions received

from CJAG, the final restoration will utilize a seed mixture that contains 40% Kentucky bluegrass (*Poa pratensis*), 30% Perennial ryegrass (*Lolium perenne*), 20% Hard fescue (*Festuca brevipila*), and 10% Creeping red fescue (*Festuca rubra*). The mixture will be broadcast at a ratio of 18 pounds/acre.

4. Prior to seeding, the topsoil will be tilled to provide a loose and suitable seed bed for germination. Two inches of straw mulch will be added as cover following the seeding. Revegetation will be monitored and supplemented as needed for 12 months. Following the 12-month period, erosion controls will be removed from the site.

5.0 SPILL PREVENTION AND RESPONSE

Due to the nature of the operations, the potential for a spill of pollutants during operations is low. The highest probability for a spill will occur during refueling operations of vehicles or equipment. Materials that pose a potential spill concern are diesel, gasoline, hydraulic fluid, and motor oil. Spill prevention and response is discussed in detail in the Environmental Protection Plan.

6.0 CONTACT INFORMATION / RESPONSIBLE PARTIES

24-hr Emergency Contact:
Name: Kimberly Vaughn (HGL)
Telephone Number: 512-658-6828
E-mail: kvaughn@hgl.com

Site Supervisor:
Name: Jesse Norman (HGL)
Telephone Number: 317-468-5214
E-mail: jnorman@hgl.com

Quality Control Specialist:
Name: Rebecca Powers
Telephone Number: 501-422-9077
E-mail: rpowers@hgl.com

HGL and its excavation subcontractor (to be determined [TBD]) will establish a stormwater management team that is responsible for the implementation of this SWPPP. The team will consist of the following personnel:

- Project Manager – Kimberly Vaughn (HGL)
- Site Supervisor – Jesse Norman (HGL)
- Quality Control Specialist (QCS) – Rebecca Powers (HGL)
- Subcontractor Foreman – (TBD)

The members of the team will have full knowledge of the SWPPP contents. The Site Supervisor and QCS will train operations personnel regarding the contents of the SWPPP, and the necessity of prompt communication of any observed deficiencies to the Site Supervisor or QCS. The excavation subcontractor will be responsible for the installation and maintenance of sedimentation and erosion control features.

The Site Coordinator will be responsible for obtaining approval for any proposed modifications to the SWPPP, and the Site Supervisor will be responsible for incorporating approved modifications. The QCS will have primary responsibility for inspecting site conditions and all sediment and erosion control features. All controls will be inspected a minimum of every 7-days and within 24-hours of any storm event of ½ inch (in) or more within a 24-hour period. Inspections will be documented in writing and kept on file and included in the Final Remedial Action Completion Report. HGL is responsible for conducting these inspections. The QCS is a qualified individual and will have the authority to initiate action should maintenance or repair of controls be needed. A copy of the inspection form is included in Appendix E of the QAPP. All inspections by the stormwater management team will be fully documented, and copies of the documentation will be maintained on site by the QCS. The Site Supervisor will have ultimate responsibility for ordering and supervising any necessary maintenance, repairs, or improvements to the sediment and erosion controls for the project.

Repairs and maintenance need to be documented in inspection reports and on the Corrective Action Log and must be conducted within three days of documentation. If an inspection reveals that existing practices are not adequate and a new practice is needed, the SWPPP will be revised, and the new practice implemented within ten days of documentation. If an inspection reveals that a control practice has not been installed, the practice must be installed within ten days. If the inspection reveals that a planned control practice is not needed, the inspection record must contain a statement of explanation as to why the control practice is not needed. HGL is responsible for all repairs, maintenance, and installation of new practices. HGL will notify the CJAG Environmental Office any time repair and/or maintenance is necessary. A copy of the Corrective Action Log is included in Appendix E of the QAPP.

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FIGURES

Figure B.4.1
Group 8 MRS
Remedial Action Plan and Site Features

Legend

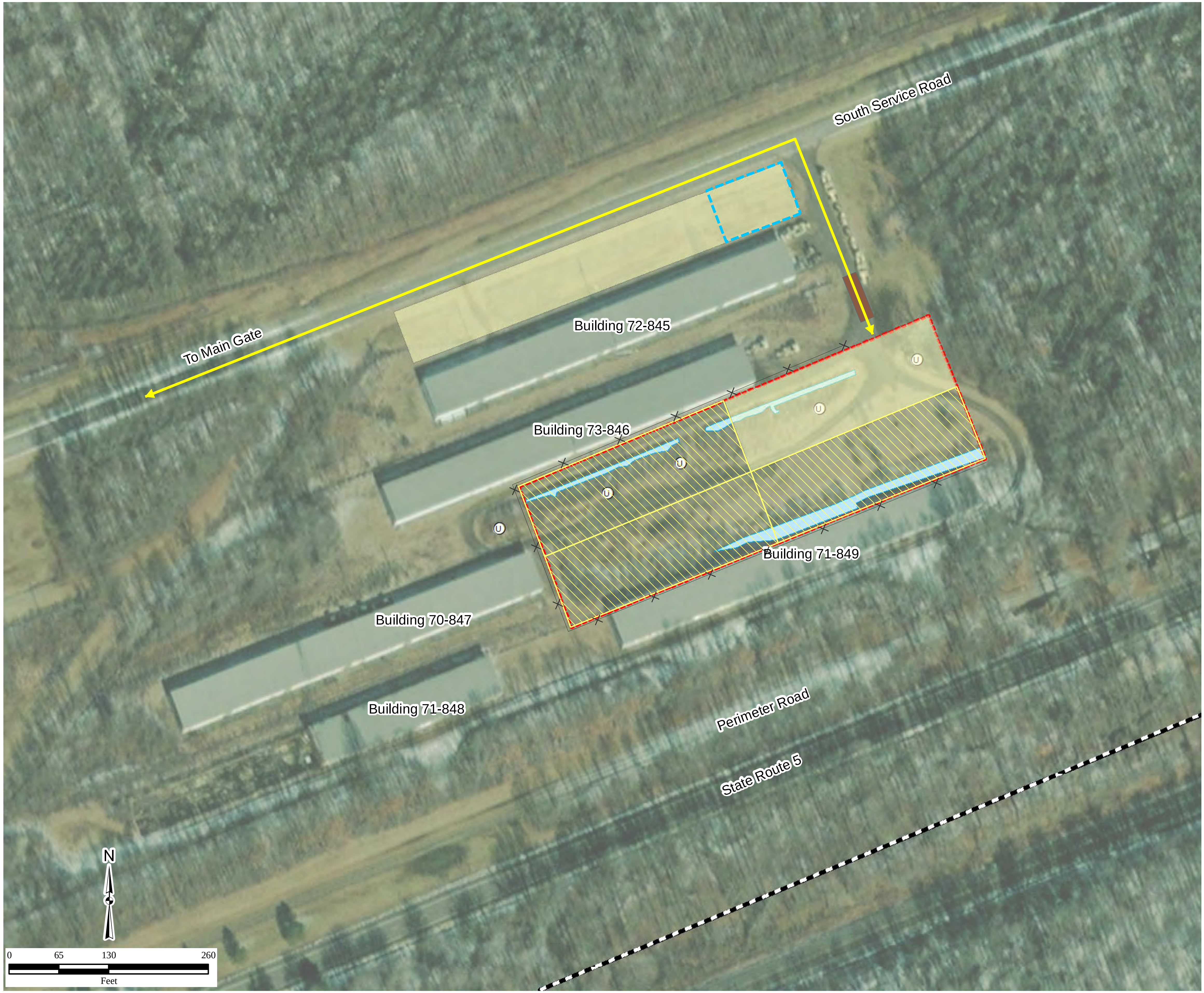
Ⓤ	Power Pole
—	Topographic Contour (2ft Interval)*
↔	Construction/Haul Route
⬚	Temporary Storage and Portable Bathroom
▨	Excavation Area
🟨	Staging/Load-out Area
🟦	Ditch
⬚	MRS
⬚	Installation Boundary

Notes:
* Contours are dated 2016 and from Portage County GIS.
Values are in feet. Only those within the MRS are labeled.

MRS=munitions response site
RVAAP=Ravenna Army Ammuntion Plant
UFP-QAPP=Uniform Federal Policy-Quality-Assurance Project Plan

*\\SRV-GST-01\hglgis\Ravenna_AAP_BM3001\Group_8\QAPP_Prelim\
(B4-01)Group8_MRS_Site_Features_Topo.mxd
12/22/2022 RR
Source: HGL,CB&I, USACE, e²M
ArcGIS Online Imagery*

Figure B.4.2
Group 8 MRS
Erosion and Sedimentation Control Measures Map



Legend

Ⓤ	Power Pole
×	Wattle/Silt Fence
↔	Construction Route
⬚	Temporary Storage and Portable Bathroom
▨	Excavation Area
🟫	Vehicle Tracking Pad
🟨	Staging/Load-out Area
🟦	Ditch
⬚	MRS
⬚	Installation Boundary

Notes:
MRS=munitions response site
RVAAP=Ravenna Army Ammuntion Plant
UFP-QAPP=Uniform Federal Policy-Quality-Assurance Project Plan

\\SRV-GST-01\hglgis\Ravenna_AAP_BM3001\Group_8\QAPP_Prelim\(B4-02)Group8_Erosion_Sed_Control_Measures.mxd
12/22/2022 RR
Source: HGL,CB&I, USACE, e²M
ArcGIS Online Imagery

APPENDIX C

WASTE MANAGEMENT PLAN

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APPENDIX C
WASTE MANAGEMENT PLAN

REMEDIAL ACTION
FORMER RAVENNA ARMY AMMUNITION PLANT
RVAAP-063-R-01 GROUP 8 MRS
MUNITIONS RESPONSE SITE
PORTAGE AND TRUMBULL COUNTIES, OHIO

Contract No. W912DR-21-D-0005
Delivery Order No. W912DR21F0327

Prepared for:

U.S. Army Corps of Engineers, Baltimore District
2 Hopkins Plaza
Baltimore, MD 21201-2930

Prepared by:
HydroGeoLogic, Inc.
11107 Sunset Hills Road, Suite 400
Reston, Virginia 20190

July 2023

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LIST OF ATTACHMENTS

LIST OF ACRONYMS AND ABBREVIATIONS

CJAG	Camp James A. Garfield Joint Military Training Center
DoD	Department of Defense
HGL	HydroGeoLogic, Inc.
IAW	in accordance with
IDW	investigation-derived waste
MD	munitions debris
MDAS	material documented as safe
MDEH	material documented as an explosive hazard
mg/L	milligrams per liter
MPPEH	material potentially presenting an explosive hazard
MRS	Munitions Response Site
RA	Remedial Action
RVAAP	Ravenna Army Ammunition Plant
RCRA	Resource Conservation and Recovery Act
TCLP	toxicity characteristic leaching procedure
UFP-QAPP	Uniform Federal Policy Quality Assurance Protection Plan
WMP	Waste Management Plan

1.0 WASTE MANAGEMENT PLAN

1.0.1 This Waste Management Plan (WMP) describes the approach, methods, and operational procedures that will be employed to manage waste generated during performance of the Remedial Action (RA). The WMP supports the RA to be conducted at the former Ravenna Army Ammunition Plant (RVAAP), now known as Camp James A. Garfield Joint Military Training Center (CJAG), for the Group 8 Munitions Response Site. This work is being conducted for the U.S. Army Corps of Engineers, Baltimore District under Contract No. W912DR-21-D-0005, Delivery No. W912DR21F0327.

1.0.2 All wastes will be stored, characterized, managed, labeled, transported, and disposed in accordance with (IAW) all appropriate Federal, State, and local regulations and statues. An example of the Waste Tracking Log that will be used for this project is presented at the end of this WMP.

1.1 WASTE DISPOSAL

All waste generated will be properly characterized, stored, transported, and disposed of in accordance with all applicable regulations and through approved channels. Although the generation of hazardous waste is not anticipated, elevated concentrations of cadmium and/or lead in soil could result in a portion of the soil cuttings and/or excavated soil being classified as Resource Conservation and Recovery Act (RCRA) hazardous waste per their associated toxicity characteristic leaching procedure (TCLP) metals regulatory limits of 1 milligram per liter (mg/L) (cadmium) and 5 mg/L (lead). The results of the pre-excavation soil characterization sampling will provide total concentrations and TCLP results for the final planned excavation areas. The availability of the pre-excavation data will assist in the appropriate management of excavated soil during on-site activities. The general types of materials expected to be generated during the munitions response activities consist primarily of soil cuttings, excavated soil, material documented as safe (MDAS), wastewater, debris, and non-ordnance related scrap. Attachment C1 to this WMP includes the Waste Management Guidelines for CJAG.

1.2 MATERIAL DOCUMENTED AS SAFE

This section describes the munitions debris (MD) storage, processing, certification, and documentation requirements for MDAS. All material potentially presenting an explosive hazard (MPPEH) that is inspected, verified, and certified to be free of explosive hazards will be classified as MDAS. MDAS generated during the project will be stored in a secure area inside locked containers. Once the field investigation is complete, the sealed containers will be shipped for proper disposal. Certification and disposal of MDAS will be monitored by the Unexploded Ordnance Quality Control Specialist using the applicable Measurement Performance Criteria. MDAS will be handled IAW HydroGeoLogic, Inc. (HGL) Standard Operating Procedure 408.504, MPPEH Inspection and Management (Appendix D).

1.2.1 MD and MDAS Storage

1.2.1.1 In order to maintain the chain of custody, the following categories of material will not be commingled. Should commingling occur, the safe-certified MD will lose its safe certification.

- MD awaiting documentation of its explosives safety status
- Material which has been certified safe (MDAS)
- Material that has been certified hazardous (material documented as an explosive hazard [MDEH])

1.2.1.2 MD and MDAS will be stored in locked containers with signage. Once certified as MDAS, the material will be placed in transport containers with container seals traceable to the transfer documentation.

1.2.2 Documentation of MDAS Certification

Certification as MDAS will be provided using a Disposal Turn-in Document DD Form 1348-1A (series). The two signatures required for the safe certification must be directly above the typed or clearly stamped or legibly printed full name, rank/rate/grade, complete organization name and address, and phone numbers (commercial and Defense Switched Network). Safe certifications shall follow the guidelines listed in the Department of Defense (DoD) Manual 4140.72 and include the following statement:

> *This certifies and verifies that the material listed has been 100 percent inspected, 100 percent re-inspected by qualified personnel, processed by DoD Explosives Safety Board-approved means, or undergone the application of expert knowledge, and to the best of our knowledge and belief, is inert and/or free of explosives or related materials and does not pose an explosive hazard.*

1.2.3 MDAS Disposal

Metal debris classified as MDAS will be collected and disposed off-site for recycling at Demil Metals located in Northbrook, Illinois. The integrity of the containers and the chain of custody will be maintained, and the DD Form 1348-1A will travel with the material through final disposition. The material will be processed through a smelter, shredder, or furnace prior to resale or release in accordance with all governing regulations. The facility will provide a return receipt indicating that the material has been received as well as a separate letter stating that the material has been destroyed by shredding or smelting, thus ensuring the proper chain of custody has been maintained.

1.3 NON-ORDNANCE RELATED SCRAP

Non-ordnance related scrap could be generated during intrusive investigations. Metal that is inspected and certified as not MD will be removed from the site and stored separately from the MD, MDAS, and MDEH. The metal will be transported for off-site disposal.

1.4 INVESTIGATION RELATED TRASH

Investigation generated trash such as used personal protective equipment, sampling equipment, miscellaneous packaging material, etc., will be disposed of as municipal waste.

1.5 EXCAVATED SOIL AND SOIL CUTTINGS

As detailed in the definable feature of work (DFW) #5 in Worksheet 17 of the Uniform Federal Policy Quality Assurance Protection Plan (UFP-QAPP), waste soil (excavated soil and soil sampling cuttings) will be generated during this RA. Additionally, DFW #2, #3, #4, and #5 within the UFP-QAPP define the waste handling and waste characterization methods. Soil cuttings generated during pre-excavation soil characterization sampling activities will be containerized in 55-gallon drums and managed as Investigation Derived Waste. One composite waste characterization sample will be collected from the soil cuttings drums (One sample per 10 drums) and submitted for off-site laboratory analysis of the required Waste Acceptance Criteria parameters. If the soil cuttings are classified as RCRA non-hazardous waste they, will be stored at a location approved by CJAG until they can be added to and disposed along with similarly-classified excavated soil. Non-hazardous waste soil will be disposed at U.S. Ecology in Detroit, Michigan.

1.6 WASTEWATER

As detailed in the DFW #5 in Worksheet 17 of the UFP-QAPP, wastewater (decontamination or excavation dewatering) may be generated during this RA. Additionally, DFW #2, #3, #4, and #5 within the UFP-QAPP define the waste handling and waste characterization methods for wastewater. Wastewater will be generated during soil sampling and excavation equipment decontamination activities. Decontamination wastewater will be containerized in 55-gallon drums and characterized in the same manner described previously for soil cuttings. Although not anticipated, wastewater generated due to excavation dewatering will be handled in the same manner as decontamination wastewater. Non-hazardous wastewater will be disposed at Clean Water Environmental in Dayton, Ohio.

1.7 HAZARDOUS WASTES

As mentioned previously, although no hazardous waste is anticipated for this project, waste characterization profiles will be developed for soil cuttings and excavated soil. If hazardous wastes are generated, disposal of will be IAW with the procedures described in the Environmental Protection Plan (Appendix B).

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ATTACHMENT C1

WASTE MANAGEMENT GUIDELINES

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ANNEX E - APPENDIX B

Waste Management Guidelines

Camp James A. Garfield Joint Military Training Center

Integrated Environmental Contingency Plan

Table of Contents

E.B. 1 - PURPOSE

Guidelines to be followed by contractors working at military installations owned by the Ohio Army National Guard (OHARNG) who are generating/shipping Hazardous or Non-Hazardous Waste. NOTE: The text in this document is taken directly from the *OHIO ARMY NATIONAL GUARD WASTE MANAGEMENT GUIDELINES* published April 30, 2020.

E.B. 2 - POLICY

The policy of the OHARNG is to comply with all local, state, federal and installation requirements.

E.B. 3 – OHARNG Waste Management POCs

NAME	JOB TITLE	OFFICE	Email
Shane Mathey	OHARNG Hazardous Waste Manager	(614) 336-7394	michael.s.mathey.nfg@mail.mil
Shane Mathey's Alternate	Shane Mathey's Environmental Supervisor	(614) 336-7395	michael.s.mathey.nfg@mail.mil
Brad Kline	CJAG Hazardous Waste Manager	(614) 336-4918	bradley.w.kline2.nfg@mail.mil
Katie Tait	RVAAP Restoration Program Hazardous Waste Manager	(614) 336-6136	kathryn.s.tait.nfg@mail.mil
B. Kline / K. Tait Alternate	Environmental Supervisor Alternate	(614) 336-6568	See above

E.B. 4 - COORDINATION

1. Coordinate all waste generation and shipments with the appropriate POC listed above or the Environmental Supervisor in their absence.

2. Notify the POC prior to waste sampling for characterization. Details about sampling activities (i.e., number of sample, analysis, etc.) must be provided.

3. All Hazardous and Non-Hazardous waste storage locations must be pre-approved by POC prior to generation.

4. Ensure all labels include: Accumulation Start Date, Contractor, and Waste Type.

5. When contractors have waste onsite, a weekly inspection and inventory must be completed and submitted to the POC.

6. All wastes shall be tracked and logged throughout the duration of the project. Contractor will provide the POC with a monthly rollup report of all waste and recycled streams generated by no later than the 10th day of the following month.

E.B. 5 - HAZARDOUS WASTE TSDF AND WASTE HAULERS

Contractors are required to utilize qualified Defense Logistics Agency (DLA) waste haulers and Treatment, Storage, and Disposal Facilities (TSDFs). The current qualified waste hauler and TSDF list can be viewed by following the “Qualified Facilities” and “Qualified Transporters” links found on the DLA Hazardous Waste Disposal Homepage:

http://www.dla.mil/DispositionServices/Offers/Disposal/HazardousWaste/HazWasteDisposal.aspx

E.B. 6 - HAZARDOUS OR NON-HAZARDOUS MANIFEST FORM INSTRUCTIONS

1. OHARNG Sites other than CJAG and RVAAP Restoration Program:
    A. The OHARNG Waste Management POC is Mr. Shane Mathey
    B. Site name and address for each facility will be provided upon request.
    C. Ohio EPA identification number for each facility will be provided upon request.

2. CJAG:
    A. The OHARNG Waste Management POC is Mr. Brad Kline
    B. Military and non-restoration operations waste Site Name is Camp Ravenna Joint Military Training Center.
    C. Site Address: 1438 State Route 534 SW, Newton Falls, Ohio 44444, (614) 336-4918.
    D. Mailing Address: Camp James A. Garfield ENV, ATTN: Brad Kline, 1438 State Route 534 SW, Newton Falls, Ohio 44444, (614) 336-4918.
    E. Ohio EPA ID#: OHD981192925.

3. RVAAP Environmental Restoration Program:
    A. The OHARNG Waste Management POC is Ms. Katie Tait
    B. Restoration Program waste Site Name is Former Ravenna Army Ammunition Plant.
    C. Site Address: 8451 State Route 5, Ravenna, Ohio 44266, (614) 336-6136.
    D. Mailing Address: Camp James A. Garfield ENV, ATTN: Katie Tait, 1438 State Route 534 SW, Newton Falls, Ohio 44444, (614) 336-6136.
    E. Ohio EPA ID#: OH5210020736

4. All OHARNG Sites

   A. Contractor's shipping Hazardous Waste must provide a Land Disposal Restriction (LDR) in accordance with 40 CFR Part 268.

   B. Profiling;

      1) The required shipping documentation (i.e., waste profile and executive summary of lab reports (if available)) need to be submitted to the appropriate OHARNG POC, or designee(s) for approval and signature prior to shipping.

      2) Results of characterization must be submitted to the appropriate OHARNG POC within 30 days after collecting sample.

   C. Manifests - Hazardous and Non-Hazardous:

      1) The waste carrier/transporter provides appropriate manifest to the contractor.

      2) The contractor is required to:

         a. Insure the OHARNG POC or designee(s) is available to sign the manifest on the scheduled day of shipment;

         b. Verify that each manifest is properly completed and signed by the OHARNG POC or designee(s);

         c. Provide the Generator copy of the manifest to Shane Mathey or designee(s); and

         d. Ensure that the original Generator copy of the manifest signed by the treatment storage disposal facility is returned to the appropriate OHARNG address within 30 days of the shipping date for Hazardous and Non-Hazardous Waste.

         e. The use of a Bill of Lading, in lieu of a waste manifest, must be approved in advance by the OHARNG POC.

E.B. 7 – SAAS, CAAS, AND CONTAINERS

1. All Satellite Accumulation Areas (SAAs) and containers will comply with 40CFR 262.34(c)(1).

2. From the time any waste is placed in a waste storage container, proper labeling must be on the container (proper labeling includes contractors name and contact information, waste type). An accumulation start date must also be included on containers stored in a Central Accumulation Areas (CAAs).

3. Any waste that is subject to Hazardous Waste Manifest Requirements of the USEPA must comply with 40 CFR Part 262.

4. Pending analysis label is to be used from the time the sample is taken and until the results are received. In no case will waste labeled pending analysis exceed 45 days.

5. For all hazardous waste containers, a container log must be maintained.

6. SAAs and CAAs must be properly documented and closed out once a project are complete.

E.B. 8 – RECORDS

1. All OHARNG Hazardous and Non-Hazardous records except for CJAG and RVAAP Restoration Program records are maintained at the Columbus environmental office, point of contact is Shane Mathey at (614) 336-7394.

2. All CJAG and RVAAP Restoration Program Hazardous and Non-Hazardous records are maintained at the CJAG environmental office, point of contacts are Katie Tait at (614) 336-6136 and Brad Kline at (614) 336-4918.

3. Due to the LQG requirement to comply with the training requirements at 40 CFR 265.16, contractors are required to meet personnel training provisions. If the amount of hazardous waste generated during a project causes an OHARNG facility to be a LQG, contractors must be properly trained on RCRA duties/requirements. Documentation of this training must be kept in the project file.

WEEKLY NON-HAZARDOUS & HAZARDOUS WASTE INSPECTION/INVENTORY SHEET

Contractor: ______________ **Month:** ___________________ **Year:** _____________ **Waste Description:** _____________

Container Nos.:

	WEEK 1	**WEEK 2**	**WEEK 3**	**WEEK 4**
	Date: **Time:**	**Date:** **Time:**	**Date:** **Time:**	**Date:** **Time:**
Point of Contact (Name / Number)				
Project Name:				
Contracting Agency and POC:				
Waste Determination: Pending Analysis, Hazardous, Non-Hazardous, etc.				
*Location on installation:				
Date Generated:				
Projected date of disposal:				
Non-Haz, Satellite, 90-day storage area				
Waste generation site:				
Number of Containers (size / type):				
Condition of Container:				
Containers closed, no loose lids, no loose bungs?	yes / no	yes / no	yes / no	yes / no
Waste labeled properly and visible (40 CFR 262.34 (c) (1):	yes / no	yes / no	yes / no	yes / no
Secondary containment	yes / no	yes / no	yes / no	yes / no
Incompatibles stored together?	yes / no	yes / no	yes / no	yes / no
Any spills?	yes / no	yes / no	yes / no	yes / no
Spill kit available?	yes / no	yes / no	yes / no	yes / no
Fire extinguisher present and charged?	yes / no	yes / no	yes / no	yes / no
Containers grounded if ignitable?	yes / no / na	yes / no / na	yes / no / na	yes / no / na
Emergency notification form/info present?	yes / no	yes / no	yes / no	yes / no
Container log binder present?	yes / no	yes / no	yes / no	yes / no
Signs posted if required?	yes / no	yes / no	yes / no	yes / no
Photo’s submitted	yes / no	yes / no	yes / no	yes / no
Printed Name:				
Signature:				

This form is required for Non-Hazardous and Hazardous waste including PCB and special waste.

CONTRACTORS ARE REQUIRED TO SUBMIT THIS FORM <u>WEEKLY</u> TO THE CJAG-ENV OFFFICE WHEN WASTE IS STORED ON SITE.

CONTRACTORS ARE ENCOURAGED TO INCLUDE PHOTOS WITH EACH WEEKLY INSPECTION SHEET WHEN WASTE IS STORED ON SITE.

*Draw detailed map showing location of waste within the site.

ATTACHMENT C2

WASTE TRACKING LOG

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WASTE TRACKING LOG

Project Name/Site: RA - RVAAP Group 8 MRS
Project No: Contract #W912DR-21-D0005; Delivery Order #W912DR21F0327
Waste Characterization Sample No(s): ____________________
Waste Characterization Sample Analyses: ____________________
Container/Date Sampled: ____________________
PO Number:

Container Type	Date Delivered	Container Designation	Accumulation Date	IDW Source Locations	Media	Amount	Storage Location	Comments/Notes

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APPENDIX D

FIELD STANDARD OPERATING PROCEDURES

HGL HS 12.1	Lock-out Tag-out Procedure
HGL SOP 201.512	Control of Hazardous Energy
HGL SOP 201.522	Excavation and Trenching
HGL SOP 201.528	Drilling Safety
HGL SOP 201.531	Forklifts and Earthmoving Equipment
HGL SOP 300.07	Environmental Database Quality Control
HGL SOP 401.501	Field Logbook Use and Maintenance
HGL SOP 408.504	MPPEH Inspection and Management
HGL SOP 408.504.F01	MPPEH Processing Checklist
HGL SOP 408.507	MEC Anomaly Avoidance Support
HGL SOP 411.02	Sampling Equipment Cleaning and Decontamination
HGL SOP 411.03	Subsurface Utility Avoidance
HGL SOP 501.05	Analog and Digital MEC Operations
HGL SOP 501.06	Vegetation Removal Operations on MMRP Sites
HGL SOP 501.08	MEC Construction Support

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Control of Hazardous Energy Program **Lockout/Tagout Procedure**	H&S Procedure No.: 12.1 Publication Date: March 2012 Revision No.: 06 Revision Date: June 2020 Review Date: June 2021

APPENDIX A
LOCKOUT/TAGOUT PROCEDURE

Equipment ______________________________ Location ______________________________

Date ______________________________ Written by ______________________________

Make Notifications

Notify affected employee(s) that a lockout is required, the reason for the lockout, and the expected duration.

Take all necessary equipment, tools, and personal protective equipment to the job site.

Consider hazards associated with the work you will be doing and gather the tools and equipment needed to do the job safely.

Machine or Equipment Shutdown

Determine all sources of energy feeding into the machine or equipment. Describe the steps needed to shut down the equipment.

Isolation and Lockout/Tagout

Describe steps needed to isolate equipment from all energy sources, including the location(s) to apply lockout/tagout devices. If a lockout device cannot be applied, list the location of the tagout device and any additional precautions taken to ensure the level of safety is equal to that of a lockout device.

Release Stored Energy

List any devices that may contain stored energy and the process to safely release or contain this energy. Examples include, but are not limited to, capacitors, springs, hydraulic/pneumatic cylinders, and pressurized piping.

Verification of Isolation

To ensure that all energy sources have been isolated; 1) Ensure that no personnel can be exposed to operating machinery or equipment, 2) Operate all controls to ensure that the equipment will not operate. List all controls that need to be tested and all indicators that should be observed to ensure the equipment has been isolated from all energy sources. CAUTION: Ensure all controls have been returned to the off or neutral position upon completion of the test.

Jogging or Cycling (If applicable)

If, during repair or maintenance activities, the machine or equipment is required to be jogged or cycled, list the location of lockout/tagout device(s) that can be removed to accomplish this. Only the lockout/tagout devices absolutely necessary to allow the equipment or machine to be jogged or cycled should be removed. After the equipment or machine has been cycled or jogged, the lockout/tagout devices shall be reapplied and above steps shall be repeated prior to commencing work.

Release from Lockout/Tagout and Restoring to Service

- Make sure all tools and other materials have been removed.
- Make sure machines are fully reassembled and guards and other safety devices have been reinstalled.
- Check the work area to ensure that all employees are clear of the equipment or machine.
- Verify that all controls are in their neutral or off position.
- Each lockout/tagout device shall be removed from each energy isolation device by the employee who applied the device.
- Indicate here any steps needed to safely re-energize the machine or equipment.
- Notify all affected employees that the lockout/tagout devices have been removed and the machine or equipment is safe for use.

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CORPORATE TECHNICAL PROCEDURE	
Approved for issue by:	
Process Owner	Steve Davis CIH, CSP Digitally signed by Steve Davis CIH, CSP Date: 2021.09.28 14:32:00 -04'00'
Corporate Quality Director	Theresa Rojas Digitally signed by Theresa Rojas Date: 2021.09.27 13:06:29 -04'00'
Control of Hazardous Energy	**Document No.: HGL SOP 201.512 (formerly H&S Procedure 12)**
	Process Category: Health and Safety
	Revision No.: 7
	Effective Date: September 27, 2021
	Last Review Date: September 27, 2021
	Next Review Date: September 2022

1.0 PURPOSE AND APPLICABILITY

The purpose of these requirements is to prevent harm to personnel due to the unexpected release of energy, particularly when employees construct, install, set up, adjust, inspect, modify, maintain, or service machines or equipment. The fundamental requirements are to shut down and lock out (LO) or tag out (TO) equipment during service, maintenance, inspection, or any other activities that expose personnel to potentially harmful releases of energy. A secondary purpose is to comply with the Control of Hazardous Energy (Lockout/Tagout) standard, 29 Code of Federal Regulations (CFR) 1910.147 and 29 CFR 1926.416, the Occupational Safety and Health Administration (OSHA) regulation that addresses electrical energy control in the construction industry.

These requirements apply to any work that exposes HGL employees and HGL temporary employees to the unplanned and potentially harmful release of energy such as electricity, pressurized fluids, steam, movement, gravity, and heat. This includes situations where personnel work on or near systems or equipment that produce, use, or store hazardous energy and that have the potential to harm personnel through the unplanned release of energy. Examples of such work include equipment inspection, testing, and maintenance tasks that require removal of protective guards. HGL subcontractors that perform this work must have their own processes that meet or exceed OSHA regulations and client requirements.

2.0 SUMMARY OF METHOD

This procedure requires at least the following actions:

- Understand the equipment to be worked on or around, the potential hazards of unplanned energy releases, and hazard controls. Only staff who have the proper tools and equipment may attempt to work this equipment.
- Shut down, isolate, de-energize, block, or otherwise control the hazard and lock all sources of energy to equipment that could cause harm by an unexpected release of energy whenever personnel remove guards or could otherwise be exposed to the hazardous energy. Use tagout devices to indicate the hazard and warn against activating the equipment when it is not possible to lock such equipment.

- Locks or tags are to be removed **only** by the individual who placed them on the equipment rendering them "Out of Service." If this is not possible the supervisor may remove locks or tags after first going through the specified removal process and verifying that no personnel will be harmed by removing the lock or tag and activating the equipment.
- Return equipment to safe status (replace guards, exit the hazardous area, etc.) prior to removing locks or tags and reactivating the equipment.
- Train staff in the applicable hazards and hazard controls.

3.0 DEFINITIONS

Affected employee **–** An employee who works in an energy control area. The affected employee cannot service, maintain, or inspect and is not responsible for carrying out the hazardous energy control program.

Authorized employee **–** An employee who services, maintains, or inspects machines or equipment. Authorized employees are responsible for carrying out the hazardous energy control procedures for their protection and the protection of others.

Energized **–** A machine or piece of equipment connected to an energy source or containing residual or stored energy.

Hazardous Energy Control Program **–** A set of written equipment-specific procedures that clearly and specifically outlines the scope, purpose, authorization, rules, and techniques to be used by staff to control hazardous energy at a specific location. LO/TO procedures and equipment may be one aspect of a hazardous energy control program.

Energy-isolating device **–** Any mechanical device that physically prevents the transmission or release of energy. Examples of energy-isolating devices include manually operated electrical circuit breakers, disconnect switches, line valves, and pipe blocks.

Energy source **–** Any source of electrical, mechanical, hydraulic, pneumatic, chemical, thermal, gravity, or other type of energy.

Lockout (LO) **–** The placement of an LO device on an energy-isolating device, according to established procedure, that ensures that the energy-isolating device and the controlled equipment cannot be operated until the LO device is removed.

Lockout device **–** Any device that uses a positive means such as a lock to hold an energy-isolating device in a safe position, preventing energizing of machinery or equipment. When properly installed, blank pipe flanges or bolted pipe slip blinds are LO devices.

Tagout (TO) **–** The placement of a TO device on an energy-isolating device, according to established procedure, warning others that the energy-isolating device and controlled equipment may not be operated until the TO device is removed.

Tagout device **–** Any prominent warning device, such as a tag and a means of attachment that can be securely fastened to an energy-isolating device according to an established procedure. The tag indicates that the machine or equipment is “Out of Service” and not to be operated until the removal of the TO device according to the hazardous energy control procedure.

4.0 HEALTH AND SAFETY WARNINGS

Failure to follow this procedure when working on or near equipment that may release energy or be activated during planned work can result in injury or death from burns, electrocution, arc flash, crushing, and struck-by incidents.

5.0 CAUTIONS

This section is not applicable to this procedure.

6.0 INTERFERENCES

This section is not applicable to this procedure.

7.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

Corporate officers, managers, and project-level staff should all be role models for safety. Other responsibilities are summarized below.

7.1 SENIOR MANAGEMENT (PRESIDENT, CHIEF OPERATING OFFICER, DIVISIONAL VICE PRESIDENTS)

- Provide adequate resources to enable safe work.
- Maintain a general awareness of procedural requirements.

7.2 CORPORATE HEALTH AND SAFETY DIRECTOR

- Approve this procedure and subsequent revisions.
- Provide support and assistance in the implementation of this procedure.

7.3 OFFICE MANAGERS

- Maintain a general awareness of the applicable portions of these requirements.

- Maintain an awareness of potentially hazardous energy sources on supervised work and ensure that adequate controls are in place.

7.4 OFFICE HEALTH AND SAFETY COORDINATORS AND SITE SAFETY AND HEALTH OFFICERS

- Maintain a general awareness of the applicable portions of these requirements.
- Maintain an awareness of hazardous energy sources in the office or on work sites.
- Verify that energy hazards are adequately controlled.

7.5 SUPERVISORS AND PROJECT MANAGERS

- Ensure that a written, equipment-specific, LO/TO procedure is developed for work in which HGL personnel may be exposed to the unplanned release of hazardous energy.
- Ensure that personnel authorized to perform LO/TO are properly trained.
- Procure a copy of the client’s LO/TO procedures for each piece of client equipment that an HGL employee may be required to service or maintain.
- Procure appropriate hazardous energy control devices for project tasks.
- Coordinate HGL’s hazardous energy control programs with those of the owner or contractor.
- Ensure that staff adheres to approved procedures.

7.6 EMPLOYEES

- Follow the hazardous energy control procedures.
- Alert supervisor if proper equipment is not available or there are uncontrolled hazards.

8.0 EQUIPMENT AND SUPPLIES

Locks and tags are used to control access to energy isolation devices and prevent inadvertent activation. Locks and tags used for hazardous energy control must meet the following requirements.

- They must be able to withstand the environment that they are exposed to for the maximum period of time the exposure is expected.
- They must identity the employee applying the device.
- They must be of a unique design or color to readily identify them as belonging to the LOTO program.
- They must not be used for anything other than lockout activities.

Locks must be substantial enough to prevent removal without the use of excessive force or unusual techniques (such as with the use of bolt cutters). Tags must be attached by substantial and non-reusable attachments such as a zip tie. Tags must warn against the hazardous condition resulting from system energization and include wording such as “DANGER - DO NOT START, OPEN, CLOSE, ENERGIZE, OPERATE.”

9.0 PROCEDURAL STEPS

9.1 BASIC REQUIREMENTS

To ensure the safety of staff, the following measures must be taken when working with hazardous energy sources:

- Protect personnel from the hazards of unplanned energy releases during tasks such as permit-required confined space entry or equipment maintenance, repair, or inspection. In general, these requirements apply when protective guards are removed from equipment or personnel enter areas that are not intended for worker occupancy. These requirements do not apply to routine work where protective guards are in place and personnel do not enter areas that are not designed for occupancy.
- Identify and assess the potential energy sources that employees may be exposed to when protective guards are removed or when personnel enter areas that are not designed for occupancy. Potential hazardous energy sources include, but may not be limited to, electricity (electrocution and arc flash), pressurized gasses, gravity (components that may fall on personnel), moving parts (agitators, blades, grinders), steam, ionizing radiation, thermal systems (heaters, chillers), and movement or engulfment by particulate matter or liquids.
- Shut down, isolate, and lock all sources of energy to equipment that have a potential to cause harm by unplanned releases of energy whenever personnel remove guards, enter areas not designed for occupancy, or otherwise create a potential exposure to the hazardous energy. Use tags to indicate the hazard and warn against activating the equipment when it is not possible to lock such equipment.
- See HGL SOP 201.511: *Electrical Safety* and H&S Procedure 26: *Confined Space Entry* for additional information.[1]
- Ensure that personnel who perform such work are properly trained and understand the equipment to be worked on or around, the potential hazards of unplanned energy releases, and hazard controls. Only staff who have the proper tools may attempt to repair or maintain equipment.

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[1] When updated, H&S Procedure 26 will be renumbered as HGL SOP 201.527.

- Ensure that locks or tags are removed by the individual that placed them. If this is not possible, the supervisor may remove them after first going through the specified removal process to ensure that workers are not endangered by removal.

When the energy isolating devices are not lockable, tagout may be used if employees receive additional training and more rigorous periodic inspections are conducted by the Site Safety and Health Officer (SSHO). For more complex energy source systems, more comprehensive procedures may need to be developed, documented, and utilized.

LO/TO devices must be specific to this purpose, must be capable of withstanding the ambient conditions, and must identify the authorized person who placed the devices. TO devices must be labeled with warnings of the hazards and specify that they are not to be removed except by the person who placed them.

9.2 EQUIPMENT-SPECIFIC LOCKOUT/TAGOUT TEMPLATE

Equipment-specific procedures are developed by experienced and knowledgeable personnel and then reviewed by the supervisor. These procedures must provide the following information:

- Administrative information such as procedure numbers and procedure development dates;
- Identification of potential energy sources(s);
- Shutdown preparation;
- Notification of affected and other employees;
- Machine or equipment shutdown;
- Machine or equipment isolation;
- Application of LO/TO devices;
- Stored energy management; and
- Isolation verification.

9.3 LOCK OR TAG REMOVAL BY AUTHORIZED EMPLOYEES

In most cases, the HGL authorized employee who applied the LO/TO is responsible for removing the lock/tag and restoring the equipment to service. The sequence of LO/TO removal steps is as follows:

- Check machine/equipment and the immediate area to ensure that nonessential items have been removed and that the equipment is operationally intact and unimpaired for successful re-energization/restart and operation.

- Ensure that employees have been removed from area.
- Remove locks/tags in the reverse order of their application during all LO/TO steps to re-energize equipment.
- Notify affected employees that servicing and maintenance have been completed and that the equipment is ready for use.
- Submit the required HGL paperwork, completed by an authorized employee, to the supervisor.

9.4 LOCK OR TAG REMOVAL BY OTHERS

When the authorized employee who applied the LO/TO device is not available to remove it, the device may be removed under the direction of the supervisor.

The supervisor must perform the following to complete the process:

- Verify that the authorized employee who locked out the equipment is not available or on the site.
- Make all reasonable efforts to contact the authorized employee to inform him/her that his/her LO/TO device will be removed.
- Visually confirm that all employees, tools, and materials are clear of the equipment.
- Remove the LO or TO device as described above.
- Ensure the authorized employee is notified that his/her lock has been removed before he/she resumes work at the facility.

9.5 GROUP LOCKOUT/TAGOUT

When more than one staff member or more than one employer services, maintains, or inspects machines or equipment covered by this procedure, they must implement the measures below to ensure that each worker is provided with a level of protection equivalent to that provided by use of a personal LO or TO device.

- The supervisor will designate a “primary” authorized employee to be responsible for all the employees working under the protection of the group LO device.
- The “primary” authorized employee is in charge of the process and will place the group LO device.
- The primary authorized employee must notify affected employees before and after LO procedures are performed.

- Each authorized employee must place his/her own personal LO/TO device on the energy isolating device and remove his/her own devices when he/she stops working on the machine or equipment.
- When an energy isolating device cannot accept locks, a multiple TO system must be used.

9.6 SHIFT OR PERSONNEL CHANGE

If LO/TO work extends over multiple shifts, the continuity of protection must be maintained. Two steps are required:

- If an LO/TO procedure will extend into the following shift, the authorized employee who placed his/her personal lock must remove it.
- The lock must immediately be replaced with the personal lock of the next authorized employee to continue the repair or maintenance on that equipment or machine.

9.7 CORD AND PLUG CONNECTED EQUIPMENT

The following procedures are used when servicing or maintaining cord and plug equipment:

- Unplug equipment from its electrical outlet.
- Maintain control of the plug to prevent it from being re-inserted in an outlet. If it is not possible to maintain direct control of the plug, utilize a secure method to lock it out such as placing a lockable cover over the plug.
- Ensure that all other forms of hazardous energy such as batteries and capacitors are also controlled.

9.8 OUTSIDE CONTRACTORS

Whenever outside service, maintenance, or inspection personnel are to be engaged in activities that could expose HGL employees to hazardous energy sources, each must inform the other of their respective LO/TO procedures. The guidance for contractor procedures is as follows:

- Verify that the contractor has an LO/TO procedure and that they provide or ensure training of their personnel.
- Ensure that all potentially affected HGL personnel are aware of the contractor's work and will not be endangered by the work.
- Coordinate with the contractor to ensure that their personnel are not endangered by HGL activities.

9.9 TAGS

Tags alone may be used without locks only when equipment cannot be locked out. The following requirements and restrictions apply to the use of tags:

- Tags are warning devices affixed to energy isolating devices and do not provide the physical restraint that is provided by a lock.
- When a tag is attached to an energy isolating device, it is not to be removed without the express consent of the authorized person responsible for it. It is never to be bypassed, ignored, or otherwise defeated.
- To be effective, tags must be legible and understandable by all the authorized, affected, and other employees whose work operations are, or may be, in the area.
- Tags and their means of attachment must be made of materials that will withstand the environmental conditions encountered in the workplace.
- Tags must be attached to energy isolating devices securely so that they cannot be detached inadvertently or accidentally during use.

9.10 TRAINING REQUIREMENTS

- Each employee who is authorized to perform equipment maintenance or other tasks that may expose personnel to hazardous energy must receive training in the recognition of applicable hazardous energy sources, the type and magnitude of the energy available in the workplace, and the methods and means necessary for energy isolation and control.
- Each employee who could be affected by exposure to a hazardous energy release must be instructed about the purpose and use of the hazardous energy control program, prohibitions relating to attempts to restart or re-energize locked out or tagged out machines or equipment, and safe clearance distances from energized equipment.
- When tagout systems are used, employees must be trained in the limitations of tags.
- Retraining must be provided when there is a change in equipment or processes that presents a new hazard.

Refer to the training requirement in HGL SOP 201.511: *Electrical Safety* for additional information.

10.0 DATA AND RECORDS MANAGEMENT

Documentation generated as a result of this procedure must be collected and maintained in accordance with document control requirements of the HGL Quality Manual. Create and retain records of at least electrical safety training, electrical hazard assessments, and hazard control plans.

11.0 QUALITY CONTROL AND QUALITY ASSURANCE

The responsible manager or site safety and health officer must perform sufficient due diligence to verify that work meets requirements.

12.0 REFERENCES

OSHA 29 CFR 1910.147, *The Control of Hazardous Energy (lockout/tagout)*

OSHA 29 CFR 1926, Subpart K, *Electrical*

HGL SOP 201.511: *Electrical Safety*

HGL H&S Procedure 26: *Confined Space Entry*[2]

13.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
0	March 2012	Initial Release
1	February 2013	Annual review
2	August 2015	Annual review
3	March 2017	Annual review
4	May 2018	Annual review
5	May 2019	Annual review
6	June 2020	Annual review
7	September 2021	Annual review with the format revised to reflect CMS Library standards and the document number changed from Health and Safety Procedure 11 to HGL SOP 201.512.

ATTACHMENTS

None.

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[2] When updated, H&S Procedure 26 will be renumbered as HGL SOP 201.527.

CORPORATE TECHNICAL PROCEDURE	
Approved for issue by:	
Process Owner	Steve Davis CIH, CSP Digitally signed by Steve Davis CIH, CSP Date: 2022.04.06 15:25:12 -04'00'
Corporate Quality Director	Theresa Rojas Digitally signed by Theresa Rojas Date: 2022.04.06 18:01:12 -04'00'

Excavation and Trenching	
	Document No.: HGL SOP 201.522 **(Formerly H&S Procedure 21)**
	Process Category: Health and Safety
	Revision No.: 9
	Effective Date: April 6, 2022
	Last Review Date: April 6, 2022
	Next Review Date: April 2023

1.0 PURPOSE AND APPLICABILITY

The purpose of this procedure is to minimize the probability of injuries and incidents associated with excavation and trenching and to comply with applicable regulations including Occupational Safety and Health Administration (OSHA) Subpart P, Excavations, and specifically 29 Code of Federal Regulations (CFR) 1926.650, 29 CFR 1926.651, and 29 CFR 1926.652. This procedure is applicable to all HydroGeoLogic, Inc. (HGL) employees and temporary employees who work in, or near, excavation or trenching operations, or who manage such operations. HGL subcontractors who perform excavation and trenching for HGL must comply with the applicable portions of this procedure and must have their own programs and procedures that are at least as protective as this procedure.

If HGL employees perform excavation work, HGL must provide and designate, in writing, a qualified Excavation Competent Person who will be in direct charge of the excavation. If a subcontractor performs the excavation work, the subcontractor must provide and designate, in writing, a qualified Excavation Competent Person. When a subcontractor is in charge of excavation work, HGL's site manager or Site Safety and Health Officer (SSHO) must be knowledgeable in excavation hazards and hazard controls to ensure that the subcontractor implements adequate hazard controls.

2.0 SUMMARY OF METHOD

Excavation and trenching are among the most hazardous construction operations. Trench cave-ins pose a significant risk and are more likely than many other excavation-related accidents to result in worker fatalities. Trench collapses cause dozens of fatalities and hundreds of injuries each year. Other potential hazards that may be encountered during excavating and trenching include falls, falling loads, hazardous atmospheres, struck-by or crushing incidents involving mobile equipment, and releases (electricity, gas, fuel) from damaged utilities.

OSHA defines an excavation as any man-made cut, cavity, trench, or depression formed by earth removal in the earth's surface. A trench is defined as a narrow excavation deeper than it is wide, and no wider than 15 feet (4.5 meters).

HGL will ensure that the following hazard controls, at a minimum, are implemented for all excavation or trenching:

- An Excavation Competent Person(s) appropriately trained to recognize excavation hazards and authorized to correct hazards must be designated in writing by the responsible manager. If the Excavation Competent Person is an HGL employee, they must be designated, in writing, by the Project Manager or Supervisor and approved by the Corporate Health and Safety Director. The Excavation Competent Person is in charge of the excavation(s), is responsible for ensuring that excavation work is carried out safely and in compliance with requirements. and must be on site during excavation activities.
- Prior to excavation, the Excavation Competent Person must conduct a visual survey to identify surface hazards or challenges such as existing buildings, sidewalks, power lines, surface drainage, or similar and must implement a plan to prevent damage to important surface features or harm to personnel due to surface features.
- Prior to excavation, the Excavation Competent Person must ensure that a good faith effort is made to identify and locate buried utilities and structures in the proposed area of excavation to prevent inadvertent contact or damage and to minimize the potential for injury to personnel. This effort will include, at minimum, contacting facility or public utility locating services and completing dig permits or similar documentation. Private utility-locating services may also be used to precisely locate utilities by using equipment such as ground-penetrating radar. If there are utilities within 5 feet of the proposed excavation, the precise location of those utilities must be determined by excavating with low-impact tools such as a hand auger, shovel, or air knife. If a utility extends through the area to be excavated, the utility must be exposed to confirm location and depth using low-impact technique(s) at least once every 10 feet. The utility must be exposed continuously, using low-impact techniques, when performing powered excavation within 5 feet of the utility. If hazardous utilities such as gas lines or high-voltage electrical lines are in or near the excavation area, the Excavation Competent Person will investigate the viability of having such utilities de-energized or de-pressurized. See HGL Standard Operating Procedure (SOP) 411.03: *Subsurface Utility Avoidance* for additional information.[1]
- The Excavation Competent Person must ensure that trenches are constructed and controlled to prevent harm to personnel by cave-in. If cave-ins are prevented by sloping or benching, they must assess the soil type (using a pocket penetrometer or equivalent device) to verify that the angle is sufficient to prevent a cave-in.
- The Excavation Competent Person must be present and actively overseeing excavation activities and must perform and document inspections of excavations at least once each day and any time conditions change, such as after a rain.
- The Excavation Competent Person must ensure that, when personnel enter trenches deeper than 5 feet, engineering controls to prevent cave-in are in place. Appropriate engineering controls may include sloping or supporting (shoring) the sides of trenches.

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[1] When updated, HGL SOP 411.03: *Subsurface Utility Avoidance* will be renumbered as HGL 505.156.

- HGL must inspect excavations managed by subcontractors at sufficient frequency to confirm that the subcontractor complies with these requirements and must require the subcontractor to make corrections if they are not in compliance.
- If Munitions and Explosives of Concern (MEC) are or may be present in the area being excavated, additional requirements apply. These requirements vary based on the probability of encountering MEC and the nature and hazard posed by the MEC. The requirements may include additional hazard controls such as escort by Unexploded Ordnance (UXO)-qualified personnel, large exclusion zones to include potential blast radius, equipment operation by UXO-qualified personnel, and specialty equipment such as remote-controlled or shielded excavators. See HGL's standard operating procedures for munitions response work for additional information.

3.0 DEFINITIONS

Aluminum Hydraulic Shoring **–** A pre-engineered shoring system designed specifically to support the sidewalls of a trench and prevent cave-ins that is comprised of aluminum hydraulic cylinders (cross braces) used in conjunction with vertical rails (uprights) or horizontal rails (wales).

Benching (Benching System) **–** A method of protecting employees from cave-ins by excavating the sides of an excavation to form one or a series of horizontal levels or steps, usually with vertical or near-vertical surfaces between levels.

Cave-In **–** The separation of a mass of soil or rock material from the side of an excavation or the loss of soil from under a trench shield or support system and the sudden falling or sliding of the material into the excavation in sufficient quantity that could entrap, bury, or otherwise injure and immobilize a person.

Class 2 High-Visibility Garment **–** As defined by the American National Standards Institute (ANSI), Class 2 must have at least 775 square inches of high-visibility material and at least 201 square inches of reflective tape.

Class 3 High-Visibility Garment **–** As defined by ANSI, Class 3 must have at least 1,240 square inches of high-visibility material and at least 310 square inches of reflective tape. These garments resemble short-sleeved T-shirts with reflective tape on the body and sleeves.

Cross Braces **–** The horizontal members of a shoring system installed perpendicular to the sides of the excavation, the ends of which bear against either uprights or wales.

Excavation **–** Any man-made cut, cavity, trench, or depression in an earth surface formed by earth removal.

Excavation Competent Person **–** One who is capable of identifying existing and predictable hazards in the surroundings or working conditions that are unsanitary, hazardous, or dangerous to employees and who has authorization to take prompt corrective measures to eliminate them. An

Excavation Competent Person must have documented classroom or on-the-job training in excavation hazards and the control of those hazards and must be designated, in writing, for the specific project or excavation.

Faces or ***Sides*** **–** The vertical or inclined earth surfaces formed because of excavation work.

Failure **–** The breakage, displacement, or permanent deformation of a structural member or connection that reduces its structural integrity and supportive capabilities. See also kickout.

Hazardous Atmosphere **–** An atmosphere that because of being explosive, flammable, poisonous, corrosive, oxidizing, irritating, oxygen deficient, toxic, or otherwise harmful, can cause death, illness, or injury.

Kickout **–** The accidental release or failure of a cross brace.

Pocket Penetrometer – A device used by the Excavation Competent Person to perform a visual classification of soils. The pocket penetrometer, or hand penetrometer, can be used to make quick estimates of unconfined compressive strength of cohesive soils, especially in field applications.

Protective System **–** A method of protecting employees from cave-ins, from material that could fall or roll from an excavation face or into an excavation, or from the collapse of adjacent structures. Protective systems include support systems, sloping and benching systems, shield systems, and others that provide the necessary protection.

Registered Professional Engineer (P.E.) **–** A person who is registered as a P.E. in the state where the work is to be performed.

Sheeting **–** The member of a shoring system that retains the earth in position and is supported by other members of the shoring system.

Shield (Shield System) **–** A structure able to withstand the forces imposed on it by a cave-in and thereby protect employees within the structure. Shields can be permanent structures or can be designed to be portable and moved along as work progresses. Additionally, shields can be either pre-manufactured or job-built in accordance with CFR 1926.652© (3) ©(c) (4). Shields used in trenches are usually referred to as “trench boxes” or “trench shields.”

Shoring (Shoring System) **–** A structure such as a metal hydraulic, mechanical, or timber that supports the sides of an excavation and is designed to prevent cave-ins.

Sloping (Sloping System) **–** A method of protecting employees from cave-ins by sloping the sides of an excavation upward and outward at an angle away from the excavation. The angle of incline required to prevent a cave-in varies with differences in soil type, environmental conditions of exposure, and application of surcharge loads.

Stable Rock **–** Natural solid mineral material that can be excavated with vertical sides and will remain intact while exposed. Unstable rock is considered stable when the rock material on the side or sides of the excavation is secured against caving-in or movement by rock bolts or by another protective system that has been designed by a registered P.E.

Structural Ramp **–** A ramp built of steel or wood usually used for vehicle access. Ramps made of soil or rock are not considered structural ramps.

Support System **–** A structure such as underpinning, bracing, or shoring that provides support to an adjacent structure, underground installation, or the sides of an excavation.

Tabulated Data **–** Tables and charts approved by a registered P.E. used to design and construct a protective system.

Trench (Trench Excavation) **–** A narrow excavation (in relation to its length) made below the surface of the ground. In general, the depth is greater than the width, but the width of a trench (measured at the bottom) is less than 15 feet (4.6 meters [m]).

Trench Box **–** See "*Shield*."

Trench Shield **–** See "*Shield*."

Type A Soil **–** Cohesive soil with an unconfined compressive strength of 1.5 tons per square foot or greater.

Type B Soil **–** Cohesive soil with an unconfined compressive strength greater than 0.5 ton per square foot but less than 1.5 tons per square foot.

Type C Soil **–** Cohesive soil with an unconfined compressive strength of 0.5 ton per square foot or less.

Uprights **–** The vertical members of a trench shoring system placed in contact with the earth and usually positioned so that individual members do not contact one another. Uprights placed so that individual members are closely spaced and in contact with or interconnected to each other are often called "sheeting."

Wales **–** Horizontal members of a shoring system placed parallel to the excavation face whose sides bear against the vertical members of the shoring system or earth.

4.0 HEALTH AND SAFETY WARNINGS

Failure to follow this procedure may result in property damage, third party claims, injuries, deaths, or regulatory violations.

5.0 CAUTIONS

This section is not applicable to this procedure.

6.0 INTERFERENCES

This section is not applicable to this procedure.

7.0 PERSONNEL QUALIFICATIONS / RESPONSIBILITIES

Corporate officers, managers, and project-level staff should all be role models for safety. Their other responsibilities are summarized below.

7.1 SENIOR MANAGEMENT (PRESIDENT, CHIEF OPERATING OFFICER, DIVISIONAL VICE PRESIDENTS)

- Maintain a general awareness of health and safety procedures.
- Provide adequate resources to perform work safely.

7.2 CORPORATE HEALTH AND SAFETY DIRECTOR

- Approve this procedure and subsequent revisions.
- Provide support and assistance in implementation.
- Monitor the overall effectiveness of the program.
- Assist with atmospheric testing and equipment selection, as needed.
- Assist with personal protective equipment selection, as needed.
- Assist with the selection of Excavation Competent Persons.
- Evaluate proposed protective systems, as needed.
- Facilitate training of affected employees and supervisors.

7.3 OFFICE MANAGERS

- Maintain a general awareness of the applicable portions of these requirements.
- Be aware of excavations conducted on supervised work and ensure that adequate controls are in place.

7.4 OFFICE HEALTH AND SAFETY COORDINATORS

- Maintain a general awareness of the applicable portions of these requirements.
- Maintain an awareness of excavation work managed by the office.
- Verify that adequate controls are in place for excavation work controlled by the office.

7.5 SUPERVISORS AND PROJECT MANAGERS

- Maintain an awareness of the applicable portions of these requirements.
- Provide the resources to facilitate safe operation.
- Ensure that employees receive appropriate training and oversight.
- Promptly address any unsafe conditions or unsafe acts.
- Conduct regular discussions with staff on matters pertaining to health and safety.
- Ensure that the excavation work is supervised by a qualified Excavation Competent Person.

7.6 SITE SAFETY AND HEALTH OFFICER

- Maintain an awareness of the applicable portions of these requirements.
- Conduct site-specific training and maintain training records.
- Verify and retain documentation of the training and written designation of the Excavation Competent Person in project records.
- Perform daily inspections of the worksite to verify that hazard controls are being implemented and are effective.
- Maintain and use excavation and trenching hazardous atmosphere testing equipment, as necessary.
- The SSHO may be the Excavation Competent Person if properly trained, experienced, and authorized, in writing, to manage the excavation work as well as to stop excavation work, if necessary.

7.7 EXCAVATION COMPETENT PERSON

- Ensure that excavation work is conducted safely and in compliance with applicable requirements.
- Survey areas of planned excavations to identify potential hazards such as water sources and adjacent structures.
- Ensure that effective measures are taken to identify and locate buried structures. Such steps may include completing dig permits, contacting utility companies or facility management, conducting instrument surveys, and daylighting utilities using low-impact techniques such as digging with a hand auger, shovel, or air knife.
- Ensure that adequate measures are taken to protect personnel from hazards due to buried structures. Such steps may include allowing only low-impact excavation techniques within 5 feet of known potentially dangerous buried structures.

- Implement effective measures to prevent trench cave-ins such as sloping or shoring if personnel need to work in trenches deeper than 5 feet. Trenches shallower than 5 feet that an Excavation Competent Person examines, determines, and documents that there is no potential for cave-in do not require protective systems; however, a fixed means of egress must be provided for trenches deeper than 4 feet.
- Perform inspections of excavations at least once each day and more often following rain or other events that might change conditions within an excavation.

7.8 EMPLOYEES

- All employees, including subcontractor personnel, who work in or around excavations, must comply with the requirements of OSHA and the U.S. Army Corps of Engineers, if they apply, and with any applicable site or facility requirements. .
- Report all hazardous practices or situations to HGL management.

8.0 EQUIPMENT AND SUPPLIES

Equipment and supplies used to meet these requirements may include shovels; hand augers; utility detecting instruments; excavation equipment such as bulldozers, backhoes, and excavators; and trench wall stabilization equipment such as trench boxes.

9.0 PROCEDURAL STEPS

9.1 UTILITY LOCATES

Prior to mobilizing to the site or initiating site work, the Project Manager (PM) (or designee) must contact local utility companies or facility utility organizations and order utility locates in the vicinity of the proposed excavation. The exact locations and depths of any utilities that are within the area of excavation, or within 5 feet of its perimeter, must be verified by low-impact excavation tools such as a shovel, hand auger, or air knife to expose any buried structures. Determine and mark the location of sewer, telephone, fuel, electric, water, or any other underground installations or wires that may be encountered during excavation work, as specified in HGL SOP 411.03 before opening an excavation. The PM must arrange, as necessary, with the appropriate utility entity for the protection, removal, shutdown, or relocation of active underground utilities.

9.2 PRE-WORK SITE INSPECTION

Before excavation begins, the Excavation Competent Person must thoroughly inspect the site to determine if there are surface features such as buildings, sidewalks, drainage systems, trees, overhead utilities, or other features that must be protected or that may pose a hazard. The Excavation Competent Person can use the HGL Excavation and Trenching Oversight Checklist (see HGL SOP 201.522.F01) to document this inspection. The Excavation Competent Person must implement steps, such as supporting or removing such features, to prevent putting employees at risk and to prevent inadvertent damage to important surface features.

9.3 PROTECTION OF UTILITIES

If utilities are known to be within the footprint of an excavation or within 5 feet of the outer edge of an excavation, the exact locations and depths of the utilities must be determined by excavating with low-impact tools such as a hand auger, shovel, or air knife. If a utility extends through the area to be excavated, the utility must be exposed to confirm its location and depth using a low-impact techniques at least once every 10 feet. Moreover, the utility must be exposed continuously, using low-impact techniques, when performing powered excavation within 5 feet of the utility.

Loose materials such as soil or gravel should not be stockpiled over buried utilities. If it is necessary to stockpile soil, gravel, or similar materials over buried utilities, the depth of utilities in the stockpile area must be determined in advance with low-impact excavation equipment such as shovels, hand augers, or air knifes, and plastic sheeting or a similar visible indicator must be used to delineate the boundary between the stockpiled material and the original ground surface.

If overhead utilities are present in the work area, provisions must be made to flag the lines and utilize signage identifying the overhead hazard. Do not allow equipment to be operated within 20 feet of uninsulated, energized overhead transmission lines. However, the distance may be decreased if the voltage is known. The required stand-off is specified in 29 CFR 1910.269.

9.4 PROTECTION OF THE PUBLIC

Prior to starting excavation, install fencing, barricades, walkways, lighting, and signage, as necessary, for the protection of the public.

- Provide guardrails, fences, or barricades on excavations adjacent to walkways, driveways, and other pedestrian or vehicle thoroughfares. Maintain warning lights or other illumination, as necessary, for the safety of the public and employees from sunset to sunrise.
- Effectively barricade or cover wells, holes, pits, shafts, and all similar hazardous excavations and post notices, as necessary, to prevent unauthorized access. Backfill all temporary excavations of this type as soon as possible.
- Provide walkways or bridges protected by standard guardrails where employees and the public are permitted to cross over excavations. Where workers in the excavation may pass under these walkways or bridges, use a standard guardrail and toe board to prevent the hazard of falling objects.

9.5 STRUCTURAL RAMPS

- An Excavation Competent Person must design structural ramps used by employees as a means of access or egress from excavations. A person qualified in structural design must design structural ramps used for access or egress of equipment, and the structural ramps must be constructed in accordance with the design.
- Ramps and runways constructed of two or more structural members must have the structural members connected securely to prevent movement or displacement.
- Structural members used for ramps and runways must have uniform thickness.
- Attach cleats or other surface treatments on the top surface of structural ramps used in place of steps to prevent slipping.

9.6 LADDERS

Locate a stairway, ladder, ramp, or other safe means of egress in trench excavations that are 4 feet (1.22 m) or more in depth to require no more than 25 feet (7.62 m) of lateral travel for employees. Extend the top of the ladder at least 3 feet above the upper level.

9.7 EXPOSURE TO VEHICULAR TRAFFIC

Provide employees exposed to vehicular traffic with, and require that they wear, high-visibility vests or other suitable garments marked with or made of reflectorized or high-visibility material. If traffic is moving at speeds under 50 mph, Class 2 vests are appropriate. If traffic is moving at speeds over 50 mph, then Class 3 vests must be worn. Provide emergency lighting, such as spotlights or portable lights, as needed, to perform work safely. Install concrete barriers or other traffic controls as designed in the traffic control plan.

9.8 EXPOSURE TO FALLING LOADS

Do not permit employees to walk or work underneath loads being handled by lifting or digging equipment. Employees are required to stand away from any vehicle being loaded or unloaded to avoid being struck by any spillage or falling materials. Operators may remain in the cabs of vehicles being loaded or unloaded when the vehicles provide adequate protection for the operator during loading and unloading operations.

9.9 WARNING SYSTEM FOR MOBILE EQUIPMENT

All mobile heavy equipment must have working backup alarms. Site personnel should never walk behind operating equipment without equipment operator visual acknowledgement or radio contact. Use a warning system consisting of barricades, hand or mechanical signals, or stop logs when mobile equipment is being operated adjacent to the edge of an excavation if the operator does not have a clear and direct view of the edge of the excavation. If possible, the grade should be away from the excavation.

9.10 HAZARDOUS ATMOSPHERES

The SSHO must test the atmosphere in excavations over 4 feet deep if a hazardous atmosphere exists or could reasonably be expected to exist, as detailed in the approved project plans. A hazardous atmosphere could be expected, for example, in excavations in landfill areas, areas where hazardous substances are stored nearby, areas with soil or groundwater contamination, or near gas pipelines.

- Take adequate precautions to prevent employee exposure to atmospheres containing less than 19.5% oxygen, greater than 10% of the lower flammable limit of flammable gas, and other hazardous atmospheres. These precautions might include providing forced ventilation of the workspace.
- Perform atmospheric testing using a properly calibrated direct reading gas monitor. Direct reading gas detector tubes or other acceptable means may also be used to test potentially toxic atmospheres. Direct reading gas monitors must be field-checked against a calibration standard each day before use and must be within ±5% of the concentration of the standard.

9.11 WALKWAYS AND GUARDRAILS

Provide walkways where employees or equipment are permitted to cross over excavations. Install guardrails where walkways are 6 feet or more above lower levels.

9.12 FALL PROTECTION

Each employee at the edge of an excavation 6 feet (1.8 m) or more in depth must be protected from falling by guardrail systems, fences, or barricades when the excavations are not readily seen because of plant growth or other visual barrier.

9.13 PROTECTION FROM WATER ACCUMULATION HAZARDS

- Employees are not permitted to enter or work in excavations that contain or are accumulating water, unless the Excavation Competent Person has inspected the excavation and determined that the hazard controls are adequate and the water poses no hazard. Precautions may include special support or shield systems to protect from cave-ins or water removal to control the level of accumulating water.
- If water is controlled or prevented from accumulating using water removal equipment, a person trained in the use of that equipment must monitor the water removal equipment and operation.
- If excavation work interrupts the natural drainage of surface water (such as streams), use diversion ditches, dikes, or other suitable means to prevent surface water from entering the excavation. Take precautions to provide adequate drainage of the area adjacent to the

excavation. The Excavation Competent Person must re-inspect excavations after each rain incident and take steps, as necessary, to protect personnel.

9.14 STABILITY OF ADJACENT STRUCTURES

The Excavation Competent Person must determine if the excavation work could affect the stability of adjoining buildings, walls, sidewalks, or other structures and implement controls sufficient to protect personnel and any important features, such as buildings.

Do not permit excavation below the level of the base or footing of any foundation, retaining wall, or pipeline that could reasonably be expected to pose a hazard to employees, except when

- A support system, such as underpinning, is provided to ensure the safety of employees and the stability of the structure;
- The excavation is in stable rock; or
- A registered P.E. has approved the determination that the structure is sufficiently removed from the excavation as to be unaffected by the excavation activity.

9.15 PROTECTION FROM FALLING OBJECTS AND LOOSE ROCKS OR SOIL

- The Excavation Competent Person must ensure that employees are protected from harm due to loose rock or soil that could pose a hazard by falling or rolling into an excavation. Do not permit excavation personnel to work above one another where the danger of falling tools, equipment, rock, or earth exists.
- Protect employees from excavated materials, equipment, or other materials that could pose a hazard by falling or rolling into excavations. Excavations into the side of a hill resulting in a vertical wall require an exclusion zone at least 1.5 times the wall height away from the base of the wall.
- Provide protection by keeping such materials or equipment at least 2 feet from the edge of excavations and by using restraining devices that are sufficient to prevent materials or equipment from falling or rolling into excavations, or by a combination of both, if necessary.
- Store materials and equipment more than 2 feet from the edge of the excavation.
- Materials piled, grouped, or stacked near the edge of an excavation must be stable and self-supporting.

9.16 INSPECTION BY EXCAVATION COMPETENT PERSON

- The Excavation Competent Person must conduct daily inspections of excavations, adjacent areas, and protective systems for evidence of a situation that could result in possible cave-ins or failure of protective systems, hazardous atmospheres, or other hazardous conditions. The Excavation Competent Person must conduct an inspection before the start of work and after every rainstorm or other hazard-increasing occurrence.

- Where the Excavation Competent Person finds evidence of a situation that could result in a possible cave-in, failure of protective systems, hazardous atmosphere, or other hazardous conditions, exposed employees must leave the hazardous area until precautions have been taken to ensure their safety.
- The Excavation Competent Person must maintain a written log of all inspections conducted, including the date, worksite location, results of the inspection, and a summary of any action taken to correct existing hazards.

9.17 PROTECTIVE SYSTEM REQUIREMENTS

9.17.1 Protection of Employees

The Excavation Competent Person must ensure that employees in trenches are protected from cave-ins by using either an adequate sloping and benching system or an adequate support or protective system. The only exceptions are as follows:

- Excavations made entirely in stable rock; or
- Excavations less than 5 feet in depth where examination of the ground by the Excavation Competent Person provides no indication of a potential cave-in.

9.17.1.1 <u>Allowable Slope or Bench Configurations for Excavations Less Than 20 Feet Deep</u>

- Slope excavations at an angle no steeper than 1.5 degrees horizontal to 1 degree vertical (34 degrees measured from the horizontal), unless one of the options listed below is used; or
- Evaluate soil types, as shown in 29 CFR 1926, Subpart P, Appendix A, and slope excavations as specified in 29 CFR 1926 Subpart P, Appendix B.

9.17.1.2 <u>Slope or Bench Designs Using Other Tabulated Data</u>

The design of sloping or benching systems for excavations less than 20 feet deep may be selected from and constructed in accordance with other tabulated data, such as tables and charts. The tabulated data used must be in written form and identify the following:

- Factors that affect the selection of a sloping or benching system;
- Limits of the use of the data, including the maximum height and angle of the slopes determined to be safe;
- Other information needed by the user to make correct selection of a protective system;

At least one copy of the tabulated data that identifies the registered P.E. who approved the data must be present at the jobsite during construction of the protective system.

9.17.1.3 Slope or Bench Design by a Registered Professional Engineer

A registered P.E. must approve sloping or benching systems designed in a manner other than those described in Sections 9.17.1.1. and 9.17.1.2 and for excavations deeper than 20 feet.

Designs must be in written form and must include at least the following information:

- The maximum height and angle of the slopes that were determined to be safe for the excavation; and
- The identity of the registered P.E.(s) who approved the design.

Maintain at least one copy of the design at the jobsite while the slope is being constructed.

9.17.2 Design of Support, Shield, and Other Protective Systems

The Excavation Competent Person must select the design of support systems, shield systems, and other protective systems. The Excavation Competent Person must oversee the system construction in accordance with the requirements detailed in the following sections.

9.17.2.1 Designs Using 29 CFR 1926, Subpart P, Appendices A, C and D

- Design timber shoring in trenches in accordance with the requirements of the OSHA guidelines.
- Design aluminum hydraulic shoring in accordance with the manufacturer's tabulated data or the requirements of the OSHA guidelines.

9.17.2.2 Designs Using Manufacturer's Tabulated Data

- Construct and use support systems, shield systems, and other protective systems designed from the manufacturer's tabulated data in accordance with all specifications, recommendations, and limitations issued or made by the manufacturer.
- Allow deviation from the specifications, recommendations, and limitations issued or made by the manufacturer only after the manufacturer issues specific written approval.
- Retain documentation of the manufacturer's specifications, recommendations, and limitations, as well as the manufacturer's written approval to deviate from the specifications, recommendations, and limitations, at the jobsite during construction of the protective system(s).

9.17.2.3 Designs Using Other Tabulated Data

Select and construct designs of support systems, shield systems, and other protective systems in accordance with tabulated data, such as tables and charts.

The tabulated data must be in written form and include all the following:

- Factors that affect the selection of a protective system drawn from such data;
- Limits of the use of such data; and
- Information needed by the user to make a correct selection of a protective system from the data.

Maintain at least one written copy of the tabulated data, including the identification of the registered P.E. who approved the data, at the jobsite during construction of the protective system.

9.17.2.4 Design by a Registered Professional Engineer

A registered P.E. must approve support systems, shield systems, and other protective systems designed in a manner other than the preceding three options.

Designs must be in written form and include the following:

- A plan indicating the sizes, types, and configurations of the materials to be used in the protective system; and
- The identity of the registered P.E. who approved the design.

Maintain at least one copy of the design at the jobsite during construction of the protective system.

9.17.3 Materials and Equipment

The Excavation Competent Person must ensure that materials and equipment used for protective systems are free from damage or defects that might affect their proper function.

Use and maintain manufactured protective systems in accordance with the recommendations of the manufacturer and in a manner that will prevent employee exposure to hazards.

When materials or equipment used for protective systems are damaged, the Excavation Competent Person or the manufacturer's representative must perform an evaluation to determine if the materials or equipment is safe for continued use.

9.17.4 Installation and Removal of Supports

The Excavation Competent Person must ensure that the following controls are implemented.

- Securely connect members of support systems to prevent sliding, falling, kick-outs, or other potential hazards.
- Install and remove support systems in a manner that protects employees from cave-ins, structural collapses, or from being struck by members of the support systems.
- Do not subject individual members of the support systems to loads exceeding those they were designed to support.
- Begin and progress removal of support systems from the bottom of the excavation. Release members slowly. If there is any indication of possible failure of the remaining members of the structure or possible cave-in of the sides of the excavation, halt the work until it can be examined by the Excavation Competent Person.
- Conduct backfilling in conjunction with the removal of support systems from excavations.
- Allow excavation of material to a level no greater than 2 feet below the bottom of the members of a support system.
- Closely coordinate installation of a support system with the excavation of trenches.

9.17.5 Shield Systems

The Excavation Competent Person must ensure that the following hazard controls are implemented.

- Do not subject shield systems to loads greater than those they are designed to withstand.
- Install shields in a manner that will restrict lateral or other hazardous movement of the shield that could occur during cave-in or unexpected soil movement.
- Protect employees from the hazard of cave-ins when they are entering or exiting the areas protected by shields.
- Do not permit employees in trenches when shields are being installed, removed, or moved vertically.
- Allow excavation of material to a level no greater than 2 feet below the bottom of the shield system only if the system is designed to resist the forces calculated for the full depth of the trench.

9.18 TRAINING

The Excavation Competent Person must ensure that personnel involved in excavation work are trained on at least the following topics:

- Work practices that must be followed during excavating or working in excavations;
- Personal protective equipment that will be required during work in excavations and the procedures that will be followed if a hazardous atmosphere exists or could reasonably be expected to develop during work on an excavation; and
- Emergency and non-entry rescue methods as well as the procedure for calling rescue services.

The Excavation Competent Person must be trained and knowledgeable in the following:

- Basic soil science, including test methods for soil adhesion (visual and manual testing) and the various soil types encountered in construction (Soil Types A, B, C, and Layered);
- The use of soil classification tools (pocket penetrometer) to properly classify soil types and determine the optimum engineering controls to be used;
- Hazardous air test equipment and procedures, including those testing instruments specific to an HGL chemically contaminated site, and the basic interpretation of results;
- Proper usage of various trenching protective devices and methods (e.g., sloping, benching, shoring and trench boxes); and
- Locating and working safely around buried underground utilities.

10.0 DATA AND RECORDS MANAGEMENT

Documentation generated as a result of this procedure must be collected and maintained in accordance with the document control requirements of the HGL Quality Manual. Specifically, maintain training and designation records for Excavation Competent Persons in project records and include daily excavation and trenching inspection forms (HGL SOP 201.522.F01) and hazardous atmospheric trench testing records (if applicable) with the project records.

11.0 QUALITY CONTROL AND QUALITY ASSURANCE

The responsible manager or site safety and health officer must perform sufficient due diligence to verify that work meets requirements.

12.0 REFERENCES

29 CFR 1926.650 *Safety and Health Regulations for Construction, Subpart P, Excavations; General Requirements.*

29 CFR 1926.650 *Safety and Health Regulations for Construction, Subpart P, Excavations; Protection Systems.*

29 CFR 1926 *Appendices A-F; Safety and Health Regulations for Construction, Subpart P, Excavations; Design of Support, Shield, and Other Protective Systems.*

29 CFR 1926.501(b)(7)(i) *Duty to Have Fall Protection.*

EM 385-1-1 *USACE Health and Safety Requirements Manual, November 2014, Section 25, Excavation and Trenching.*

HGL SOP 411.03: *Subsurface Utility Avoidance*[2]

13.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
9	April 6, 2022	Initial CMS Library Version

ATTACHMENTS

None.

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[2] When updated, HGL SOP 411.03 will be renumbered as HGL SOP 401.519.

CORPORATE TECHNICAL PROCEDURE

Approved for issue by:

Process Owner	Steve Davis CIH, CSP Digitally signed by Steve Davis CIH, CSP Date: 2022.05.02 08:27:24 -04'00'
Corporate Quality Director	Theresa Rojas Digitally signed by Theresa Rojas Date: 2022.04.29 13:27:24 -04'00'

Drilling Safety

Document No.: HGL SOP 201.528
(formerly H&S Procedure 27)
Process Category: Health and Safety
Revision No.: 6
Effective Date: April 29, 2022
Last Review Date: April 29, 2022
Next Review Date: April 2023

1.0 PURPOSE AND APPLICABILITY

The purpose of this procedure is to minimize the safety risks of drilling and drilling-related work. Detailed requirements for safe drilling are presented in project-level health and safety documents such as site safety and health plans (SSHPs) and activity hazard analyses (AHAs). This procedure applies to any work in which HGL employees or temporary employees operate drill rigs or oversee the operation of subcontractors' drill rigs.

2.0 SUMMARY OF METHOD

This procedure requires at least the following actions:

- Pre-qualify drilling subcontractors to confirm safe operation on previous work.
- Follow the applicable HGL standard operating procedures discussed in the sections below.
- Ensure that health and safety documents such as SSHPs and AHAs are in place and followed.
- Collect and retain current documentation on hazardous waste safety training and medical clearance (as applicable) for drilling subcontractor personnel .
- Perform utility clearance of drilling locations before all drilling. See HGL SOP 411.03: *Subsurface Utility Avoidance*.[1]
- Ensure that rigs remain at least 20 feet from overhead uninsulated power transmission lines unless additional controls are in place.
- Exclude unauthorized and unnecessary personnel from the drilling area and specifically from the area where drilling tools, drill flights, and the drilling mast could fall.
- Ensure that rig operators inspect rigs before use and that they have a copy of the rig operator's manual.

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[1] When updated, HGL SOP 411.03 will be renumbered as HGL SOP 401.519.

- Oversee the work of drilling subcontractors to promote a safe working environment. HGL staff should not direct the work of subcontractor employees but should interact with their management.
- Require hearing protection unless specific testing of the rig(s) in use indicates that noise levels are less than 85 dBA.
- Require all personnel in the vicinity of operating rigs to wear safety glasses and hard hats.
- Minimize dust generation caused by grouting. Grout dust can pose a risk of hazardous silica exposure.
- Ensure that rigs with exposed rotating or moving parts that could cause injury are equipped with kill switches, also referred to as “deadman” switches. All personnel working near rigs that have kill switches must know where the switches are and how to operate them.
- Confirm that kill switches are functional.

3.0 DEFINITIONS

Activity Hazard Analysis (AHA) – See Job Safety Analysis.

Air Knife – A device that directs compressed air to advance a hole. Usually used in conjunction with a vacuum truck. Generally used to safely advance a borehole through depths where underground utilities may be present but have not been precisely located.

Auger/Auger Flight – Any of various tools or devices having a helical shaft or member that are used for boring holes.

Bit – The cutting or boring element used in drilling wells.

Boom Truck – A vehicle with a crane arm used for lifting augers, casing, or other heavy equipment.

Borehole – The hole drilled by the bit. A borehole may have casing in it, it may be open (uncased), or a portion of it may be cased and a portion of it may be open.

Casing – A tubular retaining structure that is installed in the well bore to maintain the well opening.

Clearance Techniques – Application of specialized equipment to detect the presence of buried structures.

Concrete Coring – The cutting of surface concrete so drilling can be conducted in the underlying soil. This may be done with circular drill bits or flat saws.

Cuttings – The fragments of rock and soil dislodged by the bit and brought to the surface in the drilling fluid or by the rotation of the auger.

Decontamination – The act of cleaning equipment to remove unwanted materials or chemicals. Commonly performed by pressure washing, steam cleaning, or hand scrubbing with soap and water.

Direct Push – A drilling technique that uses a percussion hammer or hydraulic ram to push or hammer various sampling tools into the subsurface. Geotechnical sampling, continuous soil sampling, in situ groundwater sampling, and small-diameter well installation can be performed with these units.

Drill Rod Chuck Jaws – Hydraulic-driven equipment that holds the drill stem stable as the drill is rotated and advanced.

Drill Stem (Drill Rods) – All members in the assembly used for drilling by the rotary method from the swivel to the bit, including the Kelly, drill pipe and tool joints, drill collars, stabilizers, and various subsequent items.

Drilling Fluid – Circulating fluid that forces cuttings out of the borehole and to the surface. A mixture of clay, water, and other chemical additives is the most common drilling fluid, but boreholes can also be drilled using air, gas, or water as the drilling fluid.

Drive Hammer – A hydraulically driven hammer that advances the casing as the drill bit advances.

Grouting – Filling the annulus between the casing and borehole with liquid slurry of grout (cement and/or bentonite) and water to support the casing and prevent fluid migration between permeable zones.

Hoisting Cables and Ropes – Ropes or metal cables that are used to lift or move augers, casing, and sampling tools. They are usually attached to sheaves on the mast.

Job Safety Analysis (JSA) – Also known as an Activity Hazard Analysis. A step-by-step breakdown of a task. With each step, the hazard risks and precautions are identified. A JSA is typically a three-column document in which the first column lists the operational steps of the job or activity, the second column lists the hazards that could be encountered at each step, and the third column lists the precautions to control the hazard. Usually included in, attached to, or referenced by the health and safety plan (HSP).

Kill Switch – A safety device, normally a button or toggle stick, that will turn off the drill rig engine when actuated. It is usually located in the back of the rig near where the drilling is conducted.

Mast – A movable derrick capable of being raised as a unit, as distinguished from a standard derrick, that cannot be raised to a working position as a unit.

Rotary Drilling – A drilling method in which a hole is made by a rotating bit to which a downward force is applied and cuttings are brought to the surface. The bit is fastened to and rotated by the drill stem, which also provides a passageway through which the drilling fluid is circulated. Additional joints of drill pipe are added as drilling progresses.

Sampling Tools – Tools that capture and retrieve subsurface soil samples such as a Shelby tube, sample extruder, or split barrel sampler.

Sheaves – A grooved pulley over which tape, wire, or cable rides.

Utility Mark-Outs – The surface marking of underground utilities and structures. There are public services (one call centers) that will mark out underground utilities on public rights-of-way and private locaters that will mark out utilities on private property.

Wellhead – Equipment installed at the surface of the borehole when a casing is installed in the borehole. A wellhead may include such equipment as the casing head and tubing head.

Wire Rope Hoist or ***Draw Works*** – An arrangement of pulleys and wire rope used for lifting heavy objects, a winch or similar device.

4.0 HEALTH AND SAFETY WARNINGS

Failure to follow this procedure may result in injury or property damage.

5.0 CAUTIONS

This section does not apply to this procedure.

6.0 INTERFERENCES

This section does not apply to this procedure.

7.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

Corporate officers, managers, and project-level staff should all be role models for safety. Other responsibilities are summarized below.

7.1 SENIOR MANAGEMENT (PRESIDENT, CHIEF OPERATING OFFICER, DIVISIONAL VICE PRESIDENTS)

- Maintain a general awareness of health and safety procedure.
- Provide adequate resources to perform work safely.

7.2 CORPORATE HEALTH AND SAFETY DIRECTOR

- Approve these requirements and subsequent revisions.
- Provide support and assistance in the implementation of these requirements.

7.3 OFFICE MANAGERS

- Maintain a general awareness of the applicable portions of these requirements.
- Maintain an awareness of any drilling-related work conducted by office personnel and ensure that adequate controls are in place.

7.4 OFFICE SAFETY AND HEALTH COORDINATORS

- Maintain a general awareness of the applicable portions of these requirements.
- Maintain an awareness of drilling work managed by the office.

7.5 SUPERVISORS AND PROJECT MANAGERS

- Maintain an awareness of the applicable portions of these requirements.
- Implement these requirements on work under their control.
- Ensure that drilling subcontractors are pre-qualified to confirm safe operation on previous work.

7.6 SITE SAFETY AND HEALTH OFFICERS

- Maintain an awareness of the applicable portions of these requirements.
- Lead the morning tailgate safety meeting and ensure that all site visitors check in, have the appropriate qualifications, and receive site-specific training before entering a work area.
- Verify that drill rigs are inspected before initial use and at regular intervals on subsequent days.
- Verify that drilling hazards are controlled and that the AHAs/JSAs are on site and reviewed by all parties before drilling begins.
- Verify that any drill rig kill switches are functional.
- Verify that personnel exposed to drilling hazards wear the required personal protective equipment (PPE).
- Ensure that HGL staff who could be exposed to drilling hazards are aware of the hazards and the necessary hazard control measures.

7.7 EMPLOYEES

- Complete required training.
- Maintain an awareness of the applicable portions of these requirements.
- Follow the applicable drilling requirements.
- Wear required PPE.
- Stop or question any work that may be unsafe.
- Notify supervisor of uncontrolled hazards or incidents.

8.0 EQUIPMENT AND SUPPLIES

Equipment and supplies associated with this procedure include various types of drilling equipment. This equipment is normally provided and operated by drilling subcontractors.

9.0 PROCEDURAL STEPS

The information presented in this section is necessarily general and must be supplemented by rig operators' manuals, SOPs, SSHPs, and AHAs/JSAs. General procedural steps include the following:

- Pre-qualify drilling subcontractors to confirm safe operation on previous work. HGL's procurement organization uses a screening process that examines subcontractors' safety history and identifies subcontractors that are more likely to perform safely. The project manager should participate in this process, particularly in cases where a subcontractor's prior history reveals problems.
- Follow applicable HGL SOPs. HGL has established SOPs that address direct-push sampling (SOP 403.04), hollow-stem auger drilling and sampling (SOP 403.05), monitoring well installation (SOP 406.02.), well development (SOP 406.01), and subsurface utility avoidance (SOP 411.03).[2] The applicable SOPs should be consulted before starting drilling work.
- Ensure that health and safety documents such as SSHPs and AHAs are in place and followed. In cases where HGL hires a drilling subcontractor, the project-level health and safety documents (APP, SSHP) should be prepared by HGL. The drilling AHA or JSA should be prepared by the drilling subcontractor.
- Require that drilling subcontractors meet training and other requirements for the site and tasks. If the work is subject to the Occupational Safety and Health Administration's hazardous waste standard, collect and retain documentation on current hazardous waste training and medical clearance for subcontractor personnel.

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[2] When updated, HGL SOPs 403.04, 403.05, 406.02, 406.01, and 411.03 will be renumbered as HGL SOPs 401.507, 401.508, 401.516, 401.515, and 401.519, respectively.

- Perform utility clearance of drilling locations before drilling. Contact public “one call” services or utility companies, hire a private utility locator if drilling on private property, use unexploded ordnance (UXO)-technician support if munitions may be present, review as-built drawings, walk the area to verify that utilities have been marked and to look for signs of unidentified utilities, and excavate the first 5 feet with low-impact methods, such as a hand auger or air knife, if there is uncertainty about the locations of utilities, especially if within 25 feet of known utilities. Post-hole diggers and breaker bars are not low-impact tools. See HGL SOP 411.03: *Subsurface Utility Avoidance* for additional information.[3]
- Execute HGL’s regulatory and contractual requirements to oversee the work of contractors, make them aware of safety requirements imposed by HGL or HGL’s client, and require them to correct any unsafe conditions or behaviors. HGL personnel should not direct the work of subcontractor employees but should implement HGL’s safety responsibilities through their management chain. The subcontractor, as the employer, is directly responsible for the safety of their personnel.
- Keep drill rigs and boom trucks away from overhead power lines. Maintain at least 20 feet of clearance unless the voltage is known. If the voltage is known, maintain at least the minimum distances listed below.

Voltage (nominal, kV, Alternating Current)	Minimum Clearance Distance
Up to 50	10 ft (3 m)
51– 00	15 ft (4.6 m)
201–350	20 ft (6 m)
351–500	25 ft (7.6 m)
501–750	35 ft (10.7 m)
751–1,000	45 ft (13.7 m)
Over 1,000	As established by the utility owner/operator or Registered Professional Engineer who is a Qualified Person with respect to electrical power transmission and distribution).

- Exclude unauthorized and unnecessary personnel from the drilling area, specifically from the area where drilling tools, drill stem sections, and the drilling mast could fall, and from where contaminated materials could be brought to the surface.
- Ensure that rig operators inspect rigs before use and regularly thereafter. Confirm that the inspection includes checking for frayed or broken strands in wire rope, damaged shackles or other connections, damaged or nonlocking hoisting hooks, leaking or damaged hydraulic lines, broken welds, and malfunctioning kill switches or “deadman” switches.
- Verify that the drill rig operator’s manual accompanies the drill rig.

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[3] When updated, HGL SOP 411.03 will be renumbered as HGL SOP 401.519.

- Require hearing protection for personnel near rigs unless specific testing of the rig(s) in use indicates that noise levels are less than 85 dBA. Most drill rigs expose nearby personnel to unsafe noise levels, and this exposure requires the use of hearing protection to prevent hearing loss.
- Require that all personnel in the vicinity of operating rigs wear safety glasses, hard hats, and safety-toed boots. Personnel near moving parts should not wear loose-fitting clothing or jewelry. Personnel who perform material handling should wear heavy-duty work gloves. If skin contact with contaminants is likely, chemical-resistant gloves should be worn (this may be in addition to work gloves).
- Minimize dust generation caused by grouting. Grout dust may pose a risk of hazardous silica exposure.
- Ensure that rigs with exposed rotating or moving parts that could cause injury are equipped with kill switches, also referred to as “deadman” switches. All personnel working near rigs that have kill switches must know where the switches are and how to operate them. Personnel should stay clear of moving parts to the extent possible.
- Confirm that kill switches are functional. This should be done without an operator at the controls to preclude any potential for deception.

10.0 DATA AND RECORDS MANAGEMENT

Documentation generated as a result of this procedure must be collected and maintained in accordance with document control requirements of the HGL Quality Manual.

11.0 QUALITY CONTROL AND QUALITY ASSURANCE

The responsible manager or site safety and health officer must perform sufficient due diligence to verify that work meets requirements.

12.0 REFERENCES

- 29 CFR 1910.120, *Hazardous Waste Operations and Emergency Response*
- HGL SOP 406.01: *Well Development*[4]
- HGL SOP 406.02: *Monitoring Well Installation*[5]
- HGL SOP 403.04: *Direct-Push Sampling*[6]
- HGL SOP 403.05: *Hollow-Stem Auger Drilling and Sampling*[7]

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[4] When updated, HGL SOP 406.01 will be renumbered as HGL SOP 401.515.

[5] When updated, HGL SOP 406.02 will be renumbered as HGL SOP 401.516.

[6] When updated, HGL SOP 403.04 will be renumbered as HGL SOP 401.507.

[7] When updated, HGL SOP 403.05 will be renumbered as HGL SOP 401.508.

- HGL SOP 411.03: *Subsurface Utility Avoidance*[8]
- Environmental Remediation Drilling Safety Guideline, 2008

13.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
6	April 29, 2022	Initial CMS Library Version

ATTACHMENTS

None.

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[8] When updated, HGL SOP 411.03 will be renumbered as HGL SOP 401.519.

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HGL HydroGeoLogic, Inc Exceeding Expectations	**CORPORATE TECHNICAL PROCEDURE**	
	Approved for issue by:	
	Process Owner	Steve Davis CIH, CSP Digitally signed by Steve Davis CIH, CSP Date: 2022.05.05 07:48:12 -04'00'
	Corporate Quality Director	Theresa Rojas Digitally signed by Theresa Rojas Date: 2022.05.04 23:21:10 -04'00'
Forklifts and Earthmoving Equipment		**Document No.: HGL SOP 201.531 (formerly H&S Procedure 30)**
		Process Category: Health and Safety
		Revision No.: 6
		Effective Date: May 3, 2022
		Last Review Date: May 3, 2022
		Next Review Date: May 2023

1.0 PURPOSE AND APPLICABILITY

The purpose of this procedure is to provide a standardized process for verifying and documenting operator competency and to provide the guidance necessary for HydroGeoLogic, Inc. (HGL) staff to operate and conduct routine maintenance such as greasing equipment, cleaning filters, and fueling forklifts (powered industrial trucks), excavators, front-end loaders, skid-steers, backhoes, large tractors, and similar powered earthmoving equipment without injury to themselves or others. This procedure also provides guidance that should be used in support of HGL's oversight of subcontractors that operate such equipment.

This procedure applies to all HGL employees or temporary employees who operate forklifts or earthmoving equipment such as excavators, front-end loaders, skid-steers, and similar powered equipment that may be used for material handling, excavation, earthmoving, clearing and grubbing, and similar tasks. This procedure does not apply to the use of mobile cranes or similar specialized load-handling equipment. These requirements do not apply directly to subcontractors, but subcontractors performing work on HGL jobs are expected to implement equivalent practices and to comply with any applicable site- or project-specific requirements.

2.0 SUMMARY OF METHOD

Unsafe operation of forklifts (powered industrial trucks), and earthmoving equipment can result in fatalities, serious injuries, and property damage. To minimize the probability of such negative events, this procedure requires at least the following actions:

- Ensure that operators have received documented operator training and that this training is refreshed or reevaluated every 3 years. Training documentation should be maintained in the workplace and carried by operators. Training documentation for HGL personnel should also be uploaded to HGL's online training and certifications database.
- Ensure that operators are authorized in writing by the responsible manager using the authorization form in HGL SOP 201.531.F01 or equivalent. When subcontractors operate equipment, authorization must be issued by the operator's employer.
- Confirm and retain documentation that subcontractor equipment operators are properly qualified and are authorized in writing by their employer.

- Assess site and task hazards and plan effective hazard controls. Document assessments and hazard controls in Activity Hazard Analyses or similar documents.
- Inspect equipment daily and document the inspections on the checklist in HGL SOP 201.531.F02, or an equivalent form, such as a U.S. Army Corps of Engineers (USACE) earthmoving equipment inspection checklist.
- Perform routine maintenance (grease pins and bushings, clear air and exhaust filters of dust and debris) daily or as recommended by the operator's manual and remove flammable debris from any area where it may be exposed to high temperatures.
- Ensure that equipment has functional backup alarms.
- Ensure that operators use safety restraints.

3.0 DEFINITIONS

Attachments – Components that replace or are added to the original forks of a forklift or other earthmoving equipment. Some attachments include side shifters, slip-sheet attachments, carton clamps, multipurpose clamps, rotators, fork positioners, carpet poles, pole handlers, container handlers, and roll clamps. Any attachment on a forklift will reduce its nominal load rating, which is computed with a stock fork carriage and forks. The actual load rating may be significantly lower.

- ***Drum Handler Attachment*** – A mechanical attachment that slides onto the tines (forks). It usually has a spring-loaded jaw that grips the top lip-edge of a drum for transport. Another type grabs around the drum in a manner similar to the roll or barrel attachments.
- ***Fork Positioner*** – A hydraulic attachment that moves the tines (forks) together or apart. This removes the need for the operator to manually adjust the tines for different sized loads.
- ***Roll and Barrel Clamp Attachment*** – A mechanical or hydraulic attachment used to squeeze the item to be moved. It is used for handling barrels, kegs, or paper rolls. This type of attachment may also have a rotate function.
- ***Rotator*** – A forklift attachment that allows the tines to be rotated to aid the handling of skids that may have become excessively tilted and other specialty material handling needs.
- ***Side Shifter*** – A hydraulic attachment that allows the operator to move the tines (forks) and backrest of a fork lift laterally. This enables easier placement of a load without having to reposition the truck.

Carriage – Component to which the forks or other attachments mount. It is mounted into and moves up and down the mast rails by means of chains or by being directly attached to the hydraulic cylinder. Like the mast, the carriage may have either rollers or bushings to guide it in the interlocking mast rails.

Counterweight – Hangs off the rear of the equipment to provide lifting capacity and balance the weight of lifted loads. There is typically a blind spot immediately behind the counterweight.

Equipment – For the purposes of this procedure, this includes forklifts and earthmoving equipment such as excavators, bulldozers, front-end loaders, and skid-steers.

Mast – The vertical assembly that does the work of raising and lowering the load. It is made up of interlocking rails that also provide lateral stability. The interlocking rails may either have rollers or bushings as guides. The mast is driven hydraulically and operated by one or more hydraulic cylinders directly or using chains from the cylinders.

Overhead Guard – A metal roof supported by posts at each corner of the cab that helps protect the operator from any falling objects. On some equipment, the overhead guard is an integrated part of the frame assembly.

4.0 HEALTH AND SAFETY WARNINGS

Failure to implement these requirements could result in injuries, fatalities, and property damage.

5.0 CAUTIONS

This section does not apply to this procedure.

6.0 INTERFERENCES

This section does not apply to this procedure.

7.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

Corporate officers, managers, and project-level staff should all be role models for safety. Other responsibilities are summarized below.

7.1 SENIOR MANAGEMENT (PRESIDENT, CHIEF OPERATING OFFICER, DIVISIONAL VICE PRESIDENTS)

- Maintain a general awareness of health and safety procedure.

7.2 PROVIDE ADEQUATE RESOURCES TO PERFORM WORK SAFELY. CORPORATE HEALTH AND SAFETY DIRECTOR

- Approve these requirements and subsequent revisions.
- Provide support and assistance in the implementation of these requirements.
- Review and approve the Operator Training program(s).

7.3 OFFICE MANAGERS

- Maintain a general awareness of the applicable portions of these requirements.
- Maintain an awareness of forklift and earthmoving equipment operation associated with the office and provide support, as appropriate, to minimize risks.

7.4 OFFICE HEALTH AND SAFETY COORDINATORS

- Maintain a general awareness of the applicable portions of these requirements.
- Conduct assessments of forklift and earthmoving equipment operation associated with the office and confirm that operators are trained and operating equipment safely.

7.5 SUPERVISORS AND PROJECT MANAGERS

- Maintain an awareness of the applicable portions of these requirements.
- Implement these requirements on work under their control.
- Ensure that equipment operators are trained and operate equipment safely.
- Ensure that equipment operators read the operators' manuals for equipment they operate.
- Observe equipment operation by new or newly trained operators to confirm and document familiarity and competence with the equipment.
- Authorize operators in writing using HGL SOP 201.531.F01.

7.6 DIRECTOR OF CONSTRUCTION SERVICES

- Maintain an awareness of the applicable portions of these requirements.
- Participate in the process of authorizing HGL equipment operators by reviewing supervisors' draft operator authorizations and concurring with the authorizations or conducting additional evaluations, as appropriate.

7.7 SITE SAFETY AND HEALTH OFFICERS

- Maintain an awareness of the applicable portions of these requirements.
- Lead the morning tailgate safety meeting and ensure that all site visitors check in, have the appropriate qualifications, and receive site-specific training before entering a work area.
- Include discussion of equipment hazards and hazard controls in site-specific training when such equipment is in use.
- Include assessment of equipment operation in routine inspections when equipment is in use.

- Stop work or implement effective controls if equipment operation is unsafe.
- Collect and retain documentation of equipment training and inspections.
- Report safety problems to management.

7.8 EMPLOYEES

- Complete required training as specified in Section 9.1.
- Stop or question any work that may be unsafe, such as speeding or operation on dangerous terrain.
- Operate equipment as specified by these requirements.
- Do not operate equipment unless trained and authorized, in writing, to do so.
- Read and understand the operator’s manual for all equipment that the individual operates.
- Report unsafe conditions or operation to the appropriate health and safety representative or to the appropriate manager.

8.0 EQUIPMENT AND SUPPLIES

Equipment and supplies associated with this procedure include fork lifts, excavators, bulldozers, and similar earthmoving equipment.

9.0 PROCEDURAL STEPS

9.1 OPERATOR TRAINING

Operators must have received documented operator training, and this training must be refreshed or reevaluated every 3 years. Training consists of a combination of formal instruction, practical training (demonstrations performed by the trainer and practical exercises performed by the trainee), and evaluation of the operator’s performance in the workplace. This training can be presented by a training vendor, by an equipment rental company, or by an appropriately qualified supervisor. In some cases, prior training and/or experience will be accepted.

9.1.1 Initial Training

Initial operator training must cover the following topics:

- How to read the operator’s manual;
- Operating instructions, warnings, and precautions for the types of equipment the operator will be authorized to operate;
- Differences between the equipment and on-road vehicles such as cars and trucks;

- Controls and instrumentation: where they are located, what they do, and how they work;
- Engine or motor operation;
- Steering, safe speeds, and maneuvering;
- Visibility (including restrictions due to loading and backing);
- Fork and other attachment adjustment, operation, and use limitations;
- Types of attachments: bucket, side shifter, rotator, roll and barrel clamp, drum handler, mower, etc.;
- Vehicle capacity;
- Vehicle stability;
- Any vehicle inspection and maintenance that the operator will be required to perform;
- Refueling and/or charging and recharging of batteries;
- Operating limitations; and
- Any other operating instructions, warnings, or precautions listed in the operator's manual for the types of equipment that the employee is being trained to operate.
- Safe operation of the equipment under direct observation of the trainer or direct supervisor.
- Written authorization using HGL SOP 201.531.F01, signed by the direct supervisor and the Director of Construction Services.

9.1.2 Site-Specific Training

Site-specific operator training must cover the following topics:

- Surface conditions where the vehicle will be operated and conditions that should be avoided;
- Safe lifting capacity of the equipment to be used, the composition and weight of loads to be carried, and load stability;
- Load manipulation, stacking, and unstacking;
- Pedestrian traffic in areas where the equipment will be operated;
- Narrow aisles or roads and other restricted places where the vehicle will be operated;
- Hazardous locations where the vehicle will be operated;
- Uneven ground, ramps, and other sloped surfaces that could affect the vehicle's stability;

- Closed environments and other areas where insufficient ventilation or poor vehicle maintenance could cause a buildup of carbon monoxide or diesel exhaust; and
- Other unique or potentially hazardous environmental conditions in the workplace that could affect safe operation.

9.1.3 Refresher Training and Evaluation

Each operator must be retrained or be reevaluated at least once every 3 years to ensure that the operator still has the knowledge and skills needed to operate the equipment safely. Retraining and reevaluation must be documented, and documentation should be uploaded to HGL's online training and certifications database.

In addition to periodic retraining and reevaluation, refresher training in relevant topics must be provided to the operator when:

- The operator has been observed to operate the vehicle in an unsafe manner;
- The operator has been involved in an accident or near-miss incident;
- The operator has received an evaluation that reveals that the operator is not operating the equipment safely;
- The operator is assigned to drive a different type of equipment; and/or
- A condition in the workplace changes in a manner that could affect safe operation of the equipment.

9.1.4 Recognition of Prior Training or Experience

Employees who have prior operating experience or documented operator training can be authorized to operate equipment if the prior training and/or experience is deemed to be acceptable. The process for this authorization is described below.

1. The employee submits documentation of prior training and/or experience to the responsible manager for review. Documentation of prior experience must include a signed statement that indicates the type(s) of equipment, the period(s) of use, the types of activities, and/or the date of training and topics covered.
2. The submitted information is reviewed by the responsible manager, with assistance from the Corporate Health and Safety Director, as needed. If the employee provides acceptable documentation, the responsible manager authorizes the employee to demonstrate use of the equipment to be operated.
3. The responsible manager observes the employee while operating the equipment to assess the employee's competency with the equipment. If the employee's operation is acceptable, the responsible manager documents the successful assessment and submits the draft authorization to the Director of Construction Services for review and final

approval. The written authorization must specify the type(s) of equipment that the employee is authorized to operate. HGL SOP 201.531.F01 can be used to document the evaluation and authorization.

9.2 STANDARD OPERATING PRACTICES

The following standard operating practices must be implemented when HGL personnel operate forklifts or earthmoving equipment.

- All HGL operators must be authorized, in writing, by the responsible manager and the Director of Construction Services using HGL SOP 201.531.F01 or equivalent. Authorization of subcontractor personnel must be issued by the operator’s employer.
- Assess site and task hazards and prescribe hazard controls before initiating work. Document assessments in Activity Hazard Analyses or similar documents. These assessments must include the type and characteristics of the equipment, the attachments to be used (forks, buckets, blades, mowers, etc.), the terrain and conditions of operation, and the loads or materials to be moved or transported. The hazards and controls must be communicated to operators and to personnel who will be near the operating equipment.
- Ensure that loads are within the rated capacity of the equipment.
- Inspect equipment upon delivery or before first use and before each day’s use thereafter and conduct routine maintenance (grease pins and bushings, clear air and exhaust filters of dust and debris). Verify that no flammable debris is in the engine compartment or other areas where it could be ignited. Verify that brakes, overhead guard, controls, gauges, lights, seat belts, back up alarm, and routine operational features are in proper working order. Correct identified defects or take the equipment out of service. Document these inspections using the HGL Forklift Safety Inspection Checklist (HGL SOP 201.531.F02) or similar document.
- Use safety restraints any time the engine is running (including during non-moving lifting operations).
- Do not allow personnel under elevated loads, forks, or any attachments.
- Do not allow personnel within the swing radius of operating equipment.
- Do not allow passengers to ride on equipment unless there is designed seating with safety restraints.
- Do not use cell phones or other distracting devices while operating equipment.
- Lower forks or attachments (de-energize) when equipment is left unattended.
- Do not transport loads with forks or bucket more than 8 inches off the ground or floor surface.

- Do not modify a forklift or other equipment or replace factory-installed attachments unless the changes are assessed and can be confirmed to be safe.

10.0 DATA AND RECORDS MANAGEMENT

Documentation generated as a result of this procedure must be collected and maintained in accordance with document control requirements of the HGL Quality Manual. Activity Hazard Analyses, training records, and inspection records are retained as part of project documentation. Training records must include employee name, date of training, date of evaluation, and the trainer's name. Records of formal training should be uploaded to the corporate Training and Certification database.

11.0 QUALITY CONTROL AND QUALITY ASSURANCE

The responsible manager or site safety and health officer must perform sufficient due diligence to verify that work meets requirements.

12.0 REFERENCES

- 29 CFR 1910.178, *Powered Industrial Trucks.*
- 29 CFR 1926, *Subpart O, Motor Vehicles, Mechanized Equipment, and Marine Operations:* https://www.osha.gov/laws-regs/regulations/standardnumber/1926/1926SubpartO.
- HGL SOP 201.531.F01: *Authorization for Operation of Forklifts or Earthmoving Equipment by HGL Personnel.*
- HGL SOP 201.531.F02: *HGL Equipment Safety Inspection Checklist.*
- OSHA. *Powered Industrial Trucks eTool:* https://www.osha.gov/SLTC/etools/pit/.
- OSHA. *Safety and Health Topics; Powered Industrial Trucks:* https://www.osha.gov/SLTC/poweredindustrialtrucks/index.html.
- U.S. Army Corps of Engineers (USACE). EM 385-1-1: *USACE Safety and Health Requirements Manual, Section 18, Vehicles, Machinery and Equipment*.

13.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
6	May 3, 2022	Initial CMS Library Version

14.0 ATTACHMENTS

None.

HGL HydroGeoLogic, Inc Exceeding Expectations	**STANDARD OPERATING PROCEDURE** Approved by: Digitally signed by Rojas, Theresa Date: 2020.12.21 16:13:34 -05'00' Corporate Quality Director
Environmental Data Quality Control	**SOP No.: 300.07 (formerly 303.01)** **SOP Category: QA/QC** **Revision No.: 3** **Revision Date: December 21, 2020** **Review Date: December 2022**

1.0 PURPOSE AND APPLICABILITY

This standard operating procedure (SOP) describes quality control (QC) steps associated with the processes of entering, updating, maintaining, reproducing, delivering, and archiving data from an environmental project database. The purpose of this SOP is to provide guidance to ensure that the electronic data in databases is complete, correct, and ready for use during a project or in a deliverable. Other SOPs address the QC associated with the actual data itself, such as the review and validation of analytical data generated from the laboratory analysis of environmental media (HGL SOP No. 300.06) and the management and archiving of electronic files and records (HGL SOP No. 100.01).

This SOP applies to environmental projects for which data is stored and managed in electronic form in a project database. The procedures apply to multiple types of data, including laboratory analytical data, field-recorded data, sample location (survey) data, screening criteria, and performance criteria.

Contract requirements and/or client directives may override the procedures specified here. Deviations from this SOP must be documented in the project's quality assurance project plan or quality control plan.

2.0 SUMMARY OF METHOD

The procedures rely on a two-step QC process whenever data is entered into, modified, or extracted from a project database. An Originator performs the initial action, which could include uploading data into the project database. An independent Reviewer conducts a QC review of the Originator's work. This process is followed throughout the entire data life cycle from entry into a database through analysis, extraction, and use of the data in project deliverables (for example, report tables).

3.0 DEFINITIONS

Database: A database is any software program used to store and maintain electronic project data. Examples include general purpose software such Microsoft Access or Microsoft Excel or specialized software for managing environmental data such as EQuIS™ or gINT®.

Database Manager: The person responsible for maintaining the database and performing other functions, both routine (for example, posting data for use by project staff) and unscheduled (for example, correcting data found erroneous during other QC reviews), is the Database Manager.

Draft Copy: A draft copy is a hard copy record that is printed and provided to the reviewer for verification.

Electronic Record: Electronic records include any document or data that exists as an electronic file.

Field Data Record: Field data records are field-generated documents including logbooks, exhibits, and forms extracted from HGL SOPs or site-specific project planning documents.

Hard Copy Record: A hard copy record is a document delivered in paper form or filled out by hand.

Original Data Source: Original data sources contain the data values to be entered into the database. These can include laboratory data deliverables for analytical data or field notebooks/data sheets for field measured data. If the data is obtained from a previous study, the original data collected for that study should be used whenever possible rather than relying on reports derived from that data.

Originator: The person who performs the data entry is considered the Originator.

Reviewer: The person who performs the QC review of the Originator’s work is the Reviewer in accordance with contract requirements, project documents, and/or SOPs such as HGL’s Data Validators.

4.0 PERSONNEL QUALIFICATIONS

The Originator must be familiar with environmental data collection and analysis methods, parameters, and terminology through training and experience.

The Reviewer must be familiar with environmental data collection and analysis methods, parameters, and terminology through training and experience.

The Database Manager must be experienced with using environmental database software and with creating and maintaining project-specific databases.

5.0 EQUIPMENT AND SUPPLIES

Not applicable.

6.0 SAFETY

There are no particular safety hazards or requirements for this procedure.

7.0 PROCEDURAL STEPS

Data management QC procedures comprise four categories of data management: (1) automated data entry, (2) manual data entry, (3) modifications to existing electronic data, and (4) extractions of data from a database for use in technical analyses or reports or for delivery to the customer.

(1) Automated data entry processes include the use of data import functions for loading data that is already in electronic form into a database.

(2) Manual data entry means keyboard data entry of values into a database.

(3) Modifications to existing electronic data include the use of automated or manual procedures to modify values in the database (for example, manually updating analytical data qualifiers or using a macro to modify data).

(4) Extractions of data from a database include manual copying of values, but extractions are usually performed using automated procedures, such as export functions, database queries, and/or database reporting services.

Unless specified otherwise in contract or project documents, the following frequency of data QC is used depending on the method of data entry:

Method	QC Frequency
Automatic Data Entry, Modification, or Extraction	10%
Manual Data Entry, Modification, or Extraction	100%

7.1 DATA QUALITY CONTROL REVIEW

For those projects where changes are made directly in the database, such as the FUDSChem database, the database must be able to maintain an audit trail. Changes are reviewed by a second person before the data is released for general use.

A QC review of data can also be performed by reviewing either a hard copy printout of the data or reviewing the data in electronic form such as Excel worksheets.

Hard copy data QC is performed as follows:

- After the data has been entered, modified, or exported, the Originator provides a printout of the data, referred to as the Draft Copy, to the Reviewer.
- The Reviewer checks the Draft Copy against the original data source document.
- Data entries verified as correct and acceptable for use are marked as reviewed by highlighting, placing a checkmark by the data or using another acceptable manner to bring this to the attention of the next reviewer.

- Corrections to the Draft Copy printout are marked in ink by drawing a single line through the incorrect value. The correction is written to the side of the original entry.
- If errors are encountered during a 10 percent QC check, the Reviewer must check another 10 percent of the data. If additional errors are found, this process is repeated until no errors are found or all the data has been reviewed.
- Upon completion of the hard copy data review, the Reviewer initials and dates the Draft Copy printout and identifies the level of QC that was performed (for example, 100 percent QC or 10 percent QC).
- The Reviewer returns the Draft Copy to the Originator, who verifies the edits and provides the corrections to the Database Manager. The Database Manager incorporates the corrections into the project database.

Electronic data QC using Excel is performed as follows:

- The Originator provides an electronic copy of the data in an Excel worksheet to the reviewer.
- The Reviewer checks the data against the original data source document.
- Corrections are marked by changing the font color, highlighting them, or using another acceptable manner to bring the corrections to the attention of the next reviewer. Any changes should be documented and transmitted to the Originator, with a copy saved in the hard copy or electronic version of the project file.
- Upon completion of the review, the Reviewer saves the verified electronic file with his/her initials appended to the file name and the level of QC that was performed (for example, “Brandywine_EMI_100QC_LJ”).
- The Originator verifies any edits made by the Reviewer and provides the corrections to the Database Manager. The Database Manager incorporates the corrections into the project database.

Corrections to the database are made as follows:

- If the QC processes described above identify discrepancies between data in the project database versus data in the original source document, the Database Manager and the Originator must identify the cause of and correct the errors.
- If the error was caused by automated data processes, the Database Manager (1) corrects the coding of the automated data process and (2) notifies the Project Managers of any affected projects to determine the need for additional data QC.
- Updates and corrections to the project database are made by the Database Manager and verified by the Reviewer.

7.2 DATA USED FOR FURTHER ANALYSIS OR INTERPRETATION

Any data used for further analysis or data interpretation (for example, risk assessment, modeling, engineering design) should be verified by the end user for completeness and accuracy before each use. The appropriate QC review will vary based on the end use. Examples of the types of review that may be performed include the following:

- Ensure that all required data is included and that no “extra” or unwanted data are present.
- Verify that the data meet the required data quality objectives for the intended use. For example, data that is acceptable for use in determining a contaminant source area may not meet the validation requirements for a risk assessment.
- Verify the number of reported analytes per method.
- Review the reported units for consistency.
- Ensure that data are reasonable based on historical data or familiarity with site conditions.

If the same data is used in successive steps of an analysis, but is re-ordered, reformatted, converted to different units, or otherwise modified, 10 percent QC checks of that data against the original data should be performed because these modifications could introduce unintended changes.

8.0 INTERFERENCES

Not applicable.

9.0 DATA AND RECORDS MANAGEMENT

A record of all changes to data and records should be maintained in electronic or in hard copy form. Completion of each instance of data QC (for example, initial database entry, database modification, data use review) must be documented. This documentation is kept in the project file and updated each time a data QC is completed to provide a cumulative record that data used and/or presented in HGL deliverables has been subjected to appropriate QC review.

All hard copy or electronic records of the data QC review process must be provided to the Project Manager or designee for inclusion in the project file. These records are retained until the Project Manager has determined that these records can be discarded, subject to HGL’s document retention policies and applicable contract requirements. Under no circumstances can these records be discarded before the completion of the project.

10.0 QUALITY ASSURANCE AND QUALITY CONTROL

See Section 7.0.

11.0 REVISION HISTORY

Revision 0	April 2014	Initial Release
Revision 1	December 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	March 8, 2018	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 3	December 21, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 303.01 to 300.07 and changing the title from "Environmental Database Quality Control" to "Environmental Data Quality Control."

HGL HydroGeoLogic, Inc Exceeding Expectations	**CORPORATE TECHNICAL PROCEDURE**	
	Approved for issue by:	
	Process Owner	Jodie Johnson Digitally signed by Jodie Johnson Date: 2022.03.23 08:46:13 -07'00'
	Corporate Quality Director	Theresa Rojas Digitally signed by Theresa Rojas Date: 2022.03.23 09:12:40 -04'00'
Field Logbook Use and Maintenance		**Document No.: HGL SOP 401.501 (formerly 300.04)**
		Process Category: Services
		Revision No.: 4
		Effective Date: March 21, 2022
		Last Review Date: March 21, 2022
		Next Review Date: March 2024

1.0 PURPOSE AND APPLICABILITY

This standard operating procedure (SOP) describes the minimum requirements and procedures for the proper documentation of information in field logbooks. This procedure outlines methods, lists examples for proper data entry into a field logbook, and provides the standardized HGL format. The field logbook is the primary means for recording field activities and pertinent observations, measurements, and calculations during a project. The logbook serves as the foundation for all field data collected that will be used to evaluate the project site. Field logbooks should provide sufficient detail to demonstrate compliance with project plans and serve as evidentiary documentation during legal proceedings, if needed. Documentation must be accurate, thorough, and complete so that field activities can be reconstructed to confirm that client, regulatory, contract, and work plan requirements are met.

2.0 SCOPE AND APPLICATIONS

This procedure provides guidance for logbook use and maintenance during routine field operations on environmental projects. Applicable regulatory and client requirements should be considered when documenting field activities in logbooks. Any deviations from the methods presented herein must be approved by the assigned HGL project manager and the HGL project quality assurance/quality control officer. Project-specific requirements for field documentation typically should be provided in project planning documents.

3.0 GENERAL REQUIREMENTS

The field logbook is the primary means of documenting field activities. Logbook entries must be completed concurrent with the associated field activity and present a thorough but concise summary of the activity. All project work must be performed in accordance with the project-specific planning documents.

Any deviations from specified project requirements or work plans that occur while in the field must immediately be reported to the project manager and documented in the field logbook. If such deviations are intended for field implementation, they must be approved by the project manager and/or the relevant program manager prior to implementation, and the approval must be documented in the logbook (refer to change or variance documentation requirements in the planning documents). Deviations from requirements are documented sufficiently to re-create the modified process and/or product and associated approvals.

All field personnel present on site to conduct work related to environmental projects are responsible for documenting field activities in logbooks. If field personnel are working in teams, one team member should be assigned to document the work performed in a logbook. Documentation in logbooks must be legible, accurate, and organized. Logbooks must be maintained over the course of the project in accordance with this SOP.

In addition to logbook entries, the HGL field team leader, or approved designee, typically prepares daily logs of field activities to provide clients records of the work completed, significant events and observations, and measurements taken in the field. These daily logs rely on documentation from the logbooks. Therefore, information presented in the logbook and daily logs should match.

The HGL field team leader, or approved designee, should review logbook entries at the end of each workday to ensure that they are complete/adequate. Any deficiencies observed in the logbook and the required corrective measures should immediately be communicated. Regular review of logbooks ensures that field activities are being documented properly and establishes clear expectations for documented information. Logbook entries should be reviewed on a regular basis by the project manager or an approved designee to verify that they have been completed in accordance with this SOP.

4.0 PROCEDURE

4.1 INTRODUCTION

Field logbooks provide a means for recording and documenting observations and field activities at a site. Field logbooks are intended to provide sufficient data and observation notes to enable participants to reconstruct events that occurred while performing field activities and to refresh the memory of field personnel when drafting reports or giving testimony during legal proceedings. As such, all entries must be as factual, detailed, and as descriptive as possible so that a particular situation can be reconstructed without reliance on the memory of field crews. Field logbooks are not intended to be used as the sole source of project or sampling information. A sufficient number of logbooks are to be assigned to a project to ensure that each field team has a logbook at all times.

4.2 FIELD LOGBOOK IDENTIFICATION

Field logbooks are bound books with consecutively prenumbered pages (preferably waterproof) that cannot be removed from the binding. Field logbooks should be dedicated to the project and appropriately labeled. Logbooks are permanently assigned to a project for the duration of the contract. When not in use, the field logbooks are to be stored in site project files. If site activities stop for an extended period (2 weeks or more), field logbooks must be stored in the project files in the appropriate HGL office. The field logbooks are to be scanned on a regular basis, grouped in files by date of the field event, and stored electronically in the proper project file on SharePoint.

The following information will be clearly written on the cover of the logbook:

- Organization to which the book is assigned (HGL),
- Site name, location, and identification (ID) number,
- Project name and ID number,
- Sequential logbook number (if multiple logbooks are used on the project), and
- Start and end dates of the information contained within the logbook.

Contact information should be recorded inside the front cover in case the logbook is misplaced. The following list provides examples of useful and pertinent information that may be recorded inside the front cover (optional).

- Project contract number,
- Project manager's name and contact information,
- Serial numbers and model numbers for equipment that will be used for the project duration,
- Formulas, constants, and example calculations, and
- Other useful telephone numbers and contact information.

4.3 LOGBOOK ENTRY PROCEDURES

Each daily logbook entry should start on a new page. All entries in logbooks must be made using indelible blue or black ink. No erasures or deletions from the logbook are permitted. If an incorrect entry or error is made, the data is crossed out with a single line and then initialed and dated by the originator. Under no circumstances may the incorrect entry be erased, made illegible, or obscured so that it cannot be read. A chronological record of the daily field activities conducted should be recorded in the logbook and signed by the field personnel at the end of the daily entry. All relevant information is recorded in the logbook at the time it occurred. Time (in military or 24-hour format) is recorded next to each entry. The site name, project name, and date are included at the top of each page. No pages or spaces are left blank. At the end of each day, a diagonal line is drawn through the remaining space on the page, and the line is signed and dated.

Logbook entries should be objective, factual, clear, and concise. Entries into the logbook may contain a variety of information and will vary from project to project; however, the format, concept, and general information that will be recorded are similar. Appropriate header information must be documented on the first page of each daily entry into the logbook. At a minimum, the following information must be recorded on the first page of the logbook entry for each day:

- Date (on all pages),
- Site name, site location, project name, and project number,
- Purpose/objective of the field event and brief description of the current task or activity,
- Weather (i.e., temperature, cloud cover, humidity, wind speed and direction) at the start of day and projected for the day. Changes during the day should be documented at the time of the change,
- Names and company/agency affiliation of all field personnel, subcontractors, and visitors,
    - Include initials for relevant field personnel to reference them by initials within the logbook to streamline note taking,
- Make, model, and quantity of all HGL and subcontractor equipment on site,
- Level of personal protective equipment being used on the site, and
- Arrival and departure times.

In addition, information recorded in the field logbooks during investigation, data collection, or sampling events includes, but is not limited to, the following:

- Documentation of safety meetings (e.g., daily tailgate);
- Sample description including sample IDs, collection time and date, analytical parameters, methods and type of laboratory analyses, depth interval, volume, type and number of containers, preservative, media sampled, sample collection method (e.g., low-flow sampling), and type of sampling equipment (e.g., peristaltic pump and low-density polyethylene tubing);
- Information on field quality control samples (e.g., field duplicates, trip blanks, equipment rinsates, field blanks, and matrix spike/matrix spike duplicates [MS/MSDs]) including collection time, date, and the associated parent sample ID;
- Sample courier airbill numbers and the associated quantity of sample coolers and chains of custody numbers;
- Observations about the site and samples (e.g., odors, appearances);
- Information about any activities, extraneous to sampling activities, that could affect the integrity of the samples;
- Equipment decontamination time(s) and method(s);
- Any public involvement, visitors, or press interest, comments, or questions; as well as times present on site;
- Make and model of equipment used on site including time and date of calibration along with the calibration standard lot numbers and expiration dates, and calibration results;

- Background levels of each instrument and possible background interferences;
- Air monitoring equipment readings (e.g., breathing zone, monitoring wells, soil cuttings, specified depth intervals of soil cores);
- Verification of subsurface utility clearance (e.g., dig permits number, state one-call ticket numbers);
- Field parameters such as pH and specific conductivity as required by the sampling method and planning documents;
- Unusual observances, irregularities, or problems noted on site or with equipment used;
- Description of any deviations from the work plan or changes in the scope of work and reason(s) why;
- A photographic log that lists subject, person taking photograph, distance to subject, direction, time, photograph number, and noteworthy items for each photograph stating what feature/item the photo is documenting;
- Subcontractor progress and/or any problems encountered;
- A description of the investigation-derived waste, the quantity generated, the type of container, and the storage location;
- Numbers/titles of forms used during sampling and any information contained therein (Note that a form does not take the place of the field logbook.); and
- Upon completion of a field event, a clear entry indicating that the event has been completed (e.g., “event complete,” “end of shift,” “field team demobilized”).

Entries are be organized into easily understandable tables if possible. A sample format is shown in Attachment 1. A Logbook Quick Guide, which provides logbook entry requirements and suggestions, is included as Attachment 2. Logbooks can become contaminated when used in the field. The field team should make every effort to avoid contaminating the logbook. Logbooks can be kept in seal-top poly bags or protected with temporary plastic covers.

4.4 REVIEW

The assigned field team leader, or an approved designee, checks field logbooks for completeness and accuracy on an appropriate site-specific schedule determined by the project leader. Any discrepancies in the logbooks are noted and returned to the originator for correction. The originator or other field team member knowledgeable about the field task reviews the comments, makes appropriate revisions, and signs and dates them. The reviewer verifies that revisions have been made before placing the logbook photocopies on the project file in SharePoint.

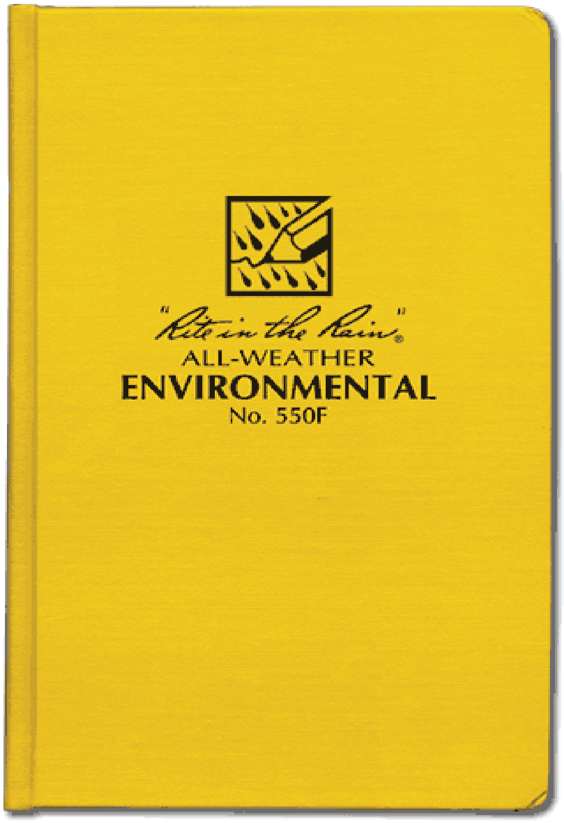
5.0 REVISION HISTORY

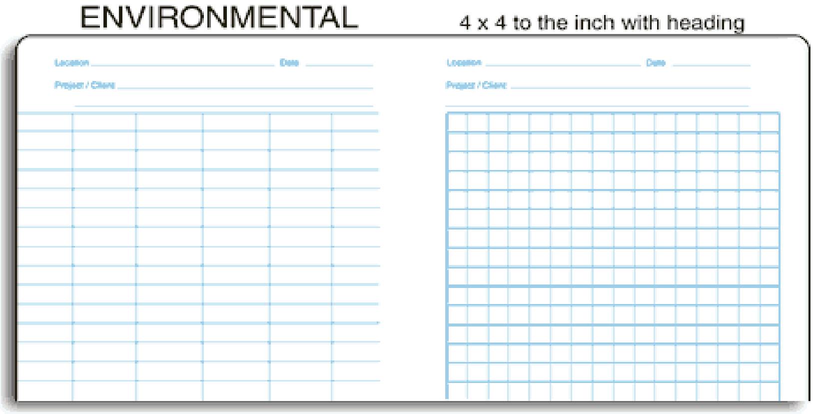
Revision Number	Revision Date	Reasons for Revision
4	March 21, 2022	Initial CMS Library Version

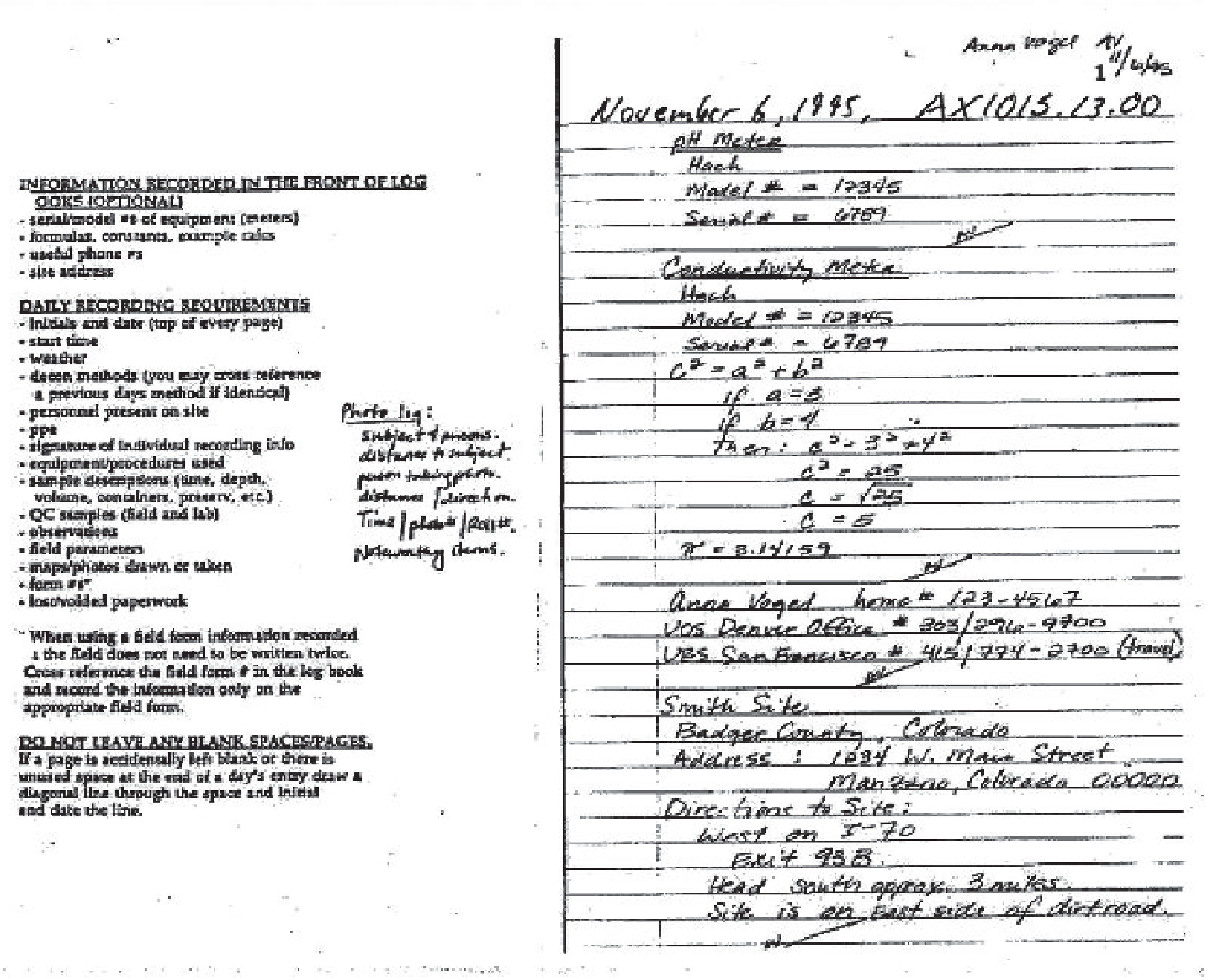
ATTACHMENTS

Attachment 1 – Example Field Logbook
Attachment 2 – Logbook Quick Guide

ATTACHMENT 1
EXAMPLE FIELD LOGBOOK


"Rite in the Rain"®
ALL-WEATHER
ENVIRONMENTAL
No. 550F


ENVIRONMENTAL
4 x 4 to the inch with heading
Location
Date
Project / Client
Location
Date
Project / Client



INFORMATION RECORDED IN THE FRONT OF LOG BOOKS (OPTIONAL)
- serial/model #s of equipment (meters)
- formulas, constants, example calcs
- useful phone #s
- site address

DAILY RECORDING REQUIREMENTS
- initials and date (top of every page)
- start time
- weather
- decon methods (you may cross reference a previous days method if identical)
- personnel present on site
- ppe
- signature of individual recording info
- equipment/procedures used
- sample descriptions (time, depth, volume, containers, preserv. etc.)
- QC samples (field and lab)
- observations
- field parameters
- maps/photos drawn or taken
- form #s
- lost/voided paperwork

When using a field form information recorded in the field does not need to be written twice. Cross reference the field form # in the log book and record the information only on the appropriate field form.

DO NOT LEAVE ANY BLANK SPACES/PAGES.
If a page is accidentally left blank or there is unused space at the end of a day's entry draw a diagonal line through the space and initial and date the line.

Photo log:
Subject of photos -
distance to subject.
person taking photo.
distance / direction.
Time / photo# / Roll#.
weather conditions.

Anna Vogel AV 11/6/95
1

November 6, 1995, AX1015.13.00

pH Meter
Hach
Model # = 12345
Serial # = 6789
AV

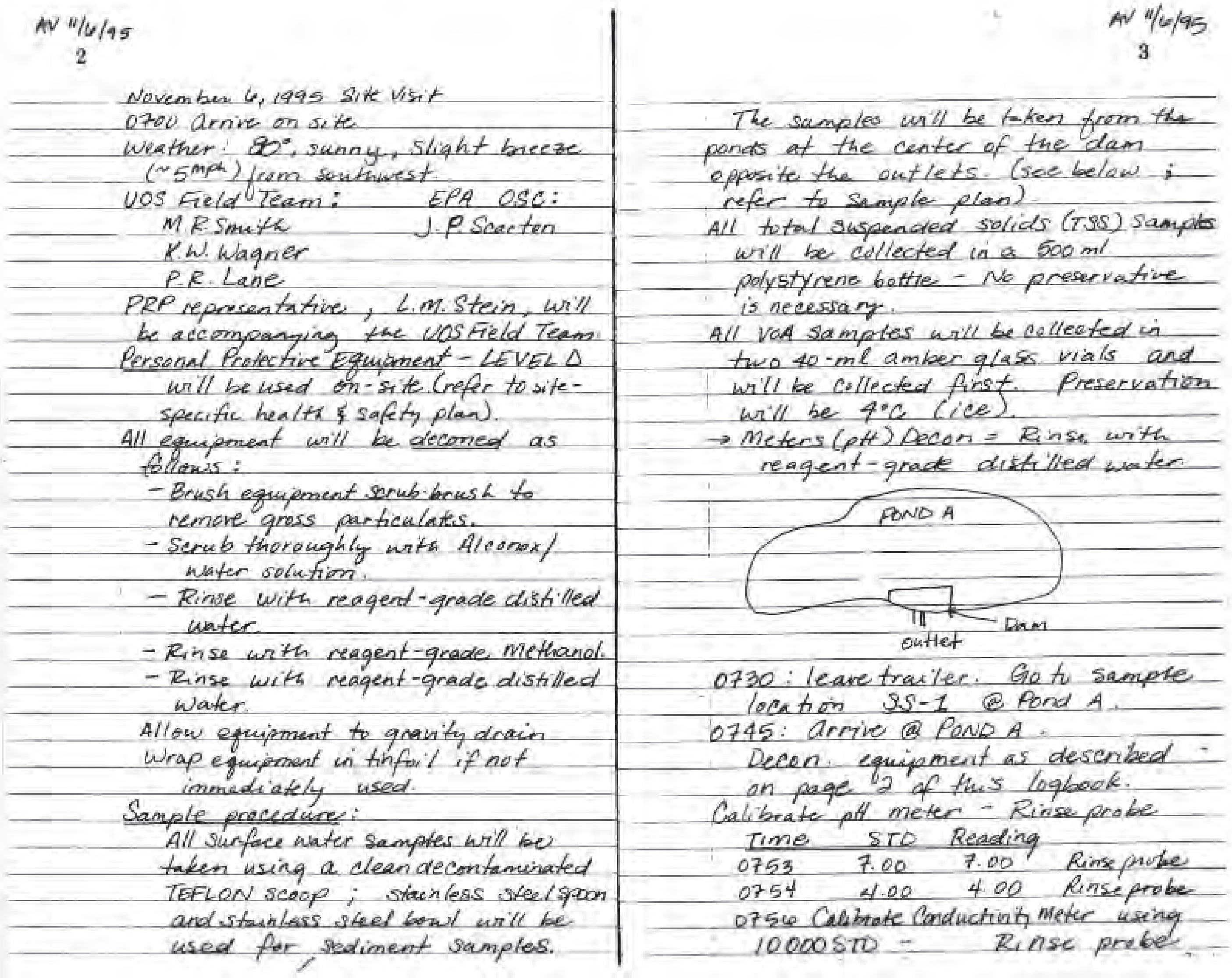
Conductivity Meter
Hach
Model # = 12345
Serial # = 6789

$c^2 = a^2 + b^2$
if $a = 3$
if $b = 4$
Then: $c^2 = 3^2 + 4^2$
$c^2 = 25$
$c = \sqrt{25}$
$c = 5$

$\pi = 3.14159$
AV

Anna Vogel home # 123-4567
VOS Denver Office # 303/296-9700
VBS San Francisco # 415/774-2700 (travel)
AV

Smith Site:
Badger County, Colorado
Address: 1234 W. Main Street
Manzano, Colorado 00000
Directions to Site:
West on I-70
Exit 95B.
Head south approx. 3 miles.
Site is on east side of dirt road.
AV



AV 11/6/95
2

November 6, 1995 Site Visit
0700 Arrive on site
Weather: 80°, sunny, Slight breeze (~5mph) from southwest.

UOS Field Team:
M.R. Smith
K.W. Wagner
P.R. Lane

EPA OSC:
J.P. Scacten

PRP representative, L.M. Stein, will be accompanying the UOS Field Team.

Personal Protective Equipment - LEVEL D will be used on-site (refer to site-specific health & safety plan).

All equipment will be deconed as follows:
- Brush equipment scrub-brush to remove gross particulates.
- Scrub thoroughly with Alconox/water solution.
- Rinse with reagent-grade distilled water.
- Rinse with reagent-grade Methanol.
- Rinse with reagent-grade distilled water.

Allow equipment to gravity drain
Wrap equipment in tinfoil if not immediately used.

Sample procedure:
All Surface water samples will be taken using a clean decontaminated TEFLON scoop; stainless steel spoon and stainless steel bowl will be used for sediment samples.

AV 11/6/95
3

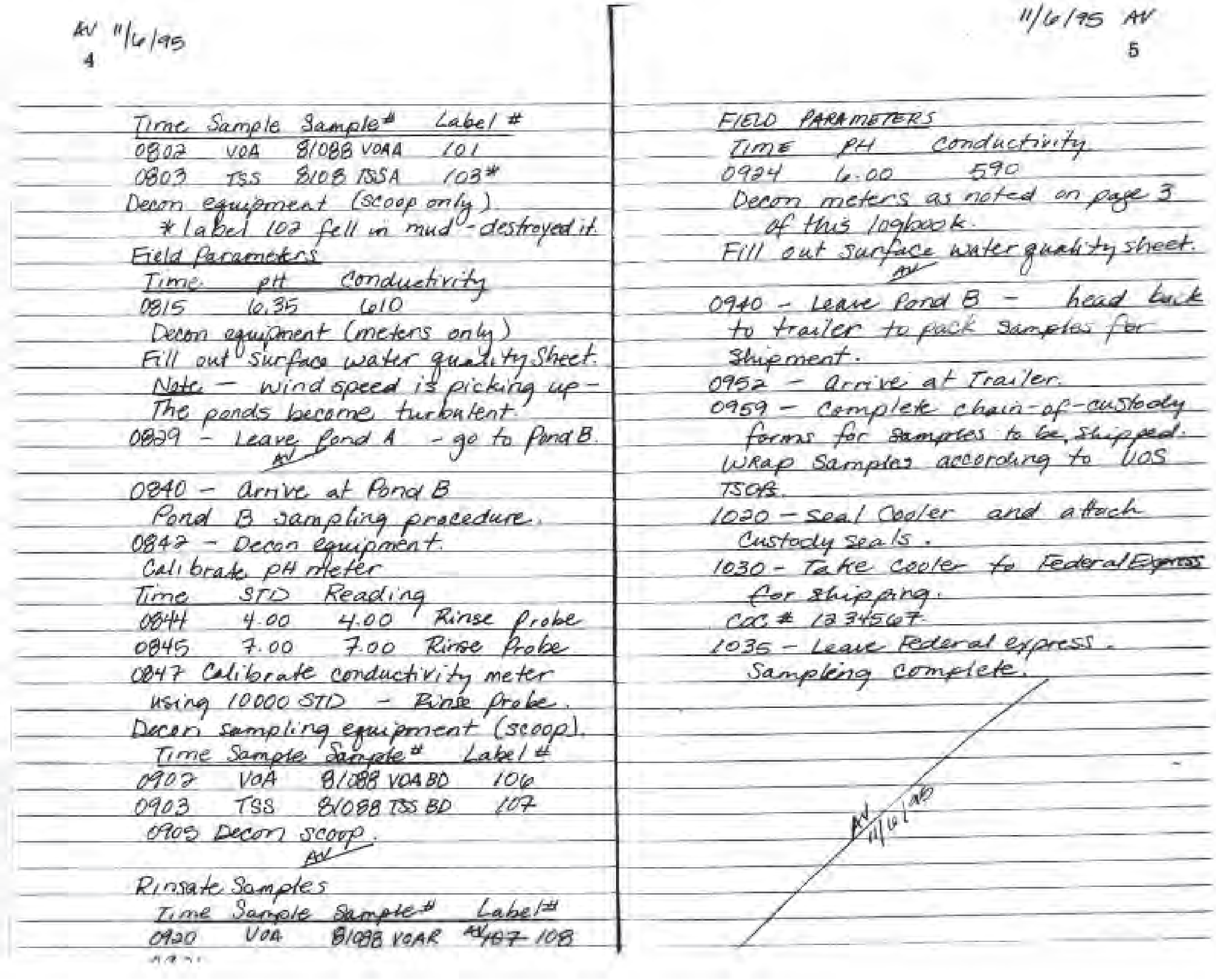
The samples will be taken from the ponds at the center of the dam opposite the outlets. (see below; refer to sample plan)
All total suspended solids (TSS) samples will be collected in a 500 ml polystyrene bottle - No preservative is necessary.
All VOA samples will be collected in two 40-ml amber glass vials and will be collected first. Preservation will be 4°C (ice).
→ Meters (pH) Decon = Rinse with reagent-grade distilled water

POND A
Dam
outlet

0730: leave trailer. Go to sample location SS-1 @ Pond A.
0745: Arrive @ POND A.
Decon. equipment as described on page 2 of this logbook.
Calibrate pH meter - Rinse probe

Time	STD	Reading	
0753	7.00	7.00	Rinse probe
0754	4.00	4.00	Rinse probe

0756 Calibrate Conductivity Meter using 1000 STD - Rinse probe



AV 11/6/95
4

Time	Sample	Sample#	Label #
0802	VOA	8/088 VOAA	101
0803	TSS	8108 TSSA	103*

Decon equipment (scoop only).
*label 102 fell in mud - destroyed it.

Field Parameters

Time	pH	Conductivity
0815	6.35	610

Decon equipment (meters only)
Fill out surface water quality Sheet.
Note - wind speed is picking up - The ponds become turbulent.
0829 - Leave Pond A - go to Pond B.
AV

0840 - Arrive at Pond B
Pond B sampling procedure.
0842 - Decon equipment.
Calibrate pH meter

Time	STD	Reading	
0844	4.00	4.00	Rinse Probe
0845	7.00	7.00	Rinse Probe

0847 Calibrate conductivity meter using 10000 STD - Rinse Probe.
Decon sampling equipment (scoop).

Time	Sample	Sample#	Label#
0902	VOA	8/088 VOA BD	106
0903	TSS	8/088 TSS BD	107

0905 Decon scoop.
AV

Rinsate Samples

Time	Sample	Sample#	Label#
0920	VOA	8/088 VOAR	AV ~~107~~ 108

11/6/95 AV
5

FIELD PARAMETERS

TIME	PH	Conductivity
0924	6.00	590

Decon meters as noted on page 3 of this logbook.
Fill out surface water quality sheet.
AV

0940 - Leave Pond B - head back to trailer to pack samples for shipment.
0952 - Arrive at Trailer.
0959 - Complete chain-of-custody forms for samples to be shipped.
Wrap Samples according to UOS TSOB.
1020 - Seal Cooler and attach Custody seals.
1030 - Take cooler to Federal Express for shipping.
COC # 1234567
1035 - Leave Federal express.
Sampling complete.

AV 11/6/95

ATTACHMENT 2
LOGBOOK QUICK GUIDE

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LOGBOOK QUICK GUIDE

TOP

Location: County/City/State
Project/Client: Project/Client Name

MINIMAL REQUIREMENTS

- times of activities (military)
- author of day’s entries
- field team members
- field team member assignments
- field activities
- EPA or other regulatory personnel observing - activities
- other personnel
- public or press visitors
- equipment used
- equipment calibration information
- serial numbers of equipment
- weather
- decontamination methods
- level of PPE
- calculations used
- sample information
  - o ID
  - o depth
  - o volume
  - o containers
  - o preservative
  - o media
  - o QC samples

LOGBOOK QUICK GUIDE

MINIMAL REQUIREMENTS (cont.)

- background levels and readings
- possible instrument interferences
- photographs
  - + number
  - + direction
  - + description
  - + photographer

OTHER REQUIREMENTS

- unusual observations
- strike through mistakes with single line
- diagonal line across unused portion of page with signature and date
- use indelible black or blue ink
- no erasable ink
- generate tables when possible for information
- leave no pages blank
- place North arrow on sketches
- leave no open lines
- staple business cards of visitors in book
- deviations from approved plans
- field forms completed

** Black text applies to all activities.*
** Red text applies to activities that include sampling.*

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	CORPORATE TECHNICAL PROCEDURE	
	Approved for issue by:	
	Process Owner	Digitally signed by Neil Feist Date: 2022.09.01 08:51:54 -05'00'
	Corporate Quality Director	Theresa Rojas Digitally signed by Theresa Rojas Date: 2022.08.31 16:12:32 -04'00'
MPPEH Inspection and Management		**Document No.: HGL SOP 408.504 (formerly 501.04)**
		Process Category: Services
		Revision No.: 6
		Effective Date: August 29, 2022
		Last Review Date: August 29, 2022
		Next Review Date: August 2024

1.0 PURPOSE AND APPLICABILITY

This standard operating procedure (SOP) describes procedures for handling material potentially presenting an explosive hazard (MPPEH), including inspection, management, safety, security, and chain of custody (CoC) certification during munitions response activities.

This SOP applies to all HydroGeoLogic, Inc. (HGL) employees involved in inspection and management processes for certifying MPPEH as either material documented as safe (MDAS) or as material documented as an explosive hazard (MDEH) before transfer within or release from U.S. Department of Defense (DoD) control. HGL employees tasked with performing these procedures must be qualified in accordance with DoD Safety Board (DDESB) Technical Paper 18, DoD Instruction (DoDI) 4140.62, and DoD Manual (DoDM) 4140.72.

Personnel who use this procedure must complete the SOP Acknowledgement Sheet (HGL SOP 408.501.F01) and submit it to the project Senior Unexploded Ordnance Supervisor (SUXOS) as evidence that they have read and understand this SOP. Project leaders retain this document in the project file.

2.0 SUMMARY OF METHOD

At project startup the MPPEH processing and munitions debris (MD) and range-related debris (RRD) storage areas are identified. Before establishing the MPPEH processing and MD and RRD storage areas, the UXO Quality Control Specialist (UXOQCS) and UXO Safety Officer (UXOSO) conduct a joint inspection of the storage areas. At the end of site activities, and when all MPPEH, MD, and RRD have been processed, the storage area can be closed.

3.0 DEFINITIONS

3.1 DEFINITIONS

Exclusion Zone (EZ): A safety zone established around a work area that contains or potentially contains munitions and explosives of concern (MEC). Only project personnel and authorized escorted visitors are allowed within the EZ. Examples of EZs include safety zones around MEC intrusive activities and safety zones where MEC is intentionally detonated.

Fuze: Devices that initiate the detonation sequence in munitions, such as (1) a device with explosive components designed to initiate a train of fire or detonation in a munition, and (2) a nonexplosive device designed to initiate an explosion in a munition. Fuzes are typically associated with munitions (for example, mortars, and bombs), but are occasionally found separately. They may contain a charge large enough to cause injury or death.

Material Documented as an Explosive Hazard (MDEH): MPPEH that cannot be documented as MDAS, that has been assessed and documented as to the maximum explosive hazards the material is known or suspected to present, and for which the CoC has been established and maintained. This material is no longer considered to be MPPEH. The MDEH characterization only addresses the explosives safety status of the material.

Material Documented as Safe (MDAS): MPPEH that has been assessed and documented as not presenting an explosive hazard and for which the CoC has been established and maintained. This material is no longer considered MPPEH.

Material Potentially Presenting an Explosive Hazard (MPPEH): Material owned or controlled by the DoD that, prior to determination of its explosives safety status, potentially contains explosives or munitions (for example, munitions containers and packaging material; MD remaining after munitions use, demilitarization, or disposal; and RRD) or potentially contains a high enough concentration of explosives that the material presents an explosive hazard (for example, equipment, drainage systems, holding tanks, piping, or ventilation ducts that were associated with munitions production, demilitarization, or disposal operations).

Excluded from MPPEH are munitions within the DoD-established munitions management system, nonmunitions-related material (for example, horseshoes, rebar, other solid objects), munitions-related solid metal fragments that do not realistically present an explosive hazard, and other items that may present explosion hazards (for example, gasoline cans, compressed gas cylinders) that are not munitions and are not intended for use as munitions.

Military Munitions: All ammunition products and components produced for or used by the armed forces for national defense and security, including ammunition products or components under the control of DoD, the Coast Guard, Department of Energy, and National Guard.

Military Munitions include confined gaseous, liquid, and solid propellants; explosives; pyrotechnics; chemical and riot control agents; smokes; and incendiaries, including bulk explosives and chemical warfare agents, chemical munitions, rockets, guided and ballistic missiles, bombs, warheads, mortar rounds, artillery ammunition, small arms ammunition, grenades, mines, torpedoes, depth charges, cluster munitions and dispensers, demolition charges, and devices and components thereof. The term also includes non-nuclear components of nuclear devices managed under the nuclear weapons program of the Department of Energy after all required sanitization operations under the Atomic Energy Act of 1954 (42 U.S.C. 2011 et seq.) have been completed (10 U.S.C. 2710(e)(3)(A)).

Military Munitions do not include wholly inert items, improvised explosive devices, and nuclear weapons, nuclear devices, and nuclear components, except as noted above.

Military Munitions Response Program (MMRP): An element of the Secretary of Defense's Defense Environmental Restoration Program that addresses the following:

- Potential explosives safety, health, and environmental issues caused by past DoD munitions related activities; and
- Potential explosives safety hazards presented by MEC, which includes UXO, discarded military munitions (DMM), and munitions constituent concentrations high enough to pose an explosive hazard and potential environmental contamination.

Minimum Separation Distance (MSD): MSD is the distance at which personnel in the open must be from an intentional or unintentional detonation.

Munitions and Explosives of Concern (MEC): Specific categories of military munitions that may pose unique explosives safety risks, including the following:

- UXO, as defined in 10 U.S.C. 101(e)(5)(A) through (C);
- DMM, as defined in 10 U.S.C. 2710(e)(2); or
- Munitions constituents (such as TNT and RDX), as defined in 10 U.S.C. 2710(e)(3), present in high enough concentrations to pose an explosive hazard.

Munitions Debris (MD): Remnants of munitions (such as fragments, penetrators, projectiles, shell casings, links, and fins) remaining after munitions use, demilitarization, or disposal. Inert munitions-related material recovered during an MEC removal.

Range-Related Debris (RRD): Debris, other than MD, collected from operational ranges or from former ranges (for example, targets such as tanks, vehicles, or other man-made structures).

Transferred within or Released from DoD Control: A situation where a receiver has taken physical custody of MDEH or MDAS from DoD and has received signed documentation that acknowledges the MDEH or MDAS material (DD Form 1348-1A, Issue Release/Receipt Document, or an equivalent document).

Unexploded Ordnance (UXO): Military munitions that meet one of the following criteria:

- Have been primed, fuzed, armed, or otherwise prepared for action;
- Have been fired, dropped, launched, or projected;
- Have been placed in such a manner as to constitute a hazard to operations, installation, personnel, or material; and
- Remain unexploded whether by malfunction, design, or any other cause.

A more detailed description of the term UXO is provided in Public Law (P.L.) 106-65, section 3031 (c)(5)(A).

UXO-Qualified Personnel: Individuals who meet the training requirements for UXO technician and personnel and have performed successfully in military Explosive Ordnance Disposal (EOD) positions or are qualified to perform in the following service contractor positions: UXO Technician II, UXO Technician III, UXOQCS, and SUXOS.

3.2 ABBREVIATIONS/ACRONYMS

CFR	Code of Federal Regulations
CoC	chain of custody
DDESB	DoD Safety Board
DMM	discarded military munitions
DoD	U.S. Department of Defense
DoDI	U.S. DoD Instruction
DoDM	U.S. DoD Manual
DQCR	Daily Quality Control Report
EM	Engineer Manual
EOD	Explosive Ordnance Disposal
EZ	exclusion zone
HGL	HydroGeoLogic, Inc.
HTRW	hazardous, toxic, or radiological waste
MD	munitions debris
MDAS	material documented as safe
MDEH	material documented as an explosive hazard
MEC	munitions and explosives of concern
MMRP	Military Munitions Response Program
MPPEH	material potentially presenting an explosive hazard

MSD	minimum separation distance
P.L.	Public Law
PM	Project Manager
QC	quality control
RRD	range-related debris
SOP	standard operating procedure
SUXOS	Senior Unexploded Ordnance Supervisor
USACE	U.S. Army Corps of Engineering
UXO	unexploded ordnance
UXOQCS	UXO Quality Control Specialist
UXOSO	UXO Safety Officer

4.0 HEALTH AND SAFETY WARNINGS

Only personnel who are qualified UXO personnel will perform inspections of MPPEH.

5.0 CAUTIONS

The UXOQCS inspects the MPPEH processing and MD/RRD storage area containers weekly. It is paramount that these inspections enforce all safeguarding and security control measures required to prevent potential co-mingling of processed material.

6.0 INTERFERENCES

It is the responsibility of the project manager (PM) to coordinate with the MMRP Operations Manager to ensure final closeout of all material demilitarization/destruction certification records.

7.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

7.1 MANAGERS AND SUPERVISORS

The PMs and field supervisors ensure that all site personnel read, understand, and follow this SOP.

The SUXOS or senior UXO-qualified individual assigned ensures that all MPPEH activities are conducted in accordance with the following:

- Defense Explosives Safety Regulation 6055.09, Edition 1, UXO, Munitions Response, Waste Military Munitions, and Material Potentially Presenting an Explosive Hazard (MPPEH);
- DoDI 4140.62, Material Potentially Presenting and Explosive Hazard;
- DoDM 4140.72, Management of Material Potentially Presenting an Explosive Hazard;
- EM 385-1-97, Explosives Safety and Health Requirements Manual; and
- All other references shown in Section 12.0 of this SOP.

HGL UXO technicians must comply with these procedures for processing MPPEH for final disposition. Only UXO-qualified personnel are authorized to perform MPPEH processing.

7.2 UXO TECHNICIANS

Immediately refer any discrepancies with procedural steps or safety issues pertaining to this SOP to the responsible supervisor for corrective action. Follow these procedures to safely conduct inspections of the exterior and interior surfaces of all recovered MPPEH and to ensure these items do not present an explosive hazard.

1. Unexploded Ordnance Sweep Personnel:
   - Marks suspected items only; may not assess a suspect item to determine its status.
2. UXO Technician I:
   - Tentatively identifies a located item as MPPEH with confirmation by a UXO Technician II or III.
3. UXO Technician II:
   - Performs an inspection of each item as it is recovered and determines the following:
     - Is the item a UXO, a DMM, MD, RRD, or other debris?
     - Does the item contain explosives hazards or other dangerous fillers?
     - Does the item require detonation?
     - Does the item require demilitarization or venting to expose dangerous fillers?
     - Does the item require removal of batteries, mercury seals, or switches; draining of engine fluids; or removal of illuminating dials and other visible liquid hazardous, toxic, or radiological waste (HTRW) materials?
   - Segregates material items requiring demilitarization or venting procedures from items ready for certification as MDAS.

- Processes any items found to contain explosive hazards or other dangerous fillers in accordance with applicable procedures.

4. UXO Technician III:
   - Performs an inspection of all items recovered by their team to determine if it free of explosives hazards or other dangerous fillers and engine fluids, illuminating dials, and other visible liquid HTRW materials.
   - Supervises detonation of items containing explosive hazards or other dangerous fillers and venting/demil procedures.
   - Supervises the consolidation of MPPEH to be containerized and sealed, ensuring that MD and RRD are segregated.
5. UXOQCS:
   - Conducts daily audits of the procedures used by UXO teams and individuals for processing MPPEH.
   - Performs or witnesses the 100 percent inspection or DDESB-approved processing of the material to “verify” that the MD and RRD are free of explosive hazards and engine fluids, illuminating dials, or other visible liquid HTRW materials as required for completion of DD Form 1348-1A, Issue Release/Receipt Document (see HGL SOP 408.504.F03).
6. UXOSO:
   - Ensures the specific procedures and responsibilities for processing MPPEH for certification as MD or RRD specified in the work plan are being followed.
   - Ensures all procedures for processing MPPEH are being performed safely and consistent with applicable regulations.
7. SUXOS:
   - Ensures that work and quality control (QC) plans specify the procedures and responsibilities for processing MPPEH for final disposition as MD or RRD.
   - Completes a Requisition and Turn-in Document, DD Form 1348-1A, for all MD and RRD to be transferred for final disposition.
   - Performs or witnesses the 100 percent reinspection or DDESB-approved processing of the material to “certify” that the MD and RRD are free of explosive hazards necessary to complete the DD Form 1348-1A.
   - Certifies all MD and RRD as free of explosive hazards, engine fluids, illuminating dials, and other visible liquid HTRW materials.
   - Ensures that inspected debris is secured in a closed, labeled, and sealed container, and document as follows:

    - The container will be closed and clearly labeled on the outside with the following information: U.S. Army Corps of Engineering (USACE) or applicable DoD component/installation name/HGL/0001/seal's unique identification. Subsequent containers will be numbered 0002, 0003, and so on.
    - The container will be closed in such a manner that a seal must be broken in order to open the container. The container will be clearly marked with the seal's identification number.
    - A document describing each container will be created with the following information: container contents, weight of container, location where MD or RRD was obtained, HGL named as contractor, names of certifying and verifying individuals, unique container identification, and seal identification. Include these documents in a separate section of the final report.
- Establishes a secure location for collecting, processing, and storing MPPEH, MD and RRD until transferred off site.
- Uses the MPPEH, MD and Range Debris Processing and Storage Area Inspection Checklist (see HGL SOP 408.504.F01) to verify the following:
    - EZs are maintained during MPPEH inspection and processing activities.
    - Adequate warning signs and boundary markers are in place during MPPEH inspection and processing activities.
    - Storage containers, drums, pallets, and tarpaulins are in good repair.
    - Storage containers and drums are properly labeled and legible.
    - Uninspected material is properly segregated from inspected material to prevent co-mingling.
    - Storage containers or drums in active use are locked to prevent uninspected material from being co-mingled with inspected material until seals are installed.
    - Demilitarized items are secured.

8.0 EQUIPMENT AND SUPPLIES

Equipment needed for the management and processing of MPPEH includes the following:

- Storage containers (drums) and drum dolly,
- Pallets and pallet jack,
- Drum seals and locks,
- Weight scales,
- Bore scopes,
- Buckets, and
- Tarps.

9.0 PROCEDURAL STEPS

9.1 MPPEH CERTIFICATION AND VERIFICATION PROCEDURES

Assess all MPPEH to determine and document its explosive safety status before transferring the material within DoD facilities or releasing it from DoD control. Authorized and technically qualified personnel must certify the MPPEH as MDAS before it can be released to the public. MPPEH procedures must comply with DoDI 4140.62, DoDM 4140.72, and Engineer Manual (EM) 385-1-97.

Adhere to the following certification and verification procedures for material suspected or determined to be MPPEH before transporting it within or out of DoD control:

1. Obtain certification and verification that a 100 percent inspection and an independent 100 percent reinspection has been completed and that the material has been determined to be free of explosives before certifying it as MDAS.
2. The SUXOS certifies that the debris is free of explosives hazards and can be classified as MDAS.
3. The UXOQCS verifies that the debris is free of explosive hazards.
4. The first signatory must be technically qualified and either a DoD employee or a DoD contractor. This signatory must have performed or witnessed the initial 100 percent inspection/certification. The second signatory must be a technically qualified U.S. citizen who is either a DoD employee or a DoD contractor. This signatory must have performed or witnessed the independent 100 percent re-inspection/verification. Each signatory must ensure the CoC was maintained before signing the explosives-safety-status documentation.
5. Use DD Form 1348-1A, Issue Release/Receipt Document, as the certification/verification document. The DD Form 1348-1A must clearly show the names of the SUXOS and the UXOQCS and contain the information listed below. (Refer to example shown in Attachment 1.)
    - <u>Block 5</u>: Document date
    - <u>Block 17</u>: Basic material content (type of metal, such as steel or mixed)
    - <u>Block 20</u>: Total weight
    - <u>Blocks 24, 25, 26, and 27</u>:
        - SUXOS and UXOQCS name
        - Company name
        - SUXOS and the UXOQCS signature
        - HGL Huntsville office addresses and telephone number
        - HGL unique identification number for each container
        - Container seal number

- Site name, city, and state of where MD or RRD was obtained

6. Enter the following certification/verification on each DD Form 1348-1A (refer to HGL SOP 408.504.F02) for MD or RRD transferred within or released from DoD control. The SUXOS and the UXOQCS must sign the form. The following statement is required on MDAS documentation, and it must identify which methodology is used: *"The material listed on this form has been inspected, processed by DDESB-approved means, or undergone the application of expert knowledge, in compliance with DoD policy, and to the best of my knowledge and belief, does not pose an explosive hazard. The explosive safety status was determined by (enter verbiage from (a), (b), or (c) depending on which process was used)."*

   (a) 100-percent visual inspection and an independent 100-percent re-inspection by qualified personnel;

   (b) Processing by a DDESB-approved method with appropriate post-processing inspection (e.g., sampling) of the material; or

   (c) Application of DoD Component-approved expert knowledge. A certification or verification statement as shown must be signed and dated by a DoD contracted person or a government employee.

7. Upon receipt of the material identified on the DD Form 1348-1A, the PM and the SUXOS ensure that the following blocks are completed by the qualified recycler:
   - Block 10: Quantity of material received
   - Block 22: Signature
   - Block 23: Date

9.2 MAINTAINING CHAIN OF CUSTODY AND FINAL DISPOSITION

Coordinate with the USACE or the appropriate DoD agency to arrange for maintaining the CoC and for final disposition of the certified and verified materials. Release the certified and verified material only to an organization that will perform the following:

1. Provide certification on company letterhead stating the following:

   "Upon receiving the unopened labeled containers each with its unique identified and unbroken seal ensuring a continued chain of custody, and after reviewing and concurring with all the provided supporting documentation, sign for having received and agreed with the provided documentation that the sealed containers contained no explosive hazards when received. The contents of these sealed containers will not be sold, traded or otherwise given to another party until the contents have been smelted and are only identifiable by their basic content."

2. Send notification and supporting documentation to HGL documenting that the sealed containers have been smelted and are now only identifiable by their basic content.

The following steps should then be taken:

- Incorporate this supporting documentation into the final report as supporting documentation for the final disposition of MD and RRD.
- If the CoC is broken, the affected MPPEH must undergo a second 100 percent inspection, then 100 percent reinspection, and be documented again to verify its explosives safety status (identified as either MD or RRD). Refer to Section 10.0 of this SOP.
- MDAS is no longer considered MPPEH as long as the CoC remains intact. A legible copy of inspection, reinspection, and documentation must accompany the material through final disposition and be maintained for a period of 3 years thereafter. Maintain this documentation as directed in Section 10.0 of this SOP.
- Document MDAS being transferred within or released from DoD control on the CoC form (see HGL SOP 408.504.F03).
- The PM contacts the receiving agent/recycler to obtain MDAS final demilitarization certification documentation.

9.3 MDAS MANAGEMENT

Dispose of all MDAS with a recycler that smelts MDAS prior to resale or release in accordance with EM 385-1-97. If it is discovered during the material transfer and shipping process that a seal has been broken and the CoC of the material cannot be verified, the material in question will be subject to reinspection following the established MPPEH processes described in Sections 9.1 and 9.2 of this SOP.

The MDAS recycler subcontractor prepares the documentation verifying the demilitarization and final disposition of the material and provides copies of all MDAS certification and CoC documentation to HGL. Refer to examples in Attachments 1, 2, and 3.

9.4 MDEH MANAGEMENT

Complete the following procedures before releasing MDEH:

- Ensure that MDEH is transferred or released only to a qualified receiver that meets the following requirements:
  - Has the licenses, permits, and site approvals, as appropriate, required to manage and dispose of the materials being received.
  - Has the facilities, capacity, and technical expertise required to safely manage the explosive hazards associated with the MDEH being received.
  - Has procedures in place for the management and disposition of MDEH in accordance with this issuance and DoDD 5134.01, DoDM 4160.21, and DoDI 4160.28.

  - Has personnel who are
    - Experienced in managing and processing hazardous materials equivalent to MDEH and meet the criteria specified in Paragraph 4.3.c of DoDM 4140.72 and
    - Trained and experienced in the identifying and safe handling of used and unused military and/or any potential explosive hazards that may be associated with the specific MPPEH.
- Advise the receiver of all the potential hazards associated with the MDEH. The receiver must agree to receive and process the material in accordance with DoDI 4140.62.
- Choose public transportation routes that comply with DoD hazardous material transportation regulations for all MDEH shipments.
- Maintain CoC and accountability records through final disposition of the MDEH. A legible copy of inspection, reinspection, and other documentation as discussed in Sections 9.1 and 9.2 must accompany MDEH through final disposition and be maintained for a period of 3 years thereafter and in accordance with Section 10.0 of this SOP.

9.5 INSPECTIONS

9.5.1 Project Startup Inspection

Before establishing the MPPEH processing and MD and RRD storage areas, the UXOQCS and UXOSO conduct a joint inspection of the storage areas and document the results of the inspection, identify any discrepancies, and note their disposition in the project Daily Quality Control Report (DQCR).

9.5.2 Routine Weekly Inspection

The UXOQCS inspects the MPPEH processing and MD/RRD storage area containers every week after they arrive and are collected at the site, using the MPPEH Processing and MD and Range Debris Storage Area Inspection Checklist (HGL SOP 408.504.F01) for conducting and documenting weekly storage area inspections.

The weekly inspection is necessary to determine if unauthorized entry into the containers has been attempted, or if unauthorized removal of the contents has occurred.

> **– NOTE –**
>
> *It is paramount that these inspections enforce all safeguarding and security control measures required for preventing potential co-mingling of processed material and ensuring the integrity of this process is properly maintained.*

The UXOQCS weekly inspection addresses the following questions:

1. Are EZs properly maintained?
2. Are adequate hazard warning signs and boundary markers in place?
3. Are storage containers and drum pallet in fair condition?
4. Are containers properly labeled and labels intact?
5. Are containers sealed and locked?
6. Are uninspected items segregated from inspected items?
7. Are demilitarized items secured?
8. Are any materials missing, or is there evidence of tampering or unauthorized entry?

The UXOQCS signs the inspection checklist upon completion of this inspection; the SUXOS reviews and signs the inspection checklist. The checklist is maintained with the project site office files and is annotated in the weekly production report and the DQCR.

The SUXOS periodically spot-checks the MPPEH processing and MD/RRD storage area containers to ensure that security, integrity, and good housekeeping of the storage area is maintained.

10.0 DATA AND RECORDS MANAGEMENT

All project personnel are responsible for documenting in detail all reports, logs, and certification and inspection forms based on their assigned level of technical responsibility. Use the forms listed below in the order shown for documenting the MPPEH inspection, certification, storage, and transfer and release of material process.

Before the transfer within DoD or release from DoD, document all verified and certified materials as follows:

- The SUXOS prepares two original copies of the following:
  - DD Form 1348-1A, Issue Release/Receipt Document (HGL SOP 408.504.F02; refer to the two examples provided as Attachment 1.
  - CoC form.
  - Obtain signatures from the SUXOS, Ordnance and Explosive Safety Specialist (or UXOQCS), and MDAS recycler in all appropriate blocks on all documents as shown in Attachments 1, 2, and 3.
- Distribute copies of the DD Form 1348-1A, Issue Release/Receipt Document, and CoC form as follows:

  - Copy 1:
    - Issued to the receiver (for example, transporter, demilitarization facility, or recycler).
  - Copy 2:
    - Mail the original to the HGL Huntsville office repository, to the attention of the MMRP Operations Manager, for retention.
    - Scan Copy 2 as a PDF file and submit it electronically to the subcontracted recycler manager and the HGL MMRP Operations Manager.
- The SUXOS coordinate the following:
- Notifying the receiver agent/qualified recycler in advance of any pickups and shipments to its facility,
- Forwarding advanced electronic copies of all completed CoC and DD Form 1348-1A documents to the receiving agent/recycler,
- Following up with the receiving agent/recycler to obtain final (signed) destruction certification documents, and
- Ensuring that either hard or electronic copies of the final demilitarization certification documents are forwarded to the HGL Huntsville office, to the attention of the MMRP Operations Manager.

11.0 QUALITY CONTROL AND QUALITY ASSURANCE

- The HGL Corporate QA Manager and MMRP Operations Manager ensure that this SOP is reviewed annually for completeness, accuracy, and safety.
- The HGL MMRP Operations Manager maintains, manages, and annually reviews this SOP for procedural, QC, and safety issues. All questions, comments, or recommendations regarding this SOP should be directed to the MMRP Operations Manager.
- PMs and supervisors ensure that all site personnel read, understand, and follow this SOP.
- Refer any discrepancies found with procedural steps or safety issues pertaining to this SOP to the responsible supervisor for corrective action.

All personnel involved in the MPPEH inspection process and management must carefully follow all safeguards and security control measures to prevent co-mingling of processed material. If suspicion arises that co-mingling has occurred, notify the SUXOS and the UXOQCS immediately. Reinspect and process the suspect material in accordance with this SOP.

Train all UXO-qualified personnel in the recognition and safe handling of used and unused military munitions and specific types of MPPEH in accordance with DoDI 4140.62, Material Potentially

Presenting an Explosive Hazard and DoDM 4140.72, Management of Material Potentially Presenting an Explosive Hazard. Qualify all UXO-qualified personnel in accordance with DDESB Technical Paper 18.

12.0 REFERENCES

U.S. Army Corps of Engineers (USACE), Engineer Manual 385-1-97, Explosives Safety and Health Requirements.

U.S. Code of Federal Regulations (CFR), Title 27, Part 55, Commerce in Explosives.

CFR, Title 29, Part 1910, Occupational Safety and Health Standards.

CFR, Title 29, Part 1926, Occupational Safety and Health Standards.

CFR, Title 40, Parts 260-299, Protection of the Environment.

CFR, Title 49, Parts 100-199, Transportation.

U.S. Department of Defense (DoD) 4145.26-M, Contractor's Safety Requirements for Ammunition and Explosives.

DoD Explosive Safety Board Technical Paper 18, Minimum Qualifications for Personnel Conducting Munitions and Explosives of Concern-Related Activities.

DoD, Defense Explosives Safety Regulation 6055.09, Edition 1.

DoD Instruction 4140.62, Material Potentially Presenting an Explosive Hazard (MPPEH).

DoD Manual 4160.28-M-1, Defense Demilitarization Manual.

DoD Manual 4140.72, Management of Material Potentially Presenting an Explosive Hazard.

13.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
6	August 29, 2022	Initial CMS Library Version updated to include requirements of DoDM 4140.72.

ATTACHMENTS

Attachment 1	DD Form 1348-1A Issue Release/Receipt Document (MD and RRD release or transfer) and DD Form 1348-1A Issue Release/Receipt Document (MD release only)
Attachment 2	Sample Certificate of Destruction and Blanket Certificate of Destruction Letters
Attachment 3	Chain of Custody Form

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ATTACHMENTS

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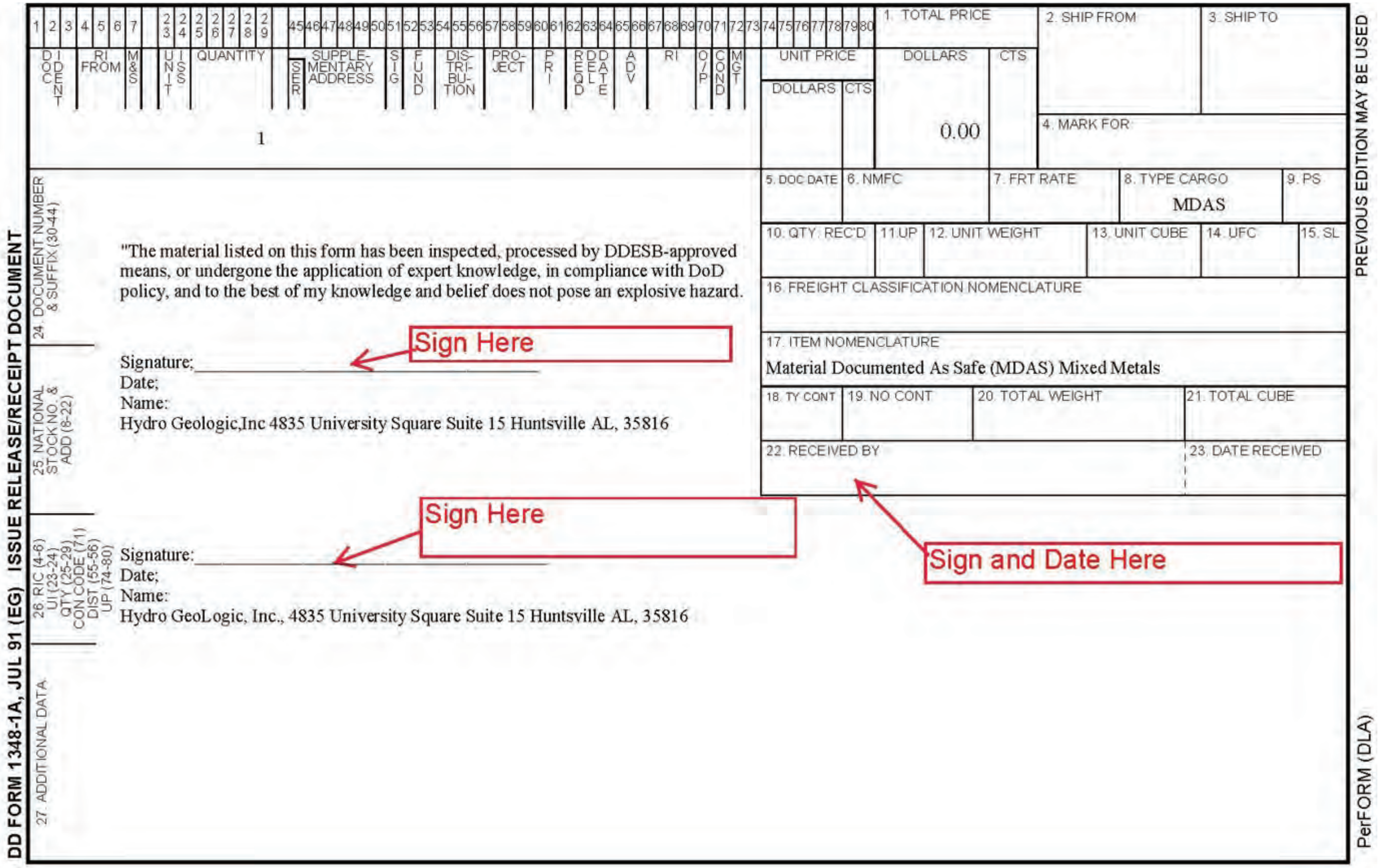
ATTACHMENT 1



DD FORM 1348-1A, JUL 91 (EG) ISSUE RELEASE/RECEIPT DOCUMENT

1 2 3	4 5 6	7	23	24	25 26 27 28 29	45	46 47 48 49 50	51	52 53	54 55 56	57 58 59	60 61	62 63 64	65 66	67 68 69	70	71	72 73	74 75 76 77 78 79 80	1. TOTAL PRICE	2. SHIP FROM	3. SHIP TO
DOC IDENT	RI FROM	M & S	UNIT	UI SS	QUANTITY	SER	SUPPLEMENTARY ADDRESS	SIG	FUND	DISTRIBUTION	PROJECT	PRI	REQD DEL DATE	ADV	RI	O/P	COND	MGT	UNIT PRICE: DOLLARS, CTS	DOLLARS, CTS		
					1															0.00	4. MARK FOR	

5. DOC DATE	6. NMFC	7. FRT RATE	8. TYPE CARGO MDAS	9. PS	
10. QTY. REC'D	11. UP	12. UNIT WEIGHT	13. UNIT CUBE	14. UFC	15. SL

16. FREIGHT CLASSIFICATION NOMENCLATURE

17. ITEM NOMENCLATURE

Material Documented As Safe (MDAS) Mixed Metals

18. TY CONT	19. NO CONT	20. TOTAL WEIGHT	21. TOTAL CUBE
22. RECEIVED BY			23. DATE RECEIVED

24. DOCUMENT NUMBER & SUFFIX (30-44)

"The material listed on this form has been inspected, processed by DDESB-approved means, or undergone the application of expert knowledge, in compliance with DoD policy, and to the best of my knowledge and belief does not pose an explosive hazard.

Sign Here

Signature;____
Date;
Name:
Hydro Geologic,Inc 4835 University Square Suite 15 Huntsville AL, 35816

25. NATIONAL STOCK NO. & ADD (8-22)

Sign Here

Sign and Date Here

26. RIC (4-6) UI (23-24) QTY (25-29) CON CODE (71) DIST (55-56) UP (74-80)

Signature:____
Date;
Name:
Hydro GeoLogic, Inc., 4835 University Square Suite 15 Huntsville AL, 35816

27. ADDITIONAL DATA

PREVIOUS EDITION MAY BE USED

PerFORM (DLA)

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ATTACHMENT 2

SAMPLE

CERTIFICATE OF DESTRUCTION

To: Mr./Mrs. ___________________,
Project Manager,
HydroGeoLogic, Inc.
(Applicable HGL office address)

From: Mr./Mrs. Debris,
President/Owner
American EOD Services, Inc.
1206 East Park Avenue
Anaconda, MT 59711-0878

Re: Demilitarization and Recycling of Material Documented as Safe (MDAS) for the Remedial Investigation at Munitions Response Site ANAD-001-R-01, Recoiless Rifle Range, Anniston Army Depot, AL, U.S. Army Corp of Engineer Contract Number W912DY-10-D-0023, Delivery Order Number 002

Upon receiving the unopened labeled containers each with its unique identified and unbroken seal ensuring a continued chained of custody, and after reviewing and concurring with all the provided supporting documentation, sign for having received and agreeing with the provided documentation that the sealed containers contained no explosive hazards when received. The contents of these sealed containers will not be sold, traded or otherwise given to another party until the contents have been smelted and are only identifiable by their basic content.

I hereby certify that the material received/shipped from Anniston Army Depot, AL was demilitarized by means of shredding and smelting by the Bonetti Explosives, LLC, Columbus, TX and were only identifiable by their basic contents; furthermore, recycling was accomplished by smelting. The material was recycled by smelting into new steel products by at Lone Star Foundries, Inc., Austin, TX an are only identifiable by their basic content.

This certifcation is made in accordance with and subject to penalties of law under the United States Code, Title 18, Section 1001, Crimes and Criminal Procedures.

SIGN HERE

SIGNATURE: ______________________________

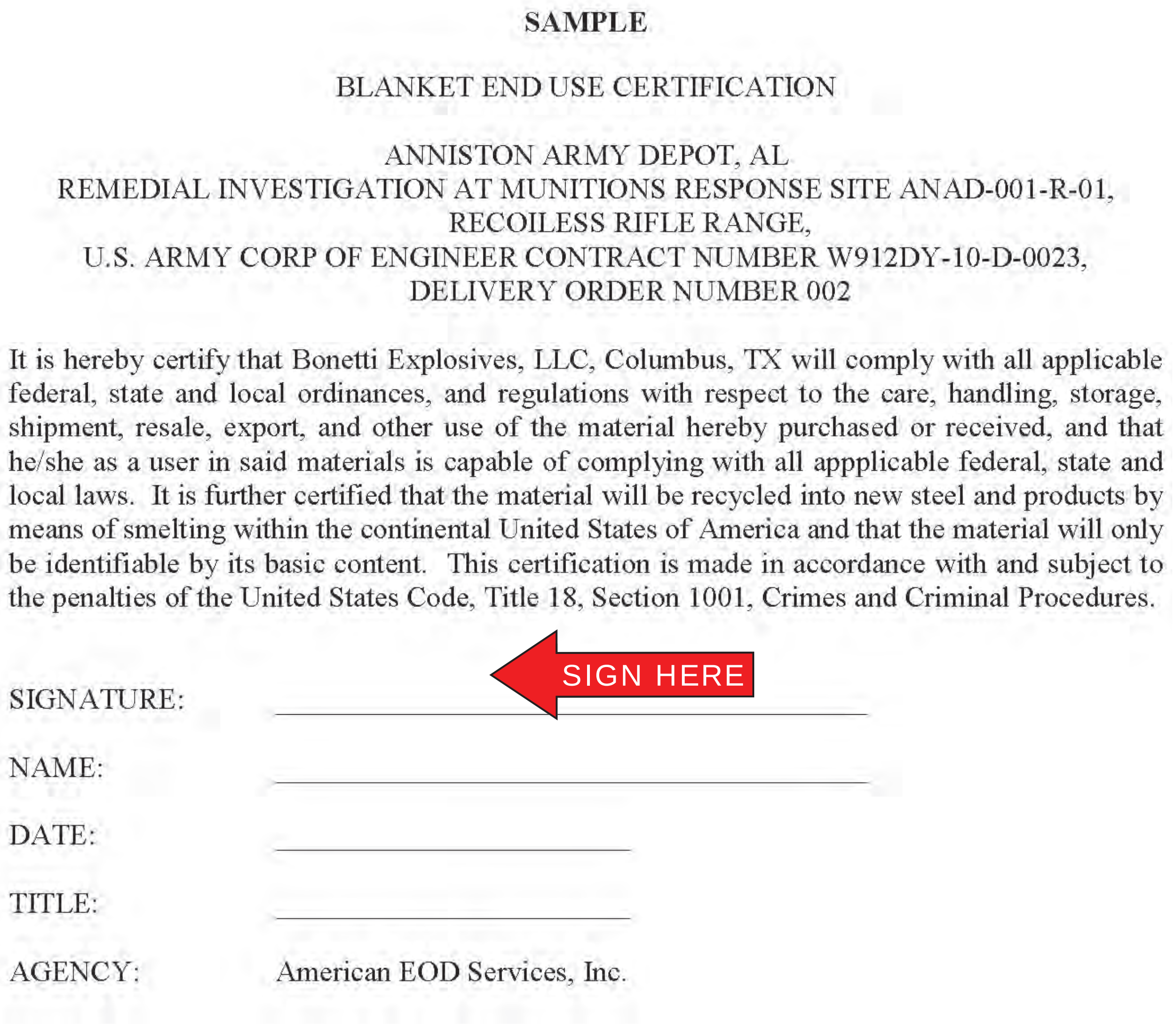
NAME: ______________________________

DATE: ______________________________

TITLE: President/Owner

AGENCY: American EOD Services, Inc.

ATTACHMENT 2 (continued)



SAMPLE

BLANKET END USE CERTIFICATION

ANNISTON ARMY DEPOT, AL
REMEDIAL INVESTIGATION AT MUNITIONS RESPONSE SITE ANAD-001-R-01,
RECOILESS RIFLE RANGE,
U.S. ARMY CORP OF ENGINEER CONTRACT NUMBER W912DY-10-D-0023,
DELIVERY ORDER NUMBER 002

It is hereby certify that Bonetti Explosives, LLC, Columbus, TX will comply with all applicable federal, state and local ordinances, and regulations with respect to the care, handling, storage, shipment, resale, export, and other use of the material hereby purchased or received, and that he/she as a user in said materials is capable of complying with all appplicable federal, state and local laws. It is further certified that the material will be recycled into new steel and products by means of smelting within the continental United States of America and that the material will only be identifiable by its basic content. This certification is made in accordance with and subject to the penalties of the United States Code, Title 18, Section 1001, Crimes and Criminal Procedures.

SIGN HERE

SIGNATURE: ______________________

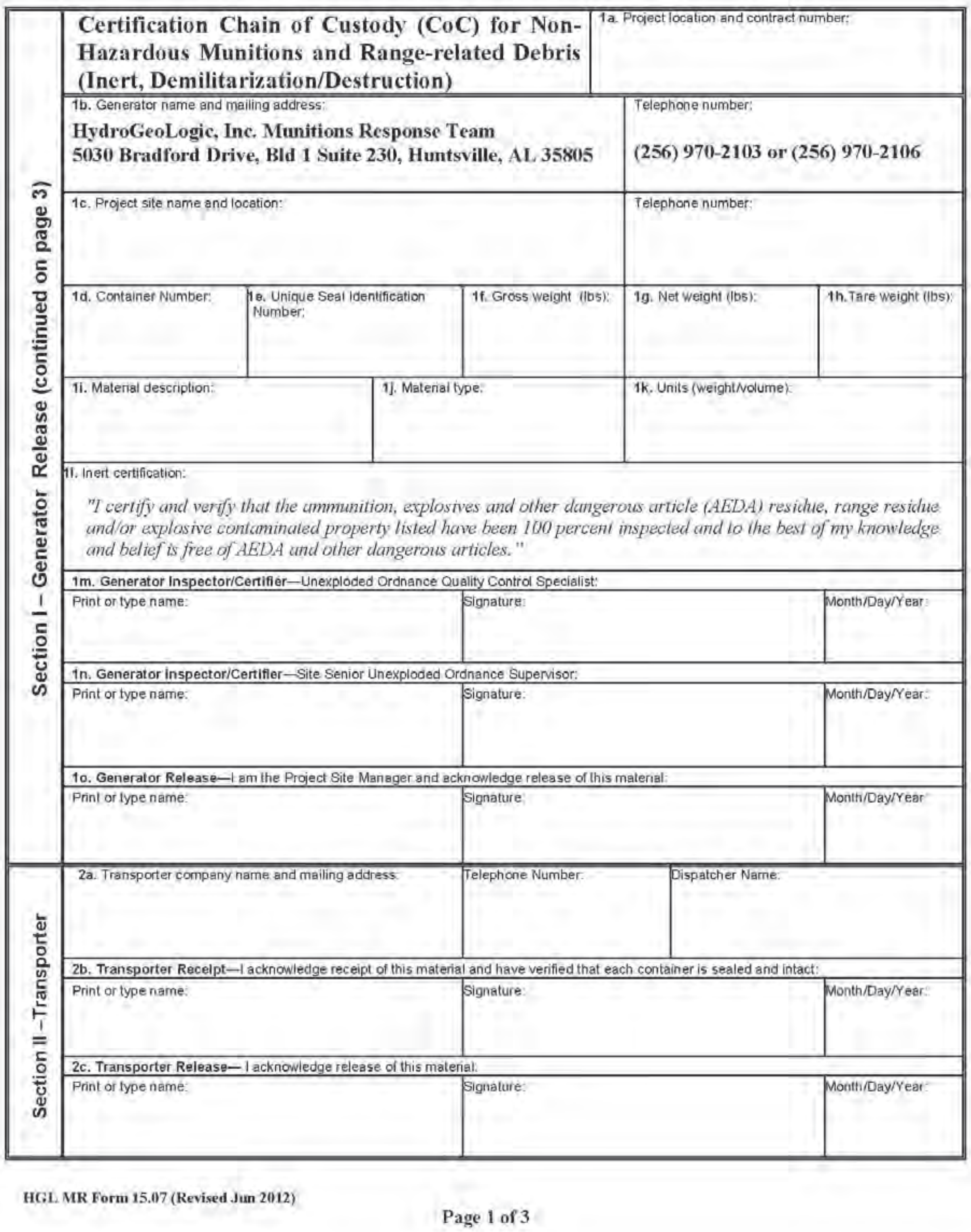
NAME: ______________________

DATE: ______________

TITLE: ______________

AGENCY: American EOD Services, Inc.

ATTACHMENT 3



Section I – Generator Release (continued on page 3)

Certification Chain of Custody (CoC) for Non-Hazardous Munitions and Range-related Debris (Inert, Demilitarization/Destruction)	1a. Project location and contract number:
1b. Generator name and mailing address: **HydroGeoLogic, Inc. Munitions Response Team** **5030 Bradford Drive, Bld 1 Suite 230, Huntsville, AL 35805**	Telephone number: **(256) 970-2103 or (256) 970-2106**
1c. Project site name and location:	Telephone number:

1d. Container Number:	1e. Unique Seal Identification Number:	1f. Gross weight (lbs):	1g. Net weight (lbs):	1h. Tare weight (lbs):

1i. Material description:	1j. Material type:	1k. Units (weight/volume):

1l. Inert certification:

"I certify and verify that the ammunition, explosives and other dangerous article (AEDA) residue, range residue and/or explosive contaminated property listed have been 100 percent inspected and to the best of my knowledge and belief is free of AEDA and other dangerous articles."

1m. Generator Inspector/Certifier—Unexploded Ordnance Quality Control Specialist:

Print or type name:	Signature:	Month/Day/Year:

1n. Generator Inspector/Certifier—Site Senior Unexploded Ordnance Supervisor:

Print or type name:	Signature:	Month/Day/Year:

1o. Generator Release—I am the Project Site Manager and acknowledge release of this material:

Print or type name:	Signature:	Month/Day/Year:

Section II –Transporter

2a. Transporter company name and mailing address:	Telephone Number:	Dispatcher Name:

2b. Transporter Receipt—I acknowledge receipt of this material and have verified that each container is sealed and intact:

Print or type name:	Signature:	Month/Day/Year:

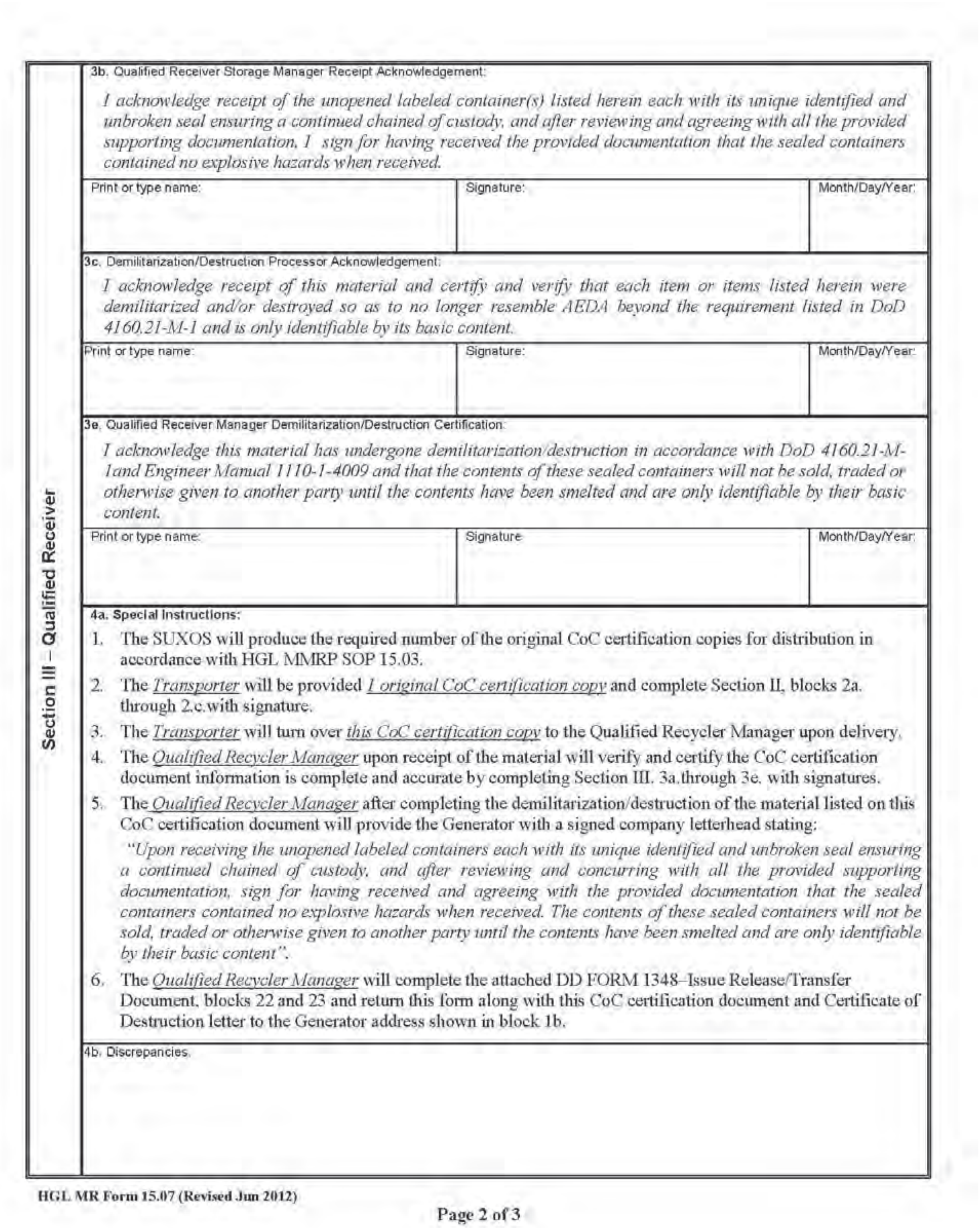
2c. Transporter Release— I acknowledge release of this material:

Print or type name:	Signature:	Month/Day/Year:

HGL MR Form 15.07 (Revised Jun 2012)

Page 1 of 3

ATTACHMENT 3 (continued)



Section III – Qualified Receiver

3b. Qualified Receiver Storage Manager Receipt Acknowledgement:

I acknowledge receipt of the unopened labeled container(s) listed herein each with its unique identified and unbroken seal ensuring a continued chained of custody, and after reviewing and agreeing with all the provided supporting documentation, I sign for having received the provided documentation that the sealed containers contained no explosive hazards when received.

Print or type name:	Signature:	Month/Day/Year:

3c. Demilitarization/Destruction Processor Acknowledgement:

I acknowledge receipt of this material and certify and verify that each item or items listed herein were demilitarized and/or destroyed so as to no longer resemble AEDA beyond the requirement listed in DoD 4160.21-M-1 and is only identifiable by its basic content.

Print or type name:	Signature:	Month/Day/Year:

3e. Qualified Receiver Manager Demilitarization/Destruction Certification:

I acknowledge this material has undergone demilitarization/destruction in accordance with DoD 4160.21-M-1and Engineer Manual 1110-1-4009 and that the contents of these sealed containers will not be sold, traded or otherwise given to another party until the contents have been smelted and are only identifiable by their basic content.

Print or type name:	Signature	Month/Day/Year:

4a. Special Instructions:

1. The SUXOS will produce the required number of the original CoC certification copies for distribution in accordance with HGL MMRP SOP 15.03.
2. The *Transporter* will be provided *1 original CoC certification copy* and complete Section II, blocks 2a. through 2.c.with signature.
3. The *Transporter* will turn over *this CoC certification copy* to the Qualified Recycler Manager upon delivery.
4. The *Qualified Recycler Manager* upon receipt of the material will verify and certify the CoC certification document information is complete and accurate by completing Section III. 3a.through 3e. with signatures.
5. The *Qualified Recycler Manager* after completing the demilitarization/destruction of the material listed on this CoC certification document will provide the Generator with a signed company letterhead stating:

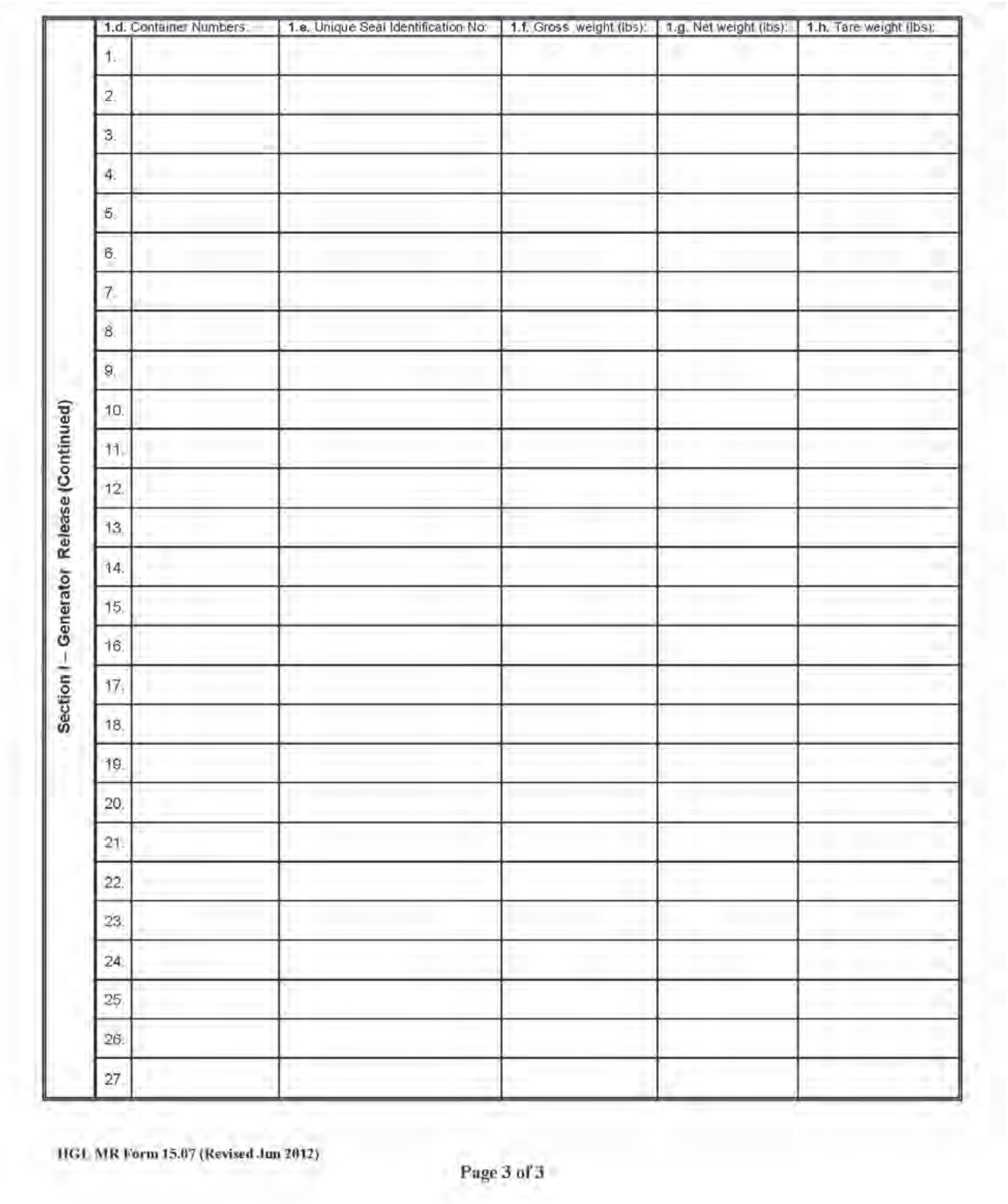
   "Upon receiving the unopened labeled containers each with its unique identified and unbroken seal ensuring a continued chained of custody, and after reviewing and concurring with all the provided supporting documentation, sign for having received and agreeing with the provided documentation that the sealed containers contained no explosive hazards when received. The contents of these sealed containers will not be sold, traded or otherwise given to another party until the contents have been smelted and are only identifiable by their basic content".
6. The *Qualified Recycler Manager* will complete the attached DD FORM 1348–Issue Release/Transfer Document, blocks 22 and 23 and return this form along with this CoC certification document and Certificate of Destruction letter to the Generator address shown in block 1b.

4b. Discrepancies.

HGL MR Form 15.07 (Revised Jun 2012)

Page 2 of 3

ATTACHMENT 3 (continued)



Section I – Generator Release (Continued)

1.d. Container Numbers:		**1.e.** Unique Seal Identification No:	**1.f.** Gross weight (lbs):	**1.g.** Net weight (lbs):	**1.h.** Tare weight (lbs):
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
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26.					
27.					

HGL MR Form 15.07 (Revised Jun 2012)

Page 3 of 3

Material Potentially Presenting an Explosive Hazard, Munitions, and Range Debris Processing Storage Area Inspection Checklist

Site (name, city, and state):		
Inspection conducted by:	Signature:	Date:
Reviewed by Senior UXO Supervisor:	Signature:	Date:

I. INSPECTION ITEM

A. MPPEH and MD Storage Areas	YES	NO	Explain Discrepancies
1. Are exclusion zones maintained during processing?	☐	☐	
2. Are adequate warning signs and boundary markers in place?	☐	☐	
3. Are containers/drums in fair condition?	☐	☐	
4. Are drum pallets in fair condition?	☐	☐	
5. Are containers properly labeled and labels intact?	☐	☐	
6. Are un-inspected items segregated from inspected items to prevent comingling?	☐	☐	
7. Are containers sealed or locked?	☐	☐	
8. Are MDEH items properly segregated and secure?	☐	☐	
9. Are demilitarization items secure?	☐	☐	
10. Is the plastic tarpaulin intact?	☐	☐	
B. RD Storage Area	**YES**	**NO**	**Explain Discrepancies**
1. Are there any pieces or parts of targets missing?	☐	☐	
2. Is there evidence of any disturbance to the Pile?	☐	☐	

HGL HydroGeoLogic, Inc Exceeding Expectations	**CORPORATE TECHNICAL PROCEDURE**	
	Approved for issue by:	
	Process Owner	Digitally signed by Neil Feist Date: 2022.09.26 12:20:18 -05'00'
	Corporate Quality Director	Theresa Rojas Digitally signed by Theresa Rojas Date: 2022.09.26 13:15:25 -04'00'
MEC Anomaly Avoidance Support		**Document No.: HGL SOP 408.507**
		Process Category: Services
		Revision No.: 3
		Effective Date: September 13, 2022
		Last Review Date: September 13, 2022
		Next Review Date: September 2024

1.0 PURPOSE AND APPLICABILITY

This standard operating procedure (SOP) describes the procedures for all HydroGeoLogic, Inc. (HGL) and subcontractor personnel providing munitions and explosives of concern (MEC) anomaly avoidance support during field operations where there is a potential for encountering MEC hazards.

Personnel who use this procedure must complete the SOP Acknowledgement Form (HGL SOP 408.501.F01) and submit it to the Senior Unexploded Ordnance Supervisor (SUXOS) and Unexploded Ordnance Quality Control Specialist (UXOQCS) as evidence that they have read and understand this procedure. Project leaders retain this documentation in the project file.

2.0 SUMMARY OF METHOD

All HGL employees tasked with performing MEC-related activities must qualify in accordance with (IAW) the Department of Defense Explosive Safety Board (DDESB) Technical Paper (TP) 18. Perform all work in a manner consistent with Occupational Safety and Health Administration-established standards and requirements. Conduct all activities in conformance with the project-specific Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP).

This MEC SOP discusses surface and subsurface anomaly avoidance procedures and techniques to be used while conducting munitions response and hazardous, toxic, and radioactive waste (HTRW)-related activities during investigative, design, and remedial actions. Anomaly avoidance techniques must be employed on properties known or suspected to contain MEC or chemical agent to avoid surface and subsurface explosive and chemical hazards. Anomaly avoidance techniques are implemented for activities that include the following:

- Surveying and mapping,
- Environmental and natural resource assessments,
- Surface and subsurface sampling,
- Boring and drilling,
- Groundwater monitoring, and
- Sign and fence installation.

3.0 DEFINITIONS

3.1 DEFINITIONS

Anomaly Avoidance: Techniques employed on property known or suspected to contain MEC or chemical agent, regardless of configuration, to avoid contact with potential surface or subsurface hazards, to allow entry into the area for the performance of required operations.

Hazardous, Toxic, and Radioactive Waste (HTRW) Activities: Activities undertaken for the following:

- The U.S. Environmental Protection Agency's Superfund program,
- The Defense Environmental Restoration Program, including Formerly Used Defense Sites (FUDS),
- Installation Restoration Program sites at active U.S. Department of Defense (DoD) facilities,
- HTRW actions associated with civil works projects, and
- Any other mission or non-mission work performed for others at HTRW sites.

HTRW actions during the investigative/design phase of an HTRW project on a site with known MEC or unknown fillers require anomaly avoidance procedures. MEC removal actions may be required in advance of HTRW activities (construction) on a HTRW project site with known MEC hazards.

Munitions and Explosives of Concern (MEC): Specific categories of military munitions that may pose unique explosives safety risks, including the following:

- Unexploded ordnance (UXO), as defined in 10 United States Code (U.S.C.) § 101(e)(5)(A) through (C);
- Discarded military munitions (DMM), as defined in 10 U.S.C. § 2710(e)(2); or
- Munitions constituents (such as TNT or RDX), as defined in 10 U.S.C. § 2710(e)(3), present in high enough concentrations to pose an explosive hazard.

Munitions Constituents: Any materials originating from UXO, DMM, or other military munitions, including explosive and non-explosive materials, and emission, degradation, or breakdown elements of such ordnance or munitions.

Munitions Debris: Remnants of munitions remaining after munitions use, demilitarization, or final disposition. Examples of munitions remnants include fragments, penetrators, projectiles, shell casings, links, and fins. Munitions debris also includes inert munitions-related material recovered during an MEC removal.

<u>*Recovered Chemical Warfare Materiel (CWM)*</u>*:* Non-stockpiled CWM previously discarded, buried, or fired and discovered either unexpectedly or during planned environmental restoration operations that DoD has either secured in place or placed under DoD control pending final disposition. CWM is normally secured in a DDESB-approved storage location or interim holding facility, pending final disposition.

<u>*Unexploded Ordnance (UXO)*</u>*:* As defined by 10 U.S.C. § 101(e)(5)(A) through (C), military munitions that

- Have been primed, fuzed, armed, or otherwise prepared for action;
- Have been fired, dropped, launched, projected, or placed in such a manner as to constitute a hazard to operations, installation, personnel, or material; and
- Remain unexploded whether by malfunction, design, or any other cause.

<u>*UXO-Qualified Personnel*</u>*:* Personnel who have performed successfully in military explosive ordnance disposal positions or are qualified to perform in the following contractor positions as listed in the Department of Labor's Service Contract Act Directory of Occupations: UXO Technician II, UXO Technician III, UXO Safety Officer, UXO Quality Control Specialist, or Senior UXO Supervisor.

3.2 ABBREVIATIONS/ACRONYMS

APP	Accident Prevention Plan
bgs	below ground surface
CWM	chemical warfare materiel
DDESB	Department of Defense Explosive Safety Board
DMM	discarded military munition
DoD	U.S. Department of Defense
DPT	direct push technology
FUDS	Formerly Used Defense Site
GPS	global positioning system
HGL	HydroGeoLogic, Inc.
HTRW	hazardous, toxic, and radioactive waste

IAW	in accordance with
MEC	munitions and explosives of concern
MMRP	Military Munitions Response Program
PA/SI	Preliminary Assessment/Site Inspection
RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine (also known as Royal Demolition Explosive)
SOP	standard operating procedure
SSHP	Site Safety and Health Plan
SUXOS	Senior Unexploded Ordnance Supervisor
TNT	trinitrotoluene
TP	Technical Paper
U.S.C.	United States Code
UXO	unexploded ordnance
UXOQCS	Unexploded Ordnance Quality Control Specialist

4.0 HEALTH AND SAFETY WARNINGS

Before entering an area requiring MEC anomaly avoidance, the UXO Technician must conduct a safety brief covering emergency procedures, operations, MEC hazards, and anomaly avoidance procedures. If MEC is encountered during any phase of work, follow the procedures in the project planning documents. In general, adhere to the following MEC safety precautions and protocols:

- Observe this cardinal principle when work may involve ordnance, explosives, ammunition, severe fire hazards, or toxic materials: limit the exposure to a minimum number of personnel, for the minimum amount of time, to a minimum amount of hazardous material consistent with a safe and efficient operation.
- DO NOT touch, move, or jar any ordnance items regardless of their markings or apparent condition.
- **Under no circumstances handle any MEC during avoidance activities or move MEC in an attempt to make a positive identification.**
- DO NOT touch, pick up, kick, or move anything that is unfamiliar or unknown.
- DO NOT roll the item over or scrape the item to identify markings.
- DO NOT approach or enter a munitions site if an electrical storm is occurring or approaching. If a storm approaches during site operations, leave the site immediately and seek shelter.

- DO NOT transmit radios or cellular phones in the vicinity of suspect MEC hazards.
- DO NOT walk across an area where the ground surface cannot be seen or that has not been cleared of MEC hazards by the UXO Technician.
- DO NOT rely on color codes for positive identification of ordnance items nor their contents.
- DO NOT drive vehicles into a suspected MEC area until anomaly avoidance techniques have been implemented.
- DO NOT be misled by markings on the MEC item stating “practice” or “dummy.” Practice ordnance can have explosive charges used to mark and/or spot the point of impact, or the item could be marked incorrectly.
- Clearly mark the location of any ordnance item found during anomaly avoidance activities so that it can be easily located and avoided.

— WARNING —

Removing or taking any munitions, explosive, or UXO or munitions-related debris from the site by any employee is strictly prohibited.

5.0 CAUTIONS

5.1 MEC ENCOUNTERED

If MEC is encountered, the UXO Technician performs the following:

- Stops the team, draws attention to the hazard, and marks the hazard with a high-visibility pin flag, paint, or surveyors tape.
- If safe to do so, attempts to identify the MEC hazard via markings and other external features such as shape, size, and external features.
- Records the locations of any MEC hazard items in a Global positioning system (GPS), if possible.
- Photographs the hazard.
- Makes notifications required in the project planning documents.

5.2 MEC DISPOSITION

The UXO escort is not authorized or equipped to perform MEC disposition. MEC discoveries must be reported to the designated personnel/agencies identified in project planning documents. If staff encounter a MEC item that cannot be avoided or that, based on its fuzing or current condition, presents an imminent hazard, the UXO escort must immediately notify the personnel/agencies designated in project planning documents.

6.0 INTERFERENCES

Not applicable.

7.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

For anomaly avoidance on a site with potential MEC, HGL provides a UXO team consisting of a minimum of two personnel, one of whom must be a UXO Technician II or above (see exception in following paragraph). The UXO Technician serves as the UXO Team Leader and has ultimate responsibility for ensuring that all MEC anomaly avoidance support activities are performed IAW this SOP, project planning documents, and/or the APP/SSHP. The UXO Technician directs all MEC anomaly avoidance support during field operations.

A UXO Technician I can provide escort duties if under the supervision of UXO-qualified personnel. (Note: Although escort by a UXO Technician I is typically performed under the supervision of UXO-qualified personnel, the responsible commander or authority may approve UXO Technician I personnel to perform escort duties without supervision. Such approval must be based on an approved risk assessment and implementation of methods to mitigate potential exposures). Escorts will help ensure that MEC on the surface and subsurface anomalies are avoided.

7.1 UXO PERSONNEL

UXO personnel perform the following:

- Provide MEC recognition, location, and explosive safety functions.
- Conduct explosive safety briefing for all site personnel and visitors.
- Conduct a surface and subsurface anomaly avoidance.
- Work closely with U.S. Army Corps of Engineers/client personnel on all MEC-related matters.
- Coordinate and report MEC discoveries IAW project planning documents.

7.2 NON-UXO PERSONNEL

Non-UXO personnel perform the following:

- Obtain training in recognizing the potential hazards associated with MEC.

- Remain with the UXO Technician at all times unless otherwise cleared to work without a UXO escort.
- Follow the instructions given by the UXO Technician if an accident occurs.

Exercise caution when walking on site and follow UXO Technician directions

8.0 EQUIPMENT AND SUPPLIES

8.1 GENERAL EQUIPMENT AND SUPPLIES

The following geophysical equipment is typically used during MEC anomaly avoidance operations:

- Magnetometers such as the Schonstedt GA 52-CX,
- Frequency-domain electromagnetic induction metal detectors such as the White's All Metals Detector, and
- Downhole monitors.

The following supplies are typically used during MEC anomaly avoidance operations:

- Flagging ribbon,
- Pin flags,
- GPS units, and
- High visibility, biodegradable spray paint.

8.2 EQUIPMENT INSPECTIONS

Staff must perform the following equipment inspections:

- Perform a daily equipment function check on all geophysical instruments and GPS equipment. Describe the performance results of the equipment check in the logbook or in an instrument maintenance and calibration log following each functionality test.
- If an equipment function check indicates that any piece of equipment is not operating correctly, and it cannot be field repaired immediately, remove the equipment from service until it can be repaired.

9.0 ANOMALY AVOIDANCE PROCEDURES

Conduct anomaly avoidance procedures during field investigation activities whenever there is a potential for encountering MEC. The purpose of the procedures is to avoid any potential surface and subsurface MEC hazards during these activities. Anomaly avoidance procedures including the following are outlined in the subsections below:

- Establishing site access routes and site boundaries, and conducting MEC avoidance surveys;
- Conducting land surveying and mapping;
- Conducting Preliminary Assessments/Site Inspections (PAs/SIs) on FUDS and Base Realignment and Closure sites;
- Conducting geophysical surveying; and
- Assessing environmental and natural resources:
    - Surface soil sampling,
    - Subsurface soil sampling,
    - Boring and drilling,
    - Groundwater monitoring, and
    - Test pits and trenches excavations.

9.1 ACCESS SURVEY AND MEC AVOIDANCE PROCEDURES

The UXO escort must conduct an access path survey for surface hazards and subsurface anomalies before any type of activities commence, including foot and vehicular traffic. The UXO escort is responsible for conducting the access survey using the following steps:

- Conduct an access survey of the footpath and/or vehicular lanes approaching and leaving work areas with known or suspected MEC. Typically, the access route will be twice as wide as the widest vehicle that will use the route.
- Conduct an access survey around the proposed work site that is large enough to support all planned operations. The size of the area will consider the maneuverability of the equipment and the space required to stage support vehicles or equipment.
- Use geophysical instrumentation capable of detecting the smallest known or anticipated MEC to locate anomalies just below the ground surface that may be encountered because of erosion from rain or because of continual foot or vehicular traffic. If the emplacement depth is greater than the detection capabilities, then the escort must complete the geophysical survey in intervals until the required depth is reached (for example, every 6 inches, 1 foot, 2 feet, and so forth).
- Clearly mark the route(s) for future entry control.
- If anomalies or surface MEC are encountered, mark the items and relocate the work area to an anomaly free area to avoid contact.
- Prohibit personnel from working outside of the surveyed areas.

9.2 CLEARING AND GRUBBING

Initial clearing and grubbing operations may be required before field activities. The objective of clearing and grubbing is to create unhindered access for the field teams. In areas with potential MEC hazards, the UXO Team must perform the following:

- Survey the proposed clearing and grubbing area with a geophysical instrument. Mark hazards with survey flagging or pin flags.
- Begin clearing and grubbing within the area established by the survey.
- Use qualified UXO Technicians to escort grubbing teams at all times.
- Exercise caution when using mechanical grubbing equipment.
- Keep the lowest part of the cutting deck of mechanized equipment at least 6 inches above ground level to avoid potential contact with any MEC hazards remaining after the initial survey.

9.3 LAND SURVEYING AND MAPPING PROCEDURES

The UXO Technician performs the following during land surveying and mapping activities:

- Conducts an access survey of the routes to and from the proposed survey site and an area around the site.
- Visually inspects the surface of each proposed survey point for any indication of MEC or MEC-related contamination.
- Uses a handheld geophysical instrument to assess the presence or absence of subsurface anomalies at the locations where survey points/stakes installation is planned. If responses indicate an anomaly, the UXO Technician disallows survey point/stake installation at that specific location and assists in selecting an alternate location.

9.4 SAMPLING AND DRILLING PROCEDURES

9.4.1 Surface Soil Sampling (Zero to 6 Inches)

The following paragraphs describe anomaly avoidance procedures for surface soil sampling between 0 and 6 inches below ground surface (bgs) in areas with potential MEC.

- Conduct a surface access survey of the routes to and from the proposed investigation site as well as of a support area around the investigation site.
- Visually inspect the surface of each proposed surface soil sampling site for any indication of MEC or MEC-related contamination.

- Survey the proposed sample locations using handheld geophysical instruments.
- Select an alternate location to collect surface soil samples if anomalies are detected at a proposed sampling location or too many anomalies are detected in a general area of interest.

9.4.2 Subsurface Soil Sampling (Below 6 Inches) and Monitoring Well Installation

The following paragraphs describe anomaly avoidance procedures for subsurface soil sampling and monitoring well installations in an area with potential MEC. Subsurface soil sampling is defined as the collection of samples below a nominal depth of approximately 6 inches with a split-spoon, Shelby tube, direct push sampler, or bucket auger (hand auger) soil sampler using drilling techniques. Drilling techniques are also used to install groundwater monitoring wells for HTRW investigations. The UXO team adheres to the following procedures:

- Conduct a surface access survey of the routes to and from the proposed investigation site as well as an area around the investigation site.
- Conduct a subsurface survey of the proposed drill hole location(s) with a handheld, geophysical instrument to detect subsurface MEC anomalies.
- Prominently mark the locations of any anomalies detected with survey flagging or non-metallic pin flags for avoidance.
- Select a new sampling or borehole location if an anomaly is detected.
- Incrementally complete the downhole geophysical survey (for example, every 2 feet) if the subsurface sampling depth is greater than the geophysical detection capabilities.

9.4.3 Incremental Geophysical Survey for Conventional MEC Avoidance

For intrusive sampling (subsurface sampling and well drilling) in areas with suspected MEC, the team completes follows this procedure:

1. **Begin the installation:**
    - Complete the access survey of the area.
    - Complete the geophysical survey and install a pilot hole at the sample or drill location if no anomalies are detected.
    - As long as no anomalies are detected, advance the pilot hole to the maximum reach of the auger or to the maximum depth of the proposed drill hole.
    - During installation, incrementally complete the downhole geophysical survey (for example, every 2 feet) if the subsurface sampling depth is greater than the geophysical detection capabilities.

- When working in impact areas, the UXO team may discontinue incremental screening once a depth of 30 feet bgs is reached or the depth of MEC penetration has been exceeded, whichever is less.
- For all other areas, incremental geophysical screening will be determined based on an assessment of the site's characteristics and history.

2. **If anomalies are detected:**

    - Stop installation immediately and backfill the pilot hole IAW project-specific procedures.
    - Direct HTRW sampling personnel to select a new location.

9.4.4 Test Pits and Trench Excavations

Test pits and trench excavations are used to identify and characterize large subsurface HTRW areas of concern. Adhere to the following procedures:

- Conduct an access survey of the routes to and from the proposed excavation locations.
- If an anomaly is detected, select a new excavation location.
- If the proposed excavation depth is greater than the geophysical instrument detection capabilities, the UXO team proceeds as follows:
    - HTRW personnel can begin excavation in 1-foot increments.
    - At the end of each 1-foot increment, the UXO team screens for anomalies. If an anomaly is detected, the HTRW team must modify the excavation to avoid the anomaly.
    - If MEC is encountered, all operations must cease. The UXO team accesses the item and follows MEC procedures detailed in the project planning documents.
    - After the MEC hazard has been removed, excavation using anomaly avoidance can continue.
- If potentially hazardous waste, debris, or drums are encountered during test pit or trenching operations, stop all excavation activities. The Site Safety Officer adheres to the following procedure:
    - Assess the situation and direct a change to the personal protective equipment for site workers, if necessary.
    - Notify the appropriate personnel IAW the project planning documents.
    - Handle wastes IAW the project planning documents.

9.4.5 Soil Sampling with Direct Push Technology

The following paragraphs describe anomaly avoidance procedures for soil sampling and use of direct push technology (DPT) in areas with potential MEC. Soil sampling with DPT typically involves manual or mechanical penetration at the desired location, followed by withdrawal and collection of a soil sample. The UXO Team adheres to the following procedure:

- Conduct a surface access survey of the routes to and from the proposed investigation site as well as an area around the investigation site.
- Follow the anomaly-avoidance procedures described above for subsurface soil sampling and monitoring well installations as follows:
  - Conduct an incremental down-hole geophysical survey for metallic anomalies.
  - Conduct actual sampling and geophysical instrument screening through the DPT borehole.
  - Backfill the sampling location IAW project-specific procedures after collection of the soil samples.

9.4.6 Groundwater Monitoring

Groundwater monitoring activities include measuring groundwater elevations, measuring free product thickness, and collecting analytical samples. Unless a path is clearly marked, HTRW sampling personnel must be escorted by UXO-qualified personnel when conducting groundwater monitoring/aquifer characterization activities in areas with potential MEC.

9.5 PRELIMINARY ASSESSMENT AND SITE INSPECTION

On sites were MEC hazards may be present, UXO Technicians perform anomaly avoidance measures to prevent non-UXO personnel conducting PA/SI work on the site from contacting MEC hazards.

10.0 DATA AND RECORDS MANAGEMENT

Personnel performing MEC anomaly avoidance activities should maintain copies of daily safety and tailgate briefings. A logbook should be maintained to record information such as daily weather events, performance results of equipment checks, visitors to the site, and instructions from government personnel.

11.0 QUALITY CONTROL AND QUALITY ASSURANCE

The Military Munitions Response Program (MMRP) Operations Manager ensures that this SOP is reviewed at least every two years for completeness, accuracy, and safety. Project Managers and supervisors ensure that all site personnel read, understand, and follow this SOP, and they must bring any discrepancies with procedural steps or safety issues pertaining to this SOP to the attention of the responsible supervisor for corrective action.

The senior UXO-qualified person on site has final on-site authority on all munitions and MEC procedures and safety issues. This individual has direct reporting and communications responsibility with all responsible authorities as directed by the HGL Project Manager.

12.0 REFERENCES

U.S. Department of Defense (DoD), Defense Explosives Safety Regulation 6055.09, Edition 1.

DoD Explosives Safety Board, Technical Paper 18, Minimum Qualifications for Personnel Conducting Munitions and Explosives of Concern-Related Activities.

U.S. Army Corps of Engineers (USACE), Engineer Manual 385-1-97, Explosives Safety and Health Requirements Manual.

13.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
3	September	Initial CMS Library Version

ATTACHMENTS

None.

HGL
HydroGeoLogic, Inc
Exceeding Expectations

STANDARD OPERATING PROCEDURE

Approved by: Dick, Jeff Digitally signed by Dick, Jeff Date: 2020.06.18 16:05:40 -04'00' Corporate Quality Manager

Sampling Equipment Cleaning and Decontamination	**SOP No.: 411.02 (formerly 2.01)**
	SOP Category: Environmental Services
	Revision No.: 5
	Revision Date: June 18, 2020
	Review Date: June 2022

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe field methods to be used for cleaning and decontaminating sampling equipment.

This procedure is specifically applicable to sampling equipment that has been used to collect environmental samples or could have been exposed to contamination that could affect worker safety and/or the integrity of the analytical results of the media sampled.

Other decontamination procedures may apply to a specific project; refer to the project-specific planning documents for project-specific decontamination methods and schedules.

Any deviations from specified requirements must be justified to and authorized by the project manager and/or the relevant program manager and discussed in the approved project plans. Deviations from requirements are documented sufficiently to re-create the modified process.

2.0 SUMMARY OF THE METHOD

This SOP describes the procedures to be followed to achieve effective decontamination as follows: (1) remove contaminants from contaminated surfaces, (2) minimize the spread of contamination to uncontaminated surfaces, (3) avoid any cross-contamination of samples, and (4) minimize personnel exposures. The intent is to accomplish the required level of decontamination while minimizing the generation of additional solid and liquid waste.

3.0 DEFINITIONS

ASTM Type II Water: This is the type of deionized reagent grade water, as defined by ASTM International, used in the final rinse of surfaces of contaminated equipment.

Equipment: Equipment comprises those items (variously referred to as “field equipment” or “sampling equipment”) that are necessary to conduct sampling activities but that do not directly contact the samples.

Laboratory Detergent: This is a standard brand of phosphate-free laboratory detergent such as Liquinox® or Luminox®. Liquinox® is a traditional anionic laboratory detergent used for general cleaning and when there is concern that harsher cleaners could affect the stability of the sampling equipment. Luminox® is a specialized detergent that can remove oils and organic contamination. It may be used in lieu of a solvent rinse step in cleaning equipment for trace contaminant sampling.

Where not specified in these procedures, either detergent is acceptable. The project-specific plans should indicate if Luminox® use is acceptable.

Organic-free Water: This is tap water that has been treated with activated carbon and deionizing units. At a minimum, the finished water must meet the analytical criteria of deionized water, and it should contain no detectable pesticides, herbicides, or extractable organic compounds and no volatile organic compounds above minimum detectable levels for a given set of analyses. Organic-free water obtained by other methods is acceptable as long as it meets the above analytical criteria.

Potable/Tap Water: Potable/tap water is provided by local city sources and is safe for consumption. Chemical analysis of the water source is not required before it is used. Deionized water or organic-free water may be substituted for tap water.

Sampling Devices: This is equipment used to acquire samples.

4.0 GENERAL REQUIREMENTS

All work is performed in accordance with the project-specific planning documents. Refer to the project-specific health and safety plan for relevant health and safety requirements. Any deviations from specified requirements must be justified to and authorized by the project manager and/or the relevant program manager. Deviations from requirements are documented sufficiently to re-create the modified process.

5.0 EQUIPMENT AND SUPPLIES

The following equipment is specific to decontamination requirements and does not include required safety equipment and field documentation described in the site-specific plans. Project-specific plans should be consulted for any additional equipment or deviations from the list below:

- Laboratory detergent,
- Brushes (not wire wound),
- Paper towels/rags,
- Squirt bottles (one for each decontamination fluid),
- 5-gallon buckets or decontamination pad/kiddie pool to contain decontamination fluids,
- Potable water,
- Deionized water,
- Drums or containers for decontamination fluids/solids,
- Drum/container waste labels,
- Sampling containers for decontamination fluid/solid sampling,
- Aluminum foil,
- Steam cleaner, and
- Generator and fuel.

6.0 PROCEDURAL STEPS

Decontamination of sampling devices is performed in a designated decontamination area, removed from any sampling or dedicated office location. This designated area must be in a location free of direct exposure to airborne and radiological surface contaminants and upwind of any field activities that could jeopardize the decontamination procedures or cross contaminate the cleaned equipment.

6.1 GENERAL

The following general rules are followed for decontamination operations:

- Contaminated or dirty sampling devices/equipment should not be stored with or above clean (decontaminated) sampling devices/equipment.
- Clean, decontaminated sampling devices should be segregated from all other equipment and supplies.
- Paint or any other coatings must be removed from any part of a sampling device that may either contact a sample or may otherwise affect sample integrity. After such coatings are removed, the sampling device must be decontaminated using the appropriate method.
- For any of the specific decontamination methods that may be used, the substitution of higher-grade water is permitted (for example, using deionized water in place of tap water). However, deionized water is less effective than tap water in rinsing away detergent during the initial rinse.
- Decontaminated sampling devices and all filled and empty sample containers are stored in locations protected from exposure to any contaminant.
- The method for decontaminating sampling devices and the exterior of sample containers that have been exposed to radioactive material is based on the material contaminated, the sample medium, the radiation levels, and the specific radionuclides to be removed.
- The release of decontaminated sampling devices and sample containers for unrestricted use is based on site-specific criteria. These site-specific criteria should be detailed in the project-specific plans.
- Rags/paper towels used during decontamination activities may become a hazardous waste and require segregation. Refer to the project-specific plans for hazardous waste disposal requirements.
- Sampling devices must be decontaminated before being used in the field to prevent potential cross-contamination of a sample.
- Sampling devices must be decontaminated between samples to prevent cross-contamination.

- Sampling devices must be decontaminated at the close of the sampling event before being taken off site.
- An acceptable alternative to cleaning and decontaminating sampling devices is using items cleaned or sterilized by the manufacturer that are discarded after one use. Care must be exercised to ensure that such previously cleaned or sterilized items do not retain residues of chemical or radioactive sterilizing agents that might interfere with analytical techniques.
- Whenever visible dirt, droplets of liquid, stains, or other extraneous materials are detected on the exterior of a sample container, the exterior surfaces must be decontaminated. This step should be performed before the container is placed in a sample cooler or shipping container.
- For sample containers used in controlled access areas, more rigorous cleaning and/or radiation monitoring may be required before removal from the site. Refer to the project-specific planning documents for details.
- Decontamination fluids/solids as well as other used cleaning supplies, such as paper towels and rags, should be treated as investigation-derived waste and managed in accordance with the project-specific planning documents.

6.2 DECONTAMINATION METHODS

The following decontamination methods are examples of some of those most commonly used in field investigations. Note that the decontamination methods described in this section are for guidance only; the project-specific planning documents and the SOPs referenced in them provide the actual procedures that must be followed. The field operations manager may need to adjust decontamination practices to fit the sampling situation and applicable requirements. All variances from the project-specific planning documents must be approved by the project manager in advance and documented. Procedures for packaging and disposing of all waste generated during decontamination are described in the project-specific planning documents.

6.2.1 Water Level Indicators

The following steps are taken to decontaminate water level indicators. Unless conditions warrant, it is only necessary to decontaminate the wetted portion of the measuring tape. It may be more practical to decontaminate the tape as it is being rewound, but with the reel several feet away from the wellhead (see project-specific planning documents):

1. Wash with detergent and tap water.
2. Rinse with tap water.
3. Rinse with deionized water.

6.2.2 Submersible Groundwater Pumps

The following procedures are taken to decontaminate submersible pumps used to collect groundwater samples. This is the general procedure for non-dedicated pumps, unless the dedicated pump is being removed from the well.

1. Disconnect and discard the previously used tubing from the pump. Wash the pump exterior with detergent and water.
2. Prepare and fill three containers with decontamination solutions consisting of Container 1, tap water and detergent solution; Container 2, a tap water rinsing solution; and Container 3, a deionized water final rinsing solution. The containers should be large enough to hold the pump and 1 to 2 liters of solution. An array of 2-foot-long 2-inch PVC pipes with bottom caps is a common arrangement. Buckets can also be used as long as the water covers the intake screen of the pump. The containers should be labeled to ensure that decontamination is completed in the correct steps. The solutions should be changed at least daily.
3. Place the pump in Container 1. Turn the pump on and circulate the detergent and water solution through the pump and then turn the pump off.
4. Place the pump in Container 2. Turn the pump on and circulate the tap water through the pump and then turn the pump off.
5. Place the pump in container 3. Turn the pump on and circulate the deionized water through the pump and then turn the pump off.
6. Disconnect the power and remove the pump from Container 3.
7. Decontaminate the power lead by washing it with detergent and water, followed by tap water and a deionized water rinse. This step may be performed before washing the pump, if desired.
8. Wind the power lead back on a reel, and place the pump and reel in a clean plastic bag.

6.2.3 Bladder Pumps

The following procedures are used to decontaminate bladder pumps that use disposable bladders. If the bladder pump being used does not have a disposable bladder, the decontamination procedures outlined in Section 6.2.2 should be used.

1. Disconnect and discard previously used tubing from the pump.
2. Completely disassemble the pump, being careful not to lose the check balls, O-rings, ferrules, or other small parts.
3. Remove and discard the pump bladder.

4. Clean all parts with tap water and detergent, using a brush if necessary to remove particulate matter and surface films.
5. Rinse thoroughly with tap water.
6. Rinse thoroughly with deionized water.
7. Install a new pump bladder.
8. Reassemble the pump and wrap it in aluminum foil or store it in a decontaminated pump storage tube.

6.2.4 Small Tools/Samplers

The following procedures are used to decontaminate small tools/samplers (e.g., stainless steel bowls, sample trowels, and hand augers).

1. Wash the tools/samplers with detergent and tap water, using a brush to remove particulate matter and surface film.
2. Rinse thoroughly with tap water.
3. Rinse thoroughly with deionized water.
4. Wrap the tools/samplers in aluminum foil or place them in a clean plastic bag.

6.2.5 Drilling and Direct-Push Technology Sampling Equipment

These procedures are used for drilling and direct-push technology (DPT) sampling activities involving the construction of monitoring wells to be used for collecting groundwater samples or for collecting soil and groundwater samples.

6.2.5.1 Drill and DPT Rig

Any portion of the drill or DPT rig or backhoe over the borehole or sample location that has come into contact with soil or groundwater (mast, backhoe bucket, drilling platform, hoist, cathead) should be steam cleaned (detergent and high-pressure hot water) between boreholes or sample locations. A decontamination pad should be constructed as specified in the project-specific plans to contain soil and decontamination fluids.

6.2.5.2 Downhole Drilling and DPT Equipment

The following is the standard procedure for field cleaning augers, drill stems, rods, tools, and associated equipment.

1. Wash the equipment with tap water and detergent, using a brush if necessary to remove particulate matter and surface film. Steam cleaning may be necessary to remove matter that

is difficult to remove with the brush. Drilling equipment that is steam cleaned should be place on racks above the floor of the decontamination pad. Hollow-stem augers, drill rods, drive casing, and other equipment that is hollow or has holes that transmit water or drilling fluids should be cleaned on the inside with vigorous brushing or steam cleaning.

2. Rinse the equipment with tap water.
3. Remove the equipment from the decontamination pad and cover it with clean plastic or reinstall the equipment on the drill rig.

6.3 QUALITY CONTROL

The effectiveness of the decontamination procedures is monitored by submitting samples of rinse water to the laboratory for low-level analyses of the parameters of interest, also referred to as equipment blanks. An attempt should be made to select different sampling devices each time devices are decontaminated to ensure that a representative sampling of all devices is obtained over the length of the project. Equipment blanks should be collected as specified in the project-specific planning documents.

7.0 RECORDS

Documentation generated as a result of this procedure is collected and recorded in a field logbook in accordance with procedures listed in SOP 300.04: *Field Logbook Use and Maintenance*.

8.0 REVISION HISTORY

Revision 0		Initial Release
Revision 1	December 2010	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2		Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 3	July 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 4	February 2018	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 5	June 18, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 2.01 to 411.02.

HGL
HydroGeoLogic, Inc
Exceeding Expectations

STANDARD OPERATING PROCEDURE

Approved by: Rojas, Theresa Digitally signed by Rojas, Theresa Date: 2020.09.29 13:37:34 -04'00' Corporate Quality Manager

Subsurface Utility Avoidance	**SOP No.: 411.03 (formerly 401.01)**
	SOP Category: Environmental Services
	Revision No.: 3
	Revision Date: September 29, 2020
	Review Date: September 2022

1.0 SCOPE AND APPLICABILITY

This procedure applies to work that involves penetrating the soil surface with powered equipment during drilling or excavation activities. It is permissible to use a client's or facility owner/operator's utility avoidance procedure in lieu of this procedure if it provides equivalent protection.

For overhead utility lines avoidance, see the following procedures:

- HGL H&S Procedure 21: *Excavation and Trenching*,
- HGL H&S Procedure 27: *Drilling Safety*,
- HGL H&S Procedure 32: *Aerial Lift and Elevated Work Platform*, and
- HGL H&S Procedure 40: *Forklifts and Earthmoving Equipment*.

1.1 SUMMARY OF METHOD

This procedure establishes the minimum requirements for avoiding damage to subsurface utilities from unintentional contact with powered equipment.

1.2 HEALTH AND SAFETY WARNINGS

This procedure is not intended to address the hazards associated with subsurface investigation activities. Consult HGL Health and Safety (H&S) Procedure 21: *Excavation and Trenching* and Procedure 27: *Drilling Safety* for safety guidance and requirements. Do not perform intrusive work in areas that may contain unexploded ordnance (UXO) without a UXO escort and clearance by qualified UXO personnel.

Follow the procedures below if a utility is damaged during work (refer to the project Health and Safety Plan or Accident Prevention Plan for project contact information):

- If a gas line has been breached, shut down all nearby equipment that might provide an ignition source.
- Evacuate the immediate area of the breach unless the breached item clearly poses no hazard to personnel, as determined by the Site Safety and Health Officer (SSHO).
- Notify the owner/manager of the utility and emergency services (as appropriate) immediately. Note that in many cases contacting the public utility locating service (using One Call, calling 811, or going online to https://call811.com) will notify the member utility. In some states it is required by law to notify the One Call service.

- If a buried electrical line is cut or damaged, call the power company emergency number for instructions.
- Notify the HGL Project Manager and H&S Director.
- Do not proceed with activities until the situation has been assessed by qualified H&S or utility owner personnel and written permission to resume work has been granted by the Project Manager and H&S Director.

1.3 PERSONNEL RESPONSIBILITIES

The Project Manager is responsible for the following:

- Obtaining any facility-specific requirements/procedures for intrusive work, such as a dig permit;
- Obtaining specifications and "as-built" drawings for any buried lines, utilities, tanks, or other structures at the site and reviewing the proposed locations for drilling or excavation relative to those structures;
- Verifying that if client or facility utility avoidance procedures are to be used, they provide protection that is equivalent to that provided by this HGL SOP;
- Arranging for additional utility location beyond One Call service, such as private utility locating subcontractors, if
    - No accurate utility maps or "as-built" drawings are available,
    - Work is being performed close to high-value or high-hazard buried utilities, or
    - Work is being performed in residential areas, inside buildings, outside of public rights-of-way, or in other locations where unmapped utilities may be present.
- Arranging for UXO escort and UXO clearance if unexploded ordnance may be present;
- Ensuring that utility owner/manager emergency phone numbers are in emergency contact lists; and
- Ensuring that arrangements and procedures for subsurface utility avoidance are addressed during the pre-mobilization readiness review. These include establishing procedures for intrusive activities within 5 feet of a utility; arranging for HGL not to be responsible for damages to subsurface utilities in accordance with the One Call service or facility liability provisions; and obtaining a written waiver from the client or site owner, if needed.

The Field Manager is responsible for the following:

- Contacting the state utility One Call service and/or facility utility program to locate and mark subsurface utilities and hazards at the worksite and to update them during the duration of the work;

- Completing the utility avoidance checklist attached to this SOP before the start of intrusive work;
- Ensuring that fieldwork involving powered drilling or excavation follows this procedure and other applicable requirements including HGL H&S procedures;
- Ensuring that site personnel are trained on the requirements of this SOP;
- Discussing utility-related emergency procedures in the pre-mobilization readiness review and daily safety briefings;
- Ensuring that all drilling or excavation locations are marked using high-visibility paint or some other recognizable and durable marking;
- Reviewing utility maps against field markings and resolving any inconsistencies or questions with the One Call service or facility utility program;
- Verifying at the start of each workday that drilling/excavation and utility markings are intact and clear, and contacting the One Call service or facility utility program to re-mark utilities if necessary;
- Understanding the utility incident reporting requirements for the state and facility where the work is done; and
- Reporting immediately any unintentional contact or damage to subsurface assets or hazards.

1.4 DATA AND RECORDS MANAGEMENT

Steps taken to avoid damaging utilities must be documented in the appropriate records such as the utility avoidance checklist, pre-drilling checklist, inspection checklist from H&S Procedure 21, field logbooks, and photographs, including photographs of the utility marks relative to the boring/excavation prior to the start of intrusive activities. Copies of utility maps, completed dig permits, and other relevant documentation must be kept at the project site and in the project files.

2.0 PROCEDURE

The Field Manager is responsible for executing this procedure on the project site and completing the Utility Avoidance Checklist in Attachment 1 before the start of intrusive work.

Before commencing intrusive work using powered equipment, contact the public utility locating service (using One Call, calling 811, or going online to https://call811.com), the facility's utility program, or a private utility contractor. Utilities not in the public right-of-way are typically not marked by the One Call service.

Complete a walk-over survey of excavation or drilling locations prior to intrusive activities and then visually confirm that known utilities have been marked as appropriate and that markings are consistent with visible cues of possible subsurface utilities including the following:

- Utility posts/line markers,
- Water shutoff valves,
- Sewer cleanouts/manhole covers,
- Discharge pipes,
- Stormwater inlets,
- Irrigation wells and pivots,
- Fire hydrants (hydrants are typically offset from the water main by several feet),
- Junction boxes,
- Electrical poles with conduit into the subsurface,
- Light poles,
- Storage tank vents,
- Transformers, and
- Cuts/patches in pavement.

Determine if proposed drilling or excavation locations are immediately between storage tanks and product dispenser systems, between storage tanks and control units or buildings, between underground storage tanks and tank air vents, between manholes and sewer connections, or between any features that are likely to be connected by a subsurface utility, and if they are, relocate the drilling/excavation locations if possible. Identify facility assets (for example, equipment, control centers, fire suppression systems, vital communication systems, hospitals, polices stations) that may be impacted or harmed if a utility is breached. Know the location of any shutoff valves in the area (for example, irrigation lines). Take photographs of all drilling and excavation locations prior to, during, and after work is complete.

Contact the One Call service or facility utility program if a utility is encountered that has not been marked or communicated to complete the locate and marking for that utility. If a utility is encountered and has not been marked or communicated by the One Call service or facility utility program, notify the Project Manager and H&S Director, who will determine the next step, such as arranging an independent utility survey and notifying the One Call service or facility utility program of the failure.

If a planned intrusive location is within 5 feet of a utility, reposition the work if feasible and request a new utility clearance by the One Call service. Consult the Project Manager before deciding to relocate a planned drilling or excavation location; obtain client approval if necessary. Keep in mind that many utility markings are approximations and that the utilities may be several feet from the markings.

For drilling operations, if it is not feasible to relocate the drilling location, excavate at least the first 5 feet (deeper if it is likely that there are deeper utilities) of boreholes with a low-impact technique such as hand augering, hydrovacing, or air knifing. Pre-excavation of boreholes using low-impact techniques must also be performed under the following conditions:

- The location of utilities is uncertain.
- The work is being done in a residential or high population commercial area.

It is permissible to omit low-impact pre-excavation of boreholes under the following conditions:

- It has been verified that no hazardous (for example, gas, liquid fuel, or electric) or mission-critical communication (for example, fiber optic) subsurface utilities exist within 25 feet of the planned drilling location and that HGL will not be responsible for damages to subsurface utilities in accordance with the One Call service or facility liability provisions; or
- A written waiver has been obtained from the client or site owner.

Situations that do not fit the above criteria should be resolved at the pre-mobilization readiness review. Decisions to forego low-impact pre-excavation of drilling boreholes are subject to Program Manager approval through the issuance of a Subsurface Utility Avoidance memorandum or Field Work Variance. The memorandum must detail the justification to forgo the procedures outlined in this SOP, H&S Procedure 21: *Excavation and Trenching* and H&S Procedure 27: *Drilling Safety*. The revised procedure must be discussed during the readiness review meeting with all task participants, and the signed memorandum must be included with the readiness review form and/or pre-drilling checklist.

Criteria for determining the need to pre-excavate boreholes are summarized below:

Criterion	Utility Location	Other Condition	Decision
a	Within 5 feet		Pre-excavate
b	Between 5 and 25 feet	Uncertain if utilities are present	Pre-excavate
c	More than 25 feet	No hazardous or high-value utilities are present	May skip pre-excavation
d	Uncertain	Residential or high-population commercial	Pre-excavate
e	Uncertain	No hazardous or high-value utilities are present; HGL liability waived	May skip pre-excavation
f	Uncertain	Not d or e	Site-specific; resolve at pre-mobilization readiness review and document in review minutes

For excavation operations, if utilities are located within the planned excavation or within 5 feet of the limits of the excavation, the precise location of those utilities must be determined by excavating with low-impact tools such as hand auger, shovel, hydrovac or air knife. This may be necessary at several locations within the excavation area to confirm that the apparent route and depth of the utility do not change. **If a utility extends throughout the area to be excavated, the utility must be exposed to confirm its location and depth at least once every 10 feet. The utility must be exposed continuously, using low impact techniques, when performing powered excavation within 5 feet of the utility.**

HGL must inspect excavations managed by subcontractors at sufficient frequency and at least daily to confirm that the subcontractor is complying with these requirements and must require the subcontractor to make corrections if they are not in compliance.

If subsurface obstructions prevent reaching a depth of 5 feet using low-impact techniques, verify that the obstruction itself is not a utility (for example, a concrete sewer pipe versus concrete rubble). Conversely, if there is a credible probability that utilities are present at depths greater than 5 feet, the low-impact excavation may be continued to greater depths. **It is not permissible to omit low-impact excavation due to a lack of suitable equipment.**

Inspect the low-impact excavation and excavated material for indications of utilities, such as the edge of a pipe visible in the sidewall of the excavation or the presence of pea gravel that may be pipe bedding. If a subsurface utility is unintentionally encountered at any time during a low-impact or powered boring or excavation, cease all work in the immediate area and contact the SSHO and Field Manager.

Any material generated during pre-excavation activities is managed in accordance with the project-specific planning documents.

Maintain and protect markings for utility locations during the work. If utility markings are weathered away or removed, or if the location or boundaries of the activity change, repeat the locating processes and replace the markings. Many utility incidents occur when the boundaries of excavations are changed or the marked utility locations wear off.

3.0 REFERENCES

HGL, H&S Procedure 21: *Excavation and Trenching*.
HGL, H&S Procedure 21.1: *Excavation and Trenching*, Appendix A, Inspection Checklist.
HGL, H&S Procedure 27: *Drilling Safety*.
HGL, H&S Procedure 32: *Aerial Lift and Elevated Work Platform*.
HGL, H&S Procedure 40: *Forklifts and Earthmoving Equipment*.

3.0 REVISION HISTORY

Revision 0	July 2016	Initial Release
Revision 1	May 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	June 1, 2018	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 3	September 29, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 401.01 to 411.03.

ATTACHMENTS

Attachment 1 – Utility Avoidance Checklist

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ATTACHMENT 1
UTILITY AVOIDANCE CHECKLIST

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UTILITY AVOIDANCE CHECKLIST

Date: ____________________ Project/Site: ______________________________

Field Manager: ______________________________

Work to be Performed: ______________________________

Consideration	Y	N	Explanation	Initial
1. Has a dig permit been obtained and approved?				
2. Has state One Call service been contacted?				
3. Has facility utility program been contacted?				
4. Has a private utility locating survey been conducted?				
5. Have as-built drawings been reviewed for utilities or subsurface hazards (e.g., USTs)?				
6. Has a visual inspection of the work area(s) been completed, including taking photographs?				
7. Have all known utilities and subsurface hazards been clearly marked?				
8. Has a visual inspection indicated the possible presence of other utilities or subsurface hazards?				
9. Are intrusive activities being conducted within 5 feet of a utility?				
10. If Item 9 is YES, can activity be relocated?				
11. Are any final drilling locations within 5 feet of a utility; are utility locations uncertain or working in residential or high population area? If YES, excavate first 5 feet using low-impact techniques				
12. Are any utilities within 5 feet of the excavation limits? If YES, determine precise location with low-impact techniques.				
13. Can drilling proceed WITHOUT excavating the upper 5 feet with low-impact techniques? Explain why.				
14. If working near overhead power lines, is a minimum clearance of 20 feet being maintained?				
15. Has written approval been granted by the Program Manager to deviate from SOP 411.03? Attach to checklist.				
Other considerations:				

HGL
HydroGeoLogic, Inc
Exceeding Expectations

STANDARD OPERATING PROCEDURE

Analog and Digital MEC Operations

SOP No.: 501.05 (formerly 506.01)
SOP Category: MMRP and Geophysics
Revision No.: 2
Revision Date: October 16, 2019
Review Date: October 2021

Approved By:

Jeff Dick
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Date: 2019.10.16 10:36:58 -04'00'

10/16/2019
Date

Jeff Dick, P.E.
Corporate Quality Manager

Digitally signed by Neil Feist
Date: 2019.10.16 09:38:49 -05'00'

Date

Neil Feist
MMRP Operations Manager

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# Analog and Digital MEC Operations	**SOP No.: 501.05 (formerly 506.01)**
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1.0 PURPOSE

This standard operating procedure (SOP) establishes standard safe practices for performing munitions and explosives of concern (MEC) operations during munitions response projects conducted by HydroGeoLogic, Inc. (HGL) unexploded ordnance (UXO)-qualified personnel.

2.0 SUMMARY OF METHOD

All HGL employees tasked with performing MEC-related activities must be qualified in accordance with (IAW) Department of Defense Explosive Safety Board Technical Paper 18. They must perform all work in a manner consistent with Occupational Safety and Health Administration-established standards and requirements, and they must conduct all activities in conformance with the project-specific Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP).

Analog data collection and clearance operations conducted on the surface or subsurface are typically executed within a grid system or along transects. Anomalies resulting from analog or digital geophysical mapping (DGM) targets are pinpointed, investigated, documented, and resolved IAW the project planning documents. When DGM targets exist, intrusive teams perform the process of anomaly resolution, which includes reacquiring the anomaly and excavating it (uncovering, identifying, documenting, and clearing). This process is followed by quality control (QC) post-dig hole checks performed by QC personnel to provide independent verification that the source of the anomaly was removed during intrusive operations. The process of anomaly resolution for DGM targets is covered in this SOP and in SOP 551.01: DGM Using a Geonics EM61-MK2.

This SOP primarily details the procedures and safety measures required for analog and digital clearance of munitions-related materials on the surface and subsurface. This SOP also details the general data collection, documentation, and reporting requirements for these tasks.

3.0 DEFINITIONS AND ABBREVIATIONS/ACRONYMS

3.1 DEFINITIONS

Essential Personnel: U.S. Department of Defense and contractor personnel necessary for the safe and efficient completion of field operations conducted in an exclusion zone (EZ). Multiple multidisciplinary MEC teams project performing project tasks may be in the EZ while MEC procedures are being performed as long as team separation distances (TSDs) are maintained.

Exclusion Zone (EZ): A safety zone established around a MEC operations work area. Only essential personnel and authorized/escorted visitors are allowed within the EZ.

- Examples of EZs include safety zones around MEC intrusive activities and safety zones where MEC is intentionally detonated.
- For chemical warfare material projects sites, the EZ is the area within the no significant effects zone.

Munitions and Explosives of Concern (MEC): Specific categories of military munitions that may pose unique explosive risks, including the following:

- UXO, as defined in 10 U.S.C. § 101(e)(5);
- Discarded military munitions, as defined in 10 U.S.C. § 2710(e)(2); or
- Munitions constituents (for example, TNT, RDX) as defined in 10 U.S.C. § 2710(e)(3), present in high enough concentrations to pose an explosive hazard.

MEC Operations: Defined as MEC identification; access procedures such as excavation, either by hand or using heavy equipment; handling of UXO, explosives or explosive items; or disposal, including movement, transportation, and final disposal of MEC.

Minimum Separation Distance (MSD): The distance that personnel in the open must maintain from an intentional or unintentional detonation.

Team Separation Distance (TSD): The distance that teams of essential personnel must be separated from one another during the conduct of MEC activities on a Military Munitions Response Program (MMRP) site. Normally this the K40 distance of the munitions with the greatest fragmentation distance (MGFD) for the site.

Unexploded Ordnance (UXO): As defined by 10 U.S.C. § 101(e)(5)(A) through (C), UXO includes military munitions that

- Have been primed, fuzed, armed, or otherwise prepared for action;
- Have been fired, dropped, launched, projected, or placed in such a manner as to constitute a hazard to operations, installations, personnel, or material; and
- Remain unexploded whether by malfunction, design, or any other cause.

A more detailed description of the term UXO is provided in Public Law 106-65, § 3031 (c)(5)(A)

3.2 ABBREVIATIONS/ACRONYMS

APP	Accident Prevention Plan
BIP	blow-in-place

DGM	digital geophysical mapping
EMM	earth moving machinery
EZ	exclusion zone
GIS	Geographical Information System
GPS	Global Positioning System
HGL	HydroGeoLogic, Inc.
IAW	in accordance with
MD	munitions debris
MEC	munitions and explosives of concern
MGFD	munitions with the greatest fragmentation distance
MMRP	Military Munitions Response Program
MPPEH	material potentially presenting an explosive hazard
MQO	measurement quality objective
MSD	minimum separation distance
PPE	personal protective equipment
QC	quality control
RTK	real time kinematic
RTS	robotic total station
SOP	standard operating procedure
SSHP	Site Safety and Health Plan
SUXOS	Senior UXO Supervisor
TSD	team separation distance
UXO	unexploded ordnance
UXOQCS	UXO Quality Control Specialist
UXOSO	UXO Safety Officer

4.0 PERSONNEL

This section describes the personnel, responsibilities, and qualifications required to implement this SOP. The following individuals are involved in operations:

- **Senior UXO Supervisor (SUXOS) –** Responsible for ensuring that all intrusive operations are performed IAW this SOP and the project planning documents.
- **UXO Safety Officer (UXOSO) –** Communicates directly with the Corporate Health and Safety Director on all matters concerning safety and ensures that all explosive safety procedures are performed IAW project planning documents.
- **UXO Field Team Leader –** Directs intrusive teams during field operations.
- **UXO Quality Control Specialist (UXOQCS) –** Communicates directly with the Quality Manager on all matters concerning quality and signs off (UXOQCS or designee) on completeness and accuracy of intrusive documentation.
- **UXO Technician –** responsible for performing intrusive operations IAW the SOPs and the project planning documents.
- **Data Processor/Analyst or Geographical Information System (GIS) Specialist –** Uploads and checks intrusive data for completeness and accuracy; works with UXO Team to resolve any discrepancies.

5.0 EQUIPMENT AND SUPPLIES

The following equipment is typically used during clearance operations:

Detector(s):

- Flux-gate magnetometers:
  - Schonstedt GA 52-CX
  - Schonstedt GA 72-CD
  - Forester FEREX 4.032
  - Ebinger MAGEX 120 LW
  - Vallon EL 1302D1 or 1303D
  - Chicago Steel Tape (Magna-Trak 102)
- Frequency-Domain Electromagnetic Induction Metal Detectors:
  - White’s All Metals Detector
  - Fisher 1266X
  - Garrett
  - Foerster Minex
  - Minelab Explorer II
- Production-Time Domain Electromagnetic Induction Metal Detectors
  - Geonics EM61 (various models)
  - Schiebel AN PSS-12

  - Vallon VMH3
  - Minelab F3

- Positioning system (if applicable): Global Positioning System (GPS) with survey areas (transects or grids) uploaded for visual reference; real time kinematic (RTK) GPS or robotic total station (RTS) for reacquisition of DGM targets.
- Measuring tapes (if local grid is to be used).

6.0 SAFETY

During MEC clearance operations, HGL personnel must adhere to the APP/SSHP and the following general safety practices:

- Conduct operations only during daylight hours.
- Allow only qualified UXO technicians to handle MEC.
- Do not conduct MEC operations until all applicable plans for the site in question are prepared and approved.
- Conduct operations on the concept of limiting exposure to the minimum number of personnel, for the minimum amount of time, to the minimum amount of MEC consistent with safe and efficient operations.
- Before taking any action on an ordnance item, definitively identify all fuzing, including fuze type by function and the physical state/condition (armed or unarmed) of the fuze (for example, burned, broken, parts exposed/sheared).

The following practices also apply:

- All personnel must attend the Daily Safety Briefing before entering the operating area.
- Anyone who observes an unsafe act or situation can stop operations.
- Any safety violation and/or unsafe act/practice must be reported to the UXOSO immediately.

6.1 MEC SAFETY PROTOCOLS

Death or injury can occur from MEC and explosives-related accidents. MEC exposed to the elements for an extended period can become more sensitive to shock, movement, and friction because the stabilizing agent in the explosives may be degraded. The general MEC safety precautions and protocols are as follows:

- Remain alert at all times for MEC and related scrap or material potentially presenting an explosive hazard (MPPEH) hazards.

- Observe the cardinal principle of limiting the exposure to a minimum number of personnel, for the minimum amount of time, to a minimum amount of hazardous material consistent with a safe and efficient operation during activities involving ordnance, explosives, ammunition, severe fire hazards, or toxic materials.
- Always assume that MEC hazards contain a live charge until determined otherwise.
- Clearly mark the location of any ordnance item found so it can be easily located and avoided.
- Follow the procedures of the approved project planning documents.
- Upon locating any MEC hazards, immediately notify the UXO technician to take appropriate measures.
- Consider MEC that has been exposed to fire as extremely hazardous. Chemical and physical changes to the contents may have occurred that render it more sensitive than its original state. DO NOT touch, move, or jar any ordnance items regardless of the markings or apparent condition. Under no circumstances handle any MEC during avoidance activities or move it in an attempt to make a positive identification.
- DO NOT touch, pick up, kick, or move anything unfamiliar or unknown.
- DO NOT roll an unknown item over or scrape the item to identify markings.
- DO NOT approach or enter a munitions site if an electrical storm is occurring or approaching. If a storm approaches during site operations, leave the site immediately and seek shelter.
- DO NOT walk across an area where the ground surface cannot be seen and that has not been cleared of MEC hazards by a UXO technician.
- DO NOT rely on color codes for positive identification of ordnance items or their contents.
- DO NOT be misled by markings on a MEC item stating, “practice bomb,” “dummy,” or “inert.” Practice ordnance can have explosive charges used to mark and/or spot the point of impact, or the item could be marked incorrectly.

– WARNING –

Removing or taking any munitions, explosives, or UXO or munitions-related debris from the site by any employee is strictly prohibited.

6.2 OTHER HAZARDS

Hazards that may be present during MEC operations may include sharp metal, industrial chemicals, and other hazards as described below:

- *Metal Debris*: Metal debris, to include munitions debris (MD) and other debris (for example, nails, banding, barbed wire) are typically encountered during intrusive operations. Wear the appropriate personal protective equipment (PPE) to protect personnel from hazards caused by sharp objects.
- *Chemicals*: Locating industrial-type chemicals is a possibility during intrusive MEC activities. If sealed drums, contaminated soils, or other suspect conditions that indicate a potential health or safety hazard are encountered, do the following:
    - Stop work and follow proper notification procedures. The SUXOS notifies the Project Manager using established notification procedures.
    - Do not continue work in the area where the hazard was discovered until the SUXOS and UXOSO evaluate the situation and confer with the Project Manager and HGL's Corporate Health and Safety Director, and all agree that it is safe to proceed.

6.3 EXCLUSION ZONES AND MINIMUM SEPARATION DISTANCES

6.3.1 Exclusion Zones

During intrusive MEC operations, only essential project personnel may be within the EZ.

- The UXOSO and UXO Team Leaders monitor and keep the EZ intact until intrusive operations are complete.
- If a MEC item larger than the identified MGFD is encountered, notify the appropriate authority and use an EZ appropriate for the found munition.

6.3.2 Essential Personnel

Only project personnel necessary for the safe and efficient completion of field operations are allowed in an EZ. Multiple multidisciplinary MEC project teams performing tasks required to execute the project may be in the EZ while MEC procedures are being performed as long as TSDs are maintained. Team locations must be closely coordinated with the SUXOS and UXOSO. Personnel are not allowed to work in the EZ without the following:

- Briefing on the use of the buddy system,
- PPE IAW the APP/SSHP,
- Applicable training and certifications,
- Understanding of the APP/SSHP, and
- Approval of SUXOS.

6.3.3 Minimum Separation Distance

6.3.3.1 Unintentional Detonations

All nonessential personnel should be evacuated from within the EZ/MSD during intrusive operations in areas with known or suspected MEC.

6.3.3.2 Intentional Detonations

Evacuate all personnel from within the EZ/MSD during intentional detonation of MEC items.

7.0 PROCEDURES

7.1 EQUIPMENT FUNCTION CHECKS

Upon arrival at the site, verify the condition and functionality of the analog detectors as follows:

- Inspect all instruments and equipment that requires maintenance and/or calibration upon arrival, regardless of source, and periodically as required in the manufacturers' equipment manual.
- Check instrument and equipment functionality to ensure operational readiness IAW project plans.
- Remove or replace equipment from service if a functionality check indicates that it is not operating correctly and that it cannot be repaired in the field immediately.
  - Remove the item from service until it can be repaired, or
  - Replace the equipment item with a like model or an approved substitute that meets the same specifications for accuracy and precision as the item removed from service.

7.1.1 Instrument Function Checks

Check analog detectors at least daily by using the instrument in a designated instrument test plot (function check area) to verify response to known target(s). Instrument settings are determined based on the response results from the test plot. After completing the function check, document the instrument type, serial number, and results IAW project planning documents. General procedures include the following:

- Turn on the instrument to warm up the system electronics.
- Ensure that the required sensitivity settings are selected.
- Spot check readings to locate a noise-free background area.
- Perform any nulling procedures.
- Perform a personnel metal interference source check.
- Survey the test strip.
- Perform a known position check for the positioning system (if applicable).
- Document instrument tests IAW project planning documents.
- Perform post-data collection QC tests and document the findings (as required).

7.2 EARTHMOVING EQUIPMENT

7.2.1 For Removing Soil Overburden

Earth moving machinery (EMM) may be used to excavate overburden from suspected MEC. Do not use EMM to excavate within 12 inches of a suspected MEC item. Once the EMM is within 12 inches of the suspected MEC, complete the excavation using hand excavation methods. Personnel who are not UXO qualified may operate the EMM only when supervised by a UXO Technician III or higher.

- If more than one EMM is to be used on site, the same MSD for multiple work teams applies.
- There is no need to harden/shield the EMM to protect its operator when EMM is used to remove soil overburden to within 12 inches from the anomaly.

7.2.2 For Intentional Excavation of MEC

If the intent of the mechanized MEC procedure is to intentionally dig up anomalies that could be MEC without practicing MEC avoidance techniques, the equipment must be hardened/armored appropriately. The operator must also be afforded protection for blast overpressure to the K24 factor. If hearing protection is used, the K18 factor can be used.

If mechanized MEC procedures are being performed, the MSD for unintentional detonations for nonessential personnel must be the maximum fragmentation range-horizontal.

7.3 ANALOG MEC CLEARANCE PROCEDURES

During analog MEC clearance activities, the UXO technicians perform the following:

- Operate the geophysical instrument at a pace that ensures that the entire lane is searched and that the instrument is able to appropriately respond to anomalies.
- Use geophysical instruments to locate and pinpoint the anomaly.

- For subsurface anomalies, carefully remove the earth overburden to expose the source of the subsurface metallic anomaly and positively identify the source of the anomaly.
- Ensure anomalies are resolved IAW the project planning documents.

The UXO Team Leader will periodically perform QC checks behind the UXO team to ensure that the MEC clearance objectives detailed in the project planning documents are achieved.

7.3.1 Analog Grids

Teams implement the MEC surface or subsurface clearance by establishing lanes and sweeping the lanes using analog geophysical instruments. Teams establish the lanes by laying lines (ropes) or other suitable means such as marking lanes with pin flags. Unless otherwise noted, lanes must be established at a maximum of 5-foot intervals to ensure 100 percent coverage of the clearance footprint. The SUXOS typically determines which techniques will be used to mark and sweep boundaries based on site conditions.

Data collected during analog removal actions typically consist of the UXO Team Leader documenting the following on grid sheets:

- Estimated weight of MD and range related debris in grid;
- Estimated weight of other debris (for example, trash, scrap metal) in grid;
- Item description, condition, location coordinates, and disposition;
- Digital photos of MEC found during the investigation; and
- Significant items such as obstacles, structures, seeds, and detonation locations.

While not typically required for MEC removal actions, some projects require detailed documentation for each individual anomaly encountered during analog investigations. Some additional requirements could include the following:

- If required in project planning documents, grid corner locations (position coordinates) must be measured and recorded with a GPS unit and uploaded to the GIS database at the end of each survey day.
- If required in project planning documents, each anomaly investigated must be documented on digital media (for example, PDA, tablet computer) or on a hard copy dig sheet. Section 7.4.2 documents the general procedures used to recover the object(s) that caused the anomaly.
- If required in project planning documents, relevant information must be transferred to the HGL GIS at the end of each analog survey day (for example, craters, cultural features, and impassable areas).

7.3.2 Analog Transects

The team will either use predetermined virtual transects preloaded on GPS or establish transects IAW project planning documents. Teams will maintain a survey speed (~1 mile per hour or adjusted to match terrain) that allows adequate detection of subsurface anomalies. Personnel will systematically sweep the area along each transect using the appropriate sensitivity settings for the detector.

While not typically required for MEC removal actions, some projects require detailed documentation for anomalies encountered during analog investigations. Some additional requirements may include the following:

- If required in project planning documents, each anomaly investigated must be documented on digital media (for example, PDA, tablet computer) or on a hard copy sheet. Section 7.4.2 documents the general procedures used to recover the object(s) that caused the anomaly.
- If required in project planning documents, relevant information must be transferred to the HGL GIS at the end of each analog survey day (for example, craters, cultural features, and impassable areas).

7.4 DIGITAL MEC CLEARANCE PROCEDURES

There are three key aspects to digital anomaly resolution: anomaly reacquisition, anomaly excavation (including reporting dig results), and post-dig verification sampling.

7.4.1 Anomaly Reacquisition

Anomaly reacquisition is a critical element because this task must physically match anomalies on dig lists with their sources. To resolve all anomalies on the dig list and achieve project-specific measurement quality objectives (MQOs), the UXO intrusive team often must clear the entire footprint of an anomaly. The UXO intrusive team typically performs the following reacquisition process when investigating anomalies with a metallic source:

- Use the approved method to navigate to the selected location, typically an RTK GPS, an RTS, or measuring tapes.
- Reproduce the DGM signal at the target location with the approved sensor referenced in the project planning documents.
- Place a plastic pin flag and/or paint the ground surface near the reacquired source (usually the coordinates of the peak intensity) and document peak intensity, offset, and direction from dig sheet coordinates.

A more thorough discussion of anomaly reacquire is presented in SOP 551.01: DGM Using a Geonics EM61-MK2.

When investigating anomalies where the source is not metal, the intended coordinates for the excavation are occupied and a visual marker (pin flag, etc.) is placed at the location to guide the start of the excavation.

7.4.2 Digital Anomaly Excavation (Metallic Anomalies)

The disposition and final location details of each anomaly excavated are normally recorded on hard copy dig sheets. The reported dig results will ultimately be reviewed by the geophysical team, which has the authority to require that additional reacquisition and/or excavation activities be performed for anomalies having characteristics ambiguously explained by the reported dig results. The UXO intrusive team typically conducts anomaly excavation as follows:

1. Excavate each anomaly from the side of anomaly location and carefully remove overburden to expose anomaly features for evaluation.
2. If suspect MEC is encountered, the UXO Technician II and UXO Technician III (team leader) determine the item’s condition. If determined to be MEC, notify the SUXOS and follow the MEC disposition guidance in the project planning documents.
3. Record the anomaly characteristics required on the dig sheets. Typical information required on the dig sheets includes the following:
    a. Team number,
    b. Date investigated,
    c. Anomaly type (for example, UXO, MD, cultural debris, seed, other (no find, grid corner nail),
    d. Source depth, approximate dimensions and/or weight,
    e. Quantity,
    f. General description of anomaly (for example, water table at 1 foot, rocky soil, hard ground),
    g. Post-dig instrument reading (for example, mV for EM61).
4. Collect, process, and dispose of the anomaly item(s) per the project planning documents. Adhere to the following procedure for source removal:
    a. If a recovered anomaly is classified as MEC, only the SUXOS and UXOSO can determine whether the item is acceptable to move.
    b. If the item **can** be safely moved, remove and relocate the item pending further disposition, depending on project planning document guidance, and inspect under

the removed item to ensure additional source(s) are removed from the anomaly footprint.

   c. If the item **cannot** be safely moved, designate it blow-in-place (BIP) and execute the BIP procedure. Do not move MEC designated as BIP for any reason. If needed, use engineering controls to decrease the possibility of damage to personnel or property. Upon disposition, inspect the anomaly location to ensure anomalies are removed from the anomaly footprint.
   d. Collect anomalies classified as MPPEH for further inspection.
5. Dispose of all MEC and MPPEH IAW the approved procedures in the project planning documents.
6. Photograph the MEC items.
7. If required, after removing the anomaly sources, ensure that the entire anomaly footprint achieves the post-dig MQO(s) requirements IAW project planning documents.

7.4.3 Digital Anomaly Excavation (Non-Metallic Anomalies)

The disposition and final location details of each anomaly excavated are normally recorded on hard copy dig sheets. The reported dig results are ultimately reviewed by the geophysical team, which has the authority to require that additional excavation activities be performed for anomalies having characteristics ambiguously explained by the reported dig results.

The team leader in charge of the excavation decides in the field when the excavation can be stopped or meets the requirements in the planning documents. The team leader compares the excavation results with historical documentation/reports in the field (e.g., predictions of subsurface properties based on geophysical survey(s)) to ensure the results are representative). Excavations generally proceed as follows:

1. Carefully remove overburden at a marked location to a depth of approximately 1 foot (or as directed by the team leader) using hand-digging tools or an excavator.
2. Watch for materials that may pose an immediate safety hazard. If any containers are identified (e.g., plastic drums, vessels, containers) notify the UXOSO before advancing the excavation.
3. During the excavation, fill out the test pit/trench log or dig sheet, document significant findings at specified depth intervals (lithology and color, relative moisture content, changes in color of soils or relative moisture, manufactured materials, etc.), and collect any required samples at the depth interval specified in the planning documents.
4. Take digital photographs of the test pit/trench and contents before any contents are removed. Record photo ID and pertinent information on the test pit/trench log or dig sheet.

   a. If the item(s) **can** be safely removed relocate the item(s) pending further disposition.
   b. If the item(s) **cannot** be safely removed advance the excavation around the item(s), if possible.

5. When the excavation is finished, sketch the shape, approximate dimensions, and orientation with respect to North. Take digital photographs of the sides and bottom of the excavation.
6. Take digital photographs of significant items removed from the excavation that might allow additional information to be gathered (e.g., date stamp, model or serial number, product type).

7.4.4 Post-Dig Anomaly Resolution

If specified in the project planning documents (e.g., QAPP, Work Plan), post-dig anomaly resolution is performed after excavation by the intrusive team by the UXOQCS (or designee) to verify that the source(s) of the anomaly have been adequately resolved and achieved the MQO for anomaly resolution. The same technology and instrument used to identify anomalies should be used to verify that the anomalies have been resolved.

The project planning documents detail the number of anomalies that required post-dig verification. The UXOQCS (or designee) implements the post-dig anomaly resolution process IAW project planning documents.

7.5 MEC AND MPPEH PROCEDURES

Upon encountering a MEC item, a minimum of one UXO Technician II and one UXO Technician III will identify and mark the item for future disposition IAW the approved project planning documents. Only the SUXOS and UXOSO, jointly, will determine if a MEC item is acceptable to move.

7.5.1 MEC Disposal

Conduct all MEC disposal procedures IAW the project specific planning documents (Work Plan, and when applicable, ESP or ESS). The SUXOS is responsible for coordinating with the proper authorities for the final disposition of all MEC hazard(s) discoveries. Refer to the following HGL SOPs for MEC disposal and explosives procedures:

- SOP 501.01: Explosive Materials Accountability and Management
- SOP 501.03 (formerly 503.01): Explosives Storage Inspections and Security
- SOP 502.01: Explosive Demolition Operations

7.5.2 MPPEH Processing

Process all MPPEH IAW the project specific planning documents. MPPEH will be processed IAW HGL SOP 504.01: MPPEH Inspection and Management, as well as any other applicable laws, regulations and restrictions.

8.0 REPORTING

Reporting of the activities associated with this SOP consist of copies of the field notes/logbooks and hard copy forms or digital files with the intrusive results. The UXO Field Team Leader must maintain a record of site conditions and metadata in either written form (for example, field logbook) or digital form (for example, tablet-based forms), and the UXOQCS must review the intrusive records for completeness and accuracy at regular intervals during project execution. Typically, the following types of information will be documented:

- Date,
- Weather,
- Field team members,
- Instrument types, serial numbers, and sensitivity settings,
- Equipment function test results,
- Potential sources of interferences,
- Intrusive results (e.g., anomaly type, depth, characteristics),
- GPS file names and track paths (if applicable), and
- Location and description of obstacles or inaccessible areas where anomalies could not be investigated.

9.0 QUALITY CONTROL

The MQOs for intrusive operations and testing of analog and digital detectors are presented in the project planning documents. Performance relative to the MQOs is assessed during the processing of the collected data. The SUXOS or UXOQCS uses the three-phase inspection process during intrusive operations.

10.0 REFERENCES

U.S. Army Corps of Engineers (USACE), Engineer Manual 200-1-15.

USACE, Engineer Manual 385-1-97.

U.S. Department of Defense, Defense Explosives Safety Regulation 6055.09, Edition 1.

U.S. Department of Defense Explosive Safety Board (DDESB), Technical Paper 16.

DDESB, Technical Paper 18.

11.0 REVISION HISTORY

Revision 0	August 2018, Initial Release.
Revision 1	Revised to reflect the replacement of DoD 6055.09-M with *Defense Explosives Safety Regulation 6055.09, Edition 1*.
Revision 2	Revised to include general procedures when using test pits/trenching to investigate non-metallic anomalies.

HydroGeoLogic, Inc
Exceeding Expectations

STANDARD OPERATING PROCEDURE

Vegetation Removal Operations on MMRP Sites	**SOP No.: 501.06 (formerly 509.01)**
	SOP Category: MMRP and Geophysics
	Revision No.: 2
	Revision Date: December 29, 2021
	Review Date: December 2022

Approved By:

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Theresa Rojas
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Date

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Neil Feist
MMRP Operations Manager

Date

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1.0 PURPOSE

This standard operating procedure (SOP) describes the procedures for safely conducting vegetation removal operations on a munitions site.

2.0 SUMMARY OF METHOD

This SOP applies to HydroGeoLogic, Inc. (HGL) employees who are involved in vegetation removal activities by manual removal, mechanized removal, or controlled burning. All HGL employees tasked with performing munitions or explosives of concern (MEC) avoidance support during vegetation removal activities must be qualified in unexploded ordnance (UXO) in accordance with (IAW) U.S. Department of Defense Explosive Safety Board (DDESB) Technical Paper (TP) 18.

HGL staff must perform all work in a manner consistent with Occupational Safety and Health Administration (OSHA) standards and requirements. Staff should refer to the project site-specific health and safety plan for relevant health and safety requirements. All activities must be conducted in conformance with the approved project planning documents, including the Accident Prevention Plan (APP), the Site Safety and Health Plan (SSHP), task-specific Activity Hazard Analyses and when applicable the DDESB-approved Explosive Safety Submission or Explosive Site Plan.

Vegetation removal is usually required to facilitate follow-on activities such as analog removal actions or digital geophysical mapping.

3.0 DEFINITIONS AND ABBREVIATIONS/ACRONYMS

3.1 DEFINITIONS

Anomaly Avoidance: Techniques employed to allow entry into a specified area to perform required operations on property known to contain or suspected of containing MEC, other munitions in high enough concentrations to pose an explosive hazard regardless of configuration, or chemical agent (CA). Anomaly avoidance techniques focus on avoiding contact with potential surface or subsurface explosives or CA hazards.

Minimum Separation Distance (MSD): The distance at which personnel in the open must be from an intentional or unintentional detonation.

Munitions and Explosives of Concern (MEC): Specific categories of military munitions that may pose unique explosive risks, including the following:

- UXO, as defined in 10 United States Code (U.S.C.) §101(e)(5).
- Discarded military munitions, as defined in 10 U.S.C. § 2710(e)(2).

- Munitions constituents (such as, TNT, RDX), as defined in 10 U.S.C. § 2710(e)(3), present in high enough concentrations to pose an explosive hazard.

Unexploded Ordnance (UXO): As defined by 10 U.S.C. § 101(e)(5)(A) through (C), military munitions that

- have been primed, fuzed, armed, or otherwise prepared for action;
- have been fired, dropped, launched, projected, or placed in such a manner as to constitute a hazard to operations, installations, personnel, or material; and
- remain unexploded whether by malfunction, design, or any other cause.

3.2 ABBREVIATIONS/ACRONYMS

APP	Accident Prevention Plan
CA	chemical agent
CFR	Code of Federal Regulations
DDESB	(U.S.) Department of Defense Safety Board
HGL	HydroGeoLogic, Inc.
IAW	in accordance with
MEC	munitions and explosives of concern
MMRP	Military Munitions Response Program
MSD	minimum separation distance
OSHA	Occupational Safety and Health Administration
PPE	personal protective equipment
SOP	standard operating procedure
SSHP	Site Safety and Health Plan
SUXOS	Senior UXO Supervisor
TP	Technical Paper
U.S.C.	United States Code
UXO	unexploded ordnance
UXOSO	UXO Safety Officer

4.0 PERSONNEL REQUIREMENTS

All HGL employees and subcontractor personnel performing vegetation removal activities must meet the training certification and qualification requirements specified by the project APP/SSHP, and possess the following:

- Site-specific MEC awareness training prior to field activities, and
- Requisite experience in the use and safety protocol for the hand-held vegetation clearance equipment and machinery used for each task.

The presence of a UXO Qualified Person who meets the requirements of DDESB TP 18 is required in the field during vegetation clearance activities to provide oversight and MEC avoidance. Field personnel must meet all requirements for the specific activity they perform as specified in 29 Code of Federal Regulations (CFR) 1910 including, but not limited to, vehicle operation, hazardous material handling, personal protective equipment (PPE), and documentation of training records related to safety and equipment operation.

5.0 TRAINING

Before beginning any vegetation removal operations, all employees and supervisors must be trained on the hazards and safety precautions associated with the vegetation removal methods as described in the APP/SSHP. Training may include the following:

- Initial use, operation, and maintenance of vegetation removal manual tools, machines, and vehicles IAW the manufacturers' directions, and acknowledgment of warnings and precautions for that equipment; and
- Recognition, prevention, and control of other safety and health hazards in the vegetation removal.

6.0 PROCEDURES AND PLANNING

Listed below are the procedures for vegetation removal.

- Disturb only the vegetation necessary and required for safe and effective access for follow-on activities.
  - In typical situations, remove vegetation to within 4 to 6 inches of the ground surface, and trim tree limbs from the ground surface to approximately 8 feet in height.
  - Remove larger deadfall that impedes the progress and safety of personnel and equipment during follow-on activities.

- Typically, leave mature trees in place. Trees of 4 inches in diameter or greater as measured from chest height are typically considered mature. This criterion is site specific and based on agreements with the project client and specified in the project planning documents.
- Dispose of vegetation or leave it in place per agreement with the client. Stipulate site-specific disposal methods in the project planning document.
- Consider site topography, terrain and soil conditions, and drainage patterns.
- Adhere to the required level of environmental and natural resource protection identified in project planning documents.

6.1 MECHANICAL VEGETATION REMOVAL METHODS

Mechanized vegetation removal operations have several advantages in that they are usually more expedient, reduce personnel exposure and the likelihood of injuries, and may reduce project costs compared to manual methods.

6.2 ROBOTIC/REMOTE MECHANICAL VEGETATION REMOVAL METHODS

Remote/robotic-controlled mechanized operations have several advantages in that, because the operator is not on board, they can be used in dangerous, steep, or uncomfortable areas. Depending on site conditions, remote/robotic equipment production can be slower and more expensive.

6.3 MANUAL VEGETATION REMOVAL METHODS

Manual vegetation removal methods can be suitable when there is limited or low-density vegetation.

6.4 CONTROLLED BURN METHODS

Controlled burns may be used to remove dense brush or clear areas of vegetation. When controlled burn methods are used on a project site, keep all personnel beyond the MSD (from the border of the area that is being burned) for the munition with the greatest fragment distance until the UXO Safety Officer (UXOSO) and Senior UXO Supervisor (SUXOS) jointly deem the area safe to enter following the burn.

– W A R N I N G –

Controlled burning of vegetation on ranges is not an approved method to clear MEC hazards.

When planning controlled burns, it is essential to coordinate with the client regarding local regulations, restrictions, and support requirements.

Controlled burns can only be conducted when authorized and approved by the appropriate agency per the project planning documents.

7.0 EQUIPMENT

Specific equipment requirements for each project are detailed in the approved project planning documents.

- The SUXOS or the UXOSO ensures that all equipment/instruments used by site personnel are working properly and that the functionality of vegetation removal equipment is verified and documented.
- Each UXO team leader/escort ensures that the team's safety equipment is inspected before beginning work each day and verifies that required equipment is available and operational.
- Staff must immediately remove from service any machinery or equipment found to be unsafe, or that exhibits a deficiency that affects the safe operation of equipment. The equipment should be properly tagged and removed from service until the unsafe conditions are corrected or the equipment is replaced.

7.1 VEGETATION REMOVAL EQUIPMENT

Consider current site conditions when deciding which equipment is needed. The following equipment is typically used for vegetation removal:

- Chainsaws,
- Weed cutters/trimmers,
- Hand saws,
- Machetes,
- Axes,
- Wood chipper,
- Brush hog (tractor or skid steer), and
- Masticating head/mulching head (skid steer or excavator).

7.2 CHAINSAWS

7.2.1 Chainsaw Operations Requirements

Each chainsaw should be adjusted and operated IAW the manufacturer's instructions. Follow the manufacturer's recommendations for chainsaw use at all times.

- Ensure that each chainsaw used on site is equipped with a functional chain brake and a protective device that minimizes chainsaw kickback. Do not remove or disable chainsaw kickback devices.
- Ensure that each gasoline or fuel-powered chainsaw is equipped with a continuous pressure throttle control system that will stop the chain when pressure on the throttle is released.
- DO NOT fuel a chainsaw if the engine is hot. Allow the engine to cool before refueling.
- Place chainsaws on the ground or in a firmly supported position before starting the machine. If necessary, use plywood or other protective measures to ensure that the chain does not contact the ground.
  - Drop starting a chainsaw is prohibited.
  - Start with the chain brake engaged.
- Control the saw with two hands during saw operations. Exceptions to this policy include adjusting the throttle or reaching for and activating the kill switch.
- Establish secure, stable footing before starting to cut. Do not use the chainsaw in a position or at a distance that could cause insecure footing, loss of balance, or an insecure grip on the saw.
- DO NOT cut directly overhead or above shoulder height.
- Carry chainsaws in a manner that prevents operator contact with the chainsaw bar or chain.
- Shut down the saw whenever it is carried farther than 50 feet or when terrain conditions, such as obstructed ground, slippery surfaces, or dense underbrush, could cause the operator to stumble, slip, or fall.

7.2.2 Chainsaw Personal Protective Equipment

Chainsaw operators must wear the following PPE or PPE detailed in the APP/SSHP:

- Leg protection constructed with cut-resistant material, such as ballistic nylon. Leg protection must cover the full length of the thigh to the top of the boot on each leg to protect against contact with a moving chainsaw blade.

- Foot protection that covers and provides support to the ankle. Each employee who operates a chainsaw must wear foot protection constructed with cut-resistant material that will protect the employee against contact with a running chainsaw (safety toe).
- Safety glasses or goggles.
- Leather gloves.
- Ear plugs.
- Hard hat with attached hearing protection and mesh face shield.

7.2.3 Chainsaw "Kickback"

Kickback is the leading cause of injury among chainsaw users. It occurs when the teeth on the chain catch on an object as they rotate around the tip of the blade. The teeth may have enough force to cause the blade to kick back violently toward the operator. The actions prone to cause kickbacks include the following:

- Striking another object with the nose of the blade,
- Starting a bore cut improperly,
- Catching the bottom or side of a saw cut during reinsertion of the blade nose or tip, and
- Cutting objects above shoulder height.

Some defenses against kickback are as follows:

- Keep the tip guard on the chainsaw.
- Keep a firm hold on the saw.
- Use a saw that has a chain-brake or kickback guard.
- Keep the saw teeth sharp since dull teeth are more likely to cause kickback.
- Never cut anything above shoulder height. At that height, the saw is difficult to control and could potentially kickback toward the head and face.

7.2.4 Chainsaw Pinching

A pinched chainsaw can cause kickback and result in injury. Chainsaw operators should always watch for potential blade pinching situations and plan accordingly. Use the base of the blade to cut branches rather than the tip of the blade and use a high chain speed when reinserting the blade in a cut or removing it from a cut.

7.2.5 Chainsaw Inspection and Maintenance

Check the following elements regularly during each day, but no less than before starting work and at the end of the workday:

- Controls: ensure proper function.
- Chains: ensure proper adjustment.
- Mufflers: ensure that they are in place and operational.
- Chain brakes and nose shielding devices: ensure that they are in place and operational.
- Cutting edges: ensure that they are properly shaped and sharp.
- Ensure that all other safety devices are in place and function properly.

If any chainsaw needs repair, remove it from service and properly tag the equipment. Repair and reinspect it before use.

7.3 BRUSH CUTTER PROCEDURES

7.3.1 Brush Cutter Safety and Operation

The following procedures must be followed to ensure safe operation of brush cutters.

- Space individuals conducting brush cutting and removal with fuel-powered brush cutters at least 50 feet apart while the brush cutters are in use.
- Greater distances than those listed above may be required if specified by the manufacturer in the manufacturer's operating manual or other applicable documents.
- Ensure that operators are wearing proper PPE per the APP/SSHP.
    - For Nylon Line – foot protection with high traction soles and ankle protection, leather gloves, safety glasses or goggles, hard hat with mesh face shield and ear muffs, and hearing protection.
    - For Metal Cutting Blades – prescribed PPE for chainsaw operations.
- Use gas-powered brush cutters/trimmers IAW the manufacturer's operating manual. Ensure that the cutters/trimmers return to idle speed or shut off when the trigger is released.
- Start the cutter/trimmer on the ground or position the unit where it is firmly supported in an open area.
- Control equipment with both hands during operations (exceptions to this policy include the minimal time needed to adjust the throttle or reach for and activate the kill switch).
- Establish stable footing with feet firmly planted.
- Keep the trimmer head below waist level.

7.3.2 Brush Cutter Inspection and Maintenance

At a minimum, include the following in brush cutter inspections:

- Inspect the brush cutter prior to use for potential hazards, such as loose belts and missing or damaged guards.
- Examine the equipment for accumulations of grass, leaves, or excessive grease to reduce fire hazard.
- Conduct routine maintenance and inspections IAW the manufacturer's operating manual.
- If any brush cutter/trimmer needs repair, remove it from service and properly tag the equipment. Repair and reinspect it before use.

7.4 WOOD CHIPPER PROCEDURES

7.4.1 Wood Chipper Operations

During wood chipping operations, it is necessary to provide a safe work environment and to comply with the appropriate safeguarding of machinery standards. In addition to adhering to manufacturer-specific safety precautions or procedures, adhere to the following requirements:

- DO NOT open chipper access covers or doors until the drum or disc is completely stopped.
- Ensure that guards are fitted and secure on the feed chute and discharge chute to prevent contact with the disc, knives, or blower blades.
- Shut down the chipper when an employee performs any service or maintenance.
- Properly chock chippers to prevent rolling or sliding.
- Verify that operators are properly trained and supervised when operating machinery.
- DO NOT wear loose items or clothing that may catch or snag on the chipper.
- Feed the chipper from the side position away from the front of the in-feed chute. Once the in-feed rollers catch the wood, move away from the feed line of the chipper.
- Feed the butt end of branches into the chipper in-feed rollers first.
- Completely stop the in-feed rollers before correcting an action or cleaning up small pieces of wood or leaves on the machine.
- Maintain in place all guarding and emergency control mechanisms and keep them operational at all times when the machine is running.

7.4.2 Wood Chipper Inspection

Inspect and maintain wood chippers IAW manufacturers' instructions, conducting at least one inspection prior to use each day. Never operate the chipper if the required controls and guards are not in place. If chipper needs repair, remove it from service and properly tag the equipment. Repair and reinspect before use.

7.5 BRUSH HOG AND MULCHING HEAD PROCEDURES

7.5.1 Brush Hog and Mulching Head Attachment Operations

Brush hogs (mechanized mower deck attachment) and masticating/mulching heads can be an effective, efficient, and safe method for clearing vegetation. Hazard control and accident prevention depend on the awareness, concern, prudence, and proper training of personnel in the operation, transport, maintenance, and storage of equipment, in addition to the design and configuration of equipment. Use the following precautions as a general guide to safe operations when using brush hogs and masticating/mulching heads:

- Keep all guards in place while when the machine is in operation.
- Permit no riders on the attachment.
- Stop the engine, disconnect the power source, and wait for all machine movement to stop before servicing, adjusting, cleaning, or unclogging the equipment. The exception is when the machinery must be kept running for proper servicing or maintenance. In this case, follow the manufacturer's guidelines on all steps and procedures necessary to safely service or maintain the equipment.
- Make sure all personnel are clear of machinery before starting the engine, engaging power, or operating the machine.
- Become familiar with all of the machine's controls and the caution, warning, and danger decals affixed to the machine before attempting to start or operate.
- Complete a walk-around inspection to check for obvious defects, such as loose mounting bolts and damaged components, before starting or operating the machine. Correct deficiencies before starting.
- Lower the implement to ground, stop the machine engine, apply the parking brake, and allow the blades to stop completely before leaving the tractor/skid steer.
- DO NOT operate the equipment near any area where people may be present unless all safety chains or other factory-approved safety shields are in good repair.
- Establish the safe personnel working distance (depending on the size and type of attachment, the ejection hazard can be upwards of several hundred feet).

- Securely fasten the seat belt and ensure that the equipment has a rollover protection system.
- Where possible, avoid operating near ditches, embankments, and holes.
- Reduce speed when turning, crossing slopes, and operating on rough, slick, or muddy surfaces.
- Stay off slopes too steep for safe operation.
- Wear PPE such as, but not limited to, protection for eyes, ears, feet, hands, and head when operating or repairing the equipment.
- Stand clear of brush hog wing(s) and attachments when raising or lowering.

7.5.2 Brush Hog and Masticating/Mulching Head Inspection and Maintenance

Inspect and maintain the attachments IAW the manufacturer's instructions with at least one inspection before use each day. The following are the guidelines for inspection and maintenance:

- Grease and lubricate machine prior to each use and at required intervals following the manufacturer's operating manual.
- Verify that driveline shields are in place and in good repair to minimize entanglement injuries caused by rotating drivelines.
- Verify that deflector shields (chains, bands, and similar items) are in good repair to minimize injuries caused by the discharge of high-speed objects.
- Inspect blades for wear (replace if necessary).
- Check blade bolts for tightness.
- Check blades and spindles to verify that no foreign objects, such as wire or steel strapping bands, are wrapped around them.
- Inspect hydraulic lines and fittings for wear or leaks (repair or replace if needed).
- Inspect wheel(s) for wear, damage, or foreign objects (repair or replace if necessary).
- Check tractor tire air pressure (refer to manufacturer operating manual).
- During operation, listen for abnormal sounds that might indicate loose parts, worn bearings, or other damage.
- Remove vegetative debris from the engine compartment often enough to prevent fires.
- Conduct the following after each use:
  - Clean all debris from the machine, especially the underside of the deck and affixed safety decals.

  - Inspect cutter for worn or damaged components (repair or replace if necessary).

7.6 TREE FELLING PROCEDURES

Refer to OSHA guidelines for tree felling operations:
https://www.osha.gov/SLTC/etools/logging/manual/felling.html.

Safety must be of utmost concern when approaching a tree to cut. According to OSHA, "More people are killed while felling trees than during any other logging activity." Cutting trees with a chainsaw is particularly hard on the eyes and ears. OSHA Regulation 29 CFR 1910.266 requires that chainsaw operators wear the following:

- Head protection,
- Hearing protection (a combination head and ear protection may be used),
- Eye and face protection,
- Protective chaps or pants, and
- Protective footwear.

7.6.1 General Tree Felling Safety and Operation

7.6.1.1 Personnel Separation Distance

Designate work areas so trees cannot fall into an adjacent occupied work area.

- The distance between occupied work areas must be at least two lengths of the trees being felled.
- Calculate the distance between adjacent occupied work areas considering the degree of slope, the density of the growth, the height of the trees, the soil structure, and other hazards reasonably anticipated at that work site.

7.6.1.2 Overhead Hazards

To the greatest extent practicable, no tree felling should be conducted near overhead electric lines, but if necessary, conduct tree felling operations near overhead electric lines IAW the requirements of Title 29 CFR § 1910.333(c)(3). The site safety officer must notify the power company immediately if a felled tree contacts a power line. Each employee must remain clear of the area until the power company advises that there are no electrical hazards.

7.6.2 Tree Felling Operations

Follow these general requirements during all tree felling operations:

- Fell or remove each tree posing a danger using mechanical or other techniques that minimize employee exposure before beginning work in the area of the danger tree.
    - Lodged trees and snags are particularly dangerous. If not removed, flag and avoid the tree.
    - DO NOT work within two tree lengths of the flagged tree unless the Site Safety and Health Officer indicates that a shorter distance will not create a hazard for an employee.
- Carefully check each tree for signs of loose bark, broken branches and limbs, or other damage before felling or removing the tree.
- Remove or hold in place accessible loose bark and other damage that may create a hazard for an employee before felling or removing the tree.
- Felling on any slope where rolling or sliding of trees or logs is reasonably foreseeable must be done uphill from, or on the same level as, previously felled trees.

Before felling each tree, the feller evaluates conditions and takes precautions to avoid creating a hazard for the feller or other site personnel. Conditions evaluated include snow and ice accumulation, the wind, the lean of the tree, dead limbs, and the location of other trees. During manual tree felling, the chainsaw operator takes the following steps:

- Establish secure footing before starting to cut. Do not use the chainsaw in a position or at a distance that could cause off balance stance, insecure footing, or loss of firm grip on the saw.
- Clear away brush or other potential obstacles that might interfere with cutting the tree or using the retreat path.
- Plan and clear a retreat path:
    - The retreat path extends diagonally away from the expected felling line at an angle of approximately 45 degrees.
    - After making the back cut, immediately shut down the saw, place the saw on the ground, and move a safe distance away from the tree on the retreat path.
- Keep employees other than the feller at least two tree lengths away when cutting a spring pole or other tree under stress. Injury is possible when the cut releases stress.

Highlights of Manual Felling Techniques

- See the following for information about tree cutting: https://www.osha.gov/SLTC/etools/logging/manual/felling/cuts.html
- An undercut must be made in each tree being felled. The undercut must be of a size so that the tree will not split and will fall in the intended direction.
- A backcut must be made in each tree being felled. The backcut must leave sufficient hinge wood to hold the tree to the stump during most of its fall so that the hinge is able to guide the tree's fall in the intended direction. Except in open face felling, the backcut must be above the level of the horizontal facecut to provide an adequate platform to prevent kickback.
- The backcut may be at or below the horizontal facecut in tree pulling operations.
- Domino felling is prohibited.

8.0 GENERAL SAFETY

8.1 ENVIRONMENTAL CONDITIONS

When environmental conditions create a hazard for employees in the performance of the job, cease all vegetation and tree removal work and move each employee to a place of safety. Potentially dangerous conditions include, electrical storms, strong winds that may affect the fall of a tree, heavy rain or snow, extreme cold, dense fog, fires, mudslides, and darkness.

8.2 MUNITIONS AND EXPLOSIVES OF CONCERN SAFETY

Maximum safety in conducting any MEC operations can be achieved through adherence to applicable safety precautions, a planned approach, intensive supervision, and MEC avoidance. UXO-qualified personnel lead a site safety briefing before operational activities begin each workday. General safety considerations applicable to all essential and nonessential personnel at project sites where MEC may be encountered include the following:

- DO NOT become complacent because of familiarity with MEC or the reported probability level of MEC.
- DO NOT conduct operations during inclement weather.
- Avoid contact with MEC.

9.0 QUALITY CONTROL

The MQOs for vegetation clearance are presented in the project planning documents. Performance relative to the MQOs is assessed during the activity by designated personnel. The

SUXOS or UXOQCS uses the three-phase inspection process during vegetation clearance operations.

This SOP will be reviewed at least every 2 years IAW HGL procedures for review of controlled documents.

10.0 REFERENCES

CFR, Title 29, Section 1910.266, Logging.

CFR, Title 29, Section 1910.333(c)(3), Selection and Use of Work Practices.

HydroGeoLogic, Inc. (HGL), HGL Standard Operating Procedure 510.01, MEC Anomaly Avoidance Support.

U.S. Army Corps of Engineers (USACE), Engineer Manual 385-1-1, Safety and Health Requirements Manual.

HGL HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE

Munitions and Explosives of Concern Construction Support	**SOP No.: 501.08 (formerly 511.01)**
	SOP Category: MMRP and Geophysics
	Revision No.: 1
	Revision Date: December 16, 2020
	Review Date: December 2022

Approved By:

Digitally signed by Rojas, Theresa
Date: 2020.12.16 14:08:03 -05'00'

Theresa Rojas
Corporate Quality Director

Date

Digitally signed by Neil Feist
Date: 2020.12.16 13:12:28 -06'00'

Neil Feist
MMRP Operations Manager

Date

Munitions and Explosives of Concern Construction Support	SOP No.: 501.08 (formerly 511.01)
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1.0 PURPOSE

This standard operating procedure (SOP) provides basic guidance and procedures for performing munitions and explosives of concern (MEC) construction support.

2.0 SUMMARY OF METHOD

All HGL employees tasked with performing MEC-related activities must qualify in accordance with (IAW) Department of Defense Explosives Safety Board (DDESB) Technical Paper (TP) 18. Perform all work in a manner consistent with Occupational Safety and Health Administration established standards and requirements. Conduct all activities in conformance with the project-specific Accident Prevention Plan (APP) and Site Safety and Health Plan (SSHP).

These procedures apply to all HydroGeoLogic, Inc. (HGL) and subcontractor personnel during MEC construction support activities in areas where there is a probability of encountering MEC.

3.0 DEFINITIONS AND ABBREVIATIONS/ACRONYMS

3.1 DEFINITIONS

Chemical Warfare Materiel (CWM): Items generally configured as a munition containing a chemical compound that is intended to kill, seriously injure, or incapacitate a person through its physiological effects. CWM includes V- and G-series nerve agents or H-series (mustard) and L-series (lewisite) blister agents in other-than-munition configurations; and certain industrial chemicals (e.g., hydrogen cyanide [AC], cyanogens chloride [CK], or carbonyl dichloride [called phosgene or CG]) configured as a military munition. Because of their hazards, prevalence, and military-unique application, chemical agent (CA) identification sets are also considered CWM.

CWM does not include the following:

- Riot control devices,
- Chemical defoliants and herbicides,
- Industrial chemicals such as AC, CK, or CG not configured as a munition,
- Smoke and other obscuration producing items,
- Flame and incendiary producing items, and
- Soil, water, debris or other media contaminated with low concentrations of CAs where no CA hazards exist.

Construction Support: Assistance provided by unexploded ordnance (UXO)-qualified personnel and/or by personnel trained and qualified for operations involving CA, regardless of configuration, during intrusive construction activities on property known or suspected to contain

MEC or CA, regardless of configuration, to ensure the safety of personnel or resources from any potential explosive or CA hazards.

Construction Support/On-Call: Construction support provided, on an as-needed basis, where the probability of encountering MEC or CA, regardless of configuration, has been determined to be low. This support can respond from off site when called or be on site and available to provide required construction support.

Construction Support/On-Site: Dedicated construction support where the probability of encountering MEC or CA, regardless of configuration, has been determined to be moderate to high.

Material Potentially Presenting an Explosive Hazard (MPPEH): Material owned or controlled by the U.S. Department of Defense (DoD) that, prior to determination of its explosives safety status, potentially contains explosives or munitions (e.g., munitions containers and packaging material; munitions debris remaining after munitions use, demilitarization, or disposal; and range-related debris) or potentially contains a high enough concentration of explosives that the material presents an explosive hazard (e.g., equipment, drainage systems, holding tanks, piping, or ventilation ducts associated with munitions production, demilitarization, or disposal operations).

Excluded from MPPEH are munitions within the DoD-established munitions management system, non-munitions related material (e.g., horseshoes, rebar, other solid objects), munitions-related solid metal fragments that do not realistically present an explosive hazard, and other items that may present explosion hazards (e.g., gasoline cans, compressed gas cylinders) that are not munitions and are not intended for use as munitions.

Munitions and Explosives of Concern (MEC): This term distinguishes the following specific categories of military munitions that may pose unique explosives safety risks:

- UXO, as defined in 10 United States Code (U.S.C.) § 101(e)(5)(A) through (C);
- Discarded military munitions, as defined in 10 U.S.C. § 2710(e)(2); or
- Munitions constituents (e.g., TNT, RDX), as defined in 10 U.S.C. § 2710(e)(3), present in high enough concentrations to pose an explosive hazard.

Munitions Debris: Nonhazardous remnants of munitions (e.g., fragments, penetrators, projectiles, shell casings, links, fins) remaining after munitions use, demilitarization, or final disposition.

Recovered Chemical Warfare Materiel (RCWM): Non-stockpile CWM previously discarded, buried, or fired and discovered either unexpectedly or during planned environmental restoration operations that DoD has either secured in place or placed under DoD control pending final disposition. CWM is normally secured in a DDESB-approved storage location or interim holding facility, pending final disposition.

Unexploded Ordnance (UXO): Military munitions that

- have been primed, fuzed, armed, or otherwise prepared for action;
- have been fired, dropped, launched, projected, or placed in such a manner as to constitute a hazard to operations, installation, personnel, or material; and
- remain unexploded either by malfunction, design, or any other cause.

For the purpose of this project, the definition of UXO is limited to items larger than 50-caliber. A more detailed description of the term UXO is provided in Public Law 106-65, § 3031 (c)(5)(A).

UXO-Qualified Personnel: Personnel who have performed successfully in military explosive ordnance disposal positions or are qualified to perform in the following contractor positions as listed in the Department of Labor's Service Contract Act Directory of Occupations: UXO Technician II, UXO Technician III, UXO Safety Officer (UXOSO), UXO Quality Control Specialist (UXOQCS), or Senior UXO Supervisor (SUXOS).

3.2 ABBREVIATIONS/ACRONYMS

AC	hydrogen cyanide
APP	Accident Prevention Plan
CA	chemical agent
CG	carbonyl dichloride
CK	cyanogens chloride
CWM	chemical warfare materiel
DDESB	Department of Defense Explosives Safety Board
DoD	(U.S.) Department of Defense
ESP	Explosives Safety Submission
ESS	Explosives Site Plan
HGL	HydroGeoLogic, Inc.
IAW	in accordance with
MEC	munitions and explosives of concern
MPPEH	material potentially presenting an explosive hazard
RCWM	recovered chemical warfare material

SOP	standard operating procedure
SSHP	Site Safety and Health Plan
SUXOS	Senior UXO Supervisor
TP	Technical Paper
U.S.C.	United States Code
USACE	U.S. Army Corps of Engineers
UXO	unexploded ordnance
UXOQCS	UXO Quality Control Specialist
UXOSO	UXO Safety Officer
WP	Work Plan

4.0 PERSONNEL

For MEC construction support, the UXO team will consist of a minimum of two UXO qualified personnel (one UXO Technician III and one UXO Technician II). Depending on the scope of work, some projects may require the two personnel to be SUXOS and UXOSO qualified. The UXO team may include additional UXO-qualified personnel depending on on-site/task-specific conditions and requirements. The senior UXO-qualified person serves as the UXO Team Leader and has ultimate responsibility for ensuring that all MEC construction support activities are performed IAW this SOP and project planning documents.

The UXO team responsibilities include the following:

- Reviewing any archival information available on the area of the proposed construction footprint in an effort to determine the probable types of MEC that may be encountered and identify specific hazards and safety precautions;
- Providing MEC recognition, location, and safety functions for the construction contractor during construction activities; and
- Conducting MEC safety briefings for site personnel and visitors.

5.0 EQUIPMENT

5.1 GEOPHYSICAL EQUIPMENT AND SUPPORT EQUIPMENT

The type of geophysical equipment depends on site conditions and the type of work being conducted. For the purposes of construction support, the following geophysical equipment is typically used:

- Magnetometers, such as the Schonstedt GA 52-CX,

- Frequency-domain electromagnetic induction metal detectors, such as the White's All Metals Detector, and
- Downhole monitors.

Additional equipment items that may be required for construction support are as follows:

- Flagging ribbon,
- Pin flags,
- High-visibility, biodegradable spray paint (as required),
- Hard hat and safety vest (construction site),
- UXO team gear, and
- MEC demolition kit.

5.2 EQUIPMENT INSPECTIONS

The following inspections must be performed:

- Perform a daily equipment function check on all geophysical instruments. Describe the performance results of the equipment check in the logbook or in an instrument maintenance and calibration log following each functionality test.
- If an equipment function check indicates that any piece of equipment is not operating correctly, and it cannot be field repaired immediately, remove the equipment from service until it can be repaired.
- Inspect all instruments and equipment items that require routine maintenance and/or calibration upon arrival and periodically as required in the manufacturers' equipment manual.

6.0 SAFETY

In general, adhere to the following MEC safety precautions and protocols:

- Observe this cardinal principle when work may involve ordnance, explosives, ammunition, severe fire hazards, or toxic materials: limit the exposure to a minimum number of personnel, for the minimum amount of time, to a minimum amount of hazardous material consistent with a safe and efficient operation.
- Always assume that MEC hazards contain a live charge until determined otherwise.
- Understand that the age or condition of a MEC hazard does not decrease the effectiveness. MEC that has been exposed to the elements for an extended period of time becomes more sensitive to shock, movement, and friction because the stabilizing agent in the explosives may be degraded.

- DO NOT touch, move or jar any ordnance items regardless of the markings or apparent condition. Under no circumstances handle any MEC during avoidance activities or moved in an attempt to make a positive identification.
- DO NOT touch, pick up, kick, or move anything that is unfamiliar or unknown.
- DO NOT roll the item over or scrape the item to identify markings.
- DO NOT approach or enter a munitions site if an electrical storm is occurring or approaching. If a storm approaches during site operations, leave the site immediately and seek shelter.
- DO NOT transmit radios or cellular phones in the vicinity of suspect MEC hazards.
- DO NOT walk across an area where the ground surface cannot be seen or that has not been cleared of MEC hazards by the UXO Technician.
- DO NOT rely on color codes for positive identification of ordnance items or their contents.
- DO NOT drive vehicles into a suspected MEC area until anomaly avoidance techniques have been implemented.
- DO NOT be misled by markings on the MEC item stating “practice” or “dummy.” Practice ordnance can have explosive charges used to mark and/or spot the point of impact, or the item could be marked incorrectly.
- Clearly mark the location of any ordnance item found during anomaly avoidance activities so that it can be easily located and avoided.

— WARNING —

Removing or taking any munitions, explosives, or UXO or munitions related debris from the site by any employee is strictly prohibited.

7.0 CONSTRUCTION SUPPORT PROCEDURES

Support during construction activities may require only MEC “on-call” (standby) or “on-site” (direct) support during intrusive activities. The level of effort for construction support is site/task-specific and determined on a case-by-case basis by the project development team in coordination with the appropriate DoD agency or service component. The UXO construction support team is responsible for the following activities:

- Meet construction contractor site management and construction personnel and conduct a general work and safety briefing.
    - Probable site hazards and safety considerations,
    - MEC construction support procedures,

  - Responsibilities and lines of authority for any MEC response, and
  - Emergency response procedures.
- Physically inspecting the construction footprint with construction contractor site management and construction contractor to identify areas of concern.
- Monitoring construction activities and, if potential MPPEH is encountered, determining the item's status and if disposal action is required.
- If part of construction support scope, providing anomaly avoidance for construction contractor activities such as the following:
  - Installation of survey points,
  - Installation of utility lines,
  - Installation of signs, poles, or structures, and
  - Downhole support while driving piles.

7.1 ON-CALL CONSTRUCTION SUPPORT (LOW PROBABILITY OF ENCOUNTERING MEC)

If it is determined that there is a "low probability" of encountering MEC at a project site, at a minimum on-call support will be required. On-call support means coordinating with UXO-qualified personnel to ensure their availability, advising them about the project, and placing them "on-call" to assist if suspected MEC is encountered during construction activities. <u>This support can respond from off site when need or be on site and available to provide required construction support</u>.

7.2 ON-SITE CONSTRUCTION SUPPORT (MODERATE TO HIGH PROBABILITY OF ENCOUNTERING MEC)

If it is determined that there is a "moderate to high probability" of encountering MEC at a project site, the activity must be conducted using anomaly avoidance techniques, or UXO-qualified personnel must attempt to identify and remove any explosive or CA hazards in the construction footprint before any other activities begin. Any actions requiring MEC removal in the construction footprint must be designed and authorized in coordination with the appropriate authorities (for example, a USACE military munitions design center).

Subsurface removals require close coordination with the client and the construction contractor. Subsurface removal actions must be accomplished IAW approved project plans (Work Plan [WP], APP/SSHP, Explosives Site Plan [ESP], and Explosives Safety Submission [ESS], if required).

8.0 MUNITIONS AND EXPLOSIVES OF CONCERN

8.1 MEC ENCOUNTERED

If MEC is discovered during construction support, the construction work crew takes the following actions:

- RECOGNIZE the hazard, RETREAT from the area, and REPORT the item to the appropriate personnel.
- Mobilize MEC construction support personnel to the project site (if not already on site).
- If MEC construction support personnel are already on site, notify the construction support team.
- If MEC is found on a “low probability” site, conduct a new probability assessment.

8.2 MEC DISPOSAL

If part of the project scope, the construction support team must dispose of MEC as specified in the project planning documents (WP, and when applicable, ESP or ESS). The senior UXO-qualified person is responsible for coordinating with the proper authorities for the final disposition of all MEC hazards discoveries. If construction support personnel are tasked with MEC destruction, address the following considerations in the project planning documents:

- Teams conducting MEC destruction activities must consist of at least three personnel, with a minimum of two UXO-qualified personnel (one UXO Technician III and one UXO Technician II).
- Teams follow the MEC disposal and explosives procedures detailed in the applicable project planning documents (WP, Explosive Management Plan, ESS, ESP).
    - The plan includes guidance on acquisition, receipt, storage, transportation, inventory, and loss/theft of explosives.
    - Explosives must be used IAW federal, state, and local laws and regulations.
- Refer to the following HGL SOPs for MEC disposal and explosives procedures:
    - SOP 501.01: Explosive Materials Accountability and Management,
    - SOP 502.01: Explosive Demolition Operations, and
    - SOP 501.03: Explosives Storage Inspections and Security.

8.3 MPPEH DISPOSITION

If MPPEH processing is included in the construction support scope of work, ensure that the project is staffed with personnel qualified to certify and verify MPPEH (SUXOS and UXOQCS).

If required, MPPEH procedures must be detailed in the project planning documents. MPPEH must be processed IAW HGL SOP 501.04: MPPEH Inspection and Management, as well as any other applicable laws, regulations, and restrictions.

8.4 RECOVERED CHEMICAL WARFARE MATERIAL

Any time that recovered chemical warfare material (RWCM) or munitions with unknown fillers are identified, the MEC construction support team must notify the agencies designated in the project planning documents. When RWCM or munitions with unknown fillers are encountered during MEC construction support, all work must immediately cease. Project personnel withdraw along paths upwind from the discovery. The site is secured to prevent unauthorized access, and personnel providing security position themselves as far upwind as possible while still securing the area.

9.0 QUALITY CONTROL

The Military Munitions Response Program (MMRP) Operations Manager ensures that this SOP is reviewed at least every 2 years for completeness, accuracy, and safety. Project Managers and supervisors ensure that all site personnel read, understand, and follow this SOP, and they must bring any discrepancies with procedural steps or safety issues pertaining to this SOP to the attention of the responsible supervisor for corrective action.

The senior UXO-qualified person on site has final on-site authority on all munitions and MEC procedures and safety issues. This individual has direct reporting and communications responsibility with all responsible authorities as directed by the HGL Project Manager.

10.0 REFERENCES

Department of Defense Explosives Safety Board (DDESB), DESR 6055.09, Defense Explosives Safety Regulation.

DoD Explosives Safety Board, Technical Paper 18, Minimum Qualifications for Personnel Conducting Munitions and Explosives of Concern-Related Activities.

U.S. Army Corps of Engineers (USACE), Engineer Manual 385-1-97, Explosives Safety and Health Requirements Manual.

11.0 REVISION HISTORY

Revision 0	August 22, 2018	Initial Release
Revision 1	December 16, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 511.01 to 501.08.

APPENDIX E

FORMS

ATF Form 5400.5 Report of Missing or Loss-Explosive Materials
DD Form 1348-1A Fillable
Drilling Log (ENG Form 1836)
Erosion Control Corrective Action Log
Erosion Control Inspection Form
Form 200-1-24 CJAG First Responder Reporting
HAZWOPER 3-Day OJT
HGL Incoming Container Inspection Form Rolloff
Incident Report
OSHA300form1-1-04-Form
Root Cause Analysis Form
Demo Shot Record
ENG Form 3394
Demo Summary Sheet
Manufacturer of Explosives Record of Acquisition
Daily Summary of Magazine Transactions
MEC-UXO Accountability Record
Explosive Material Disposition Record
Preparatory Phase Checklist
Initial Phase Checklist
Noncompliance Corrective Action Report
DQCR
Safety Meeting Attendance
Tailgate Safety Meeting Log
Visitor Log
Vehicle Inspection Log
Explosive Security Survey Checklist
Quality-Safety Assurance Field Audit Checklist
Ravenna Field Change Request Form
Medical Assessment-Work Capacity Form
Daily Production Report
Heavy Equipment Inspection Checklist

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OMB No. 1140-0026 (07/31/2014)

U.S. Department of Justice
Bureau of Alcohol, Tobacco, Firearms and Explosives

Report of Theft or Loss-Explosive Materials

For ATF Use Only

Date Received

Date Faxed to JSOC & Field Division

Unique Identifier

Case Number

To Be Completed By Person Making Report

Upon discovery of any theft or loss of any of your explosive materials:

- First, contact ATF toll free at 1-800-461-8841 between 8:00 a.m. - 5:00 p.m. EST or after hours and weekends contact ATF at 1-800-800-3855 to report the theft or loss;
- Second, contact your local law enforcement office to report the theft or loss to obtain a police report; and
- Third, complete this form and attach any additional reports, sheets or invoices necessary to provide the required information, and fax the form with additional material(s) to the ATF U.S. Bomb Data Center (USBDC) at 866-927-4570 or email to USBDC@atf.gov.

1. Date

2. Type of Report *(Check one)*: ☐ Theft ☐ Loss ☐ Attempted Theft/Suspicious Activity ☐ Supplement

3. Full Name of Person Making the Report *(Last, First, Middle)*

4a. Licensee or Permittee Name

4b. Federal Explosives License or Permit Number

5a. Office Address *(Street Address, City, State, and Zip Code)*

5b. Telephone Number

5c. E-mail Address

6. Actual Location of Theft or Loss *(If different from item 5a)*

7. Theft or Loss	Date	Time
a. Discovered		
b. When Was the Magazine Last Checked		
c. Occurred *(Show approximate if exact not known)*		
d. Reported to ATF by Telephone		
e. Reported to Local Authorities		

8. Name of Local Law Enforcement Officer to Whom Reported

9. Name and Address of Local Authority to Whom Reported

10. Telephone Number

11. Police Report Number

12. Explosive Materials Lost or Stolen *(Attach invoices or additional sheets, if necessary)*

a. Manufacturer and/or Importer	**b. Brand Name**	**c. Date Shift Code**	**d. Size** *(Length & Diameter)*	**e. Quantity** *(Pounds of Explosives, Number of Dets)*	**f. Type and Description** *(Dynamite, Blasting Agents, Detonators, etc. Include for each type, size, MS delay or length of legwire, as applicable)*

13. Theft or Loss Occurred from *(Check applicable box on each row)*

a. Magazine Type:

☐ 1 ☐ 2 ☐ 2 Det. Box ☐ 3 Day Box ☐ 4 ☐ 5

☐ Outdoor ☐ Indoor

☐ Permanent ☐ Portable ☐ Mobile Truck ☐ Mobile Trailer

☐ Overnight Storage ☐ Day Storage

b. Types of Locks *(Check all that apply)*:

☐ Padlock ☐ Mortise ☐ 3-Point ☐ Puck Lock ☐ Other *(Explain)* __________

c. Location Description/Type:

☐ Licensed/Permitted Premises ☐ Remote Storage ☐ Work Site ☐ In Transit ☐ During Operations

14. Method of Entry:

☐ Door   Was a Key Used? ☐ Yes ☐ No   Suspected Employee-Involved Theft? ☐ Yes ☐ No

☐ Wall(s) ☐ Roof ☐ Floor/Bottom

Lock(s) Defeated? *(If yes, check additional appropriate boxes)* ☐ Yes ☐ No

☐ Lock Shackle Cut *(How?)* __________

☐ Lock Left Unlocked

☐ Keyway Drilled Out

☐ Other *(Explain)* __________

☐ Lock Pried, Twisted or Levered

☐ Lock Picked or Shimmed

☐ Lock Body Drilled Out or Cut

Manufacturer and Model of Lock:	Location of Magazine Keys: ☐ Office ☐ Employee ☐ Other *(Address)* __________	Are All Keys Accounted For? ☐ Yes ☐ No

15. Hood Defeated? *(If yes, check all applicable)* ☐ Yes ☐ No

☐ Hood Cut

☐ Other *(Explain)* __________

☐ Hood Removed

☐ Hood Broken

Hood Width *(Inches)* __________   Hood Length (Inches) __________

Hood Depth *(Inches)* __________   Hood Thickness (Inches) __________

16. Other Information Pertinent to the Theft, Loss or Suspicious Activity *(Any details you can provide)*

16a. Was Theft or Loss Disclosed During an ATF Inspection or Being Reported as a Result of Inspection? ☐ Yes ☐ No

16b. Additional Security Measures in Place?

☐ Alarm ☐ Security System/Service ☐ Fencing ☐ Lighting ☐ Other *(Explain)* __________

17. Signature and Title of Person Making Report	18. Date

Reporting Instructions

Email or fax this completed form to the ATF address listed below or call if no fax is available:

Bureau of Alcohol, Tobacco, Firearms and Explosives
U.S. Bomb Data Center
99 New York Ave., N.E. 8.S-295
Washington, DC 20226
Toll Free Fax: 1-866-927-4570
Email Address: USBDC@atf.gov

Questions regarding the completion of this form should be referred to the U.S. Bomb Data Center toll free at 1-800-461-8841.

Privacy Act Information

The following information is provided pursuant to section 3 of the Privacy Act of 1974 (5 U.S.C. § 522a(e)(3).

1. **Authority.** Solicitation of this information is made pursuant to Title XI of the Organized Crime Control Act of 1970 (18 U.S.C. Chapter 40). Disclosure of a theft or loss of explosive materials is mandatory pursuant to 18 U.S.C. § 842(k) for any person who has knowledge of such theft or loss from his stock.

2. **Purpose.** The purpose for the collection of this information is to give ATF notice of the theft or loss of explosive materials, and to furnish ATF with the pertinent facts surrounding such theft or loss. In addition, the information is used to confirm and verify prior notification of this theft or loss of explosive materials.

3. **Routine Uses.** The information will be used by ATF to aid in the administration of laws within its jurisdiction concerning the regulation of explosive materials and other related areas. In addition, the information may be disclosed to other Federal, State, foreign, and local law enforcement of laws within their jurisdiction. System of records notice Justice/ATF-008 Regulatory Enforcement Record System FR Vol.68 No.16 Page 3558 dated January 24, 2003.

4. **Effects of not supplying information requested.** 18 U.S.C. § 842(k) makes it unlawful for any person, who has knowledge of the theft or loss of explosive materials from his stock, to fail to report such theft or loss within twenty-four hours of discovery thereof, to the Secretary and to appropriate local authorities. The penalty for violation of this section is a fine of not more than $1,000 or imprisonment for not more than one year, or both. 18 U.S.C. § 844(b).

Paperwork Reduction Act Notice

This request in accordance with the Paperwork Reduction Act of 1995. The purpose of this information collection is to report the theft or loss of explosive materials. The information is used for investigative purposes by ATF officials. This information is mandatory by statute. (18 U.S.C. § 842)

The estimated average burden associated with this collection of information is 1 hour and 48 minutes per respondent or recordkeeper, depending on individual circumstances. Comments concerning the accuracy of this burden estimate and suggestions for reducing this burden should be addressed to Reports Management Officer, Document Services, Bureau of Alcohol, Tobacco, Firearms and Explosives, Washington, DC 20226.

An agency may not conduct or sponsor, and a person is not required to respond to, a collection of information unless it displays a currently valid OMB control number.

DD FORM 1348-1A, JUL 91 (EG) ISSUE RELEASE/RECEIPT DOCUMENT

1 2 3	4 5 6	7		23 24	25 26 27 28 29		45 46 47 48 49 50	51	52 53	54 55 56	57 58 59	60 61	62 63	64 65 66	67 68 69	70 71	72 73	74 75 76 77 78 79 80
DOC / IDENT	RI FROM	M & S		UNIT / ISS	QUANTITY	SER	SUPPLEMENTARY ADDRESS	SIG	FUND	DISTRIBUTION	PROJECT	PRI	REQD DEL DATE	ADV	RI	O/P / COND	MGT	UNIT PRICE: DOLLARS / CTS

1. TOTAL PRICE
DOLLARS
CTS

2. SHIP FROM

3. SHIP TO

4. MARK FOR

24. DOCUMENT NUMBER & SUFFIX (30-44)

"This certifies and verifies that the material listed has been 100 percent inspected and to the best of our knowledge and belief, is inert and/or free of explosives or related materials"

25. NATIONAL STOCK NO. & ADD (8-22)

DoD Contractor Title: Senior UXO Supervisor (SUXOS)
Print Name:
Signature:____
Company: HydroGeoLogic, Inc.,
Address: 4835 University Square Suite 15 Huntsville, AL 35816
Phone:

26. RIC (4-6) UI (23-24) QTY (25-29) CON CODE (71) DIST (55-56) UP (74-80)

DoD Contractor Title: UXO Quality Control Specialist (UXOQCS)
Print Name:
Signature:____
Company: HydroGeoLogic, Inc.,
Address: 4835 University Square Suite 15 Huntsville, AL 35816
Phone:

27. ADDITIONAL DATA

Container and Serial Number:

5. DOC DATE	6. NMFC	7. FRT RATE	8. TYPE CARGO MDAS	9. PS

10. QTY. REC'D	11.UP	12. UNIT WEIGHT	13. UNIT CUBE	14. UFC	15. SL

16. FREIGHT CLASSIFICATION NOMENCLATURE

17. ITEM NOMENCLATURE
Mixed Metal - Steel, Brass and Copper,

18. TY CONT	19. NO CONT	20. TOTAL WEIGHT	21. TOTAL CUBE

22. RECEIVED BY	23. DATE RECEIVED

PREVIOUS EDITION MAY BE USED

PerFORM (DLA)

1. HOLE NUMBER

DRILLING LOG For use of this form, see EM 1110-1-1804; the proponent agency is CECW-EG.	2. DIVISION	3. INSTALLATION	SHEET OF SHEETS

4. PROJECT	13. SIZE AND TYPE OF BIT
	14. DATUM FOR ELEVATION SHOWN *(TBM or MSL)*
5. LOCATION *(Coordinates or Station)*	15. MANUFACTURERS DESIGNATION OF DRILL
6. DRILLING AGENCY	16. TOTAL NO. OF OVER BURDEN SAMPLES TAKEN — a. DISTURBED — b. UNDISTURBED
7. HOLE NUMBER *(as shown on drawing title and title number)*	17. TOTAL NUMBER CORE BOXES
8. NAME OF DRILLER	18. ELEVATION GROUND WATER
	19. DATE HOLE *(YYYYMMDD)* — a. STARTED — b. COMPLETED
9. DIRECTION OF HOLE ☐ a. VERTICAL ☐ b. INCLINED c. DEG. FROM VERT.	20. ELEVATION TOP OF HOLE *(feet)*
10. THICKNESS OF OVERBURDEN	21. TOTAL CORE RECOVERY FOR BORING %
11. DEPTH DRILLED INTO ROCK	22. SIGNATURE OF INSPECTOR
12. TOTAL DEPTH OF HOLE	

23. ELEVATION	a. DEPTH	b. LEGEND	c. CLASSIFICATION OF MATERIALS *(Description)*	d. % CORE RECOVERY	e. BOX OR SAMPLE NUMBER	f. REMARKS *(Drilling Time, water loss, depth of weathering, etc., if significant)*
				%		
				%		
				%		
				%		
				%		
				%		
				%		

ENG FORM 1836, MAR 1971 PREVIOUS EDITIONS ARE OBSOLETE. *(Translucent)*

PROJECT

HOLE NUMBER

VERSION 1.0

Inspection Form - Erosion Control

Project Name: Group 8 MRS Remedial Action

Project Location: Former Ravenna/CJAG

Date of Inspection (MMDDYYYY):

Contract Number: W912DR-21-D-0005

Date	Type Device	Issues (include qty)	Recommendation	Action Taken	Resolved? (Y/N)

Inspector:

Signature:

Date:

Construction Site Inspection Checklist for OHC000005

By making use of some simple Best Management Practices (BMPs) a construction site operator can do his or her share to protect Ohio's water resources from the harmful effects of sediment. The topography of the site and the extent of the construction activities will determine which of these practices are applicable to any given site, but the BMPs listed here are applicable to most construction sites. For details on the installation and maintenance of these BMPs, please refer to the current ***Rainwater and Land Development***, *Ohio EPA's Standards for Storm Water Management Land Development and Urban Stream Protection*. The manual is available at http://epa.ohio.gov/dsw/storm/technical_guidance.

Temporary Stabilization

This is the most effective BMP. All disturbed areas that will lie dormant for over 14 days must be stabilized within 7 days of the date the area becomes inactive. The goal of temporary stabilization is to provide cover, quickly. Areas within 50 feet of a stream must be stabilized within 2 days of inactivity. This is accomplished by seeding with fast-growing grasses then covering with straw mulch. Apply only mulch between November 1 and March 31. To minimize your costs of temporary stabilization, leave natural cover in place for as long as possible. Only disturb areas you intend to work within the next 14 days.

Construction Entrances

Construction entrances are installed to minimize off-site tracking of sediments. A stone access drive should be installed at every point where vehicles enter or exit the site. Every individual lot should also have its own drive once construction on the lot begins.

Sediment Ponds

Sediment ponds are required for construction areas with concentrated runoff or when the design capacity of silt fence or inlet protection is exceeded. There are two types of sediment ponds: sediment basins and sediment traps. A sediment trap is appropriate where the contributing drainage area is 5 acres or less. The outlet is an earthen embankment with a simple stone spillway. A sediment basin is appropriate for drainage areas larger than 10 acres. The outlet is an engineered riser pipe with a skimmer or similar device used to dewater the pond at the surface. Often a permanent storm water management pond, such as a retention or detention basin, can be modified to act as a sediment basin during construction. All sediment ponds must be installed within 7 days of first grubbing the area they control, provide a minimum dewatering zone of 67 cubic yards per acre of total contributing drainage area and a sediment settling zone of 34 cubic yards per disturbed acre below the level of the outlet. Sediment basins must be designed to drain the dewatering zone over a 48-hour period.

Sediment Barriers

This is typically used at the perimeter of a disturbed area. It’s only for small drainage areas on relatively flat slopes or around small soil storage piles. Not suitable where runoff is concentrated in a ditch, pipe or through streams. For large drainage areas where flow is concentrated, collect runoff in diversion berms or channels and pass it through a sediment pond prior to discharging it from the site. Combination barriers constructed of silt fence supported by straw bales or silt fence embedded within rock check dams may be effective within small channels. As with all sediment controls, sediment barriers must be capable of pooling runoff so that sediment can settle out of suspension. Sediment barriers must be installed within 7 days of first grubbing the area it controls.

Inlet Protection

This must be installed on all yard drains and curb drains when these inlets do not drain to a sediment trap or basin. Even if there is a sediment trap or basin, inlet protection is still recommended, as it will increase the overall sediment removal efficiency. These are best used on roads with little or no traffic. If working properly, inlet protection will cause water to pond. If used on curb inlets, streets will flood temporarily during heavy storms. Check with your municipality before installing curb inlet protection. They may prefer an alternate means of sediment control such as silt fence or ponds.

Permanent Stabilization

All areas at final grade must be permanently stabilized within 7 days of reaching final grade. This is usually accomplished by using seed and mulch, but special measures are sometimes required. This is particularly true in drainage ditches or on steep slopes. These measures include the addition of topsoil, erosion control matting, rock rip-rap or retaining walls. Permanent seeding should be done March 1 to May 31 and August 1 to September 30. Dormant seeding can be done from November 20 to March 15. At all other times of the year, the area should be temporarily stabilized until a permanent seeding can be applied.

Non-Sediment Pollution Control

Although sediment is the pollutant of greatest concern on most construction sites, there are other sources of pollution. Most of these BMPs are easy to implement with a little bit of planning and go a long way toward keeping your site clean and organized. Please be sure to inform all contractors how these BMPs affect their operations on the site, particularly those that will be working near a stream.

Inspection Sheet

INSPECTIONS MUST BE CONDUCTED ONCE EVERY 7 DAYS AND WITHIN 24 HOURS OF A 0.5" OR GREATER RAINFALL. ALL SEDIMENT CONTROLS MUST BE INSTALLED PRIOR TO GRADING AND WITHIN 7 DAYS OF FIRST GRUBBING

GENERAL INSPECTION INFORMATION

Construction Site Inspection Date: ______________ Inspector Name: ______________________________________

Inspector Title: ______________________________ Qualifications/Certifications: _____________________________

Storm Events of the Last 7 Days

Storm Event Date	Storm Event Time	Storm Event Duration	Total Rainfall Amount	Discharge Occur? (Y/N)
_______________	_______________	__________________	_________(inches)	___________________
_______________	_______________	__________________	_________(inches)	___________________
_______________	_______________	__________________	_________(inches)	___________________
_______________	_______________	__________________	_________(inches)	___________________

Weather Information at the Time of Inspection

Temperature _______ Climate (Sunny, Cloudy, Rain)? __________ Is Storm Water Being Discharged? __________

Sketch or Small Site Map

Along with a narrative inspection log, Ohio EPA recommends the inspector use a sketch or a reduced photocopy of the site plan showing the location of storm water outfalls and storm drain inlets as well as the location and types of control measures. Problems observed at these locations, or at other locations on the construction site, should be highlighted and any corrective measures undertaken should be drawn in and noted in detail on the front side of the sketch. This method will also be helpful as the permittee is required to update the SWP3 to reflect current site conditions.

CONSTRUCTION ENTRANCES

Key things to look for ...

	Yes	No
1. Has the drive been constructed by placing geotextile fabric under the stone?	☐	☐
2. Is the stone 2-inch diameter?	☐	☐
3. Has the stone been placed to a depth of 6 inches, with a width of 10 feet and a length of at least 50 feet (30 feet for entrances onto individual sublots)?	☐	☐
4. If the drive is placed on a slope, has a diversion berm been constructed across the drive to divert runoff away from the street or water resource?	☐	☐
5. If drive is placed across a ditch, was a culvert pipe used to allow runoff to flow across the drive?	☐	☐

Note areas where repairs or maintenance is needed or where this practice needs to be applied:

SEDIMENT PONDS

Key things to look for ...

	Yes	No
1. Are concentrated flows of runoff directed to a sediment pond?	☐	☐
2. Is sheet-flow runoff from drainage areas that exceed the design capacity of silt fence (generally 0.25 acre or larger) directed to a sediment pond?	☐	☐
3. Is runoff being collected and directed to the sediment pond via the storm sewer system or via a network of diversion berms and channels?	☐	☐
4. Is the sediment pond dewatering zone appropriately sized (67 cubic yards per acre of total drainage area)?	☐	☐
5. Is the sediment pond sediment settling zone appropriately sized (34 cubic yards per acre of disturbed area)?	☐	☐
6. Is the sediment basin designed to be dewatered at the surface through the use of a skimmer or another similar surface water dewatering device?	☐	☐
7. Is the sediment basin designed so that the dewatering zone will drain in no less time than 48 hours?	☐	☐
8. Have the embankments of the sediment pond and the areas that lie downstream of the pond been stabilized?	☐	☐
9. For sediment traps, is there geotextile under the stone spillway and is the spillway saddle-shaped?	☐	☐
10. For sediment traps, which dewater 100% between storms, is the dewatering pipe end-capped, no larger than 6 inches in diameter, perforated and double-wrapped in geotextile?	☐	☐
11. Is the length-to-width ratio between inlet(s) and outlet at least 2:1? **NOTE**: If not, a baffle should be added to lengthen the distance.	☐	☐
12. Is the depth from the bottom of the basin to the top of the primary spillway no more than 3 to 5 feet?	☐	☐
13. For a modified storm water pond being used as a sediment pond, is the connection between the riser pipe and the permanent outlet water-tight?	☐	☐
14. Was the basin installed prior to grading the site?	☐	☐
15. Is it time to clean-out the sediment pond to restore its original capacity? Generally, sediment should be removed from the sediment settling zone once it's half-full. Stabilize the dredged sediments with seed and mulch.	☐	☐

Note areas where repairs or maintenance is needed or where this practice needs to be applied:

SEDIMENT BARRIERS

Key things to look for ...

	Yes	No
1. Is the silt fence at least 4" to 6" into the ground?	☐	☐
2. Is the silt fence trench backfilled to prevent runoff from cutting underneath the fence?	☐	☐
3. Is the silt fence pulled tight so it won't sag when water builds up behind it?	☐	☐
4. Are the ends brought upslope of the rest of the silt fence so as to prevent runoff from going around the ends?	☐	☐
5. Is the silt fence placed on a level contour? If not, the fence will only act as a diversion.	☐	☐
6. Have all the gaps and tears in the silt fence been eliminated.	☐	☐
7. Is the sediment barrier controlling an appropriate drainage area? Refer to Chapter 6 of ***Rainwater*** manual. **RULE OF THUMB**: Design capacity for 100 linear feet of sediment barrier is 0.5 acres for slopes < 2%, 0.25 acres for slopes 2% to 20%, & 0.125 acres for slopes 20% or more. Generally, no more than 0.25 acres should lie behind 100 feet of sediment barrier at 2% to 20% slope, i.e., the distance between the barrier and the top of the slope behind it should be no more than 125 feet. The allowable distance increases on flatter slopes and decreases for steeper slopes. **All non-silt fence sediment barriers must be at least 12-inches in diameter.**	☐	☐

Note areas where repairs or maintenance is needed or where this practice needs to be applied:

INLET PROTECTION

Key things to look for ...

	Yes	No
1. Does water pond around the inlet when it rains?	☐	☐
2. Has the fabric been replaced when it develops tears or sags?	☐	☐
3. For curb inlet protection, does the fabric cover the entire grate, including the curb window?	☐	☐
4. For yard inlet protection, does the structure encircle the entire grate?	☐	☐
5. Is the fabric properly entrenched or anchored so that water passes through it and not under it?	☐	☐
6. For yard inlet protection, is the fabric properly supported to withstand the weight of water and prevent sagging? The fabric should be supported by a wood frame with cross braces, or straw bales.	☐	☐
7. Is sediment that has accumulated around the inlet removed on a regular basis?	☐	☐

Note areas where repairs or maintenance is needed or where this practice needs to be applied:

TEMPORARY STABILIZATION

Key things to look for ...

	Yes	No
1. Are there any areas of the site that are disturbed, but will likely lie dormant for over 14 days?	☐	☐
2. Have all dormant, disturbed areas been temporarily stabilized in their entireties?	☐	☐
3. Have disturbed areas outside the silt fence been seeded or mulched?	☐	☐
4. Have soil stockpiles that will sit for over 14 days been stabilized?	☐	☐
5. Has seed and mulch been applied at the proper rate? In general, seed is applied at 3 to 5 lbs per 1000 sq ft and straw mulch is applied at 2-3 bales per 1000 sq ft.	☐	☐
6. Has seed or mulch blown away? If so, repair.	☐	☐

Note areas where repairs or maintenance is needed or where this practice needs to be applied:

PERMANENT STABILIZATION

Key things to look for ...

	Yes	No
1. Are any areas at final grade?	☐	☐
2. Has the soil been properly prepared to accept permanent seeding?	☐	☐
3. Has seed and mulch been applied at the appropriate rate (see Chapter 7 of the ***Rainwater*** manual)?	☐	☐
4. If rainfall has been inadequate, are seeded areas being watered?	☐	☐
5. For drainage ditches where flow velocity exceeds 3.5 ft/s from a 10-year, 24-hour storm has matting been applied to the ditch bottom?	☐	☐
6. If the flow velocity exceeds 5.0 ft/s, has the ditch bottom been stabilized with rock rip-rap? **NOTE**: Rock check dams may be needed to slow the flow of runoff.	☐	☐
7. Has rock rip-rap been placed under all storm water outfall pipes to prevent scouring in the receiving stream or erosion of the receiving channel?	☐	☐
8. For sites with steep slopes or fill areas, is runoff from the top of the site conveyed to the bottom of the slope or fill area in a controlled manner so as not to cause erosion?	☐	☐

Note areas where repairs or maintenance is needed or where this practice needs to be applied:

NON-SEDIMENT POLLUTION CONTROL

Key things to look for ...

	Yes	No
1. Has an area been designated for washing out concrete trucks? Washings must be contained on site within a bermed area until they harden. The washings should never be directed toward a watercourse, ditch or storm drain.	☐	☐
2. Is waste and packaging disposed of in a dumpster? Do not burn them on site.	☐	☐
3. Are fuel tanks and drums of toxic and hazardous materials stored within a diked area or trailer and away from any watercourse, ditch or storm drain?	☐	☐
4. Are streets swept as often as necessary to keep them clean and free from sediment? NOTE: Sediment should be swept back onto the lot - not down the storm sewers.	☐	☐
5. Are stockpiles of soil or other materials stored away from any watercourse, ditch or storm drain?	☐	☐
6. Have stream crossings been constructed entirely of non-erodible material?	☐	☐
7. If an area of the site is being dewatered, is it being pumped from a sump pit or is the discharge directed to a sediment pond? **NOTE**: if you must lower ground water, the water may be discharged to the receiving stream as long as the water remains clean. Be sure not to co-mingle the clean ground water with sediment-laden water or to discharge it off-site by passing it over disturbed ground.	☐	☐

Note areas where repairs or maintenance is needed or where this practice needs to be applied:

QRG 2.2 FIRST RESPONDER REPORTING FORM

Collect as much of the information on the top half of this form as possible before making initial notification. Complete the top and bottom of the form before turning in to Range Operations.

Name of individual reporting spill: ____________________

When did the spill occur (Date and Time)? ____________________

Spill Location (Building or area name / number, indoors or out; if vehicle involved, type and bumper number):
____

What was spilled? ____________________ **How much was spilled?** ____________________

Rate at which material is currently spilling. ____________________

Extent of spill travel? ____________________

Did the spill reach water (ditch, creek, stream, pond, well head)? ____________________

Number of injured personnel and type injuries, if applicable. ____________________

Do you need the Fire Department to respond to protect life, property, and environment? ____________________

Unit: ____________________ **State:** __________ **Report Date & Time:** ____________________

On Scene Coordinator Name and Grade: ____________________ **Phone:** ____________________

How did the spill occur (be specific)? ____________________

____

What remedial action was taken? ____________________

____

Was soil and absorbent material generated? __________ **How much?** ____________________

What is the location of the soil and absorbents? ____________________

Was the Environmental Office contacted (yes or No, date and time)? ____________________

Who did you talk to in the Environmental Office? ____________________

Was the site cleared by the Env. Office (Yes or No, date and time)? ____________________

Who cleared the site (name and grade, date and time)? ____________________

Initial information is critical. Get as much information as you can, but don't hesitate to make the initial notification if a spill is moving or worsening rapidly!

This form must be completed for all releases and turned-in to CJAG Range Operations within 24 hours.

FIRST RESPONDER SPILL/RELEASE RESPONSE ACTIONS

Units or contractors performing training or other operations at Camp James A. Garfield shall be responsible for adhering to the provisions identified in the Integrated Environmental Contingency Plans (IECP). A copy of the IECP may be obtained from the Camp James A. Garfield Environmental Supervisor. Following discovery of a spill (any size), the procedures outlined below shall be executed where applicable:

1. **If necessary, initiate evacuation of the immediate area.**
2. **Notify Camp James A. Garfield Range Operations via two-way radio or by calling (614) 336-6041, and report information contained on the "First Responder Reporting Form" if it is known or can reasonably be determined. This form has been copied on the opposite side of this page. If Range Operations cannot be reached, contact a Camp James A. Garfield OSC (listed below).**
3. **Stop spill flow when possible without undue risk of personal injury.**
4. **If trained, contain the spill using available spill response equipment or techniques.**
5. **Make spill scene OFF LIMITS to unauthorized personnel.**
6. **Restrict all sources of ignition when flammable substances are involved.**
7. **Report to the OSC upon his/her arrival to the scene. Turn in a completed copy of the Camp James A. Garfield First Responder Form to Range Operations for ALL releases, even ones cleaned up by the reporter.**

TELEPHONE NUMBER

When Camp James A. Garfield Range Operations is *not available*, the OSC *must be contacted* by the discoverer/first responder following a release if it is in water, at or above a reportable quantity (25 gallons or more of POL), a hazardous or extremely hazardous substance, a hazardous waste, or involves fire, explosion, or is otherwise a major incident.

NAME	JOB TITLE	OFFICE	24 HOUR
Camp James A Garfield Range Operations	Operations and Training	(614)336-6041	(614) 202-5783
Tim Morgan (Primary OSC)	Environmental Supervisor	(614)336-6568	(330)322-7098
Brad Kline (Alternate OSC)	Environmental Specialist	(614)336-4918	Contact Alternate
Katie Tait (Alternate OSC)	Environmental Specialist	(614)336-6136	Contact Alternate
Joint Forces Command (Alternate POC)	OHARNG Emergency Center	(888)637-9053	(888)637-9053

Off-site (from Camp James A. Garfield area code 614 phones)

Ravenna Dispatch ...…………………………………..…. 9-1-330 296-6486

SEE REVERSE FOR FIRST RESPONDER REPORTING FORM

3-DAY ON-SITE TRAINING
&
SITE HAZARD INFORMATION TRAINING LOG

Site Name:	**Location:**
Contract Number:	**Deliver Order Number:**
UXOSO:	

The site personnel listed below have received Site Hazard Information Training as specified in 29 CFR 1910.120(i) and have participated in three-days of supervised on-site training as required by 29 CFR 1910.120(e)(3)(i). The Site Hazard Information Training included information related to the nature, level, and degree of exposure likely to result during participation in site operations. The 3-Day Training has included: a description of the site chain-of-command; use/care/maintenance of PPE; personnel and equipment procedures; safe work practices; medical/training requirements; and emergency response procedures.

Name (printed)	Signature	Organization	Date Started	Date Completed

HGL
HydroGeoLogic, Inc

Incoming Rolloff Inspection Form

Project: ______________________

Rolloff Arrival Date: ______________________

Rolloff Number: ______________________

Inspector/Company: ______________________

Inspection Date: ______________________

	Y	N
Is the liner included and/or installed?		
Is there any residual contamination/waste/material in or on rolloff? (Look under liner if installed.)		
Have any old labels been removed?		
Is the tarp in good condition (no rips or holes)?		
Are the bows in good condition?		
Are the tie downs/tarp straps in good condition?		
Are there enough tie downs in place?		
Are all the wheels are intact?		
Is there any rust or holes in the rolloff (sides, bottom)?		

Please include photos of any unsatisfactory items.

Comments (if rejected include why) :

Rolloff Accepted: Signature of Inspector ______________________

Rolloff Rejected: Signature of Inspector ______________________

HGL INCIDENT REPORT

Section 1 – General Information

Date of Occurrence	**Date Reported**	**Reported to whom?**	**Time of Occurrence**
Employee Name	**Work Address**	**City, State, Zip Code**	**Work Phone Number**
Date of Birth	**Home Address**	**City, State, Zip Code**	**Home Phone Number**

Occupation (Title)	**Full time** ☐ **Part time** ☐ **Temporary** ☐

Location of Occurrence	**Address**	**City, State, Zip Code**

Description of Incident (include what employee was doing, work process, cause, injury and body part)

Witness(es)	**Address**	**City, State, Zip Code**	**Work Phone Number**

Was First Aid given on-site? Yes ☐ **No** ☐ **By whom?**

Was employee taken to hospital? Yes ☐ **No** ☐ **Ambulance Yes** ☐ **No** ☐
If so, provide name, address, and phone number of hospital and name of attending physician below:

Name of Hospital:	**Address:**	**City, State, Zip Code:**	**Phone Number:**

Attending Physician:

Did employee seek medical attention other than an emergency room? Yes ☐ **No** ☐
If so, provide practice name, address, phone number and name of attending physician below:

Practice Name:	**Address:**	**City, State, Zip Code:**	**Phone Number:**

Attending Physician:

Did employee lose time on the job? Yes ☐ **No** ☐ **If so, how many days after the initial injury date? _____**

Was employee assigned light duty? Yes ☐ **No** ☐ **If so, how many days after the initial injury date? _____**

Supervisor (print):	**Signature:**	**Date:**
Employee (print):	**Signature:**	**Date:**
Witness (print):	**Signature:**	**Date:**
Witness (print):	**Signature:**	**Date:**
Director, Health & Safety:	**Signature:**	**Date:**

OSHA's Form 300 (Rev. 01/2004)

Log of Work-Related Injuries and Illnesses

Attention: This form contains information relating to employee health and must be used in a manner that protects the confidentiality of employees to the extent possible while the information is being used for occupational safety and health purposes.

Year 20__ __

U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

You must record information about every work-related death and about every work-related injury or illness that involves loss of consciousness, restricted work activity or job transfer, days away from work, or medical treatment beyond first aid. You must also record significant work-related injuries and illnesses that are diagnosed by a physician or licensed health care professional. You must also record work-related injuries and illnesses that meet any of the specific recording criteria listed in 29 CFR Part 1904.8 through 1904.12. Feel free to use two lines for a single case if you need to. You must complete an Injury and Illness Incident Report (OSHA Form 301) or equivalent form for each injury or illness recorded on this form. If you're not sure whether a case is recordable, call your local OSHA office for help.

Establishment name ______________________________

City ______________________ *State* ______________

Identify the person			***Describe the case***			***Classify the case*** **CHECK ONLY ONE box for each case based on the most serious outcome for that case:**				**Enter the number of days the injured or ill worker was:**		**Check the "Injury" column or choose one type of illness:** (M)					
								Remained at Work									
(A) Case no.	(B) Employee's name	(C) Job title (*e.g., Welder*)	(D) Date of injury or onset of illness	(E) Where the event occurred (*e.g., Loading dock north end*)	(F) Describe injury or illness, parts of body affected, and object/substance that directly injured or made person ill (*e.g., Second degree burns on right forearm from acetylene torch*)	**Death** (G)	**Days away from work** (H)	**Job transfer or restriction** (I)	**Other record-able cases** (J)	**Away from work** (K)	**On job transfer or restriction** (L)	**Injury** (1)	Skin disorder (2)	Respiratory condition (3)	Poisoning (4)	Hearing loss (5)	All other illnesses (6)
____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
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____	____________	________	___/___ month/day	________	________________	☐	☐	☐	☐	____ days	____ days	☐	☐	☐	☐	☐	☐
					Page totals ➤	____	____	____	____	____	____	____	____	____	____	____	____
												Injury (1)	Skin disorder (2)	Respiratory condition (3)	Poisoning (4)	Hearing loss (5)	All other illnesses (6)

Be sure to transfer these totals to the Summary page (Form 300A) before you post it.

Public reporting burden for this collection of information is estimated to average 14 minutes per response, including time to review the instructions, search and gather the data needed, and complete and review the collection of information. Persons are not required to respond to the collection of information unless it displays a currently valid OMB control number. If you have any comments about these estimates or any other aspects of this data collection, contact: US Department of Labor, OSHA Office of Statistical Analysis, Room N-3644, 200 Constitution Avenue, NW, Washington, DC 20210. Do not send the completed forms to this office.

Page ___ ***of*** ___

OSHA's Form 300A (Rev. 01/2004)

Summary of Work-Related Injuries and Illnesses

Year 20__ __

U.S. Department of Labor

Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

All establishments covered by Part 1904 must complete this Summary page, even if no work-related injuries or illnesses occurred during the year. Remember to review the Log to verify that the entries are complete and accurate before completing this summary.

Using the Log, count the individual entries you made for each category. Then write the totals below, making sure you've added the entries from every page of the Log. If you had no cases, write "0."

Employees, former employees, and their representatives have the right to review the OSHA Form 300 in its entirety. They also have limited access to the OSHA Form 301 or its equivalent. See 29 CFR Part 1904.35, in OSHA's recordkeeping rule, for further details on the access provisions for these forms.

Number of Cases

Total number of deaths	Total number of cases with days away from work	Total number of cases with job transfer or restriction	Total number of other recordable cases
______	______	______	______
(G)	(H)	(I)	(J)

Number of Days

Total number of days away from work	Total number of days of job transfer or restriction
______	______
(K)	(L)

Injury and Illness Types

Total number of . . .
(M)

(1) Injuries ______
(2) Skin disorders ______
(3) Respiratory conditions ______
(4) Poisonings ______
(5) Hearing loss ______
(6) All other illnesses ______

Post this Summary page from February 1 to April 30 of the year following the year covered by the form.

Public reporting burden for this collection of information is estimated to average 58 minutes per response, including time to review the instructions, search and gather the data needed, and complete and review the collection of information. Persons are not required to respond to the collection of information unless it displays a currently valid OMB control number. If you have any comments about these estimates or any other aspects of this data collection, contact: US Department of Labor, OSHA Office of Statistical Analysis, Room N-3644, 200 Constitution Avenue, NW, Washington, DC 20210. Do not send the completed forms to this office.

Establishment information

Your establishment name ____________________

Street ____________________

City ______________ State ______ ZIP ________

Industry description (*e.g., Manufacture of motor truck trailers*)

____

Standard Industrial Classification (SIC), if known (*e.g., 3715*)

___ ___ ___ ___

OR

North American Industrial Classification (NAICS), if known (e.g., 336212)

___ ___ ___ ___ ___ ___

Employment information *(If you don't have these figures, see the Worksheet on the back of this page to estimate.)*

Annual average number of employees ____________

Total hours worked by all employees last year ____________

Sign here

Knowingly falsifying this document may result in a fine.

I certify that I have examined this document and that to the best of my knowledge the entries are true, accurate, and complete.

____
Company executive Title

( ) - / /
Phone Date

OSHA's Form 301
Injury and Illness Incident Report

Attention: This form contains information relating to employee health and must be used in a manner that protects the confidentiality of employees to the extent possible while the information is being used for occupational safety and health purposes.

U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

This *Injury and Illness Incident Report* is one of the first forms you must fill out when a recordable work-related injury or illness has occurred. Together with the *Log of Work-Related Injuries and Illnesses* and the accompanying *Summary*, these forms help the employer and OSHA develop a picture of the extent and severity of work-related incidents.

Within 7 calendar days after you receive information that a recordable work-related injury or illness has occurred, you must fill out this form or an equivalent. Some state workers' compensation, insurance, or other reports may be acceptable substitutes. To be considered an equivalent form, any substitute must contain all the information asked for on this form.

According to Public Law 91-596 and 29 CFR 1904, OSHA's recordkeeping rule, you must keep this form on file for 5 years following the year to which it pertains.

If you need additional copies of this form, you may photocopy and use as many as you need.

Completed by ________________________________

Title ________________________________

Phone (_______)_______--__________ **Date** ____/ ____/ ____

Information about the employee

1) **Full name** ________________________________

2) **Street** ________________________________

City ____________________ **State** ________ **ZIP** __________

3) **Date of birth** _____ / _____ / _____

4) **Date hired** _____ / _____ / _____

5) ☐ **Male**
☐ **Female**

Information about the physician or other health care professional

6) **Name of physician or other health care professional** ______________________

____

7) **If treatment was given away from the worksite, where was it given?**

Facility ________________________________

Street ________________________________

City ____________________ **State** ________ **ZIP** __________

8) **Was employee treated in an emergency room?**
☐ **Yes**
☐ **No**

9) **Was employee hospitalized overnight as an in-patient?**
☐ **Yes**
☐ **No**

Information about the case

10) **Case number from the *Log*** __________________ *(Transfer the case number from the Log after you record the case.)*

11) **Date of injury or illness** _____ / _____ / _____

12) **Time employee began work** __________________ **AM / PM**

13) **Time of event** __________________ **AM / PM** ☐ **Check if time cannot be determined**

14) ***What was the employee doing just before the incident occurred?*** **Describe the activity, as well as the tools, equipment, or material the employee was using. Be specific. *Examples:* "climbing a ladder while carrying roofing materials"; "spraying chlorine from hand sprayer"; "daily computer key-entry."**

15) ***What happened?*** **Tell us how the injury occurred. *Examples:* "When ladder slipped on wet floor, worker fell 20 feet"; "Worker was sprayed with chlorine when gasket broke during replacement"; "Worker developed soreness in wrist over time."**

16) ***What was the injury or illness?*** **Tell us the part of the body that was affected and how it was affected; be more specific than "hurt," "pain," or sore." *Examples:* "strained back"; "chemical burn, hand"; "carpal tunnel syndrome."**

17) ***What object or substance directly harmed the employee?*** ***Examples:*** **"concrete floor"; "chlorine"; "radial arm saw." *If this question does not apply to the incident, leave it blank.***

18) ***If the employee died, when did death occur?*** **Date of death** _____ / _____ / _____

Public reporting burden for this collection of information is estimated to average 22 minutes per response, including time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Persons are not required to respond to the collection of information unless it displays a current valid OMB control number. If you have any comments about this estimate or any other aspects of this data collection, including suggestions for reducing this burden, contact: US Department of Labor, OSHA Office of Statistical Analysis, Room N-3644, 200 Constitution Avenue, NW, Washington, DC 20210. Do not send the completed forms to this office.

ROOT CAUSE ANALYSIS

Project:			
Subject:			
Date:		**RCA No.:**	

Description of Condition Found:
Actual Cause:
Action Taken to Correct Condition: (Corrective Action should address root cause of condition found, not symptoms)
Action Taken to Prevent Recurrence:
Action Taken to Monitor Effectiveness of Corrective Action: (Generate data as proof. State the monitoring method put in place and who is responsible for reviewing data.)

Preparer	*Project Title/Role*	*Date*
Lead Technical Representative	*Project Title/Role*	*Date*
HGL Approver	*Project Manager*	*Date*

DEMOLITION SHOT RECORD

Site Name/Location:		Date:
Shot Location (Anomaly ID No./GPS Coord.)	Demolition Supervisor:	State License # (if applicable):
Type of UXO/MEC Destroyed, Vented, or Burned:	Firing Method:	Time of Shot:
Direction and Distance to Nearest Building, Road, Utility Line, etc.:	Temp: ________ Wind Dir./Speed: __________ Ceiling: _______ Clouds/Sun: _____________	
Type and Amount of Tamping Used:	Mat or Other Protection Used (list):	
Seismographic/Sound Level Meter Used: Yes ☐ No ☐	Readings/Results:	

Demolition Materials Used

Description	Amount	Description	Amount
Perforator		Time Fuze	
Det Cord		Squibs	
Electric Detonator		Black/Smokeless Powder	
Non-Electric Detonator		Two Component	
Non-El Detonator		Other (list)	

Certification

I certify that the explosives listed were used for their intended purpose, and that the UXO/MEC listed were rendered inert/destroyed.

Signature of Demolition supervisor:____ Date: ________________

DEMOLITION SHOT RECORD

Site Name/Location:		Date:
Shot Location (Anomaly ID No./GPS Coord.)	Demolition Supervisor:	State License # (if applicable):
Type of UXO/MEC Destroyed, Vented, or Burned:	Firing Method:	Time of Shot:
Direction and Distance to Nearest Building, Road, Utility Line, etc.:	Temp: ________ Wind Dir./Speed: __________ Ceiling: _______ Clouds/Sun: _____________	
Type and Amount of Tamping Used:	Mat or Other Protection Used (list):	
Seismographic/Sound Level Meter Used: Yes ☐ No ☐	Readings/Results:	

Demolition Materials Used

Description	Amount	Description	Amount
Perforator		Time Fuze	
Det Cord		Squibs	
Electric Detonator		Black/Smokeless Powder	
Non-Electric Detonator		Two Component	
Non-El Detonator		Other (list)	

Certification

I certify that the explosives listed were used for their intended purpose, and that the UXO/MEC listed were rendered inert/destroyed.

Signature of Demolition supervisor:____ Date: ________________

(For safety staff only)	REPORT NO.	EROC CODE	**UNITED STATES ARMY CORPS OF ENGINEERS ACCIDENT INVESTIGATION REPORT** For use of this form, see Help Menu and USACE Supplement to AR 385-40 The proponent agency is CESO	REQUIREMENT CONTROL SYMBOL: CEEC-S-8 *(R2)*

1. ACCIDENT CLASSIFICATION

PERSONNEL CLASSIFICATION	INJURY/ILLNESS/FATAL	PROPERTY DAMAGE		MOTOR VEHICLE INVOLVED	DIVING
GOVERNMENT ☐ CIVILIAN ☐ MILITARY	☐	☐ FIRE INVOLVED	☐ OTHER	☐	☐
☐ CONTRACTOR	☐	☐ FIRE INVOLVED	☐ OTHER	☐	☐
☐ PUBLIC	☐ FATAL ☐ OTHER			☐	

2. PERSONAL DATA

a. NAME *(Last, First MI.)*	b. AGE	c. SEX ☐ MALE ☐ FEMALE	d. SOCIAL SECURITY NUMBER	e. GRADE

f. JOB SERIES/TITLE

g. DUTY STATUS AT TIME OF ACCIDENT

☐ ON DUTY ☐ TDY

☐ OFF DUTY

h. EMPLOYMENT STATUS AT TIME OF ACCIDENT

☐ ARMY ACTIVE ☐ ARMY RESERVE ☐ VOLUNTEER

☐ PERMANENT ☐ FOREIGN NATIONAL ☐ SEASONAL

☐ TEMPORARY ☐ STUDENT

☐ OTHER *(Specify)* ______________________

3. GENERAL INFORMATION

a. DATE OF ACCIDENT *(YYYYMMDD)*

b. TIME OF ACCIDENT *(Military Time)* hrs.

c. EXACT LOCATION OF ACCIDENT

d. CONTRACTOR'S NAME

(1) PRIME

(2) SUBCONTRACTOR

e. CONTRACT NUMBER ______________________

☐ CIVIL WORKS ☐ MILITARY

☐ OTHER *(Specify)* ______________

f. TYPE OF CONTRACT

☐ CONSTRUCTION ☐ SERVICE

☐ A/E ☐ DREDGE

☐ OTHER *(Specify)* ______________

g. HAZARDOUS/TOXIC WASTE ACTIVITY

☐ SUPERFUND ☐ DERP

☐ IRP ☐ OTHER *(Specify)*

4. CONSTRUCTION ACTIVITIES ONLY *(Fill in line and corresponding code number in box from list - see help menu)*

a. CONSTRUCTION ACTIVITY (CODE) ______________________ # ☐

b. TYPE OF CONSTRUCTION EQUIPMENT (CODE) ______________________ # ☐

5. INJURY/ILLNESS INFORMATION *(Include name on line and corresponding code number in box for items e, f & g - see help menu)*

a. SEVERITY OF ILLNESS/INJURY (CODE) ______________________ # ☐

b. ESTIMATED DAYS LOST

c. ESTIMATED DAYS HOSPITALIZED

d. ESTIMATED DAYS RESTRICTED DUTY

e. BODY PART AFFECTED

PRIMARY ______________________ (CODE) # ☐

SECONDARY ______________________ (CODE) # ☐

f. NATURE OF ILLNESS / INJURY (CODE) ______________________ # ☐

g. TYPE AND SOURCE OF INJURY/ILLNESS

TYPE ______________________ (CODE) # ☐

SOURCE ______________________ (CODE) # ☐

6. PUBLIC FATALITY *(Fill in line and correspondence code number in box - see help menu)*

a. ACTIVITY AT TIME OF ACCIDENT (CODE) ______________________ # ☐

b. PERSONAL FLOTATION DEVICE USED?

☐ YES ☐ NO ☐ N/A

7. MOTOR VEHICLE ACCIDENT

a. TYPE OF VEHICLE	b. TYPE OF COLLISION	c. SEAT BELTS	USED	NOT USED	NOT APPLICABLE
☐☐ PICKUP/VAN ☐☐ AUTOMOBILE ☐☐ TRUCK ☐☐ OTHER *(Specify)* ________________	☐ SIDE SWIPE ☐ HEAD ON ☐ REAR END ☐ BROADSIDE ☐ ROLL OVER ☐ BACKING ☐ OTHER *(Specify)* ________________	(1) FRONT SEAT	☐	☐	☐
		(2) REAR SEAT	☐	☐	☐

8. PROPERTY MATERIAL INVOLVED

a. NAME OF ITEM	b. OWNERSHIP	c. AMOUNT OF DAMAGE
(1)		
(2)		
(3)		

9. VESSEL/FLOATING PLANT ACCIDENT *(Fill in line and correspondence code number in box from list - see help menu)*

a. ACTIVITY AT TIME OF ACCIDENT	(CODE)	a. ACTIVITY AT TIME OF ACCIDENT	(CODE)
________________	#	________________	#

10. ACCIDENT DESCRIPTION *(Use additional paper, if necessary, see attached page 4.)*

11. CAUSAL FACTOR*(s)* *(Read instructions before completing)*

a. *(Explain YES answers in item 13)*	YES	NO
DESIGN: Was design of facility, workplace or equipment a factor?	☐	☐
INSPECTION/MAINTENANCE: Were inspection & maintenance procedures a factor?	☐	☐
PERSON'S PHYSICAL CONDITION: In your opinion, was the physical condition of the person a factor?	☐	☐
OPERATING PROCEDURES: Were operating procedures a factor?	☐	☐
JOB PRACTICES: Were any job safety/health practices not followed when the accident occurred?	☐	☐
HUMAN FACTORS: Did any human factors such as, size or strength of person, etc., contribute to accident?	☐	☐
ENVIRONMENTAL FACTORS: Did heat, cold, dust, sun, glare, etc., contribute to the accident?	☐	☐
CHEMICAL AND PHYSICAL AGENT FACTORS: Did exposure to chemical agents, such as dust, fumes, mists, vapors or physical agents, such as noise, radiation, etc., contribute to accident?	☐	☐
OFFICE FACTORS: Did office setting such as, lifting office furniture, carrying, stooping, etc., contribute to the accident?	☐	☐
SUPPORT FACTORS: Were inappropriate tools/resources provided to properly perform the activity/task?	☐	☐
PERSONAL PROTECTIVE EQUIPMENT: Did the improper selection, use or maintenance of personal protective equipment contribute to the accident?	☐	☐
DRUGS/ALCOHOL: In your opinion, was drugs or alcohol a factor to the accident?	☐	☐
b. WAS A WRITTEN JOB/ACTIVITY HAZARD ANALYSIS COMPLETED FOR TASK BEING PERFORMED AT TIME OF ACCIDENT? *(If yes, attach a copy.)*	☐	☐

12. TRAINING

a. WAS PERSON TRAINED TO PERFORM ACTIVITY/TASK?	b. TYPE OF TRAINING	c. DATE OF MOST RECENT FORMAL TRAINING *(YYYYMMDD)*
☐ YES ☐ NO	☐ CLASSROOM ☐ ON JOB	

13. FULLY EXPLAIN WHAT ALLOWED OR CAUSED THE ACCIDENT; INCLUDE DIRECT AND INDIRECT CAUSES *(See instruction for definition of direct and indirect causes.) (Use additional paper, if necessary)*

a. DIRECT CAUSE*(s) (Attach additional sheets as needed, See page 4)*

b. INDIRECT CAUSE*(s) (Attach additional sheets as needed, See page 5)*

14. ACTION*(s)* TAKEN, ANTICIPATED OR RECOMMENDED TO ELIMINATE CAUSE*(s)*		
DESCRIBE FULLY *(Attach additional sheets as necessary, See page 5)*		
15. DATES FOR ACTIONS IDENTIFIED IN BLOCK 14.		
a. BEGINNING *(YYYYMMDD)*		b. ANTICIPATED COMPLETION *(YYYYMMDD)*
c. DATE SIGNED *(YYYYMMDD)*	d. TITLE OF SUPERVISOR COMPLETING REPORT	e. CORPS SIGNATURE, SUPERVISOR COMPLETING REPORT
c. DATE SIGNED *(YYYYMMDD)*	d. TITLE OF SUPERVISOR COMPLETING REPORT	e. CONTRACTOR SIGNATURE, SUPERVISOR COMPLETING REPORT
f. ORGANIZATION IDENTIFIER *(Division, Branch, Section, etc.,)*		g. OFFICE SYMBOL
16. MANAGEMENT REVIEW *(1st)*		
a. ☐ CONCUR b. ☐ NONCONCUR c. COMMENTS		
DATE *(YYYYMMDD)*	TITLE	SIGNATURE
17. MANAGEMENT REVIEW *(2nd - Chief Operations, Construction, Engineering, etc.,)*		
a. ☐ CONCUR b. ☐ NONCONCUR c. COMMENTS		
DATE *(YYYYMMDD)*	TITLE	SIGNATURE
18. SAFETY AND OCCUPATIONAL HEALTH OFFICE REVIEW		
a. ☐ CONCUR b. ☐ NONCONCUR c. ADDITIONAL ACTIONS/COMMENTS		
DATE *(YYYYMMDD)*	TITLE	SIGNATURE
19. COMMAND APPROVAL		
COMMENTS		
DATE *(YYYYMMDD)*	COMMANDER SIGNATURE	

10. ACCIDENT DESCRIPTION *(Continuation)*

13a. DIRECT CAUSE*(s) (Continuation)*

13b. INDIRECT CAUSE*(s) (Continuation)*

14. ACTION*(s)* TAKEN, ANTICIPATED, OR RECOMMENDED TO ELIMINATE CAUSE*(s) (Continuation)*

GENERAL. Complete a separate report for each person who was injured, caused, or contributed to the accident *(excluding uninjured personnel and witnesses)*. Use of this form for reporting USACE employee first-aid type injuries not submitted to the Office of Workers' Compensation Programs *(OWCP)* shall be at the discretion of the FOA commander. Please type or print legibly. Appropriate items shall be marked with an "X" in box(es). If additional space is needed, provide the information on a separate sheet and attach to the completed form. Ensure that these instructions are forwarded with the completed report to the designated management reviewers indicated in sections 16 and 17.

INSTRUCTIONS FOR SECTION 1 - ACCIDENT CLASSIFICATION

(Mark All Boxes That Are Applicable)

a. GOVERNMENT. Mark "CIVILIAN" box if accident involved government civilian employee; mark "MILITARY" box if accident involved U.S. military personnel.

(1) INJURY/ILLNESS/FATALITY - Mark if accident resulted in any government civilian employee injury, illness, or fatality that requires the submission of OWCP Forms CA-1 *(injury)*, CA-2 *(illness)* or CA-6 *(fatality)* to OWCP; mark if accident resulted in military personnel lost-time or fatal injury or illness.

(2) PROPERTY DAMAGE - Mark the appropriate box if accident resulted in any damage of $1000 or more to government property *(including motor vehicles)*.

(3) VEHICLE INVOLVED - Mark if accident involved a motor vehicle, regardless of whether "INJURY/ILLNESS/FATALITY" or "PROPERTY DAMAGE" are marked.

(4) DIVING ACTIVITY - Mark if the accident involved an in-house USACE diving activity.

b. CONTRACTOR.

(1) INJURY/ILLNESS/FATALITY - Mark if accident resulted in any contractor lost-time injury/illness or fatality.

(2) PROPERTY DAMAGE - Mark the appropriate box if accident resulted in any damage of $1000 or more to contractor property *(including motor vehicles)*.

(3) VEHICLE INVOLVED - Mark if accident involved a motor vehicle, regardless of whether "INJURY/ILLNESS/FATALITY" or "PROPERTY DAMAGE" are marked.

(4) DIVING ACTIVITY - Mark if the accident involved a USACE Contractor diving activity.

c. PUBLIC.

(1) INJURY/ILLNESS/FATALITY - Mark if accident resulted in public fatality or permanent total disability. *(The "OTHER" box will be marked when requested by the FOA to report an unusual non-fatal public accident that could result in claims against the government or as otherwise directed by the FOA Commander)*.

(2) VOID SPACE - Make no entry.

(3) VEHICLE INVOLVED - Mark if accident resulted in a fatality to a member of the public and involved a motor vehicle, regardless of whether "INJURY/ILLNESS/FATALITY" is marked.

(4) VOID SPACE - Make no entry.

INSTRUCTIONS FOR SECTION 2 - PERSONAL DATA

a. NAME - *(MANDATORY FOR GOVERNMENT ACCIDENTS. OPTIONAL AT THE DISCRETION OF THE FOA COMMANDER FOR CONTRACTOR AND PUBLIC ACCIDENTS)*. Enter last name, first name, middle initial of person involved.

b. AGE - Enter age.

c. SEX - Mark appropriate box.

d. SOCIAL SECURITY NUMBER - *(FOR GOVERNMENT PERSONNEL ONLY*) Enter the social security number *(or other personal identification number if no social security number issued)*.

e. GRADE - *(FOR GOVERNMENT PERSONNEL ONLY)* Enter pay grade. Example: 0-6; E-7; WG-8; WS-12; GS-11; etc.

f. JOB SERIES/TITLE - For government civilian employees enter the pay plan, full series number, and job title, e.g., GS-O810/Civil Engineer. For military personnel enter the primary military occupational specialty *(PMOS)*, e.g., 15A30 or 11G50. For contractor employees enter the job title assigned to the injured person, e.g., carpenter, laborer, surveyor, etc.

g. DUTY STATUS - Mark the appropriate box.

(1) ON DUTY - Person was at duty station during duty hours or person was away from duty station during duty hours but on official business at time of the accident.

(2) TDY - Person was on official business, away from the duty station and with travel orders at time of accident. Line-of-duty investigation required.

(3) OFF DUTY - Person was not on official business at time of accident.

h. EMPLOYMENT STATUS - *(FOR GOVERNMENT PERSONNEL ONLY)* Mark the most appropriate box. If "OTHER" is marked, specify the employment status of the person.

INSTRUCTION FOR SECTION 3 - GENERAL INFORMATION

a. DATE OF ACCIDENT - Enter the month, day, and year of accident.

b. TIME OF ACCIDENT - Enter the local time of accident in military time. Example: 1430 hrs *(not 2:30 p.m.)*.

c. EXACT LOCATION OF ACCIDENT - Enter facts needed to locate the accident scene, *(installation/project name, building number, street, direction and distance from closest landmark, etc.)*.

d. CONTRACTOR NAME

(1) PRIME - Enter the exact name *(title of firm)* of the prime contractor.

(2) SUBCONTRACTOR - Enter the name of any subcontractor involved in the accident.

e. CONTRACT NUMBER - Mark the appropriate box to identify if contract is civil works, military, or other: if "OTHER" is marked, specify contract appropriation on line provided. Enter complete contract number of prime contract, e.g., DACW 09-85-C-0100.

f. TYPE OF CONTRACT - Mark appropriate box. A/E means architect/engineer. If "OTHER" is marked, specify type of contract on line provided.

g. HAZARDOUS/TOXIC WASTE ACTIVITY *(HTW)* - Mark the box to identify the HTW activity being performed at the time of the accident. For Superfund, DERP, and Installation Restoration Program *(IRP)* HTW activities include accidents that occurred during inventory, predesign, design, and construction. For the purpose of accident reporting, DERP Formerly Used DoD Site *(FUDS)* activities and IRP activities will be treated separately. For Civil Works O&M HTW activities mark the "OTHER" box.

INSTRUCTIONS FOR SECTION 4 - CONSTRUCTION ACTIVITIES

a. CONSTRUCTION ACTIVITY - Select the most appropriate construction activity being performed at time of accident from the list below. Enter the activity name and place the corresponding code number identified in the box.

CONSTRUCTION ACTIVITY LIST

1. MOBILIZATION
2. SITE PREPARATION
3. EXCAVATION/TRENCHING
4. GRADING (EARTHWORK)
5. PIPING/UTILITIES
6. FOUNDATION
7. FORMING
8. CONCRETE PLACEMENT
9. STEEL ERECTION
10. ROOFING
11. FRAMING
12. MASONRY
13. CARPENTRY
14. ELECTRICAL
15. SCAFFOLDING/ACCESS
16. MECHANICAL
17. PAINTING
18. EOUIPMENT/MAINTENANCE
19. TUNNELING
20. WAREHOUSING/STORAGE
21. PAVING
22. FENCING
23. SIGNING
24. LANDSCAPING/IRRIGATION
25. INSULATION
26. DEMOLITION

b. TYPE OF CONSTRUCTION EQUIPMENT - Select the equipment involved in the accident from the list below. Enter the name and place the corresponding code number identified in the box. If equipment is not included below, use code 24, "OTHER", and write in specific type of equipment.

CONSTRUCTION EQUIPMENT

1. GRADER
2. DRAGLINE
3. CRANE *(ON VESSEL/BARGE)*
4. CRANE *(TRACKED)*
5. CRANE *(RUBBER TIRE)*
6. CRANE *(VEHICLE MOUNTED)*
7. CRANE *(TOWER)*
8. SHOVEL
9. SCRAPER
10. PUMP TRUCK *(CONCRETE)*
11. TRUCK *(CONCRETE/TRANSIT MIXER)*
12. DUMP TRUCK *(HIGHWAY)*
13. DUMP TRUCK *(OFF HIGHWAY)*
14. TRUCK *(OTHER)*
15. FORKLIFT
16. BACKHOE
17. FRONT-END LOADER
18. PILE DRIVER
19. TRACTOR *(UTILITY)*
20. MANLIFT
21. DOZER
22. DRILL RIG
23. COMPACTOR/VIBRATORY ROLLER
24. OTHER

INSTRUCTIONS FOR SECTION 5 - INJURY/ILLNESS INFORMATION

a. SEVERITY OF INJURY/ILLNESS - Reference paragraph 2-10 of USACE Supplement 1 to AR 385-40 and enter code and description from list below.

NOI	NO INJURY
FAT	FATALITY
PTL	PERMANENT TOTAL DISABILITY
PPR	PERMANENT PARTIAL DISABILITY
LWD	LOST WORKDAY CASE INVOLVING DAYS AWAY FROM WORK
NLW	RECORDABLE CASE WITHOUT LOST WORKDAYS
RFA	RECORDABLE FIRST AID CASE
NRI	NON-RECORDABLE INJURY

b. ESTIMATED DAYS LOST - Enter the estimated number of workdays the person will lose from work.

c. ESTIMATED DAYS HOSPITALIZED - Enter the estimated number of workdays the person will be hospitalized.

d. ESTIMATED DAYS RESTRICTED DUTY - Enter the estimated number of workdays the person, as a result of the accident, will not be able to perform all of their regular duties.

e. BODY PART AFFECTED - Select the most appropriate primary and when applicable, secondary body part affected from the list below. Enter body part name on line and place the corresponding code letters identifying that body part in the box.

GENERAL BODY AREA	CODE	BODY PART NAME
ARM/WRIST	AB	ARM AND WRIST
	AS	ARM OR WRIST
TRUNK, EXTERNAL MUSCULATURE	B1	SINGLE BREAST
	B2	BOTH BREASTS
	B3	SINGLE TESTICLE
	B4	BOTH TESTICLES
	BA	ABDOMEN
	BC	CHEST
	BL	LOWER BACK
	BP	PENIS
	BS	SIDE
	BU	UPPER BACK
	BW	WAIST
	BZ	TRUNK OTHER
HEAD, INTERNAL	C1	SINGLE EAR INTERNAL
	C2	BOTH EARS INTERNAL
	C3	SINGLE EYE INTERNAL
	C4	BOTH EYES INTERNAL
	CB	BRAIN
	CC	CRANIAL BONES
	CD	TEETH
	CJ	JAW
	CL	THROAT, LARYNX
	CM	MOUTH
	CN	NOSE
	CR	THROAT, OTHER
	CT	TONGUE
	CZ	HEAD OTHER INTERNAL
ELBOW	EB	BOTH ELBOWS
	ES	SINGLE ELBOW
FINGER	F1	FIRST FINGER
	F2	BOTH FIRST FINGERS
	F3	SECOND FINGER
	F4	BOTH SECOND FINGERS
	F5	THIRD FINGER
	F6	BOTH THIRD FINGERS
	F7	FOURTH FINGER
	F8	BOTH FOURTH FINGERS
TOE	G1	GREAT TOE
	G2	BOTH GREAT TOES
	G3	TOE OTHER
	G4	TOES OTHER
HEAD, EXTERNAL	H1	EYE EXTERNAL
	H2	BOTH EYES EXTERNAL
	H3	EAR EXTERNAL
	H4	BOTH EARS EXTERNAL
	HC	CHIN
	HF	FACE
	HK	NECK/THROAT
	HM	MOUTH/LIPS
	HN	NOSE
	HS	SCALP
KNEE	KB	BOTH KNEES
	KS	KNEE
LEG, HIP, ANKLE,	LB	BOTH LEGS/HIPS/ ANKLES/
BUTTOCKS		
BUTTOCK	LS	SINGLE LEG/HIP/ ANKLE/BUTTOCK
HAND	MB	BOTH HANDS
	MS	SINGLE HAND
FOOT	PB	BOTH FEET
	PS	SINGLE FOOT
TRUNK, BONES	R1	SINGLE COLLAR BONE
	R2	BOTH COLLAR BONES
	R3	SHOULDER BLADE
	R4	BOTH SHOULDER BLADES
	RB	RIB
	RS	STERNUM *(BREAST BONE)*
	RV	VERTEBRAE *(SPINE; DISC)*
	RZ	TRUNK BONES OTHER
SHOULDER	SB	BOTH SHOULDERS
	SS	SINGLE SHOULDER
THUMB	TB	BOTH THUMBS
	TS	SINGLE THUMB
TRUNK, INTERNAL ORGANS	V1	LUNG, SINGLE
	V2	LUNGS, BOTH
	V3	KIDNEY, SINGLE
	V4	KIDNEYS, BOTH
	VH	HEART
	VL	LIVER
	VR	REPRODUCTIVE ORGANS
	VS	STOMACH
	VV	INTESTINES
	VZ	TRUNK, INTERNAL; OTHER

f. NATURE OF INJURY/ILLNESS - Select the most appropriate nature of injury/illness from the list below. This nature of injury/illness shall correspond to the primary body part selected in 5e, above. Enter the nature of injury/illness name on the line and place the corresponding CODE letters in the box provided.
* The injury or condition selected below must be caused by a specific incident or event which occurred during a single work day or shift.

GENERAL NATURE CATEGORY	CODE	NATURE OF INJURY NAME
*TRAUMATIC INJURY OR DISABILITY	TA	AMPUTATION
	TB	BACK STRAIN
	TC	CONTUSION; BRUISE; ABRASION
	TD	DISLOCATION
	TF	FRACTURE
	TH	HERNIA
GENERAL NATURE CATEGORY	CODE	NATURE OF INJURY NAME
	TK	CONCUSSION
	TL	LACERATION, CUT
	TP	PUNCTURE
	TS	STRAIN, MULTIPLE
	TU	BURN, SCALD, SUNBURN
	TI	TRAUMATIC SKIN DISEASES/ CONDITIONS INCLUDING DERMATITIS
	TR	TRAUMATIC RESPIRATORY DISEASE
	TQ	TRAUMATIC FOOD POISONING
	TW	TRAUMATIC TUBERCULOSIS
	TX	TRAUMATIC VIROLOGICAL/INFECTIVE/ PARASITIC DISEASE
	T1	TRAUMATIC CEREBRAL VASCULAR CONDITION/STROKE
	T2	TRAUMATIC HEARING LOSS
	T3	TRAUMATIC HEART CONDITION
	T4	TRAUMATIC MENTAL DISORDER, STRESS; NERVOUS CONDITION
	T8	TRAUMATIC INJURY - OTHER *(EXCEPT DISEASE, ILLNESS)*

** A nontraumatic physiological harm or loss of capacity produced by systemic infection; continued or repeated stress or strain; exposure to toxins, poisons, fumes, etc.; or other continued and repeated exposures to conditions of the work environment over a long period of time. For practical purposes, an occupational illness/disease or disability is any reported condition which does not meet the definition of traumatic injury or disability as described above.

GENERAL NATURE CATEGORY	**CODE**	**NATURE OF INJURY NAME**
**NON-TRAUMATIC ILLNESS/DISEASE OR DISABILITY		
RESPIRATORY DISEASE	RA	ASBESTOSIS
	RB	BRONCHITIS
	RE	EMPHYSEMA
	RP	PNEUMOCONIOSIS
	RS	SILICOSIS
	R9	RESPIRATORY DISEASE, OTHER
VIROLOGICAL, INFECTIVE & PARASITIC DISEASES		
	VB	BRUCELLOSIS
	VC	COCCIDIOMYCOSIS
	VF	FOOD POISONING
	VH	HEPATITIS
	VM	MALARIA
	VS	STAPHYLOCOCCUS
	VT	TUBERCULOSIS
	V9	VIROLOGICAL/INFECTIVE/ PARASITIC - OTHER
DISABILITY, OCCUPATIONAL	DA	ARTHRITIS, BURSITIS
	DB	BACK STRAIN, BACK SPRAIN
	DC	CEREBRAL VASCULAR CONDITION; STROKE
	DD	ENDEMIC DISEASE *(OTHER THAN CODE TYPES R&S)*
	DE	EFFECT OF ENVIRONMENTAL
CONDITION		
	DH	HEARING LOSS
	DK	HEART CONDITION
	DM	MENTAL DISORDER, EMOTIONAL STRESS, NERVOUS CONDITION
	DR	RADIATION
	DS	STRAIN, MULTIPLE
	DU	ULCER
	DV	OTHER VASCULAR CONDITIONS
	D9	DISABILITY, OTHER
SKIN DISEASE OR CONDITION		
	SB	BIOLOGICAL
	SC	CHEMICAL
	S9	DERMATITIS, UNCLASSIFIED

g. TYPE AND SOURCE OF INJURY/ILLNESS *(CAUSE)* - Type and Source Codes are used to describe what caused the incident. The Type Code stands for an ACTION and the Source Code for an OBJECT or SUBSTANCE. Together, they form a brief description of how the incident occurred. Where there are two different sources, code the initiating source of the incident *(see example 1, below)*. Examples:

(1) An employee tripped on carpet and struck his head on a desk. TYPE: 210 *(fell on same level)* SOURCE: 0110 *(walking/working surface*).

NOTE: This example would NOT be coded 120 *(struck against)* and 0140 *(furniture)*.

(2) A Park Ranger contracted dermatitis from contact with poison ivy/oak.

TYPE: 510 *(contact)* SOURCE: 0920 *(plant)*

(3) A lock and dam mechanic punctured his finger with a metal sliver while grinding a turbine blade.

TYPE: 410 *(punctured by)* SOURCE: 0830 *(metal)*

(4) An employee was driving a government vehicle when it was struck by another vehicle.

TYPE: 800 *(traveling in)* SOURCE: 0421 *(government-owned vehicle, as driver)*

NOTE: The Type Code 800, "Traveling In" is different from the other type codes in that its function is not to identify factors contributing to the injury or fatality, but rather to collect data on the type of vehicle the employee was operating or traveling in at the time of the incident.

Select the most appropriate TYPE and SOURCE identifier from the list below and enter the name on the line and the corresponding code in the appropriate box.

CODE	**TYPE OF INJURY NAME**
	STRUCK
0110	STRUCK BY
0111	STRUCK BY FALLING OBJECT
0120	STRUCK AGAINST
	FELL, SLIPPED, TRIPPED
0210	FELL ON SAME LEVEL
0220	FELL ON DIFFERENT LEVEL
0230	SLIPPED, TRIPPED *(NO FALL)*
	CAUGHT
0310	CAUGHT ON
0320	CAUGHT IN
0330	CAUGHT BETWEEN
	PUNCTURED, LACERATED
0410	PUNCTURED BY
0420	CUT BY
0430	STUNG BY
0440	BITTEN BY
	CONTACTED
0510	CONTACTED WITH *(INJURED PERSON MOVING)*
0520	CONTACTED BY *(OBJECT WAS MOVING)*
	EXERTED
0610	LIFTED, STRAINED BY *(SINGLE ACTION)*
0620	STRESSED BY *(REPEATED ACTION)*
	EXPOSED
0710	INHALED
0720	INGESTED
0730	ABSORBED
0740	EXPOSED TO
0800	TRAVELING IN

CODE	**SOURCE OF INJURY NAME**
0100	BUILDING OR WORKING AREA
0110	WALKING/WORKING SURFACE *(FLOOR, STREET, SIDEWALKS, ETC.)*
0120	STAIRS, STEPS
0130	LADDER
0140	FURNITURE, FURNISHINGS, OFFICE EQUIPMENT
0150	BOILER, PRESSURE VESSEL
0160	EQUIPMENT LAYOUT *(ERGONOMIC)*
0170	WINDOWS, DOORS
0180	ELECTRICITY

0200	ENVIRONMENTAL CONDITION
0210	TEMPERATURE EXTREME *(INDOOR)*
0220	WEATHER *(ICE, RAIN, HEAT, ETC.)*
0230	FIRE, FLAME, SMOKE *(NOT TOBACCO)*
0240	NOISE
0250	RADIATION
0260	LIGHT
0270	VENTILATION
0271	TOBACCO SMOKE
0280	STRESS *(EMOTIONAL)*
0290	CONFINED SPACE
0300	MACHINE OR TOOL
0310	HAND TOOL *(POWERED; SAW, GRINDER, ETC.)*
0320	HAND TOOL *(NONPOWERED)*
0330	MECHANICAL POWER TRANSMISSION APPARATUS
0340	GUARD, SHIELD *(FIXED, MOVEABLE, INTERLOCK)*
0350	VIDEO DISPLAY TERMINAL
0360	PUMP, COMPRESSOR, AIR PRESSURE TOOL
0370	HEATING EQUIPMENT
0380	WELDING EQUIPMENT
0400	VEHICLE
0411	AS DRIVER OF PRIVATELY OWNED/RENTAL VEHICLE
0412	AS PASSENGER OF PRIVATELY OWNED/RENTAL VEHICLE
0421	DRIVER OF GOVERNMENT VEHICLE
0422	PASSENGER OF GOVERNMENT VEHICLE
0430	COMMON CARRIER *(AIRLINE, BUS, ETC.)*
0440	AIRCRAFT *(NOT COMMERCIAL)*
0450	BOAT, SHIP, BARGE
0500	MATERIAL HANDLING EQUIPMENT
0510	EARTHMOVER *(TRACTOR, BACKHOE, ETC.)*
0520	CONVEYOR *(FOR MATERIAL AND EQUIPMENT)*
0530	ELEVATOR, ESCALATOR, PERSONNEL HOIST
0540	HOIST, SLING CHAIN, JACK
0550	CRANE
0551	FORKLIFT
0560	HANDTRUCK, DOLLY
0600	DUST, VAPOR, ETC.
0610	DUST *(SILICA, COAL, ETC.)*
0620	FIBERS
0621	ASBESTOS
0630	GASES
0631	CARBON MONOXIDE
0640	MIST, STEAM, VAPOR, FUME
0641	WELDING FUMES
0650	PARTICLES *(UNIDENTIFIED)*
0700	CHEMICAL, PLASTIC, ETC.
0711	DRY CHEMICAL - CORROSIVE
0712	DRY CHEMICAL - TOXIC
0713	DRY CHEMICAL - EXPLOSIVE
0714	DRY CHEMICAL FLAMMABLE
0721	LIQUID CHEMICAL - CORROSIVE
0722	LIQUID CHEMICAL - TOXIC
0723	LIQUID CHEMICAL - EXPLOSIVE
0724	LIQUID CHEMICAL - FLAMMABLE
0730	PLASTIC
0740	WATER
0750	MEDICINE
0800	INAMINATE OBJECT
0810	BOX, BARREL, ETC.
0820	PAPER
0830	METAL ITEM, MINERAL
0831	NEEDLE
0840	GLASS
0850	SCRAP, TRASH
0860	WOOD
0870	FOOD
0880	CLOTHING, APPAREL, SHOES
0900	ANIMATE OBJECT
0911	DOG
0912	OTHER ANIMAL
0920	PLANT
0930	INSECT
0940	HUMAN *(VIOLENCE)*
0950	HUMAN *(COMMUNICABLE DISEASE)*
0960	BACTERIA, VIRUS *(NOT HUMAN CONTACT)*
1000	PERSONAL PROTECTIVE EQUIPMENT
1010	PROTECTIVE CLOTHING, SHOES, GLASSES, GOGGLES
1020	RESPIRATOR, MASK
1021	DIVING EQUIPMENT
1030	SAFETY BELT, HARNESS
1040	PARACHUTE

INSTRUCTIONS FOR SECTION 6 - PUBLIC FATALITY

a. ACTIVITY AT TIME OF ACCIDENT - Select the activity being performed at the time of the accident from the list below. Enter the activity name on the line and the corresponding number in the box. If the activity performed is not identified on the list, select from the most appropriate primary activity area *(water related, non-water related or other activity)*, the code number for "Other", and write in the activity being performed at the time of the accident.

WATER RELATED RECREATION

1. Sailing
2. Boating-powered
3. Boating-unpowered
4. Water skiing
5. Fishing from boat
6. Fishing from bank dock or pier
7. Fishing while wading
8. Swimming/supervised area
9. Swimming/designated area
10. Swimming/other area
11. Underwater activities *(skin diving, scuba, etc.)*
12. Wading
13. Attempted rescue
14. Hunting from boat
15. Other

NON-WATER RELATED RECREATION

16. Hiking and walking
17. Climbing *(general)*
18. Camping/picnicking authorized area
19. Camping/picnicking unauthorized area
20. Guided tours
21. Hunting
22. Playground equipment
23. Sports/summer *(baseball, football, etc.)*
24. Sports/winter *(skiing, sledding, snowmobiling etc.)*
25. Cycling *(bicycle, motorcycle, scooter)*
26. Gliding
27. Parachuting
28. Other non-water related

OTHER ACTIVITIES

29. Unlawful acts *(fights, riots, vandalism, etc.)*
30. Food preparation/serving
31. Food consumption
32. Housekeeping
33. Sleeping
34. Pedestrian struck by vehicle
35. Pedestrian other acts
36. Suicide
37. "Other" activities

b. PERSONAL FLOTATION DEVICE USED - If fatality was water-related was the victim wearing a person flotation device? Mark the appropriate box.

INSTRUCTIONS FOR SECTION 7 - MOTOR VEHICLE ACCIDENT

a. TYPE OF VEHICLE - Mark appropriate box for each vehicle involved. If more than one vehicle of the same type is involved, mark both halves of the appropriate box. USACE vehicle(s) involved shall be marked in left half of appropriate box.

b. TYPE OF COLLISION - Mark appropriate box.

c. SEAT BELT - Mark appropriate box.

INSTRUCTIONS FOR SECTION 8 - PROPERTY/MATERIAL INVOLVED

a. NAME OF ITEM - Describe all property involved in accident. Property/material involved means material which is damaged or whose use or misuse contributed to the accident. Include the name, type, model; also include the National Stock Number *(NSN)* whenever applicable.

b. OWNERSHIP - Enter ownership for each item listed. *(Enter one of the following: USACE; OTHER GOVERNMENT; CONTRACTOR; PRIVATE)*

c. $ AMOUNT OF DAMAGE - Enter the total estimated dollar amount of damage *(parts and labor)*, if any.

INSTRUCTIONS FOR SECTION 9 - VESSEL/FLOATING PLANT ACCIDENT

a. TYPE OF VESSEL/FLOATING PLANT - Select the most appropriate vessel/floating plant from list below. Enter name and place corresponding number in box. If item is not listed below, enter item number for "OTHER" and write in specific type of vessel floating plant.

VESSEL/FLOATING PLANTS

1. ROW BOAT
2. SAIL BOAT
3. MOTOR BOAT
4. BARGE
5. DREDGE/HOPPER
6. DREDGE/SIDE CASTING
7. DREDGE/DIPPER
8. DREDGE/CLAMSHELL, BUCKET
9. DREDGE/PIPE LINE
10. DREDGE/DUST PAN
11. TUG BOAT
12. OTHER

b. COLLISION/MISHAP - Select from the list below the object*(s)* that contributed to the accident or were damaged in the accident.

COLLISION/MISHAP

1. COLLISION W/OTHER VESSEL
2. UPPER GUIDE WALL
3. UPPER LOCK GATES
4. LOCK WALL
5. LOWER LOCK GATES
6. LOWER GUIDE WALL
7. HAULAGE UNIT
8. BREAKING TOW
9. TOW BREAKING UP
10. SWEPT DOWN 0N DAM
11. BUOY/DOLPHIN/CELL
12. WHARF OR DOCK
13. OTHER

INSTRUCTIONS FOR SECTION 10 - ACCIDENT DESCRIPTION

DESCRIBE ACCIDENT - Fully describe the accident. Give the sequence of events that describe what happened leading up to and including the accident. Fully identify personnel and equipment involved and their role*(s)* in the accident. Ensure that relationships between personnel and equipment are clearly specified. Continue on blank sheets if necessary and attach to this report.

INSTRUCTIONS FOR SECTION 11 - CAUSAL FACTORS

a. Review thoroughly. Answer each question by marking the appropriate block. If any answer is yes, explain in item 13 below. Consider, as a minimum, the following:

(1) DESIGN - Did inadequacies associated with the building or work site play a role? Would an improved design or layout of the equipment or facilities reduce the likelihood of similar accidents? Were the tools or other equipment designed and intended for the task at hand?

(2) INSPECTION/MAINTENANCE - Did inadequately or improperly maintained equipment, tools, workplace, etc. create or worsen any hazards that contributed to the accident? Would better equipment, facility, work site or work activity inspections have helped avoid the accident?

(3) PERSON'S PHYSICAL CONDITION - Do you feel that the accident would probably not have occurred if the employee was in "good" physical condition? If the person involved in the accident had been in better physical condition, would the accident have been less severe or avoided altogether? Was over exertion a factor?

(4) OPERATING PROCEDURES - Did a lack of or inadequacy within established operating procedures contribute to the accident? Did any aspect of the procedures introduce any hazard to, or increase the risk associated with the work process? Would establishment or improvement of operating procedures reduce the likelihood of similar accidents?

(5) JOB PRACTICES - Were any of the provisions of the Safety and Health Requirements Manual *(EM 385-1-1)* violated? Was the task being accomplished in a manner which was not in compliance with an established job hazard analysis or activity hazard analysis? Did any established job practice *(including EM 385-1-1)* fail to adequately address the task or work process? Would better job practices improve the safety of the task?

(6) HUMAN FACTORS - Was the person under undue stress *(either internal or external to the job)*? Did the task tend toward overloading the capabilities of the person; i.e., did the job require tracking and reacting to many external inputs such as displays, alarms, or signals? Did the arrangement of the workplace tend to interfere with efficient task performance? Did the task require reach, strength, endurance, agility, etc., at or beyond the capabilities of the employee? Was the work environment ill-adapted to the person? Did the person need more training, experience, or practice in doing the task? Was the person inadequately rested to perform safely?

(7) ENVIRONMENTAL FACTORS - Did any factors such as moisture, humidity, rain, snow, sleet, hail, ice, fog, cold, heat, sun, temperature changes, wind, tides, floods, currents, dust, mud, glare, pressure changes, lightning, etc., play a part in the accident?

(8) CHEMICAL AND PHYSICAL AGENT FACTORS - Did exposure to chemical agents *(either single shift exposure or long-term exposure)* such as dusts, fibers *(asbestos, etc.)*, silica, gases *(carbon monoxide, chlorine, etc.,)*, mists, steam, vapors, fumes, smoke, other particulates, liquid or dry chemicals that are corrosive, toxic, explosive or flammable, by products of combustion or physical agents such as noise, ionizing radiation, non-ionizing radiation *(UV radiation created during welding, etc.)* contribute to the accident/incident?

(9**)** OFFICE FACTORS **-** Did the fact that the accident occurred in an office setting or to an office worker have a bearing on its cause? For example, office workers tend to have less experience and training in performing tasks such as lifting office furniture. Did physical hazards within the office environment contribute to the hazard?

(10) SUPPORT FACTORS - Was the person using an improper tool for the job? Was inadequate time available or utilized to safely accomplish the task? Were less than adequate personnel resources *(in terms of employee skills, number of workers, and adequate supervision)* available to get the job done properly? Was funding available, utilized, and adequate to provide proper tools, equipment, personnel, site preparation, etc.?

(11) PERSONAL PROTECTIVE EQUIPMENT - Did the person fail to use appropriate personal protective equipment *(gloves, eye protection, hard-toed shoes, respirator, etc.)* for the task or environment? Did protective equipment provided or worn fail to provide adequate protection from the hazard*(s)*? Did lack of or inadequate maintenance of protective gear contribute to the accident?

(12) DRUGS/ALCOHOL - Is there any reason to believe the person's mental or physical capabilities, judgment, etc., were impaired or altered by the use of drugs or alcohol? Consider the effects of prescription medicine and over the counter medications as well as illicit drug use. Consider the effect of drug or alcohol induced "hangovers".

b. WRITTEN JOB/ACTIVITY HAZARD ANALYSIS - Was a written Job/Activity Hazard Analysis completed for the task being performed at the time of the accident? Mark the appropriate box. If one was performed, attach a copy of the analysis to the report.

INSTRUCTIONS FOR SECTION 12 - TRAINING

a. WAS PERSON TRAINED TO PERFORM ACTIVITY/TASK? - For the purpose of this section "trained" means the person has been provided the necessary information *(either formal and/or on-the-job (OJT) training)* to competently perform the activity/task in a safe and healthful manner.

b. TYPE OF TRAINING - Mark the appropriate box that best indicates the type of training; *(classroom or on-the-job)* that the injured person received, before the accident happened.

c. DATE OF MOST RECENT TRAINING - Enter YYYYMMDD of the last formal training completed that covered the activity task being performed at the time of the accident.

INSTRUCTIONS FOR SECTION 13 - CAUSES

a. DIRECT CAUSES - The direct cause is that single factor, which most directly lead to the accident. See examples below.

b. INDIRECT CAUSES - **I**ndirect causes are those factors which contributed to but did not directly initiate the occurrence of the accident.

Examples for section 13:

a. Employee was dismantling scaffold and fell 12 feet from unguarded opening.

Direct cause: failure to provide fall protection at elevation. Indirect causes: failure to enforce USACE safety requirements; improper training/motivation of employee *(possibility that employee was not knowledgeable of USACE fall protection requirements or was lax in his attitude towards safety)*; failure to ensure provision of positive fall protection whenever elevated; failure to address fall protection during scaffold dismantling in phase hazard analysis.

b. Private citizen had stopped his vehicle at intersection for red light when vehicle was struck in rear by USACE vehicle. *(Note: USACE vehicle was in proper/safe working condition).*

Direct cause: failure of USACE driver to maintain control of and stop USACE vehicle within safe distance.

Indirect cause: failure of employee to pay attention to driving *(defensive driving).*

INSTRUCTIONS FOR SECTION 14 - ACTION TO ELIMINATE CAUSE*(s)*

DESCRIPTION - Fully describe all the actions taken, anticipated, and recommended to eliminate the cause(s) and prevent reoccurrence of similar accidents/ illnesses. Continue on blank sheets of paper if necessary to fully explain and attach to the completed report form.

INSTRUCTIONS FOR SECTION 15 - DATES FOR ACTION

a. **BEGIN DATE** - Enter the date YYYYMMDD when the corrective action(s) identified in section 14 will begin.

b. **COMPLETE DATE** - Enter the date YYYYMMDD when the corrective action(s) identified in section 14 will be completed.

c. **DATE SIGNED** - Enter YYYYMMDD that the report was signed by the responsible supervisor.

d.e.. **TITLE AND SIGNATURE -** Enter the title and signature of supervisor completing the accident report. For a GOVERNMENT employee accident/illness the immediate supervisor will complete and sign the report. For PUBLIC accidents the USACE Project Manager/Area Engineer responsible for the USACE property where the accident happened shall complete and sign the report. For CONTRACTOR accidents the Contractor's project manager shall complete and sign the report and provide to the USACE supervisor responsible for oversight of that contractor activity. This USACE supervisor shall also sign the report. Upon entering the information required in 15c., 15d., 15e., 15f. and 15g. below, the responsible USACE supervisor shall forward the report for management review as indicated in section 16.

f. **ORGANIZATION NAME** - For GOVERNMENT employee accidents enter the USACE organization name *(Division, Branch, Section, etc.)* of the injured employee. For PUBLIC accidents enter the USACE organization name for the person identified in block 15d. For CONTRACTOR accidents enter the USACE organization name for the USACE office responsible for providing contract administration oversight.

g. **OFFICE SYMBOL** - Enter the latest complete USACE Office Symbol for the USACE organization identified in block 15f.

INSTRUCTIONS FOR SECTION 16 - MANAGEMENT REVIEW (1st)

1ST REVIEW - Each USACE FOA shall determine who will provide 1st management review. The responsible USACE supervisor in section 15d. shall forward the completed report to the USACE office designated as the 1st Reviewer by the FOA. Upon receipt, the Chief of the Office shall review the completed report, mark the appropriate box, provide substantive comments, sign, date, and forward to the FOA Staff Chief *(2nd review)* for review and comment.

INSTRUCTIONS FOR SECTION 17 - MANAGEMENT REVIEW (2nd)

2ND REVIEW - The FOA Staff Chief *(i .e., FOA Chief of Construction, Operations, Engineering, Planning, etc.)* shall mark the appropriate box, review the completed report, provide substantive comments, sign, date, and return to the FOA Safety and Occupational Health Office.

INSTRUCTIONS FOR SECTION 18 - SAFETY AND OCCUPATIONAL HEALTH REVIEW

3RD REVIEW - The FOA Safety and Occupational Health Office shall review the completed report, mark the appropriate box, ensure that any inadequacies, discrepancies, etc. are rectified by the responsible supervisor and management reviewers, provide substantive comments, sign, date and forward to the FOA Commander for review, comment, and signature.

INSTRUCTION FOR SECTION 19 - COMMAND APPROVAL

4TH REVIEW - The FOA Commander shall *(to include the person designated Acting Commander in his absence)* review the completed report, comment if required, sign, date, and forward the report to the FOA Safety and Occupational Health Office. Signature authority shall not be delegated.

Summary of Demolition Operations

Demolition Date	Demolition Location			Munition(s) Destroyed			
	Location Name	Easting	Northing	Item ID	Anomaly Type (1)	MEC/MD Type (2)	Nomenclature

(1) List whether the item is confirmed MEC or is classified as MPPEH.

(2) List whether the item is determined to be UXO or DMM, remained classified as MPPEH, or was determined to be MD (i.e., inert) after the demolition shot.

Manufacturer of Explosives Record of Acquisition

Site Name/Location:	**HGL License Number:** 1-AL-089-20-0F-00632
Project Site Activity: MMRP Project	**Supervisor's Name/Position:**

Date of Manufacture or Other Acquisition	**Lot Number or Manufacturer's Marks of Identification**	**Brand Name, Nomenclature or Description and Size-(when mixing binary materials)**	**Quantity Acquired**	**Name, Address, and License or Permit Number of Distributor**

HGL MR Form 15.02 (Revised August 2017)

Page __________ of __________.

Daily Summary of Magazine Transactions
(Magazine Data Card)

Nomenclature/Brand Name of Manufacturer:	**Lot Number/Manufacturer Mark:**	**Hazard Class/Division:**
Site Location/Name:	**GPS Coordinate:**	**HGL License Number:** **1-AL-089-20-0F-00632**

Date	Quantity In	Quantity Out	Current Balance	Action/Purpose (Receipt/Issue/Inventory)	Printed Name

HGL MR Form 15.03 (Revised August 2017)

Page __________ of __________.

Munitions and Explosives of Concern/Unexploded Ordnance Accountability Record

Site Location:			
Contract Number:		**Delivery/Task Order Number:**	

Grid Coordinates	Anomaly No.	Identification	Date Located	Date Disposed	Method of Disposal	Comments

Reviewed and Accepted (print name)	**Senior UXO Supervisor Signature**	**Date**

HGL MR Form 15.04 (Nov 2010)

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Explosive Material Disposition Record – Bill of Lading

Date:	**Site Name/Location:**	**Grid #/Area or GPS coordinate:**	**Distributee Explosive License #:** 1-AL-089-20-0F-00632

This form will be used as a Bill of Lading anytime explosives are transported in a HGL vehicle under DOT regulations and kept with the explosives driver in the vehicle until the explosives are issued, expended, or returned to storage.

SECTION I. Explosive Material Issued for Demilitarization/Demolition Operations:

Lot Number/Manufacturer's Marks of Identification	Brand Name, Nomenclature, or Description	Quantity	Hazard/ Class	Receipt Initials

Print Explosive Driver Name: N/A	**Explosive Driver Signature:** N/A

SECTION II. Explosive Material Expended by Detonation:

Lot Number/Manufacturer's Marks of Identification	Brand Name, Nomenclature, or Description	Quantity	Hazard/ Class	Receipt Initials

SECTION III. Explosive Material Returned to Storage:

Lot Number/Manufacturer's Marks of Identification	Brand Name, Nomenclature, or Description	Quantity	Hazard/ Class	Receipt Initials

Demolition Supervisor Signature:	**SUXOS Signature:**

PREPARATORY PHASE INSPECTION FORM

Contract No.:	**Project:**	**Date:**

1. Definable Feature of Work (Project Specific)

☐ Mobilization
☐ Location Surveying & Mapping
☐ Anomaly Reacquisition
☐ Vegetation Removal
☐
☐
☐
☐

☐ MEC Surface Removal
☐ Geophysical Data Processing
☐ Intrusive Investigation
☐ Geophysical Data Collection
☐
☐
☐
☐

☐ Anomaly Reacquisition
☐ MEC Subsurface Removal
☐ Munitions Debris/Scrap Disposal
☐ MEC Disposal
☐
☐
☐
☐

2. References (Planning Document Section)

3. Personnel Present

Government Rep Notified:	☐ Yes	☐ No	☐ N/A	**Hours in Advance:**

Name	**Position**	**Organization**

4. Submittals:

Review submittals and/or submittal log. Have all submittals been approved?	☐ Yes		☐ No
If No, what items have not been submitted?			
Are all materials on hand?	☐ Yes	☐ No	☐ N/A
If No, what items are missing?			
Check approved submittals against delivered material. (This should be done as material arrives.) Comments:			

5. Material Storage:

Are materials stored properly?	☐ Yes	☐ No	☐ N/A
If No, what action is taken?			

6. Specifications:

Review each paragraph of specifications.

Discuss procedures for accomplishing the work:

7. Preliminary Work and Permits:

Is preliminary work correct and permits on file?	☐ Yes	☐ No	☐ N/A
If No, what action is taken?			

8. Testing:

Identify test to be performed, frequency, and by whom:

PREPARATORY PHASE INSPECTION FORM

When is it required?			
Where is it required?			
Review Testing Plan:			
Has the test facility been approved?	☐ Yes	☐ No	☐ N/A
If not, what action has been taken?			
9. Results of Surveillance			
☒ Acceptable	☐ Unacceptable	Deficiency #:	NCR #:
Conducted By:		Signature:	

INITIAL PHASE INSPECTION FORM

Contract No.:	**Project:**	**Date:**

1. Definable Feature of Work (Project Specific)

☐ Mobilization	☐ MEC Surface Removal	☐ Anomaly Reacquisition
☐ Location Surveying & Mapping	☐ Geophysical Data Processing	☐ MEC Subsurface Removal
☐ Anomaly Reacquisition	☐ Intrusive Investigation	☐ Munitions Debris/Scrap Disposal
☐ Vegetation Removal	☐ Geophysical Data Collection	☐ MEC Disposal
☐	☐	☐
☐	☐	☐

2. References (Planning Document Section)

3. Planned Attendants

Name	**Position**	**Organization**

4. Observed Condition/Activities:

A. Materials being used are in strict compliance with the contract plans and specifications:	☐ Yes	☐ No	☐ N/A
If not explain:			
B. Procedures and/or work methods witness are in strict compliance with the contract specifications:	☐ Yes	☐ No	☐ N/A
If not explain:			
C. Workmanship is acceptable:	☐ Yes	☐ No	☐ N/A
State areas where improvement is needed:			
D. Safety violation noted:	☐ Yes	☐ No	☐ N/A
If yes, corrective action taken:			

5. Results of Surveillance

☒ Acceptable	☐ Unacceptable	Deficiency #:	NCR #:
Conducted By:		Signature:	

Noncompliance and Corrective Action Report

Report No.:	**Site Name and Location:**	**Gird No. (when required):**
Prepared by:	**Position:**	**Date:**

Nonconformance

Representative Notified:	**Date Notified:**	**Date Corrective Action Plan Due:**

Description of the Nonconformance:

Potential Harm:

Cause:

Corrective Action Plan

Corrective Actions:

Actions to Prevent Recurrence:

Estimated Completion Date:	
Signature:	**Date:**
Corrective Action Approval Signature:	**Date:**
Agency Representative Signature :	**Date :**

Corrective Action Closure

Comments:

Approval Signature:	**Date:**

HGL MEC Form 15.13

Daily Quality Control Report

Contract No:	**Delivery/Task Order:**

Site/Installation Name:	**City:**	**State:**	**Date:**

Site Management

Employer:	**Position:**	**Name:**	**Activity:**
HGL	Project Manager		Management
HGL	Senior UXO Supervisor		Management
HGL	Senior Geophysicist		Geo Supervisor
HGL	UXO Quality Control Specialist		Quality Control
HGL	UXO Safety Officer		Safety

Team ONE

HGL	TIII		UXO
HGL	TII		UXO
HGL	TII		UXO
HGL	TII		UXO
HGL	TII		UXO
HGL	TI		UXO
HGL	TI		UXO

Team TWO

Parsons	Geophysicist		DGM
Parsons	UXO Escort		Avoidance
Parsons	UXO Escort		Avoidance
Parsons	Geophysicist		DGM
Parsons	Geophysicist		DGM

Team THREE

Team FOUR

Daily Quality Control Report

Worked performed today by subcontractors:

3. Inspections performed (include name of team present, specifications, plans and submittals required for definable feature of work [DFOW]). Indicate 3-Phase inspection level with by inserting: Preparatory = P; Initial = I; Follow-up = F.			
Phase	**Team name:**	**DFOW** **(Insert project specific DFOWs)**	**Comments**
	Site	Mobilization	Complete
F	CMS Biologist	Environmental Survey and Beach Monitoring	Ongoing
	Site	Site Preparation	Complete
F	Parsons Geo	Conduct Validation Seeding, QC Seeding, and Construct IVS	Ongoing
F	Parsons Geo	Assemble and Verify Correct Operation of Geophysical Sensor to be used for the Detection Survey (IVS Data Collection)	Ongoing
F	Parsons Geo	Conduct Detection Survey	Ongoing
F	Parsons Geo	Conduct Detection Survey Processing and Target Selection	Ongoing
F	Parsons Geo	Validate Dynamic Survey and Cued Target List	Ongoing
F	Parsons Geo	Assemble Advanced Geophysical Sensor and Test Sensor at IVS	Ongoing
F	Parsons Geo	Collect Cued Data	Ongoing
F	Parsons Geo	Conduct Cued Data Processing	Ongoing
	Parsons Geo	Classify Anomalies and Make Dig/No-Dig Decisions	
	Parsons Geo	Validate Cued Survey and Classification	
F	UXO 1	Intrusive Investigation	Ongoing
F	UXO 1	Verify Intrusive Results	Ongoing

Daily Quality Control Report

		Conduct Final DUA	
F	**UXO 1**	**Analog Removal**	**Ongoing**
F	**UXO 1/SUXOS**	**MPPEH/MEC Handling, Certification, and Disposal**	**Ongoing**
P, I		**Demobilization**	
		MC Sampling	

Grid #	**Grid #**	**Grid #**	**Grid #**	**Grid #**	**Grid #**	**Grid #**	**Grid #**

GRID INSPECTIONS PERFORMED:

	QC inspections completed to date:				QA inspections completed to date:			
	Pass	**Fail**	**Total**		**Pass**	**Fail**	**Total**	
	0	0	0		0	0	0	

General Site Inspection	**Team (indicate by: UXO = U; or Geo = G; and No:**					**Pass**	**Fail**	**NA**
Proper work attire (PPE)	U	G				X		
Equipment calibration check	U	G						X
Vehicle condition	U	G				X		
Equipment condition	U	G				X		
Emergency equipment	U	G				X		
Proper grid layout	U	G						X
Proper search techniques	U	G						X
Team leader daily log	U	G				X		
SUXOS daily log	U							X
GIS and map data		G						X
Exclusion zone	U							X
Field office interior	U					X		
Field office exterior	U					X		
Proper demolition operations								X
Safety violations						None		

4. Soil samples taken:

Post-Detonation: ☐ No ☐ Yes ☒ None required

5. Verbal instructions received by the Government representative or client and actions taken:

None

6. Non-conformances/deficiencies reported:

None

CERTIFICATION: *I certify the above information is complete and correct and that I, or my representative, have inspected all work identified on this report performed by HGL and our subcontractor(s) and have determined to the best of my knowledge and belief that noted work activities are in compliance with the plans and specifications, except as may be noted above.*

Daily Quality Control Report

UXO Quality Control Specialist	Signature

Safety Meeting/Training Attendance Log

Date:		**Time:**		**Conducted by:**	David Briggs

Site Name/Location:			
Contract Number:	Click here to enter	**Delivery/Task Order Number:**	
Project Manager:		**Site Manager** (when applicable):	N/A
(Senior) UXO Supervisor:		**Site Safety Officer/Unexploded Ordnance Safety Health Officer:**	

Training Provided:
☐ Initial Site Hazard
☐ Weekly Safety Training
☐ Daily Safety Meeting
☐ Task/Hazard Specific
☐ Other:

Weather Conditions: ☐ Fair ☐ Poor
Temperature (Low/High): to °F
Wind speed: mph
Direction:
Precipitation: %
Humidity: %

I. TRAINING TOPICS COVERED

☐ Planned Site Activities
☐ Demolition Operations
☐ Site Controls
☐ Exclusion Zone/Personnel Limits
☐ Site Communications
☐ Physical Hazards
☐ Heat or Cold Stress
☐ Biological Hazards
☐ Chemical Hazards
☐ Routes of Chemical Exposure
☐ Chemical Exposure Symptoms
☐ Level/Type of PPE
☐ Respirator Use
☐ Decontamination Procedures
☐ Emergency Procedures/Route
☐ First Aid Procedures
☐ Buddy Team Procedures
☐ Other (describe topic(s) below)

Explain: ____
____.

Hospital/Clinic:	**Address:**	**Phone:**

II. SITE PERSONNEL / TRAINING ATTENDEES (Continued on 2nd page)

	Name	Signature	Company
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			

III. SAFETYBRIEF / TRAINING VERIFICATION

I certify that the personnel listed on this roster have received the safety and health training described above.

________________________________ ________________________________

Site Manager or Senior UXO Supervisor

Site Safety and Health Officer or UXO Safety Officer

HGL MR Form 15.18

Page ______ of ______.

Safety Meeting and Training Attendance Log

II. SITE PERSONNEL / TRAINING ATTENDEES (continued from 1st page)			
	Name	Signature	Company
11.			
12.			
13.			
14.			
15.			
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50.			

HGL MEC Form 15.18 (Oct 2010)

Page ______ of ______.

Tailgate Safety Meeting Log

HGL MR Form 15.19

Date:	**Time:**	**Team No:**
Site Name/Location:		**Grid/Location:**

1. SAFETY TOPICS DISCUSSED:

☐ Site Description
☐ Site Controls
☐ Personal Protective Equipment
☐ Emergency Procedures / Equipment
☐ Site Evacuation
☐ Physical/Biological Hazards
☐ Heat or Cold Stress
☐ Communication/Radio Procedure
☐ Environmental Concerns/Hazards
☐ Emergency Procedures/Route
☐ First Aid Procedures
☐ Injury Reporting
☐ Safe Work Practices
☐ Other:
☐ Other:
☐ Other:

2. TASK OPERATION AND REMARKS:

____

____

3. ATTENDEES:

	Print Name	Signature	Company
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			
11.			
12.			
13.			
14.			

Meeting Conducted by:	**Signature:**

Health and Safety Site Visitor Log

Project Name:		**Project Number:**	Click here to enter	**Delivery/Task Order:**	
Site Name:		**Location:**			

This form shall be used to track entry into and departure from the ***EXCLUSION ZONE, CONTAMINATION REDUCTION ZONES, OR OTHER WORK ZONES*** on all HydroGeoLogic, Inc. sites. All Personnel shall sign in and out on the form by printing their name, initializing the form and noting the time in/out.

Date	**Name**	**Representing**	**Purpose of visit**	**Escort Required**		**Equipment/ PPE Level**	**Time**	
				Yes	**No**		**In**	**Out**

Health and Safety Site Visitor Log

Date	Name	Representing	Purpose of visit	Escort		Equipment/ PPE Level	Time	
				Yes	No		In	Out

HGL MR Form 15.21 (Sep 2008)

Page ______ of ______.

Vehicle Safety Inspection Checklist

Site name/location:		
Inspection date:	**Supervisor:**	**Rental company:**
Mileage:	**Vehicle make:**	**Vehicle license number:**

1. DOCUMENTATION:	**Pass**	**Fail**	**5. BRAKES:**	**Pass**	**Fail**
Registration and License Plate			Hand/Emergency		
Insurance			Service		
Emergency Route Map & Phone #s					
2. TIRES:	**Pass**	**Fail**	**6. BELTS:**	**Pass**	**Fail**
Pressure			Proper tension		
Condition			Condition		
3. EQUIPMENT:	**Pass**	**Fail**	**7. GENERAL:**	**Pass**	**Fail**
Fire extinguishers ✱			Windshield		
First Aid/CPR/Burn Kits			Windshield Wipers		
Emergency Route Directions/Map			Windows (Condition/Operation)		
Vehicle Registration/Rental Contract			Seat Belts		
HGL Insurance Coverage Card			Steering		
Eyewash Kits			Horn		
Spare Tire			Gas Cap		
Tire Changing Equipment			Mirrors		
Tie downs✱			Door/Window Handles/Latches		
Chocks ✱			Cleanliness		
Placards ✱			Exhaust System ✱		
4. FLUID LEVELS:	**Pass**	**Fail**	**8. LIGHTS:**	**Pass**	**Fail**
Oil			Headlights (high & low)		
Coolant			Brake Lights		
Brake			Parking		
Steering			Back-up		
Transmission			Turn Signals		
Windshield Wiper			Emergency Flashers		
Fluid Leaks			Interior Lights		
5. Oil Change (mileage):					

Notes:

1. To be used weekly for all vehicles EXCEPT explosive carriers that must be inspected prior to each explosives transport.
2. Items marked with an ✱ are required for explosive carriers and must be inspected prior to each use IAW HGL MR SOP 15.00 Section 4.6.
3. All forms with failures must note the deficiencies and forward a copy of this form within two working days to the Site Safety and Health Officer

Description of deficiencies:

Corrective actions to be taken:

Inspection conducted by:	**Printed name:**	**Signature:**
Deficiencies corrected by	**Printed name:**	**Signature:**

HGL MR Form 15.23

Explosive Storage and Security Survey Checklist

Project Site (name, city and state):		
Inspection conducted by/position:	**Signature**	**Date**
UXOQCS or UXOSO:	**Signature**	**Date**
Reviewed by SUXOS:	**Signature**	**Date**

	YES	NO	NA	COMMENTS
1. **Publications**	**YES**	**NO**	**NA**	**COMMENTS**
a. ATF Federal Explosive Law & Regulations, ATF P 5400.7,	☐	☐	☐	
b. HGL SOP 15.00 Explosive Accountability & Management	☐	☐	☐	
c. HGL SOP 15.02 Explosive Storage, Inspection & Security	☐	☐	☐	
2. **Explosive Storage**	**YES**	**NO**	**NA**	**COMMENTS**
a. Proper explosive storage magazines, Type 2 conforming to BATF standard	☐	☐	☐	
b. Placards. Each magazine properly placarded with DOT Haz Class/Division symbol	☐	☐	☐	
c. Explosive compatibility groups. Separated into the appropriate Haz Class/Division	☐	☐	☐	
d. Physical Security survey conducted and documented	☐	☐	☐	
e. Locks met BATF standards	☐	☐	☐	
f. Key control system established and functional	☐	☐	☐	
g. Lightening Protection.	☐	☐	☐	
1) Magazine constructed of minimum 3/16 inch metal	☐	☐	☐	
2) Magazine grounded	☐	☐	☐	
3) Magazine located 6 feet from nearest fence	☐	☐	☐	
4) Installation/Client/Property Owner standards met	☐	☐	☐	
h. Fire Protection. Minimum size/type fire extinguisher located within 30 feet of storage magazine	☐	☐	☐	
1) Proper fire division symbol at entrance to storage site	☐	☐	☐	
5) Fire fighting control plan established in APP/SSHP	☐	☐	☐	
6) Area surrounding magazine free of rubbish, brush, dry grass, trees for a minimum of 25 foot.	☐	☐	☐	
i. Magazine location site meets IBD/PTR distances	☐	☐	☐	
j. Commercial explosives being stored in DoD facilities require DoD HC/SCG approval	☐	☐	☐	

k.	Adequate earth cover used to meet IBD & PTR distances	☐	☐	☐	
3.	**Explosive Accountability & Management**	**YES**	**NO**	**NA**	**COMMENTS**
a.	Explosive accountability and management responsibilities and organization established	☐	☐	☐	
b.	Explosive material purchase/receipt signature authority on-hand	☐	☐	☐	
c.	Accountability records & tracking established	☐	☐	☐	
d.	MEC final disposition accountability tracking records established	☐	☐	☐	
e.	Lost, missing and stolen procedures in place	☐	☐	☐	
f.	Disaster preparedness plan in place	☐	☐	☐	
g.	Receipt procedures accounting for each item of explosives properly documented on-site	☐	☐	☐	
h.	Individuals authorized to receive issue and transport Identified and granted explosive access by the BATF FELC.	☐	☐	☐	
4.	**Explosive Transportation**	**YES**	**NO**	**NA**	**COMMENTS**
a.	Hazardous waste manifest on-hand and maintained	☐	☐	☐	
b.	Explosive Transport Vehicle	☐	☐	☐	
	1) Vehicle inspection checklist on hand	☐	☐	☐	
	2) Proper DOT placards, lettering, and/numbering on hand	☐	☐	☐	
	3) Operators licensed (CDL/HazMat endorsement)	☐	☐	☐	
	4) First aid kit on board vehicle	☐	☐	☐	
	5) Communication protocols established	☐	☐	☐	
	6) Day boxes on hand	☐	☐	☐	

Munitions Response Project
Quality–Safety Assurance Field Audit/Self Assessment Checklist

PROJECT SITE: ______________________ **AUDIT DATE:** ______________________

CONTRACT NO./DO/TO: ______________________ **CONDUCTED BY:** ______________________

1. REVIEW SCOPE OF WORK (DO/TO), ESS/ESP AND WORK PLAN	**REFERENCES**	**YES**	**NO**	**N/A**	**COMMENTS**
a. Clearance areas & objectives clearly identified	PWS; WP; DoD 6055.9-M; ESS/ESP	☐	☐	☐	
b. Check for changes to WP are up to date and change pages posted?	WP	☐	☐	☐	
c. Proper depth of clearance identified?	WP, ESS/ESP	☐	☐	☐	
d. Proper target MEC identified	WP, ESS/ESP	☐	☐	☐	
e. DDESB-approved ESS/ESP:	ESS/ESP, EM 385-1-97	☐	☐	☐	
1) Do current site conditions comply/met the ESS/ESP requirements?		☐	☐	☐	
2) Is an amendment required base on current or foreseen changes to MEC/explosive demolition operations?		☐	☐	☐	
f. Is the munition with the greatest fragmentation distance (MGFD) identified?	WP, ESS/ESP	☐	☐	☐	
g. Detection depth and target depth specified	WP, ESS/ESP	☐	☐	☐	
h. EZ/MSDs identified in the WP?	WP, ESS/ESP	☐	☐	☐	
i. Corrective action standards established?	WP, HGL MR Form 15.13	☐	☐	☐	
2. DOCUMENTATION/PUBLICATION REQUIREMENTS		**YES**	**NO**	**N/A**	**COMMENTS**
a. Approval letter for WP?		☐	☐	☐	
b. Approval letter for notice to proceed?		☐	☐	☐	
c. Maps in the WP identifying the proper EZ	WP; ESS/ESP	☐	☐	☐	
d. Approval letter, FAA (NOTAM)/Notice to Mariners?	AR 95-10/AFR 11-208/OPNAVINST 3721.20B	☐	☐	☐	

e. Certificate of grounding, lightning protection for explosive storage facilities	DoD 6055.9-M; HGL SOP 15.02	☐	☐	☐	
f. WP on-site?		☐	☐	☐	
g. DDESB-approved ESS/ESP on-site?	PWS; DoD 6055.9-M	☐	☐	☐	
h. All associated work task SOPs on-site?		☐	☐	☐	
i. Medical clearance (personal physical exam)?	29 CFR 1910.120 (f)	☐	☐	☐	
j. Current copy of USACE UXO resume database on-site?		☐	☐	☐	
k. UXO technician letter submitted to USACE contracting officer (when required)?		☐	☐	☐	
l. EM 385-1-1on-site?		☐	☐	☐	
m. EM 385-1-97 on-site?		☐	☐	☐	
n. APP/SSHP on-site?		☐	☐	☐	
o. Recording, reporting and implementing lessons learned procedures established?		☐	☐	☐	
p. Security clearances/site access passes obtained?		☐	☐	☐	
q. Dig permits approved and on-site?		☐	☐	☐	
3. QUALITY CONTROL PLAN/FILES ESTABLISHED	WP; HGL SOP 15.22	**YES**	**NO**	**N/A**	**COMMENTS**
a. Three-Phase QC training established?		☐	☐	☐	
1) QC Preparatory checklist completed?	HGL MR Form 15.10	☐	☐	☐	
2) QC Initial checklist completed?	HGL MR Form 15.11	☐	☐	☐	
3) QC Final checklist completed?	HGL MR Form 15.11	☐	☐	☐	
b. Daily QC Reports?	HGL MR Form 15.14	☐	☐	☐	
c. QC Surveillance Reports completed weekly for each field team?	HGL MR Form 15.15	☐	☐	☐	

d. Daily site production report?	HGL MR Form 15.27	☐	☐	☐	
e. Government acceptance of work record on file?		☐	☐	☐	
4. APP OR SSHP	EM 385-1-1; PWS	**YES**	**NO**	**N/A**	**COMMENTS**
a. Emergency notification list posted & available?	WP, APP/SSHP	☐	☐	☐	
b. Emergency routes maps available & issued to each team?	WP, APP/SSHP	☐	☐	☐	
c. Hazard analysis & risk assessment for all tasks/equipment operations identified?	APP/SSHP and AHAs	☐	☐	☐	
d. All training requirements met**:**	29 CFR 1910.120(e), HGL SOP 15.23	☐	☐	☐	
1) General site workers HAZWOPER qualified and possess current 8-hour annual refresher?	29 CFR 1910.120(e), HGL SOP 15.23	☐	☐	☐	
1) All site workers received site specific hazards awareness training and indoctrination IAW with the provisions of the APP/SSHP?	HGL SOP 15.23	☐	☐	☐	
2) All safety and hazards awareness training and meetings properly documented?	HGL MR Form 15.18	☐	☐	☐	
e. All personnel on-site in proper PPE for tasked performed?	EM 385-1-1, 29 CFR 1910.120/134 & 132, APP/SSHP	☐	☐	☐	
f. Minimum of 2 personnel on-site First Aid/CPR certified?	EM 385-1-1, Section 03.A.03	☐	☐	☐	
g. First aid equipment immediately available to field teams?	EM 385-1-1; SSHP	☐	☐	☐	
h. Emergency eye-washes/showers comply with ANSI Standards?	ANSI Z-385.1	☐	☐	☐	
i. Fire extinguishers (specific type, size and location)?	APP/SSHP and EM 385-1-1	☐	☐	☐	
j. Ongoing safety & health training program?	HGL SOP 15.23	☐	☐	☐	
k. Work tasks identified in hazard analysis?	APP/SSHP	☐	☐	☐	
l. Do field team supervisors conduct and document daily task hazard safety briefing?	APP/SSHP and HGL Form 15.19	☐	☐	☐	
m. All approved MSDS(s) on-site?	APP/SSHP and HGL SOP 15.23	☐	☐	☐	
n. List of all MSDS inventory items and location on-site?	APP/SSHP and HGL SOP 15.23	☐	☐	☐	

o. Adequate site control during MEC operations?	WP and ESS/ESP	☐	☐	☐	
p. Visitors/safety brief log current and updated?	WP, HGL SOP 15.23	☐	☐	☐	
5. TECHNICAL MANAGEMENT PLAN	WP	**YES**	**NO**	**N/A**	**COMMENTS**
a. Procedure for discovery of MEC?	WP and ESS/ESP	☐	☐	☐	
b. Project grid size, layout and lane width established?	WP/PWS	☐	☐	☐	
c. Procedures established for changed site conditions	WP	☐	☐	☐	
d. Proper test sources/test plot established	WP	☐	☐	☐	
e. Organization chart current and indicates personnel assignments, duties and responsibilities	WP	☐	☐	☐	
f. MPPEH reporting procedures established	PWS; DoD 60559-M; DoD 4160.21-M; DoD 4140.62; EM 385-1-97	☐	☐	☐	
g. MPPEH inspection/certification procedures established	DoD 60559-M; DoD 4160.21-M; DoD 4140.62; EM 385-1-97; ESS/ESP, HGL SOP 15.03	☐	☐	☐	
h. Managing, reporting, venting and disposing of munitions debris and range-related debris established	DoD 60559-M; DoD 4160.21-M; DoD 4140.62; EM 385-1-97; ESS/ESP, HGL SOP 15.03	☐	☐	☐	
i. Limitations posed and ability of detection systems chosen?		☐	☐	☐	
6. LOCATION, MAPPING CULTURAL SURVEY PLAN	EM 385-1-97; HGL SOP 15.12	**YES**	**NO**	**N/A**	**COMMENTS**
a. Professional land surveyor/cultural survey required?		☐	☐	☐	
b. Survey teams received MEC avoidance and site safety brief?		☐	☐	☐	
c. Survey teams have UXO Technician II or above escort provided for MEC anomaly avoidance?		☐	☐	☐	
d. Grid stake, locations swept with geophysical equipment before driving stakes in ground?		☐	☐	☐	
e. Coordinates/survey notes recorded?		☐	☐	☐	

Munitions Response Project
Quality–Safety Assurance Field Audit/Self Assessment Checklist

7. VEGETATION REMOVAL	WP; SSHP; HGL SOP 15.10	**YES**	**NO**	**N/A**	**COMMENTS**
a. Vegetation removal localized as required?		☐	☐	☐	
b. UXO team pre-sweeps and marks MEC hazards?		☐	☐	☐	
c. Equipment operated to prevent impact with possible surface MEC/UXO?		☐	☐	☐	
d. Cutting does not present impalement hazard?		☐	☐	☐	
e. UXO Technician II or abovel monitoring cutting operation?		☐	☐	☐	
f. Discovered MEC hazards clearly identified and area marked appropriately?		☐	☐	☐	
8. QUALITY CONTROL PLAN	WP; APPLICABLE DID; HGL SOP 15.23	**YES**	**NO**	**N/A**	**COMMENTS**
a. QC operational/audits conducted WP?		☐	☐	☐	
b. QC grid/transects established and inspection pattern adequate?		☐	☐	☐	
c. QC training conducted and documented (preparatory, initial and follow-on surveillance)?		☐	☐	☐	
d. Pass/fail criteria clearly established?		☐	☐	☐	
e. QC check results recorded?		☐	☐	☐	
9. FACILITIES	**EM 385-1-1**	**YES**	**NO**	**N/A**	**COMMENTS**
a. Adequate work space, equipment and sanitary facilities?		☐	☐	☐	
b. Good housekeeping practices, no fire hazards or tripping hazards present?		☐	☐	☐	
c. Approved and suitable containers for flammable, toxic and explosive materials?	Section 9	☐	☐	☐	
d. Approved/adequate Explosive Storage Facilities?	DoD 6555.9-M; 27 CFR Part 555, Subpart K; ESS/ESP	☐	☐	☐	
e. Fire/emergency exits clear/unbarred?	Sections 1, 9, & 26	☐	☐	☐	
f. Personnel occupancy limits maintained and posted (on-site office trailers)		☐	☐	☐	

g. Adequate site security and facilities properly secured after hours		☐	☐	☐	
h. Toilets and washing facilities?	EM 385-1-1 Section 02.B - 02.C	☐	☐	☐	
i. Trailers and temporary structures properly anchored?	EM 385-1-1, Section 04	☐	☐	☐	
10. EQUIPMENT		**YES**	**NO**	**N/A**	**COMMENTS**
a. Automatic External Defibrillator (AED) readily available to field crews at remote locations?	EM 385-1-1, Section 03	☐	☐	☐	
b. At least one dry chemical or CO_2 fire extinguisher, minimum rating 5-BC on-site	EM 385-1-1 Section 16	☐	☐	☐	
c. Tools appropriate and serviceable		☐	☐	☐	
d. Appropriate weather monitoring devices on-hand (handheld weather station; lightning detector)?		☐	☐	☐	
e. PPE present, serviceable/utilized?	EM 385-1-1, Section 05	☐	☐	☐	
f. Equipment calibrated with last and next cal date annotated?	Operators Manual	☐	☐	☐	
g. Survey equipment inspected/serviceable?	Operators Manual	☐	☐	☐	
h. Heavy equipment inspected/serviceable?	Operators Manual, EM 385-1-1 Section 16	☐	☐	☐	
i. Two separate means of communications, radio(s)/cell phone, land line(s)?		☐	☐	☐	
j. Geophysical equipment on-hand, serviceable & identified in WP?	EM 1110-14009; Operators Manual	☐	☐	☐	
k. Copy of manufacturer's instructions & recommendations maintained with all hand and power Tools?	EM 385-1-1, Section 13	☐	☐	☐	
11. EXPLOSIVE STORAGE	27 CFR PART 555, Subpart K; AFMAN 91-201, AR 190-11; NAVSEA OP 5; HGL SOP 15.00	**YES**	**NO**	**N/A**	**COMMENTS**
a. Proper explosive storage magazines, Type 2 conforming to ATF standard?	27 CFR §555.206 (ATF P 5700.04)	☐	☐	☐	
b. Placards. Each magazine properly placarded with DOT Haz Class/Division symbol?		☐	☐	☐	
c. Explosive compatibility groups. Separated into the appropriate Haz Class/Division?		☐	☐	☐	

d.	Physical Security survey conducted and documented?	HGL SOP 15.00 and 15.02	☐	☐	☐	
e.	Security locks met ATF standards?	27 CFR §555.208.(a)(4) (ATF P 5700.04)	☐	☐	☐	
f.	Key control system established and functional?	HGL SOP 15.00 and 15.02	☐	☐	☐	
g.	Lightening Protection”		☐	☐	☐	
4)	Magazine constructed of minimum 3/16 inch metal?	2 7 CFR Part 555, Subpart K, NFPA 780	☐	☐	☐	
5)	Magazine grounded?	NFPA 780	☐	☐	☐	
6)	Magazine located 6.5 feet from nearest fence?		☐	☐	☐	
7)	BRAC, IRP, FUDS and active installation standards met?		☐	☐	☐	
h.	Fire Protection. Minimum size/type fire extinguisher located within 30 feet of storage magazine:		☐	☐	☐	
1)	Proper fire division symbol at entrance to storage site?		☐	☐	☐	
8)	Fire fighting control plan established & posted?		☐	☐	☐	
9)	Area surrounding magazine free of rubbish, brush, dry grass, trees for a minimum of 25 foot?		☐	☐	☐	
i.	Magazine site location meets IBD/PTR distances?		☐	☐	☐	
j.	Commercial explosives being stored in DoD facilities require DoD HC/SCG approval?	DA PAM 385-64; AR 385-64; AF 91-210; NAVSEA OP 5	☐	☐	☐	
k.	Adequate earth cover used to meet IBD & PTR distances?		☐	☐	☐	
12.	**EXPLOSIVE MANAGEMENT PLAN**	27 CFR Part 555; 49 CFR; DoD 6055.9-M; AFMAN 91-201; AR 190-11; NAVSEA OP 5; DIDs; HGL SOP 15.00, WP	**YES**	**NO**	**N/A**	**COMMENTS**
a.	Explosive accountability and management responsibilities and organization?		☐	☐	☐	
b.	Explosive material purchase/receipt signature authority on-hand?		☐	☐	☐	
c.	Inventory conducted once every 7 days?		☐	☐	☐	
d.	Special inventories conducted annually or as prescribed?		☐	☐	☐	

e. Accountability records maintained?		☐	☐	☐	
f. MEC final disposition/accountability records maintained?	HG L MR Form 15.04	☐	☐	☐	
g. Key control/security log maintained?	HGL MR Form 15.26	☐	☐	☐	
h. Lost, missing and stolen procedures in place?		☐	☐	☐	
i. Disaster preparedness plan in place?		☐	☐	☐	
j. Each explosive item identified in equipment plan (not required on FFP task orders)?		☐	☐	☐	
k. Initial receipt procedures & documentation on-site?		☐	☐	☐	
l. Explosives specified within WP being used at project site?		☐	☐	☐	
m. Receipt procedures accounting for each item of explosives properly documented on-site?		☐	☐	☐	
n. Individuals authorized to receive, issue and transport identified and granted explosive access by the ATF FELC?	EPQ ATF Form 5400.28	☐	☐	☐	
o. Reconciliation, receipt documents on-site?		☐	☐	☐	
p. Duplicate DSMT-magazine data cards (magazine/facility)?	HGL SOP 15.00, HGL MR Form 15.02	☐	☐	☐	
13. TRANSPORTATION OF EXPLOSIVE MATERIALS	DOT 49 CFR PARTS 100 TO 199; AR 385-64; EM 385-1-1 SEC 29; HGL SOP 15.00, WP, ESS/ESP	**YES**	**NO**	**N/A**	**COMMENTS**
a. Hazardous waste manifest on-hand and maintained?	HGL MR Form 15.06	☐	☐	☐	
b. MEC hazard properly classified?	DoD TB 700-2	☐	☐	☐	
c. Approved transportation plan?	ESS/ESP	☐	☐	☐	
d. MEC transportation training IAW DOT and State requirements?	49 CFR, Part 172	☐	☐	☐	
e. Explosive Transport Vehicle:	HGL SOP 15.22	☐	☐	☐	
1) Pre-operational inspection properly conducted?	HGL SOP 15.22	☐	☐	☐	
2) Proper DOT placards, lettering, and/numbering?	HGL MR Form 15.06	☐	☐	☐	

3) Operators licensed (CDL/HazMat endorsement)?	HGL MR Form 15.06	☐	☐	☐	
4) Explosive transport compatibility requirements maintained?	HGL MR Form 15.06	☐	☐	☐	
5) Cargo properly loaded, blocked and braced?	HGL MR Form 15.06	☐	☐	☐	
6) First aid kit on board vehicle?	HGL MR Form 15.06	☐	☐	☐	
7) Has communication capability?	HGL MR Form 15.06	☐	☐	☐	
8) Blasting caps stored separate and in appropriate container (cap box)?	HGL MR Form 15.06	☐	☐	☐	
9) NEW limits maintained for each transport container?	HGL MR Form 15.06	☐	☐	☐	
f. No Flame-producing devices in vehicle or on persons?	HGL MR Form 15.06	☐	☐	☐	
14. MEC OPERATIONAL PLAN	WP; EP 1110-1-18	**YES**	**NO**	**N/A**	**COMMENTS**
a. Is there a centrally located catalog of MEC ID guides associated to the MRA/MRS available to all UXO Technicians on-site?		☐	☐	☐	
b. Defined methodology followed:		☐	☐	☐	
a. SUXOS conducted physical check prior to MEC clearance/sweep operations?		☐	☐	☐	
b. Daily health and safety meeting conducted and documented by the UXOSO/SSHO?	APP/SSH, HGL SOP 15.23	☐	☐	☐	
c. Geophysical/magnetometer equipment used:	Applicable SOPs	☐	☐	☐	
1) Pre-operational checks conducted?		☐	☐	☐	
2) Calibration/test strip used?		☐	☐	☐	
3) Calibration/test results annotated in team and equipment test log by serial number?	WP, HGL MR Form 15.16	☐	☐	☐	
d. SUXOS on-site supervising removal operation?	WP, ESS/ESP	☐	☐	☐	
e. Operational teams operating IAW:	WP; ESS/ESP	☐	☐	☐	
1) UXO Supervisor conducted physical check MEC clearance/sweep prior to operation?		☐	☐	☐	

2) Supervisor conducted pre-operational tail-gate safety briefing?		☐	☐	☐	
3) Supervisor ensures public barricades established prior to MEC operation?		☐	☐	☐	
4) Supervisor ensures all personnel are wearing the required PPE?		☐	☐	☐	
5) Supervisor ensures proper grid selected?		☐	☐	☐	
6) Supervisor ensures individual MEC lanes established?		☐	☐	☐	
7) Team using correct detector/magnetometer instruments & model specified in WP?		☐	☐	☐	
8) Detector instruments checked against test source daily prior to MEC operations?		☐	☐	☐	
9) Results of detector check, by individual instrument serial # annotated in field log book and on equipment maintenance/ calibration log?		☐	☐	☐	
10) Supervisor ensures proper sensitivity setting based on check sources?		☐	☐	☐	
11) Anomalies/contacts are clearly marked and logged?		☐	☐	☐	
12) Supervisor ensures clearance/sweep operation results recorded?		☐	☐	☐	
13) All MPPEH, RRD, MD & CD examined by at least a one UXO Technician III and one UXO Technician II?		☐	☐	☐	
14) Supervisor ensures all MEC-MDEH clearly marked IAW WP/ESS/ESP?		☐	☐	☐	
a) MPPEH processing IAW	PWS/SOW, EM 1110-104009/EM 385-1-97, ESS/ESP and HGL SOP 15.03	☐	☐	☐	
b) Supervisor ensures MD, RRD, CD is collected and inspected?	HGL SOP 15.03	☐	☐	☐	
c) QC operation IAW?	WP	☐	☐	☐	
15) Results of MEC operation recorded?	WP, HGL SOP 15.01	☐	☐	☐	
f. All MEC/UXO is clearly marked and logged?	WP	☐	☐	☐	
g. Non-MD being collected?	WP; HGL SOP 15.03	☐	☐	☐	
h. MD inspected, vented and segregated?	WP; ESS or ESP; HGL SOP 15.03;	☐	☐	☐	

Munitions Response Project
Quality–Safety Assurance Field Audit/Self Assessment Checklist

4. MPPEH MANAGEMENT	DOT 49 CFR, DoD 6055.9-M, DoD 4140.62, EM 385-1-97, HGL SOP 15.03	**YES**	**NO**	**N/A**	**COMMENTS**
a. Are UXO Technicians involved in the MPPEH inspection/certification process properly training and understand their responsibilities?		☐	☐	☐	
b. Have all UXO Technicians performing MPPEH inspection/certification sign the SOP? acknowledgement?		☐	☐	☐	
c. Project startup procedures being followed?		☐	☐	☐	
d. Is the MPPEH inspection and certification process being properly followed?		☐	☐	☐	
e. Are inspected material and uninspected material properly segregated and procedures in place to prevent comingling?		☐	☐	☐	
f. Are storage container properly sealed, locked and secured from general access?		☐	☐	☐	
g. Are storage container properly labeled to distinguish between inspected material and uninspected material?		☐	☐	☐	
h. Weekly and spot check inspection performed?		☐	☐	☐	
i. Is chain of custody, certification, transfer/release records accurate and complete?	HGL MR Forms 15.07/15.08 and DD FORM 1348–1A	☐	☐	☐	
5. DISPOSAL OPERATIONS PLANNED ON-SITE	WP; ESS/ESP; HGL SOP 15.01	**YES**	**NO**	**N/A**	**COMMENTS**
a. MEC/UXO/MDEH disposal method IAW approved procedures?		☐	☐	☐	
b. Adequate security for explosive operation?		☐	☐	☐	
c. Disposal notification list available?		☐	☐	☐	
d. All necessary notifications made?		☐	☐	☐	
e. Movement of MEC items, or is MEC consolidation feasible IAW ESS/ESP		☐	☐	☐	
f. Protective measures/tamping being used/appropriate for MEC being destroyed?		☐	☐	☐	
g. Disposal procedures IAW:	60A-1-1-31	☐	☐	☐	
1) Misfire procedures properly performed (electric)?		☐	☐	☐	

2) Misfire procedures properly performed (non-electric)?		☐	☐	☐	
3) Misfire procedures properly performed (remote firing device)?		☐	☐	☐	
6. MUNITIONS CONSTITUENTS (MC) SAMPLING AND ANALYSIS PLAN	DOT 49 CFR, DoD 6055.9-M, HGL SOP 15.14/15	**YES**	**NO**	**N/A**	**COMMENTS**
a. Key personnel identified?		☐	☐	☐	
b. Quality assurance/control responsibilities identified?		☐	☐	☐	
c. Collection sample procedures established and followed?		☐	☐	☐	
d. Local courier identified?		☐	☐	☐	
e. Are local field logs maintained?		☐	☐	☐	
7. TRANSPORTATION PLAN	DOT 49 CFR, DoD 6055.9-M, HGL SOPs 15.14 and 15.15	**YES**	**NO**	**N/A**	**COMMENTS**
a. Pre-operational vehicle inspection logged/recorded?		☐	☐	☐	
a. Are vehicles operators licensed for specific vehicle types?		☐	☐	☐	
b. All vehicles licensed for specific vehicle type?		☐	☐	☐	
c. Fire fighting & first aid equipment on board?		☐	☐	☐	
d. Cargo properly segregated/blocked & braced?		☐	☐	☐	
e. Proper DOT placarding used?		☐	☐	☐	

Munitions Response Project
Quality–Safety Assurance Field Audit/Self Assessment Checklist

8. FIELD AUDIT/SELF ASSESSMENT FEEDBACK		
	Describe the condition or issue	**Feedback Response**
1)		
2)		
3)		
4)		
5)		
6)		
7)		
8)		
https://sharepoint.hgl.com/docs/Munitions%20Response/UXO%20Personnel/01%20-%20UXOQCS/Form%2015.40%20Quality-Safety%20Assurance%20Field%20Audit%20Checklist.docx		

By signing below, I acknowledge being notified of the results of this audit:

POSITION	**PRINTED NAME**	**SIGNATURE**	**DATE**
UXOSO/SSHO:			
UXOQCS:			
Site Manager/SUXOS:			
Project Manager:			
UXO Quality/Safety Manager:			
Senior UXO Operations Manager:			

FIELD CHANGE REQUEST

FIELD CHANGE REQUEST NO.:
PROJECT:
CONTRACT NUMBER: W912DR-21-D-0005, Delivery Order W912DR21F0327
BRIEF DESCRIPTION:

REQUESTOR IDENTIFICATION
NAME/TITLE: ORGANIZATION: HGL
PHONE:

SIGNATURE DATE

BASELINE IDENTIFICATION
BASELINE(S) AFFECTED ☐Cost ☐Scope ☐Milestone ☐Method of Accomplishment

AFFECTED DOCUMENT (TITLE, NUMBER AND SECTION):

DESCRIPTION OF CHANGE:

JUSTIFICATION:

IMPACT OF NOT IMPLEMENTING REQUEST:

PARTICIPANTS AFFECTED BY IMPLEMENTING REQUEST:

COST ESTIMATE:

Estimator Signature: ________________ ________________
Date

FIELD CHANGE REQUEST

FIELD CHANGE REQUEST NO.:
PROJECT:
CONTRACT NUMBER: W912DR-21-D-0005, Delivery Order W912DR21F0327
BRIEF DESCRIPTION:

PREVIOUS FCR AFFECTED ☐YES ☐NO; IF YES, FCR NO.

APPROVAL SIGNATURES:

USACE Representative	____________________	____________________ Date
Ohio EPA Project Coordinator	____________________	____________________ Date
HGL H&S Manager (if applicatle)	____________________	____________________ Date

Medical Assessment/Work Capacity Form

TO BE COMPLETED BY SUPERVISOR

Employee Name – Last	First	M.I.	Today's Date
Department		Shift	Employee is ☐ Regular ☐ Temporary
Date of Incident		Has an incident report been filed? ☐ Yes Date _________ ☐ No	
Reason for visit			
Supervisor Name (print)	Supervisor's Signature		Contact Information

TO BE COMPLETED BY HEATLH CARE PROVIDER

Employee states this incident is ☐ Work-related ☐ Non-work related

Medical Evaluation

Emergency Contact ____
Name Phone Number

Medical Assessment

Medical Assessment ☐ Suture _________________ ☐RX __________________________ ☐ Tetanus Shot

☐ Other treatment/medications prescribed or given:

Medical Referral To ____

Reason ____

☐ Return to work with NO restrictions or limitations.

☐ Return to work with restrictions or limitations to begin ___________________ until _________________________

☐ Time off work to begin ________________________ until __________________________

Work restrictions/limitations are ☐ Temporary ☐ Permanent

Next appointment:

At ____________________________ with ______________________________________ Date _________________ Time ________________
(clinic) (Care Provider)

Nature of restrictions/limitations:

1. ☐ No repetitive gripping with _________________ more than _______hours per day.
2. ☐ No lifting over _________lbs, to a height of _______________, over ___________ per day.
3. ☐ No pushing or pulling over _______lbs more than _________________hours per day.
4. ☐ Walking and standing limited to ____________minutes/hours per day.
5. ☐ Sitting not to exceed _______________ minutes without a ____________ break to ambulate.
6. ☐ Stooping, bending, twisting, or kneeling limited to _________ times/hour.
7. ☐ No working with the neck, or wrists bent or twisted for more than _________hours per day.
8. ☐ Work limited to _____________ hours per day.
9. ☐ Use of respiratory protection – APR, SCBA, etc. (specify which) ______________ limited to ____________________
10. ☐ Use of chemical protective coveralls, gloves, booties, (specify which) ___________ limited to _____________________

Other restrictions/limitations

Supervisor notified ☐ No ☐ Yes, via ☐ Phone ☐ Message ☐ In Person Date ____________ Time ________________

Authorization to Release/Obtain Medical Information

I hereby authorized the undersigned to share, provide, exchange and discuss medical information and records concerning this injury or illness with my doctor or health care provider and others deemed necessary including medical doctors, physical therapists, and nurse practitioners. I further authorize the health care provider to discuss essential medical information with my supervisor if this injury/illness requires time off from work and/or work restrictions. If I am claiming this incident to be the result of an occupational injury/illness, I authorize providing a copy of this report with company personnel having a legitimate need for the information. I hereby understand and agree to abide by the medical or work restrictions outlined above.

Health Care Provider Signature	Date	Employee Signature

Contract No: Click here to enter **Date:** Click here to enter

Task Order No:

1. Location of Work:	

2. Personnel Summary:

Position/Company if not HGL	**Position/Team Assigned**	**Name**
SUXOS		
UXOSO/UXOQCS		
UXO III		
UXO II		
UXO II		
UXO II		
UXO II		
UXO I		
UXO I		

3. Field Manpower Summary

Position	**Total Positions Assigned**	**Hours Per Position**	**Daily Total Hours**
SUXOS			
UXOSO/ UXOQCS			
UXO III			
UXO II			
UXO I			
Subcontractors			

4. Work Summary
Project totals may be estimated daily and periodically updated once data is processed.

Work Activity	**Daily Total**	**Project Total**
Anomalies Dug		
Grids Completed (By Teams)		
MEC Discovered		
MDAS Pounds		
Other Debris Pounds		

Summary of work activities:

-

5. Inspections: QC/QA inspection data may lag as data transfer is completed. Once QC/QA data received, totals will be updated.

Inspection	**Total Daily**		**Total Project**	
	Pass	**Fail**	**Pass**	**Fail**
UXOQCS QC Complete				
Grids QA'd (OESS)				

Contract No: Click here to enter **Date:** Click here to enter

Task Order No:

6. Explosive Usage:

Item	Quantity	Comments

7. MEC Summary:

Item	Grid	Classification			Disposition
		UXO	DMM	MC	

8. HGL Equipment:

Item Description	Quantity	Comments
Demo Kit		
RFD		
Garretts		
Schonstedt		
Day Box		

9. Rental Equipment: (rental equipment descriptions will be updated throughout project)

Item Description	Quantity	Vendor	Start Date - End Date
Portable Toilet			
Truck SUXOS			
Truck UXOSO/QCS			
Truck Team			
Skid Steer w/ Rotary Drum			

10. Visitors

N/A

11. Discrepancies.

N/A

12. Guidance or Instructions Received from Client.

N/A

13. Attachment Summary.

Attachment 1: Site Pictures

14. Signature.

CERTIFICATION: I certify the above information is complete and correct, and that I have determined to the best of my knowledge and belief that noted work activities are in compliance with the plans and specifications, except as may be noted above.

Contract No: Click here to enter **Date:** Click here to enter

Task Order No:

Site Manager/SUXOS	

Forklifts and Earthmoving Equipment Inspection Checklist	H&S Procedure No.: 30.1 Publication Date: December 2012 Revision No.: 02 Revision Date: November 2017 Review Date: November 2018

HGL EQUIPMENT SAFETY INSPECTION CHECKLIST

The purpose of the operator's inspection is to ensure that the equipment and its attachments are in safe operating order prior to use. The inspection must be conducted prior to each shift. While the regulations do not require documentation of operator inspections, such documentation is strongly encouraged as a means of verifying an adequate and complete inspection. The form on the next page (or an equivalent form) may be used to guide the operator in conducting a pre-use inspection. Inspection items are further described below.

Inspection Goals

Overhead and hand guard	• No broken welds, missing bolts, or other damage
Hydraulic cylinders	• No leaks or damage
Mast or boom assembly	• No broken welds, cracked or bent areas, or worn or missing stops
Lifting bars, straps, cables, chains and rollers	• No excessive wear, damage, fraying, kinks, rust, etc. • Adequately sized anchor shackles • Adequate lubrication
Tow hook	• Engage and release smoothly • Safety catch works properly
Forks, shovel, other	• No cracks, gouges, holes, excessive wear • No mismatching
Tires	• No gouges, chunking, or bond failure • Adequate tread • Proper inflation • No missing lugs
Battery	• Properly installed and secured • Adequately charged • No leakage • Covers and caps in place • Cables in good condition
Hydraulic fluid	• Proper level
Gauges	• Working properly
Steering	• Smooth, without binding or excess play • Power steering operates properly
Brakes	• Pedal moves freely without binding and does not go all the way to the floor • Parking brake works correctly (vehicle does not move when engaged)
Lights	• All warning and working lights function properly
Horn	• Sounds
Backup alarm	• Operates as intended
Control levers	• Moves smoothly
Safety seat, belt, switch, doors, and/or interlocks	• Operate as intended
Propane/fuel tank/hose	• No apparent cracks, checking, kinking, fraying of the hoses • Connector properly seated • Tank secured
Engine oil	• Proper level
Engine coolant	• Proper level
Transmission fluid	• Proper level
Windshield wipers	• Work properly
Attachments	• Operate smoothly with no hesitation

Forklifts and Earthmoving Equipment Inspection Checklist

H&S Procedure No.: 30.1
Publication Date: December 2012
Revision No.: 02
Revision Date: November 2017
Review Date: November 2018

FACILITY OR LOCATION____ DATE/SHIFT ____________________________

OPERATOR ____ VEHICLE NUMBER ______________________

Instructions: Check (3) each item below as “Satisfactory” (S) or “Unsatisfactory” (U) or “Not Applicable” (NA). Add any pertinent comments in the space provided for each item checked “Unsatisfactory.” Include additional pages of explanation as necessary.

Date																								
Time																								
Operator Initials																								
Inspection Item	S	U	NA	S	U	NA	S	U	NA	S	U	NA	S	U	NA	S	U	NA	S	U	NA	S	U	NA
Overhead and hand guard																								
Hydraulic cylinders																								
Mast or boom assembly																								
Lifting bars, straps, cables, chains and rollers																								
Tow hook																								
Forks, shovel																								
Tires																								
Battery																								
Hydraulic fluid																								
Gauges																								
Steering																								
Brakes																								
Lights																								
Horn																								
Backup alarm																								
Control levers																								
Safety seat, belt, switch, doors, and/or interlocks																								
Load handling attachments																								
Fuel tanks/hose																								
Engine oil																								
Engine coolant																								
Transmission fluid																								
Windshield wipers																								
Attachments																								

Date	Item of Concern	Corrective Action Taken

Inspection must be done before each shift or before equipment is used for that day.

Operator Signature ____ Date: __________________

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APPENDIX F

LABORATORY STANDARD OPERATING PROCEDURES AND CERTIFICATIONS

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ANALYSIS OF POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

Prepared by: Norm Farmer Date: 12/21/2020

Approved by: Naresh Jiawan Date: 12/21/2020

Annual Review

Reviewed by: ______________________ Date: __________

Reviewed by: ______________________ Date: __________

Reviewed by: ______________________ Date: __________

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TITLE: ANALYSIS OF POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

REFERENCES: SW846 8082A

REVISED SECTIONS: 3.4 and 7.4.1.2

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the polychlorinated biphenyl (PCB) concentrations in water, solid, and waste matrices utilizing a gas chromatograph equipped with an electron capture detector.

1.1.2 PCBs for this method are reported as Aroclors. Aroclors are multi-component mixtures consisting of various chlorinated biphenyl congeners. Quantitation is based on individual Aroclor standards. For the purpose of this SOP, the term Aroclor and PCB may be used interchangeably. The following compounds can be reported by this method:

Aroclor 1016
Aroclor 1242
Aroclor 1260
Aroclor 1221
Aroclor 1248
Aroclor 1262
Aroclor 1232
Aroclor 1254
Aroclor 1268

1.1.3 The Lower Limit of Quantitation (LLOQ) or Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. LLOQs may vary depending on matrix complications and volumes. LLOQs for this method are in the range of 0.5 ug/l for aqueous samples, 33 ug/kg for solid samples, 1ug/wipe for surface wipe samples, and 10 mg/kg for oil samples. Solid matrices are reported on a dry weight basis.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the LLOQ. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported LLOQ.

1.1.5 The LLOQ for each analyte is evaluated on an annual basis for each matrix and instrument. The LLOQ verifications are prepared by spiking a clean matrix at 0.5 to 2 times the current LLOQ level. This LLOQ verification is carried through the same preparation and analytical procedures as the samples. Recovery of the analytes should be within the established limits. The DOD QSM requirements for Limit of Detection (LOD) and Limit of Quantitation (LOQ) verifications are different. See SOP QA020 for complete requirements for MDL, LOD, LOQ, and LLOQ.

1.1.6 Compounds detected at concentrations between the LLOQ and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I” qualifier. Some program or project specifications may require that no values below the LLOQ be reported.

1.1.7 For DOD projects refer to QSM 5.0, Table 1; or QSM 5.x Table B-1 for additional method requirements and data qualifying guidance.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8082A.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with SGS - Orlando SOP OP008, OP009, or OP033.

1.2.4 The extracts are analyzed on a gas chromatograph equipped with dual electron capture detectors.

1.2.5 Manual integrations are performed in accordance with SOP QA029.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter or 250ml bottles are used for aqueous samples and 4oz jars are recommended for solid samples.

2.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be stored at ≤ 6°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Interferences from phthalate esters can be eliminated by using plastic-free solvent containers and solvent rinsed glassware.

3.3 Other organic compounds, including chlorinated pesticides, chlorinated hydrocarbons, phenols, and phthalate esters may be co-extracted by this method. Many of these interferences can be removed by sulfuric acid cleanup.

3.4 Interferences from sulfur compounds can be eliminated using an activated copper powder cleanup. Regional samples are generally high in sulfur content; therefore, all sample extracts may be sulfur cleaned prior to analysis.

3.5 The DDT analogs (DDT, DDE, and DDD) are frequently co-extracted with the PCBs. A single point DDT analog standard must be analyzed periodically to show if any PCB peaks are subject to interference.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the LLOQ.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Hexane – pesticide grade or equivalent

5.2 PCB Stock standards – Traceable to Certificate of Analysis

5.3 Surrogate standard – TCMX and Decachlorobiphenyl

5.4 DDT analogs standard – Mixture of 4,4’-DDT, 4,4’-DDE, and 4,4’-DDD

6.0 APPARATUS

6.1 Gas Chromatograph – Agilent Technologies 6890 or 7890 with 7683 Autosampler

Suitable gas chromatograph equipped with a split-splitless injection port and electron capture detectors.

Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Data System – Agilent Technologies MS Chemstation rev. DA 03.0x or EA 02.0x.

6.2.1 A computer system interfaced to the gas chromatograph that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.2.2 Data is archived to a backup server for long term storage.

6.3 Dual CLP PEST/PEST2 or equivalent: 30m X 0.53mm X 0.50/0.42um
Dual MR1/MR2 or equivalent: 30m X 0.53mm X 0.5um

6.4 Gas-tight syringes and class "A" volumetric glassware for dilutions of standards and extracts.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at ≤ 6°C, or as recommended by the manufacturer. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the GC STD Summary in the Active SOP directory.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to one year or the vendor's expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with hexane. The hold time for intermediate standards is six months or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicates analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for PCB 1016 and 1260 are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the LLOQ and the remaining standards define the working range of the detector.

Standards for the remaining PCBs are made at a single level near the low to mid-point of the curve. **NOTE:** Some projects may require that samples with positive detects for any of the remaining PCBs be reanalyzed and quantitated using a 5-point calibration.

Calibration standard concentrations for PCB 1016 and PCB 1260 are verified by the analysis of an initial calibration verification (ICV) standard. Additional ICV standards for the other PCBs may be required.

7.2 Gas Chromatograph Conditions

2ul autosampler injection Splitless or 2:1 split injection

Carrier gas – UHP Hydrogen (3.8 ml/min) or (8.0 ml/min)

Detector gas – UHP Nitrogen (45-90 ml/min)

Injection port temperature – 200 °C Detector temperature – 325 °C

Oven program – 125 °C for 0.5 minutes
35 °C/min to 250 °C for 0 minutes
25 °C/min to 310 °C for 3 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Sample Preparation

7.3.1 Water Samples

A 250ml or 1000ml aliquot of sample is extracted with methylene chloride utilizing separatory funnel extraction. The extract is solvent exchanged into hexane, concentrated to 5.0ml, sulfur cleaned, and acid cleaned.

7.3.2 Solid Samples

A 15-gram aliquot of sample is extracted with hexane:acetone utilizing pulse sonication or microwave extraction. The extract is concentrated, adjusted to 5ml with hexane, sulfur cleaned, and acid cleaned.

7.3.3 Surface Wipe Samples

A wipe sample is extracted with hexane utilizing a wrist shaker. The extract may then be sulfur cleaned or acid cleaned.

7.3.4 Oil Samples

A one-gram aliquot of sample is diluted in hexane to 10ml. The extract may then be sulfur cleaned or acid cleaned.

7.4 Gas Chromatographic Analysis

Instrument calibration consists of three major sections:

Initial Calibration Procedures
Daily Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, the chromatographic system must be calibrated, retention time windows must be determined, pattern recognition standards must be analyzed, and DDT analogs interference check must be analyzed.

7.4.1.1 External Standard Calibration

Prime the GC by injecting a conditioning standard, this is a high-level PCB standard. This will aid in deactivating the injection port and column.

Quantitation for this method is based on the response of individual peaks in each individual PCB standard. Three to six major peaks from each Aroclor are used for quantitation purposes. Each individual PCB peak is assigned the concentration of the total PCB standard.

A minimum 5-point calibration curve is created for PCB 1016, PCB 1260, TCMX, and Decachlorobiphenyl. PCB 1016 and 1260 are used to establish linearity for all of the PCBs.

The remaining PCBs are calibrated at a single level in the low to mid-point range of the PCB 1016/1260 curve. The single point standards are also used for pattern recognition.

NOTE: DoD QSM projects require a 5-point calibration for all PCBs.

Historically, many analytical methods have relied on linear models of the calibration relationship, where the instrument response is directly proportional to the amount of a target compound. The linear model has many advantages including simplicity and ease of use. However, given the advent of new detection techniques and because many methods cannot be optimized for all the analytes to which they may be applied, the analyst is increasingly likely to encounter situations where the linear model neither applies nor is appropriate. The option of using non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to compensate for detector saturation or avoid proper instrument maintenance.

NOTE: Because of this concern, select programs including SC DHEC do not support the use of non-linear regressions.

Calibration factors (CF) for PCBs are determined at each concentration by dividing the total area (or height) of the PCB (3 to 6 major peaks) by the concentration of the standard. Calibration factors for the surrogate are determined at each concentration by dividing the area (or height) of the peak by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the calibration factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively, a calibration curve of response vs. amount can be plotted. This method allows for the use of average response factors, linear regressions, and non-linear regressions. Linear regressions may be unweighted or weighted as 1/x or $1/x^2$. If the correlation coefficient (r) is $\geq$0.995 ($r^2 \geq 0.990$) then the curve can be used to quantitate target analytes in the samples.

NOTE: If a non-linear calibration curve (quadratic) is used for PCB 1016 and/or 1260, then the remaining PCBs such as 1221, 1232, 1242, 1248, 1254, 1262, and 1268 must be calibrated using a multipoint technique.

Regardless of which calibration model is chosen, the laboratory should visually inspect the curve plots to see how the individual calibration points compare to the plot.

Alternatively, either of the two techniques described below may be used to determine whether the calibration function meets acceptable criteria. These involve refitting the calibration data back to the model. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

% ERR = (xi-x'i) / xi * 100

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at calibration level i, in mass or concentration units.

Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (%RSE)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at 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.

The %RSE acceptance limit criterion is ≤20%.

7.4.1.2 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV must be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be ≤ 20%. If the ICV does not meet this criteria, a second standard must be prepared. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, analyze an ICV prepared from a third source or lot. Determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

For any DoD QSM project, if samples must be analyzed with a target analyte having a %D > 20%, then the data must be qualified accordingly.

7.4.1.3 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Individual PCB peaks must fall within the appropriate retention time windows; however, Aroclor identification is based primarily on individual peak ratios and pattern recognition.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. 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 or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations.

The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. The analyst should monitor the retention times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential “hits”. The analyst will then review these “hits” and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

7.4.1.4 DDT Analogs Interference Check

Analyze a DDT analogs standard consisting of DDT, DDE, and DDD at a concentration near the midpoint of the PCB calibration curve. Overlay the DDT analogs standard with the mid-level PCB standard (and single point standards) and determine if there are any interferences. Note on the run log which PCB peaks co-elute with the DDT series. Instrument conditions should be optimized to minimize interferences.

The analyst must be aware of these co-eluting peaks when quantitating samples that may contain DDT series. It may be necessary to omit a co-eluting peak in order to obtain the most accurate value.

7.4.2 Daily Calibration Procedures

Prime the GC by injecting a conditioning standard, this is a high-level PCB standard. This will aid in deactivating the injection port and column.

Analyze a continuing calibration verification standard (section 7.4.3) for PCB 1016 and PCB 1260. Then analyze the PCBs that are calibrated at a single concentration level.

The single point standards must be analyzed every 24 hours; they do not have to be analyzed at the same frequency as the CCV standard. For DoD QSM projects the CCVs for the other PCBs should be analyzed at the same frequency as PCB 1016 and PCB 1260.

NOTE: DoD QSM projects require CCVs for each PCB to be analyzed throughout the sequence.

7.4.3 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for PCB 1016 and PCB 1260 are prepared at various concentrations (CCV standards for other PCBs may be required); at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally, a CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| should be ≤ 20% for each analyte.

If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated. If the second standard meets criteria, then the system is considered in control and results may be reported.

Rationale for second standard such as instrument maintenance, clipped column, remade standard, etc. must be documented in the run log or maintenance log. Reanalysis of second standard without valid rationale may require the analysis of a third standard (in which case both the second and third standard would have to pass).

NOTE: For any DoD QSM project, if the second standard meets criteria, then a third standard must be analyzed. If the third standard also meets criteria, then the system is considered in control and results may be reported.

If the |%D| is greater than 20%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: For any DoD QSM project, if samples must be reported with a target analyte having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

NOTE: Samples that are being reported to the State of California require that the |%D| ≤ 15% for each analyte.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed, or the data must be qualified.

7.4.4 Sample Extract Analysis

7.4.4.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Conditioning Standard
Initial Calibration Standards (or Initial CCV)
Single Point Standards
QC Extracts
Sample Extracts
CCV Standards

7.4.4.2 Two microliters (same amount as standards) of extract is injected into the GC by the autosampler. A splitless injection technique is used. The data system then records the resultant peak responses and retention times.

7.4.4.3 Tentative identification of an analyte occurs when the peaks from the sample extract fall within the established retention time windows for a calibrated compound on the primary column.

7.4.4.4 If the peaks of interest fall within the retention time windows on the confirmation column, the identification is confirmed. Quantitation of the analyte on the primary and confirmation column should agree within 40%. If the difference is greater than 40% and no obvious reason can be found, the higher result should be reported and flagged as "estimated"; otherwise, the result from the primary column should be reported.

7.4.4.5 Pattern comparisons should also be used to aid in the identification of the multipeak analytes.

7.4.4.6 If the compound identification does not confirm on a dissimilar column, then the result should be reported as ND or "U".

7.4.4.7 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.4.8 If peak or pattern identification is prevented by the presence of interferences, further cleanup may be required, or the extract must be diluted so that the interference does not mask any analytes.

7.5 Maintenance and Trouble Shooting

7.5.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.

7.5.2 All instrument maintenance must be documented in the appropriate "Instrument Repair and Maintenance" log. The log will include such items as problem, action taken, correction verification, date, and analyst.

7.5.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

7.5.4 PC and software changes must be documented in the "Instrument Repair and Maintenance" log. Software changes may require additional validation.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Surrogates

9.1.1 Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl are used as the surrogate standards to monitor the efficiency of the extraction and clean-up procedures.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for both surrogates for the results to be acceptable.

9.1.2 If the surrogate recoveries are not within the established control limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or surrogate solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.1.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, reanalyze the sample. **NOTE: If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary; however, the resulting data must be qualified accordingly.** If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis must be reported on the final report.

9.2 Method Blank

9.2.1 The method blank is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high-level samples. The method blank must be free of any analytes of interest or interferences at ½ the required LLOQ to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination must be investigated and documented. The sample results can be reported without qualification. **NOTE**: **For samples reported to SC DHEC or DoD the associated sample results must still be reported with the B qualifier**.

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination must be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination must be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable.

NOTE: A secondary check against 70-130% limits must be performed for all analytes reported to SC DHEC.

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.3.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary, and the sample results can be reported without qualification.

9.3.2.4 **If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable; however, the resulting data must be qualified accordingly**.

9.3.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.3.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.4 Matrix Spike and Matrix Spike Duplicate

9.4.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.4.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.4.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.4.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are an indication of the sample matrix effects.

9.4.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

10.0 CALCULATIONS

The amount of the PCB in the samples is determined by averaging the concentration of the major peaks for each individual PCB. The MS Chemstation software will automatically sum and average the peaks used for each individual Aroclor.

The concentration of each individual PCB peak in the original sample is calculated as follows:

PCB Water (ug/l) = $(\text{CONC}_{\text{inst}}) \text{ X } (V_F / V_I) \text{ X DF}$

PCB Soil (ug/kg) = $[(\text{CONC}_{\text{inst}}) \text{ X } (V_F / W_I) \text{ X DF}] / \%\text{solids}$

$CONC_{inst}$ = Instrument concentration calculated from the initial calibration using mean CF, curve fit, or single point
DF = Dilution Factor
V_F = Volume of final extract (ml)
V_I = Volume of sample extracted (ml)
W_I = Weight of sample extracted (g)
%solids = Dry weight determination in decimal form

All soils are reported on a dry weight basis.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Program, which includes the use of safety glasses, gloves, and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample must be treated as a potential health hazard. Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment must be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis and standards preparation are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 REFERENCES

SW846 Method 8000D Revision 4, July 2014

SW846 Method 8082A Revision 1, February 2007

ANALYSIS OF POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

SOP Acknowledgement Form

I have read and understand this SOP. I will not knowingly deviate from this approved SOP without approval of the Department Supervisor, QA Officer, or Technical Director. If I notice any discrepancies between this SOP and the routine procedure, I will notify the Department Supervisor so that either the SOP or procedure can be changed. Furthermore, I understand that this SOP is property of SGS North America Inc. – Orlando and may not be printed nor duplicated in any manner.

Internal SOPs referenced within this SOP: OP008, OP009, OP033, GC001, QA020, QA029

Print Name	**Signature**	**Date**

Print the SOP Acknowledgement Form, sign, and submit to the SGS Orlando QA department.

ANALYSIS OF ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

Prepared by: Norm Farmer Date: 12/21/2020

Approved by: Naresh Jiawan Date: 12/21/2020

Annual Review

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TITLE: ANALYSIS OF ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

REFERENCES: SW846 8081B

REVISED SECTIONS: 3.4, 7.4.1.3 and 7.4.4.7

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of specific organochlorine pesticides in water and solid matrices utilizing a gas chromatograph equipped with an electron capture detector.

1.1.2 The following compounds can be reported by this method:

Aldrin	Dieldrin
Alpha-BHC	Endosulfan I
Beta-BHC	Endosulfan II
Delta-BHC	Endosulfan Sulfate
Gamma-BHC (Lindane)	Endrin
Chlordane (Technical)	Endrin Ketone
Alpha-chlordane	Endrin Aldehyde
Gamma-chlordane	Heptachlor
4,4'-DDD	Heptachlor Epoxide
4,4'-DDE	Methoxychlor
4,4'-DDT	Toxaphene

1.1.3 The Lower Limit of Quantitation (LLOQ) or Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. LLOQs may vary depending on matrix complications and volumes. LLOQs for the single peak pesticides in this method are in the range of 0.05 to 0.10 ug/l for aqueous samples and 1.7 to 3.3 ug/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the LLOQ. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported LLOQ.

1.1.5 The LLOQ for each analyte is evaluated on an annual basis for each matrix and instrument. The LLOQ verifications are prepared by spiking a clean matrix at 0.5 to 2 times the current LLOQ level. This LLOQ verification is carried through the same preparation and analytical procedures as the samples. Recovery of the analytes should be within the established limits. The DOD QSM requirements for Limit of Detection (LOD) and Limit of Quantitation (LOQ) verifications are different. See SOP QA020 for complete requirements for MDL, LOD, LOQ, and LLOQ.

1.1.6 Compounds detected at concentrations between the LLOQ and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I” qualifier. Some program or project specifications may require that no values below the LLOQ be reported.

1.1.7 For DOD projects refer to QSM 5.0, Table 1; or QSM 5.x Table B-1 for additional method requirements and data qualifying guidance.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8081B.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with SGS - Orlando SOP OP008 and OP009.

1.2.4 The extracts are analyzed on a gas chromatograph equipped with dual electron capture detectors.

1.2.5 Manual integrations are performed in accordance with SOP QA029.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter or 250ml bottles are used for aqueous samples and 4oz jars are recommended for solid samples.

2.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be stored at ≤ 6°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Interferences from phthalate esters can be eliminated by using plastic-free solvent containers and solvent rinsed glassware.

3.3 Other organic compounds, including PCBs, chlorinated hydrocarbons, phenols, and phthalate esters may be co-extracted by this method.

3.4 Interferences from sulfur compounds can be eliminated using an activated copper powder cleanup. Regional samples are generally high in sulfur content; therefore, all sample extracts should be sulfur cleaned prior to analysis.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the LLOQ.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Hexane – pesticide grade or equivalent

5.2 Pesticide stock standards – Traceable to Certificate of Analysis

5.3 Surrogate standard – TCMX and Decachlorobiphenyl

6.0 APPARATUS

6.1 Gas Chromatograph – Agilent Technologies 6890 or 7890 with 7683 Autosampler

Suitable gas chromatograph equipped with a split-splitless injection port and electron capture detectors.

Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Data System – Agilent Technologies MS Chemstation rev. DA 00.01, DA 03.0x or EA 02.0x.

6.2.1 A computer system interfaced to the gas chromatograph that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.2.2 Data is archived to a backup server for long term storage.

6.3 Dual MR1/MR2 (multiresidue) Column or equivalent: 30m X 0.53mm X 0.50um
Dual CLP/CLP2 Column or equivalent: 30m X 0.50mm X 0.5/0.42um

6.4 Gas-tight syringes and class “A” volumetric glassware for dilutions of standards and extracts.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at ≤ 6°C, or as recommended by the manufacturer. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the GC STD Summary in the Active SOP directory

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a “Certificate of Analysis” with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor’s expiration date. Once opened, the hold time is reduced to one year or the vendor’s expiration date (whichever is shorter)

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with hexane. The hold time for intermediate standards is six months or the vendor’s expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicates analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the single peak pesticides are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the LLOQ and the remaining standards define the working range of the detector.

Standards for the multipeak pesticides (Toxaphene and Technical Chlordane) are made at a single level near the RL. **NOTE:** Some projects may require that samples with positive detects for either toxaphene or technical chlordane be reanalyzed and quantitated using a 5-point calibration.

Calibration standard concentrations for the single peak pesticides are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Gas Chromatograph Conditions

4ul autosampler injection Splitless or 2:1 split injection

Carrier gas – UHP Helium (5-10 ml/min ramped flow)

Detector gas – UHP Nitrogen (45 - 90 ml/min)

Injection port temperature – 190 - 200 °C Detector temperature – 325 - 350 °C

Oven program – 120 °C for 0 minutes
45 °C/min to 200 °C
12.5 °C/min to 230 °C
20 °C/min to 320 °C for 2.0 minutes

Pressure Program – 5.0 ml/min for 1.0 minute
1.0 ml/min to 10.0 ml/min for 0 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Sample Preparation

7.3.1 Water Samples

A 250ml or 1000ml aliquot of sample is extracted with methylene chloride utilizing separatory funnel extraction. The extract is solvent exchanged into hexane, concentrated to 5.0ml, and sulfur cleaned.

7.3.2 Solid Samples

A 15-gram aliquot of sample is extracted with hexane:acetone utilizing pulse sonication or microwave extraction. The extract is concentrated, adjusted to 5.0ml with hexane, and sulfur cleaned.

7.4 Gas Chromatographic Analysis

Instrument calibration consists of three major sections:

Initial Calibration Procedures
Daily Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, injection port inertness must be verified, the chromatographic system must be calibrated, and retention time windows must be determined.

7.4.1.1 Breakdown Check

Prime the GC by injecting a conditioning standard; this is a pesticide standard that is approximately 20 times the concentration of the mid-level standard. This will aid in deactivating the injection port and column.

Endrin and DDT breakdown must be verified at the start of each 12-hour shift. Inject a DDT/Endrin standard that is at a concentration near the mid-point of the curve. Calculate the percent breakdown of each analyte as follows (height may be used instead of area):

$$\%DDT_{BREAKDOWN} = \frac{\text{(DDE Area + DDD Area) X 100}}{\text{(DDE Area + DDD Area + DDT Area)}}$$

$$\%Endrin_{BREAKDOWN} = \frac{\text{(Endrin ketone Area + Endrin aldehyde Area) X 100}}{\text{(Endrin ketone Area + Endrin aldehyde Area + Endrin Area)}}$$

The percent breakdown for both DDT and endrin must not exceed 15%. If breakdown exceeds 15%, instrument maintenance must be performed before any samples can be analyzed.

7.4.1.2 External Standard Calibration

A minimum 5-point calibration curve is created for the single peak pesticides, TCMX, and Decachlorobiphenyl. SGS Orlando routinely performs a 6-point calibration to maximize the calibration range.

The low point may be omitted from the calibration table for any compound with an LLOQ set at the level two standard. Additionally, the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

An entire level may be omitted provided that a minimum of 5 points remain. There must be technical justification to omit an entire level. This must be documented in the run log.

Toxaphene and chlordane (technical) are calibrated at a single level near the RL. **NOTE:** DoD QSM projects require at 5-point calibration for technical chlordane and toxaphene if they are being reported.

Historically, many analytical methods have relied on linear models of the calibration relationship, where the instrument response is directly proportional to the amount of a target compound. The linear model has many advantages including simplicity and ease of use. However, given the advent of new detection techniques and because many methods cannot be optimized for all the analytes to which they may be applied, the analyst is increasingly likely to encounter situations where the linear model neither applies nor is appropriate. The option of using non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to compensate for detector saturation or avoid proper instrument maintenance.

NOTE: Because of this concern, select programs including SC DHEC do not support the use of non-linear regressions.

Calibration factors (CF) for the single peak pesticides and surrogates are determined at each concentration by dividing the area (or height) of each compound by the concentration of the standard.

Quantitation for technical chlordane is based on the individual peaks in the chlordane standard. Three to six major peaks are used for quantitation purposes. Each individual chlordane peak is assigned the concentration of the total chlordane standard. Toxaphene may be quantitated by individual peaks (4 to 6 major peaks) or by total area. For the individual peak approach, each individual toxaphene peak is assigned the concentration of the total toxaphene standard. See method 8081B for additional information.

Calibration factor (CF) for chlordane is determined by dividing the total area (or height) of chlordane (3 to 6 major peaks) by the concentration of the standard. The calibration factor for the toxaphene is determined by dividing the total area (height cannot be used) of Toxaphene (4 to 6 major peaks) by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the calibration factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively, a calibration curve of response vs. amount can be plotted. This method allows for the use of average response factors, linear regressions, and non-linear regressions. Linear regressions may be unweighted or weighted as $1/x$ or $1/x^2$. If the correlation coefficient (r) is ≥ 0.995 ($r^2 \geq 0.990$) then the curve can be used to quantitate target analytes in the samples. Regardless of which calibration model is chosen, the laboratory should visually inspect the curve plots to see how the individual calibration points compare to the plot.

Alternatively, either of the two techniques described below may be used to determine whether the calibration function meets acceptable criteria. These involve refitting the calibration data back to the model. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

% ERR = (xi-x'i) / xi * 100

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at calibration level i, in mass or concentration units.

Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (%RSE)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at 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.

The %RSE acceptance limit criterion is ≤20%.

7.4.1.3 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV must be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be ≤ 20%. If the ICV does not meet this criteria, a second standard must be prepared. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, analyze an ICV prepared from a third source or lot.

Determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

For any DoD QSM project, if samples must be analyzed with a target analyte having a %D > 20%, then the data must be qualified accordingly.

7.4.1.4 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. 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 or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations.

The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. The analyst should monitor the retention times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential "hits". The analyst will then review these "hits" and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

7.4.2 Daily Calibration Procedures

Prime the GC by injecting a conditioning standard; this is a pesticide standard that is approximately 20 times the concentration of the mid-level standard. This will aid in deactivating the injection port and column.

Analyze a DDT/Endrin breakdown standard as described in section 7.4.1.1. This standard must be analyzed at the start of each 12-hour shift.

Analyze a continuing calibration verification standard (section 7.4.3) for the single peak pesticides. Then analyze the Technical Chlordane and Toxaphene standards that are calibrated at a single concentration level. The single point standards must be analyzed every 12 hours; they do not have to be analyzed at the same frequency as the CCV standard.

For DoD QSM projects the Technical Chlordane and Toxaphene standards must be analyzed at the beginning of the 12-hour window. Additional standards may be analyzed through the run.

7.4.3 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the single peak pesticides are prepared at various concentrations; at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally, a CCV must be analyzed after every 10 samples. If toxaphene or technical chlordane is known to be present at the site or if it a DoD QSM project, the analyst must analyze additional CCVs for these analytes.

The percent difference (%D) for each analyte of interest will be monitored. The $|\%D|$ should be $\leq 20\%$ for each analyte.

If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated. If the second standard meets criteria then the system is considered in control and results may be reported.

Rationale for second standard such as instrument maintenance, clipped column, remade standard, etc must be documented in the run log or maintenance log. Reanalysis of second standard without valid rationale may require the analysis of a third standard (in which case both the second and third standard would have to pass).

NOTE: For any DoD QSM project, if the second standard meets criteria, then a third standard must be analyzed. If the third standard also meets criteria, then the system is considered in control and results may be reported.

If the |%D| is greater than 20%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: For any DoD QSM project, if samples must be reported with a target analyte having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

NOTE: Samples that are being reported to the State of California require that the |%D| ≤ 15% for each analyte.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed, or the data must be qualified.

7.4.4 Sample Extract Analysis

7.4.4.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Conditioning Standard
DDT/Endrin Breakdown Standard
Initial Calibration Standards (or Initial CCV)
Single Point Standards
QC Extracts
Sample Extracts
CCV Standards

7.4.4.2 Four microliters (same amount as standards) of extract is injected into the GC by the autosampler. A split or splitless injection technique is used. The data system then records the resultant peak responses and retention times.

7.4.4.3 Tentative identification of an analyte occurs when the peaks from the sample extract fall within the established retention time windows for a calibrated compound on the primary column.

7.4.4.4 If the peaks of interest fall within the retention time windows on the confirmation column, the identification is confirmed. Quantitation of the analyte on the primary and confirmation column should agree within 40%. If the difference is greater than 40% and no obvious reason can be found, the higher result should be reported and flagged as "estimated"; otherwise, the result from the primary column should be reported.

7.4.4.5 Pattern comparisons should also be used to aid in the identification of the multipeak analytes.

7.4.4.6 If the compound identification does not confirm on a dissimilar column, then the result should be reported as ND or "U".

7.4.4.7 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.4.8 If peak identification is prevented by the presence of interferences, further cleanup may be required, or the extract must be diluted so that the interference does not mask any analytes.

7.5 Maintenance and Trouble Shooting

7.5.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.

7.5.2 All instrument maintenance must be documented in the appropriate "Instrument Repair and Maintenance" log. The log will include such items as problem, action taken, correction verification, date, and analyst.

7.5.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

7.5.4 PC and software changes must be documented in the "Instrument Repair and Maintenance" log. Software changes may require additional validation.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Surrogates

9.1.1 Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl are used as the surrogate standards to monitor the efficiency of the extraction and clean-up procedures.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for both surrogates for the results to be acceptable.

9.1.2 If the surrogate recoveries are not within the established control limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, surrogate solutions or internal standards. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.1.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, reanalyze the sample. **NOTE: If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary; however, the resulting data must be qualified accordingly.** If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis must be reported on the final report.

9.2 Method Blank

9.2.1 The method blank is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high-level samples. The method blank must be free of any analytes of interest or interferences at ½ the required LLOQ to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination must be investigated and documented. The sample results can be reported without qualification. **NOTE**: **For samples reported to SC DHEC or DoD the associated sample results must still be reported with the B qualifier**.

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination must be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination must be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is either de-ionized water or sodium sulfate (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable. The large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of the established control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary.

Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A marginal exceedance in the Blank Spike is defined as a recovery being outside of 3 standard deviations but within 4 standard deviations of the mean.

The number of allowable marginal exceedances is based on the number of analytes in the Blank Spike. Marginal Exceedances must be random. If the same analyte exceeds the BS control limits repeatedly, it is an indication of a systematic problem and corrective action must be taken.

Marginal exceedances are not permitted for analytes that are deemed to be “Compounds of Concern” for a specific project. “Compounds of Concern” are different from “Target Compounds”. “Target Compounds” are all analytes that are being reported for a site where “Compounds of Concern” are those analytes expected to be present at the site.

The number of allowable marginal exceedances is as follows:

1) 11-30 analytes in BS, 1 analyte allowed in ME range;

2) < 11 analytes in BS, no analytes allowed in ME range

NOTE: SC DHEC does not recognize the concept of Marginal Exceedances. Additionally, a secondary check against 70-130% limits must be performed for all analytes reported to SC DHEC.

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.3.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary, and the sample results can be reported without qualification.

9.3.2.4 **If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable; however, the resulting data must be qualified accordingly**.

9.3.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.3.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.4 Matrix Spike and Matrix Spike Duplicate

9.4.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.4.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.4.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.4.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are an indication of the sample matrix effects.

9.4.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If more than 33% of the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

10.0 CALCULATIONS

The concentration of each single peak pesticide or toxaphene (if using the total area technique) in the original sample is calculated as follows:

Water (ug/l) = $(CONC_{inst}) \times (V_F / V_I) \times DF$

Soil (ug/kg) = $[(CONC_{inst}) \times (V_F / W_I) \times DF] / \%solids$

$CONC_{inst}$ = Instrument concentration calculated from the initial calibration using mean CF or curve fit.
DF = Dilution Factor
V_F = Volume of final extract (ml)
V_I = Volume of sample extracted (ml)
W_I = Weight of sample extracted (g)
%solids = Dry weight determination in decimal form

All soils are reported on a dry weight basis.

The amount of chlordane or toxaphene (if using the multipeak technique) in the samples is determined by averaging the concentration of the major peaks for each multipeak pesticide. The MS Chemstation software will automatically sum and average the peaks used for each multipeak pesticide.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Program, which includes the use of safety glasses, gloves, and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample must be treated as a potential health hazard. Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment must be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis and standards preparation are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 REFERENCES

SW846 Method 8000D Revision 4, July 2014

SW846 Method 8081B Revision 2, February 2007

ANALYSIS OF ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

SOP Acknowledgement Form

I have read and understand this SOP. I will not knowingly deviate from this approved SOP without approval of the Department Supervisor, QA Officer, or Technical Director. If I notice any discrepancies between this SOP and the routine procedure, I will notify the Department Supervisor so that either the SOP or procedure can be changed. Furthermore, I understand that this SOP is property of SGS North America Inc. – Orlando and may not be printed nor duplicated in any manner.

Internal SOPs referenced within this SOP: OP008, OP009, GC001, QA020, QA029

Print Name	Signature	Date

Print the SOP Acknowledgement Form, sign, and submit to the SGS Orlando QA department.

ANALYSIS OF CHLORINATED HERBICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

Prepared by: Norm Farmer Date: 12/30/2020

Approved by: Naresh Jiawan Date: 12/30/2020

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TITLE: ANALYSIS OF CHLORINATED HERBICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

REFERENCES: SW846 8151A

REVISED SECTIONS: 1.1.2 and 7.4.1.2

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of specific chlorinated herbicides in water and solid matrices utilizing a gas chromatograph equipped with an electron capture detector.

1.1.2 The following compounds can be reported by this method:

Dalapon
2,4-D
2,4,5-TP (Silvex)
MCPP
Dicamba
2,4-DB
Dinoseb
MCPA
Dichloroprop
2,4,5-T
Pentachlorophenol

1.1.3 The Lower Limit of Quantitation (LLOQ) or Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. LLOQs may vary depending on matrix complications and volumes. LLOQs for the chlorinated herbicides are in the range of 0.1 to 2.5 ug/l for aqueous samples and 3.3 to 83 ug/kg for solid samples. LLOQs for MCPP and MCPA are in the range of 100 ug/l for aqueous samples and 3300 ug/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the LLOQ. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported LLOQ.

1.1.5 The LLOQ for each analyte is evaluated on an annual basis for each matrix and instrument. The LLOQ verifications are prepared by spiking a clean matrix at 0.5 to 2 times the current LLOQ level. This LLOQ verification is carried through the same preparation and analytical procedures as the samples. Recovery of the analytes should be within the established limits. The DOD QSM requirements for Limit of Detection (LOD) and Limit of Quantitation (LOQ) verifications are different. See SOP QA020 for complete requirements for MDL, LOD, LOQ, and LLOQ.

1.1.6 Compounds detected at concentrations between the LLOQ and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I” qualifier. Some program or project specifications may require that no values below the LLOQ be reported.

1.1.7 For DOD projects refer to QSM 5.0, Table 1; or QSM 5.x Table B-1 for additional method requirements and data qualifying guidance.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8151A.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with SGS - Orlando SOP OP037 and OP038.

1.2.4 The extracts are analyzed on a gas chromatograph equipped with dual electron capture detectors.

1.2.5 Manual integrations are performed in accordance with SOP QA029.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter or 250ml bottles are used for aqueous samples and 4oz jars are recommended for solid samples.

2.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be stored at ≤ 6°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Interferences from phthalate esters can be eliminated by using plastic-free solvent containers and solvent rinsed glassware.

3.3 Other organic compounds, including organic acids, chlorinated phenols and phthalate esters may be co-extracted by this method.

3.4 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, 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.

3.5 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

3.6 Sample extracts must be dry prior to methylation or else poor recoveries will be obtained.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the LLOQ.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Hexane – pesticide grade or equivalent

5.2 Herbicide stock standards – Traceable to Certificate of Analysis

5.3 Surrogate standard – DCAA

6.0 APPARATUS

6.1 Gas Chromatograph – Agilent Technologies 6890 or 7890 with 7683 Autosampler

Suitable gas chromatograph equipped with a split-splitless injection port and electron capture detectors.

Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Data System – Agilent Technologies MS Chemstation rev. DA 03.0x or EA 02.0x.

6.2.1 A computer system interfaced to the gas chromatograph that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.2.2 Data is archived to a backup server for long term storage.

6.3 Dual CLP/CLP2 Column or equivalent: 30m X 0.32mm X 0.32/0.25um

6.4 Suitable gas-tight syringes and class "A" volumetric glassware for dilutions of standards and extracts.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at ≤ 6°C, or as recommended by the manufacturer. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the GC STD Summary in the Active SOP directory.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to one year or the vendor's expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with hexane. The hold time for intermediate standards is six months or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicates analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the herbicides are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the LLOQ and the remaining standards define the working range of the detector.

Calibration standard concentrations for the herbicides are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Gas Chromatograph Conditions

1ul autosampler injection

Carrier gas – UHP Hydrogen (5.0 ml/min constant flow)

Detector gas – UHP Nitrogen (45 - 90 ml/min)

Injection port temperature – 250 °C Detector temperature – 325 °C

Oven program – 55 °C for 0.5 minute
35 °C/min to 190 °C for 1 minute
20 °C/min to 300 °C for 1 minute

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Sample Preparation

7.3.1 Water Samples

A 250ml or 1000ml aliquot of sample is extracted with diethyl ether utilizing separatory funnel extraction. The extract is concentrated, esterified, and brought to 5.0ml volume with hexane.

7.3.2 Solid Samples

A 15-gram aliquot of sample is extracted with hexane and acetone utilizing a microwave extractor. The extract is concentrated, esterified, and brought to 5.0ml volume with hexane.

7.4 Gas Chromatographic Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, the chromatographic system must be calibrated, and retention time windows must be determined.

7.4.1.1 External Standard Calibration

A minimum 5-point calibration curve is created for the herbicides and DCAA. SGS Orlando routinely performs a 6-point calibration to maximize the calibration range.

The low point may be omitted from the calibration table for any compound with an LLOQ set at the level two standard. Additionally, the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

An entire level may be omitted provided that a minimum of 5 points remain. There must be technical justification to omit an entire level. This must be documented in the run log.

Historically, many analytical methods have relied on linear models of the calibration relationship, where the instrument response is directly proportional to the amount of a target compound. The linear model has many advantages including simplicity and ease of use. However, given the advent of new detection techniques and because many methods cannot be optimized for all the analytes to which they may be applied, the analyst is increasingly likely to encounter situations where the linear model neither applies nor is appropriate. The option of using non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to compensate for detector saturation or avoid proper instrument maintenance.

NOTE: Because of this concern, select programs including SC DHEC do not support the use of non-linear regressions.

Calibration factors (CF) for the herbicides and surrogates are determined at each concentration by dividing the area (or height) of each compound by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the calibration factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively, a calibration curve of response vs. amount can be plotted. This method allows for the use of average response factors, linear regressions, and non-linear regressions. Linear regressions may be unweighted or weighted as $1/x$ or $1/x^2$. If the correlation coefficient (r) is ≥ 0.995 ($r^2 \geq 0.990$) then the curve can be used to quantitate target analytes in the samples. Regardless of which calibration model is chosen, the laboratory should visually inspect the curve plots to see how the individual calibration points compare to the plot.

Alternatively, either of the two techniques described below may be used to determine whether the calibration function meets acceptable criteria. These involve refitting the calibration data back to the model. Both %

Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

% ERR = (xi-x’i) / xi * 100

x’i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at calibration level i, in mass or concentration units.

Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (%RSE)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

x’i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at 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.

The %RSE acceptance limit criterion is ≤20%.

7.4.1.2 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV must be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be ≤ 15%. If the ICV does not meet this criteria, a second standard should be prepared. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, analyze an ICV prepared from a third source or lot. Determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

NOTE: For any DoD QSM project, the %D for all target analytes should be ≤ 20%. If samples must be analyzed with a target analyte having a %D > 20%, then the data must be qualified accordingly.

If the ICV still does not meet criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.4.1.3 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. 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 or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations.

The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. The analyst should monitor the retention times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential “hits”. The analyst will then review these “hits” and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

7.4.2 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the herbicides are prepared at various concentrations; at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally, a CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| should be ≤ 15% for each analyte.

If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated. If the second standard meets criteria, then the system is considered in control and results may be reported.

Rationale for second standard such as instrument maintenance, clipped column, remade standard, etc. must be documented in the run log or maintenance log. Reanalysis of second standard without valid rationale may require the analysis of a third standard (in which case both the second and third standard would have to pass).

NOTE: For any DoD QSM project, if the second standard meets criteria, then a third standard must be analyzed. If the third standard also meets criteria, then the system is considered in control and results may be reported.

If the |%D| is greater than 15%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: For any DoD QSM project, the %D for all target analytes should be ≤ 20%. If samples must be reported with a target analyte having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed, or the data must be qualified.

7.4.3 Sample Extract Analysis

7.4.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Conditioning Standard
Initial Calibration Standards (or Initial CCV)
QC Extracts
Sample Extracts
CCV Standards

7.4.3.2 One microliter (same amount as standards) of extract is injected into the GC by the autosampler. A splitless injection technique is used. The data system then records the resultant peak responses and retention times.

7.4.3.3 Tentative identification of an analyte occurs when the peaks from the sample extract fall within the established retention time windows for a calibrated compound on the primary column.

7.4.3.4 If the peaks of interest fall within the retention time windows on the confirmation column, the identification is confirmed. Quantitation of the analyte on the primary and confirmation column should agree within 40%. If the difference is greater than 40% and no obvious reason can be found, the higher result should be reported and flagged as “estimated”; otherwise, the result from the primary column should be reported.

7.4.3.5 If the compound identification does not confirm on a dissimilar column, then the result should be reported as ND or “U”.

7.4.3.6 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.3.7 If peak identification is prevented by the presence of interferences, further cleanup may be required, or the extract must be diluted so that the interference does not mask any analytes.

7.5 Maintenance and Trouble Shooting

7.5.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.

7.5.2 All instrument maintenance must be documented in the appropriate “Instrument Repair and Maintenance” log. The log will include such items as problem, action taken, correction verification, date, and analyst.

7.5.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

7.5.4 PC and software changes must be documented in the “Instrument Repair and Maintenance” log. Software changes may require additional validation.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Surrogates

9.1.1 DCAA (2,4-Dichlorophenylacetic acid) is used as the surrogate standard to monitor the efficiency of the extraction and clean-up procedures.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

$$\% \text{ Recovery} = (\text{Sample Amount} / \text{Amount Spiked}) \times 100$$

The percent recovery must fall within the established control limits for the results to be acceptable.

9.1.2 If the surrogate recoveries are not within the established control limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or surrogate solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.1.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, reanalyze the sample. **NOTE: If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary; however, the resulting data must be qualified accordingly.** If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis must be reported on the final report.

9.2 Method Blank

9.2.1 The method blank is either de-ionized water or acidified sodium sulfate (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high-level samples. The method blank must be free of any analytes of interest or interferences at ½ the required LLOQ to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination must be investigated and documented. The sample results can be reported without qualification. **NOTE**: **For samples reported to SC DHEC or DoD the associated sample results must still be reported with the B qualifier**.

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination must be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination must be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is either de-ionized water or acidified sodium sulfate (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable.

NOTE: A secondary check against 70-130% limits must be performed for all analytes reported to SC DHEC.

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.3.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary, and the sample results can be reported without qualification.

9.3.2.4 **If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable; however, the resulting data must be qualified accordingly**.

9.3.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.3.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.4 Matrix Spike and Matrix Spike Duplicate

9.4.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.4.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions or internal standards. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.4.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.4.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are an indication of the sample matrix effects.

9.4.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If more than 33% of the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

10.0 CALCULATIONS

The concentration of each chlorinated herbicide in the original sample is calculated as follows:

Water (ug/l) = $(\text{CONC}_{\text{inst}}) \text{ X } (V_F / V_I) \text{ X DF}$

Soil (ug/kg) = $[(\text{CONC}_{\text{inst}}) \text{ X } (V_F / W_I) \text{ X DF}] / \%\text{solids}$

$\text{CONC}_{\text{inst}}$ = Instrument concentration calculated from the initial calibration using mean CF or curve fit.
DF = Dilution Factor
V_F = Volume of final extract (ml)
V_I = Volume of sample extracted (ml)
W_I = Weight of sample extracted (g)
%solids = Dry weight determination in decimal form

All soils are reported on a dry weight basis.

If calibration standards have been prepared in the same manner as the samples (e.g., as acid herbicides and have undergone esterification) then the calculation of concentration above should be used. However, if the calibration is performed using standards made from methyl ester compounds (compounds not esterified by application of this method) then the calculation of concentration must include a correction for the molecular weight of the methyl ester versus the acid herbicide. This correction may be accounted for in the calibration table. See the GC STD Summary for the corrected concentrations.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Program, which includes the use of safety glasses, gloves, and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample must be treated as a potential health hazard. Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment must be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis and standards preparation are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 REFERENCES

SW846 Method 8000D Revision 4, July 2014

SW846 Method 8151A Revision 1, December 1996

ANALYSIS OF CHLORINATED HERBICIDES BY GAS CHROMATOGRAPHY, ELECTRON CAPTURE DETECTOR

SOP Acknowledgement Form

I have read and understand this SOP. I will not knowingly deviate from this approved SOP without approval of the Department Supervisor, QA Officer, or Technical Director. If I notice any discrepancies between this SOP and the routine procedure, I will notify the Department Supervisor so that either the SOP or procedure can be changed. Furthermore, I understand that this SOP is property of SGS North America Inc. – Orlando and may not be printed nor duplicated in any manner.

Internal SOPs referenced within this SOP: OP037, OP038, GC001, QA020, QA029

Print Name	Signature	Date

Print the SOP Acknowledgement Form, sign, and submit to the SGS Orlando QA department.

ANALYSIS OF NITROAROMATICS, NITRAMINES, AND NITRATE ESTERS BY HPLC METHOD SW-846 8330B

Prepared by: Norm Farmer Date: 01/20/2021

Approved by: Mike Eger Date: 01/22/2021

Annual Review

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TITLE: ANALYSIS OF NITROAROMATICS, NITRAMINES, AND NITRATE ESTERS BY HPLC METHOD SW-846 8330B

REFERENCES: SW846 8330B

REVISED SECTIONS: 2.1.2, 7.3.1, 7.4.1.2 and 7.4.2

1.0 SUMMARY, SCOPE AND APPLICATION

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of specific nitroaromatics, nitramines, and nitrate esters in water and solid matrices utilizing an HPLC equipped with a diode array detector.

1.1.2 The following compounds can be reported by this method:

Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	
1,3-Dinitrobenzene	1,3,5-Trinitrobenzene
2,6-Dinitrotoluene	2,4,6-Trinitrotoluene
2,4-Dinitrotoluene	2,4-diamino-6-Nitrotoluene
2-amino-4,6-Dinitrotoluene	2,6-diamino-4-Nitrotoluene
4-amino-2,6-Dinitrotoluene	2-amino-6-Nitrotoluene
Nitrobenzene	2-amino-4-Nitrotoluene
o-Nitrotoluene	4-amino-2-Nitrotoluene
m-Nitrotoluene	2,4-Diaminotoluene
p-Nitrotoluene	2,6-Diaminotoluene
DNX	3,5-Dinitroaniline
MNX	Nitroglycerine
TNX	PETN

1.1.3 The Lower Limit of Quantitation (LLOQ) or Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. LLOQs may vary depending on matrix complications and volumes. LLOQs for this method are in the range of 0.2 to 2.0 ug/l for aqueous samples and 100 to 1000 ug/kg for solid samples.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the LLOQ. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported LLOQ. Note: MDL verifications for 8330B soils must be spiked prior to grinding.

1.1.5 The LLOQ for each analyte is evaluated on an annual basis for each matrix and instrument. The LLOQ verifications are prepared by spiking a clean matrix at 0.5 to 2 times the current LLOQ level. This LLOQ verification is carried through the same preparation and analytical procedures as the samples. Recovery of the analytes should be within the established limits. The DOD QSM requirements for Limit of Detection (LOD) and Limit of Quantitation (LOQ) verifications are different. See SOP QA020 for complete requirements for MDL, LOD, LOQ, and LLOQ.

1.1.6 Compounds detected at concentrations between the LLOQ and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I” qualifier. Some program or project specifications may require that no values below the LLOQ be reported

1.1.7 For DOD projects refer to 5.0, Table 3; or QSM 5.x Table B-3 for additional method requirements and data qualifying guidance.

1.2 Summary

1.2.1 This method is adapted from SW846 Method 8330B.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with SGS – Orlando SOP OP018 and OP046.

1.2.4 The extracts are analyzed on an HPLC equipped with a diode array detector.

1.2.5 Manual integrations are performed in accordance with SOP QA029.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Aqueous samples shall be collected in amber glass bottles with Teflon lined caps. One-liter or 250ml bottles are recommended for aqueous samples.

2.1.2 Soil samples shall be collected by multi-incremental sampling or by the collection of discreet samples. This can result in sample sizes of one to two kilograms. ISM samples shall be collected in heavy duty 1 or 2-gallon Ziplock bags. It is recommended that the samples be double bagged to prevent punctures. Discreet samples are collected in 8oz jars.

2.1.3 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. Soil samples can be stored at room temperature after they have been air dried. The extracts must be stored at ≤ 6°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts should be analyzed as soon as possible but must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. All of these materials must be demonstrated to be free from interferences.

3.3 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 and acidified to pH < 3. Samples and extracts must not be exposed to temperatures above room temperature.

3.4 High levels of 4-amino-2-nitrotoluene may interfere with the surrogate 3,4-dinitrotoluene on the (Zorbax Extend C-18) primary column. In such instances, the surrogate recovery should be calculated from the confirmation column.

3.5 3,5-dinitroaniline partially co-elutes with o-nitrotoluene and p-nitrotoluene on the confirmation column.

3.6 When analyzing the RDX breakdown analytes, TNX may partially coelute with HMX on the primary column and DNX may partially co-elute with HMX on the confirmation columns.

3.7 Samples from sites using alkaline hydrolysis to remediate explosives may have a high basic pH. These samples and extracts must be neutralized prior to analysis to prevent damage to the analytical column.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the LLOQ.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Proficiency Test Sample (PT): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The PT sample is generally prepared by an outside vendor. This method requires that the PT sample go through the entire preparatory procedure including sieving and grinding. PT sample recoveries are used to document laboratory and method performance.

4.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.12 Sample Triplicate (TRP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.13 Grinding Blank (GB): An aliquot of blank sand that is processed through the ring and puck mill between different samples. It is used to monitor for carry over between samples ground with the same bowl set.

4.14 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.15 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Water – HPLC grade or equivalent

5.2 Acetonitrile – HPLC grade or equivalent

5.3 Methanol – HPLC grade or equivalent

5.4 Explosives stock standards – Traceable to Certificate of Analysis

5.5 Surrogate standards – 3,4-Dinitrotoluene

6.0 APPARATUS

6.1 HPLC – Agilent Technologies 1100 or 1260

Suitable HPLC equipped with an autosampler, pump, and diode array detector.

Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Data System – Agilent Technologies LC Chemstation rev. A 10.01 or B 04.03
Agilent Technologies MS Chemstation rev. DA 00.01 or EA 02.01

6.2.1 A computer system interfaced to the HPLC that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.2.2 The software must allow for quantitation at multiple wavelengths. Additionally, the software should allow for the viewing of the entire UV Spectra acquired over the analytical run. Comparisons can then be made between spectra from standards and samples.

6.2.3 Data is archived to a backup server for long term storage.

6.3 Primary Column – Zorbax Extend C-18 3.5u – 4.6mm X 100mm or equivalent

6.4 Confirmation Column– Zorbax Bonus RP (amine bonded C-18) 5u - 4.6mm X 250mm or equivalent

6.5 Gas-tight syringes, syringe filters, and class “A” volumetric glassware for dilutions of standards and extracts.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the HPLC Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at ≤ 6°C, or as recommended by the manufacturer. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the HPLC STD Summary in the Active SOP directory.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a “Certificate of Analysis” with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor’s expiration date. Once opened, the hold time is reduced to one year or the vendor’s expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with acetonitrile. The hold time for intermediate standards is six months or the vendor’s expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicate analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the explosives are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. Calibration standards are prepared in 75/25 (v/v) water/acetonitrile. The low standard is at a concentration at or below the LLOQ and the remaining standards define the working range of the detector.

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 HPLC Conditions

7.2.1 HPLC-BB/PP Conditions - Primary Column – (Extend C-18)

100 ul autosampler injection

Mobile phase – Gradient: Water (A), Methanol (B)

Time (min)	Solvent A	Solvent B
0-1.5	79%	21%
1.5-5.0	72%	28%
5.0-10.0	72%	28%
10.0-15.0	65%	35%
15.0-18.0	50%	50%
18.0-19.0	50%	50%
19.0-19.5	0%	100%
19.5-23.0	0%	100%

Column temperature: 43.0 °C Constant Flow: 2.0 ml/min

Diode Array Detector – Set to acquire and process data at 254-nm wavelengths using a 10-nm bandwidth. Secondary wavelength may be set to 214-nm. The 254-nm wavelength switches to 270-nm just prior to the elution of the nitrotoluenes. All data from 200-nm to 450-nm wavelengths is stored for spectral evaluation.

HPLC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.2.2 HPLC-GG/PP Conditions - Confirmation Column – (Bonus RP)

100 ul autosampler injection

Mobile phase – Gradient: Water (A), Methanol (B)

Time (min)	Solvent A	Solvent B
0-1.00	41%	59%
1.00-1.25	50%	50%
1.25-16.0	47%	53%
16.0-19.5	41%	59%
19.5-22.0	41%	59%

Column temperature: 22.0-23.0 °C Constant Flow: 0.75-0.8 ml/min

Diode Array Detector – Set to acquire and process data at 254-nm wavelengths using a 10-nm bandwidth. Secondary wavelength may be set to 214-nm. All data from 200-nm to 450-nm wavelengths is stored for spectral evaluation.

HPLC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3. Sample Preparation

7.3.1 Water Samples (extracted)

A 250ml or 1000ml aliquot of sample is extracted utilizing a solid phase cartridge. The cartridge is eluted with acetonitrile. The final volume is then adjusted to 5ml or 10ml with reagent water.

7.3.2 Solid Samples

A 10-gram aliquot of sample is extracted with 20ml of acetonitrile utilizing a platform shaker. The final volume is then adjusted to 50ml with reagent water. The extract is filtered through a .45um Teflon syringe filter to remove any particulate.

7.4. HPLC Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, the HPLC system must be calibrated, and retention time windows must be determined.

7.4.1.1 External Standard Calibration

A minimum 5-point calibration curve is created for the explosives and surrogates. SGS Orlando routinely performs a 7-point calibration to maximize the calibration range.

The low point may be omitted from the calibration table for any compound with an RL set at the level two standard. Additionally, the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

An entire level may be omitted provided that a minimum of 5 points remain. There must be technical justification to omit an entire level. This must be documented in the run log.

Historically, many analytical methods have relied on linear models of the calibration relationship, where the instrument response is directly proportional to the amount of a target compound. The linear model has many advantages including simplicity and ease of use. However, given the advent of new detection techniques and because many methods cannot be optimized for all the analytes to which they may be applied, the analyst is increasingly likely to encounter situations where the linear model neither applies nor is appropriate. The option of using non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to compensate for detector saturation or avoid proper instrument maintenance.

NOTE: Because of this concern, select programs including SC DHEC do not support the use of non-linear regressions.

Calibration factors (CF) for the explosives and surrogates are determined at each concentration by dividing the area of each compound by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the response factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD $\leq$ 15%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively, a calibration curve of response vs. amount can be plotted. This method allows for the use of average response factors, linear regressions, and non-linear regressions. Linear regressions may be unweighted or weighted as $1/x$ or $1/x^2$. If the correlation coefficient (r) is ≥ 0.995 ($r^2 \geq 0.990$) then the curve can be used to quantitate target analytes in the samples. Regardless of which calibration model is chosen, the laboratory should visually inspect the curve plots to see how the individual calibration points compare to the plot.

Alternatively, either of the two techniques described below may be used to determine whether the calibration function meets acceptable criteria. These involve refitting the calibration data back to the model. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$\% \text{ ERR} = (x_i - x'_i) / x_i \; * \; 100$

x'_i = Measured amount of analyte at calibration level i, in mass or concentration units.

x_i = True amount of analyte at calibration level i, in mass or concentration units.

Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (%RSE)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at 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.

The %RSE acceptance limit criterion is ≤15%.

7.4.1.2 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV must be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be ≤ 20%. If the ICV does not meet this criteria, a second standard must be prepared. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, analyze an ICV prepared from a third source or lot. Determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

For any DoD QSM project, if samples must be analyzed with a target analyte having a %D > 20%, then the data must be qualified accordingly.

NOTE: Second source standards may not be available for TNX, DNX, and MNX.

7.4.1.3 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. 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 or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations. HPLC retention times tend to shift more than GC retention times.

The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh heavily in the interpretation of the chromatograms. The analyst should monitor the retention times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential “hits”. The analyst will then review these “hits” and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

7.4.2 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the explosives are prepared at various concentrations; at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally, a CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| should be ≤ 20% for each analyte.

If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated. If the second standard meets criteria, then the system is considered in control and results may be reported.

Rationale for second standard such as instrument maintenance, fresh mobile phase, remade standard, etc. must be documented in the run log or maintenance log. Reanalysis of second standard without valid rationale may require the analysis of a third standard (in which case both the second and third standard would have to pass).

NOTE: For any DoD QSM project, if the second standard meets criteria, then a third standard must be analyzed. If the third standard also meets criteria, then the system is considered in control and results may be reported.

If the |%D| is greater than 20%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reportable. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: For any DoD QSM project, if samples must be reported with a target analyte having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed, or the data must be qualified.

7.4.3 Sample Extract Analysis

7.4.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Initial Calibration Standards (or Initial CCV)
QC Extracts

Sample Extracts
CCV Standards

7.4.3.2 One hundred microliters (same amount as standards) of extract is injected into the HPLC by the autosampler. The data system then records the resultant peak responses and retention times.

7.4.3.3 Tentative identification of an analyte occurs when the peaks from the sample extract fall within the established retention time windows for a calibrated compound.

7.4.3.4 The diode array detector is capable of spectral evaluation; however, the UV spectra for some explosive analytes are not unique. Confirmation by reanalysis on a dissimilar column is required for positive identification. Confirmation analysis should be performed at the same dilution(s) as the primary analysis.

7.4.3.5 If the peaks of interest fall within the retention time windows on the confirmation column, the identification is confirmed. Quantitation of the analyte on the primary and confirmation column should agree within 40%. For LIMS to be able to calculate the difference (%RPD) between primary and confirmation results, the same dilution(s) must be run on each column.

If the difference is greater than 40% and no obvious reason can be found, the higher result should be reported and flagged as “estimated”; otherwise, the result from the primary column should be reported.

7.4.3.6 If the compound identification does not confirm, then the result should be reported as ND or “U”.

7.4.3.7 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.3.8 If peak identification is prevented by the presence of interferences, further cleanup may be required, or the extract must be diluted so that the interference does not mask any analytes. Analysis on the confirmation column may also be beneficial.

7.5. Maintenance and Trouble Shooting

7.5.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.

7.5.2 All instrument maintenance must be documented in the appropriate “Instrument Repair and Maintenance” log. The log will include such items as problem, action taken, correction verification, date, and analyst.

7.5.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

7.5.4 PC and software changes must be documented in the “Instrument Repair and Maintenance” log. Software changes may require additional validation.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

DoD QSM projects require the analysis of a sample triplicate (TRP), a proficiency test sample (PT) and equipment grinding blanks (GB).

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), matrix spike duplicate (MSD), and sample duplicate (DUP). DoD QSM projects require the analysis of a sample triplicate (TRP), a proficiency test sample (PT) and equipment grinding blanks (GB).

9.1 Surrogates

9.1.1 3,4-Dinitrotoluene is used as the surrogate standard to monitor the efficiency of the extraction.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

$$\% \text{ Recovery} = (\text{Sample Amount} / \text{Amount Spiked}) \times 100$$

The percent recovery must fall within the established control limits for the results to be acceptable.

9.1.2 If the surrogate recovery is not within the established control limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or surrogate solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.1.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, reanalyze the sample. **NOTE: If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary; however, the resulting data must be qualified accordingly.** If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis must be reported on the final report.

9.2 Method Blank

9.2.1 The method blank is either de-ionized water or cleaned sand (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high-level samples. The method blank must be free of any analytes of interest or interferences at ½ the required LLOQ to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination must be investigated and documented. The sample results can be reported without qualification. **NOTE**: **For samples reported to SC DHEC or DoD the associated sample results must still be reported with the B qualifier.**

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination must be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination must be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is either de-ionized water or cleaned sand (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable. The large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of the established control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary.

Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A marginal exceedance in the Blank Spike is defined as a recovery being outside of 3 standard deviations but within 4 standard deviations of the mean.

The number of allowable marginal exceedances is based on the number of analytes in the Blank Spike. Marginal Exceedances must be random. If the same analyte exceeds the BS control limits repeatedly, it is an indication of a systematic problem and corrective action must be taken.

Marginal exceedances are not permitted for analytes that are deemed to be “Compounds of Concern” for a specific project. “Compounds of Concern” are different from “Target Compounds”. “Target Compounds” are all analytes that are being reported for a site where “Compounds of Concern” are those analytes expected to be present at the site

The number of allowable marginal exceedances is as follows:

1) 11-30 analytes in BS, 1 analyte allowed in ME range;

2) < 11 analytes in BS, no analytes allowed in ME range.

NOTE: SC DHEC does not recognize the concept of Marginal Exceedances. Additionally, a secondary check against 70-130% limits must be performed for all analytes reported to SC DHEC.

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.3.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary, and the sample results can be reported without qualification.

9.3.2.4 **If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable; however, the resulting data must be qualified accordingly**.

9.3.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.3.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.4 Proficiency Test Sample (DoD QSM ISM soil projects)

9.4.1 The proficiency test sample is a bulk volume soil sample. The PT sample is prepared by an outside vendor at specific analyte concentrations. The PT sample is taken through all preparatory procedures including drying, sieving, grinding, and subsampling.

The surrogate standard is added prior, and the PT sample is then extracted with the other samples to monitor the efficiency of the entire procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (PT Sample Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the Vendors established control limits for the results to be acceptable.

9.4.2 If the PT sample recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.4.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.4.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary, and the sample results can be reported without qualification.

9.4.2.4 If the recovery of an analyte in the PT sample is high and the associated sample is non-detect, the data may be reportable. For any DoD QSM projects the resulting data must be qualified accordingly.

9.4.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.4.2.6 If the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.5 Matrix Spike and Matrix Spike Duplicate

9.5.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.5.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.5.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.5.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.5.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are an indication of the sample matrix effects.

9.5.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If the RPDs fall outside of the established control limits, the sample, MS and/or MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary.

9.6 Sample Duplicate

The sample duplicate is a replicate sample aliquot to which the surrogate standard has been added. Sample duplicate is then extracted and taken through all cleanup procedures along with the other samples to monitor the precision of the extraction procedure.

Sample and sample duplicate results for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| Sample Result – DUP Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If the RPDs fall outside of the established control limits, the sample and/or DUP should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary.

9.7 Sample Triplicate (DoD QSM ISM soil projects)

The sample triplicate is an additional replicate sample aliquot to which the surrogate standard has been added. Sample triplicate is then extracted and taken through all cleanup procedures along with the other samples to monitor the precision of the extraction procedure.

Sample, sample duplicate, and sample triplicate results for each analyte are used to calculate the percent relative standard deviation (%RSD) for each compound.

%RSD = [Standard Deviation of the Result / Average Result] X 100

The %RSD for results above the reporting limit must not be greater than 20%. If the %RSD is greater than 20%, the sample, DUP, and/or TRP should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the %RSD is still greater than 20%, the department supervisor shall review the data and determine if any further action is necessary.

9.8 Grinding Blanks (DoD QSM ISM soil projects)

9.8.1 The grinding blanks (GB) are aliquots of blank sand that are processed through the ring and puck mill between different samples. They are used to monitor for carry over between samples ground with the same bowl set. The grinding blanks for each bowl set may be composited prior to analysis.

9.8.2 The grinding blanks must be free of any analytes of interest or interferences at ½ the required LLOQ to be acceptable. If the grinding blanks are not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated grinding blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the individual grinding blanks (non-composited), reanalyzing the samples or qualifying the results with a “B” or “V” qualifier. This must be approved by the department supervisor.

9.8.3 If the grinding blank is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

10.0 CALCULATIONS

The concentration of each explosive in the original sample is calculated as follows:

Water (ug/l) = $(\mathrm{CONC}_{\mathrm{inst}}) \times (V_F / V_I) \times \mathrm{DF}$

Soil (ug/kg) = $[(\mathrm{CONC}_{\mathrm{inst}}) \times (V_F / W_I) \times \mathrm{DF}]$

$\mathrm{CONC}_{\mathrm{inst}}$ = Instrument concentration calculated from the initial calibration using mean CF or curve fit
DF = Dilution Factor
V_F = Volume of final extract (ml)
V_I = Volume of sample extracted (ml)
W_I = Weight of sample extracted (g)

Soils are air dried prior to extraction; therefore, %solids are not used in the calculation.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses, gloves, and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample must be treated as a potential health hazard. Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment must be used by all analysts.

11.2 Pollution Prevention

Wastewater, methanol, and acetonitrile from the instrument are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 REFERENCES

SW846 Method 8000D Revision 4, July 2014

SW846 Method 8330A Revision 1, February 2007

SW846 Method 3535A Draft Revision 1, November 2000

SW846 Method 8330B Revision 2, October 2006

DoD Quality System Manual, Version 5.1, January 2017

DoD Quality System Manual, Version 5.3, May 2017

ANALYSIS OF NITROAROMATICS, NITRAMINES, AND NITRATE ESTERS BY HPLC METHOD SW-846 8330B

SOP Acknowledgement Form

I have read and understand this SOP. I will not knowingly deviate from this approved SOP without approval of the Department Supervisor, QA Officer, or Technical Director. If I notice any discrepancies between this SOP and the routine procedure, I will notify the Department Supervisor so that either the SOP or procedure can be changed. Furthermore, I understand that this SOP is property of SGS North America Inc. – Orlando and may not be printed nor duplicated in any manner.

Internal SOPs referenced within this SOP: OP018, OP046, GC001, QA020, QA029

Print Name	Signature	Date

Print the SOP Acknowledgement Form, sign, and submit to the SGS Orlando QA department.

MICRO DISTILLATION OF CYANIDE IN AQUEOUS AND SOLID MATRICES

Prepared by:	Svetlana Izosimova	Date:	08/08/2016
Approved by:	Luis Jimenez	Date:	08/08/2016

Annual Review

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TITLE: MICRO DISTILLATION OF CYANIDE IN AQUEOUS AND SOLID MATRICES

REFERENCES: EPA Method 335.4, revision 1.0, 1993; SW846 9012B, 2004, DoD QSM Ver. 5.0; TNI 2009 Standards

REVISED SECTIONS: DoD 4.2 replaced with DoD 5.0 throughout the document; Sec. 6.1 – addition of Ascorbic acid; Secs. 7..9, 7.10 - added apparatus detail; QC frequency added to Table 1; Standard levels are reconciled with table in GN115; re-branded to SGS Accutest throughout document

1.0 SCOPE AND APPLICATION

1.1 This method is applicable for the distillation of aqueous, solid, or liquid waste samples for cyanide. All samples must be taken through the distillation process before they can be analyzed for cyanide.

2.0 SUMMARY

2.1 Cyanide is 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 ion. The sample is distilled in a micro distillation unit. The sample is treated to remove interference from oxidizing agents, sulfides, and nitrites.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. See determinative method.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Since this SOP commingles SW-846 9012B and EPA 335.4, experimental aqueous MDLs must be determined semiannually for this method, as outlined in EPA 335.4. Refer to SOP QA020, current revision, for further details.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or samples loaded on an instrument within the same 12-hour shift, which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS). This is prepared from a source different from one used for the initial calibration.

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process. Both MS and MSD prepared from a source different from one used for the initial calibration.

4.10 Method Detection Limits (MDLs) MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. This definition is qualitative in nature and does not evaluate an acceptable quantitative limit for method performance. MDLs should be determined semiannually for this method. Refer to SOP QA020, current revision.

4.11 Reagent Blank: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. For methods requiring no preparation step, the reagent blank is equivalent to the method blank.

4.12 Reagent Grade: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents, which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

4.13 Reagent Water: Water that has been generated by any method, which would achieve the performance specifications for ASTM Type II water.

4.14 Reference Material: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

4.15 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.16 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the SGS Accutest Health and Safety Program which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 Cyanide is very toxic. Samples should be kept basic until time of distillation to prevent the formation of HCN. Make sure that the distillation flasks are tightly closed and leak free during the distillation process.

5.3 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of MSDS should be made available to all personnel involved in these analyses.

6.0 PRESERVATION AND HOLDING TIME

6.1 All samples should be preserved with NaOH to a pH of $\geq$12 and stored in a refrigerator at 4°C. **Ascorbic acid should be added in the field only if necessary** to prevent decomposition of the cyanides by oxidizing agents such as chlorine. The pH for each sample should be checked before the distillation process is started.

6.2 **All samples should be analyzed within 14 days of the date of collection.**

7.0 APPARATUS

7.1 Distillation apparatus. Micro Distillation block, Lachat part number A17102/202 or equivalent.

7.1.1 Maintenance and troubleshooting are limited to verification of temperature reading on block. Temperature reading verified quarterly with NIST thermometer.

7.2 MicroDistillation tubes and caps, Lachat part number A17117

7.3 Autopipetters, calibrated as per QA006, current revision

7.4 Volumetric Pipets, class A.

7.5 Volumetric flasks, class A.

7.6 50 ml graduated cylinders, Class A.

7.7 Lead Acetate paper.

7.8 Wide range pH paper capable of measuring pH values from 0 to 14.

7.9 Top-loading balance, OHAUS ADVPRO or equivalent, accurate to 0.01 g

7.10 Analytical balance, OHAUS ADVPRO or equivalent, capable of accurately weighing to the nearest 0.0001g. Balances are calibrated annually by a third-party vendor, verified daily with Class 1 weights.

7.11 Class 1 weights.

7.12 Potassium-iodide starch indicating paper

8.0 REAGENTS

All reagents should be made from ACS grade reagents unless otherwise noted. Deionized water should be used whenever water is needed as in all preparation of reagents, record the amounts used, the lot numbers of the reagents, the date made, the expiration date, storage requirements, and your initials in the reagent log book.

8.1 7.11 M sulfuric acid/ 0.79 M magnesium chloride: In the hood place a 500 ml beaker on a top loading balance with a 400g capacity or greater. Tare the balance; place 110.8g of DI water into the beaker. Then add and dissolve 32.2g of magnesium chloride hexahydrate (MgCl2. 6H2O). Slowly add 139g of sulfuric acid (in small increments). The solution will become hot.

8.2 Sulfuric acid, concentrated.

8.3 Sulfamic acid (NH_2SO_3H).

8.4 1000 ppm primary and secondary CN Stock: Purchased commercially as certified solution. The solution is accompanied by Certificate of Analysis (CoA). CoA is examined for accuracy and completeness of the information, including verification of the titrant normality. Stability of the reagent is monitored according to manufacturer's specifications. For further details on standard traceability refer to SOP QA017, current revision.

Alternatively this standard can be made by dissolving 2.51 g of KCN and 2 g KOH in 900 ml of DI water and then diluting to a final volume of 1 liter with DI water. Standardize with 0.0192 N $AgNO_3$ following the procedure outlined in the standardization section below. If it is not purchased as a certified solution, the cyanide stock should be standardized a minimum of once per week per procedure outlined in sec. 10.

8.4.1 If an external check / ICV / spiking solution is to be prepared use the same procedure as outlined in 8.5, but use a different lot of KCN.

8.5 0.100 ppm CN spiking solution. Add 10.0 uL of 1000 ppm stock solution to a 100 ml volumetric flask. Dilute to volume with DI water.

8.6 Intermediate Cyanide Solution, 10.0 mg/L. Pipet 1.0 ml of 1000 mg/l stock solution of CN into a 100 ml volumetric flask containing approximately 50 ml of .25 N NaOH. Dilute to a final volume with .25 N NaOH and mix well.

8.7 Cyanide Standard Solutions. Suggested standard concentrations are shown below. All standards are made up using the above intermediate solution (10.0 mg/L) as outlined in the table below. All the standards must be diluted to a final volume with .25 N NaOH. Alternate standard concentrations may be used as long as all method requirements are met. **Standards should have a final concentration of 0.25 N NaOH. These standards should be made fresh daily before distillation or analysis.**

ml of 10.0 mg/l CN	Final Volume	Concentration (mg/l)
5.0 ml	100 ml	0.50 mg/l
3.0 ml	100 ml	0.30 mg/l
2.0 ml	100 ml	0.20 mg/l
1.0 ml	100 ml	0.10 mg/l
0.50 ml	100 ml	0.050 mg/l
0.10 ml	100 ml	0.010 mg/l
0.00 ml	100 ml	0.000 mg/l

8.8 Standard Silver Nitrate Solution, 0.0192 N. Crush approximately 5 g of silver nitrate crystals and dry to constant weight at 40°C. Weigh out 3.2647 g of dried silver nitrate into a 1 liter flask containing approximately 800 ml of DI water. Dilute to a final volume of 1 liter with DI water and mix well.

8.9 Benzalrhodanine indicator. Dissolve 20 mg p-dimethylaminobenzalrhodanine in 100 ml acetone.

8.10 Sodium Hydroxide, 1.25 N. Dissolve 50.0 g of sodium hydroxide to approximately 800 mL of DI water. Mix well and then dilute to a final volume of 1000 mL.

8.11 Sodium Hydroxide trapping solution, 0.95 N. Add 19.0 g of sodium hydroxide to approximately 400 ml of DI water. Mix well and then dilute to a final volume of 500 ml.

8.12 Cadmium Carbonate, powdered.

8.13 pH Adjusted Blank Solution. For method blanks and spike blanks, add 1.25 N solution of NaOH dropwise to DI water to adjust the pH to $\geq$12.

9.0 INTERFERENCES

9.1 Most interferences are eliminated by the distillation procedure. Oxidizing agents such as chlorine are removed using the ascorbic acid preservative. Sulfides will distill over, but can be removed by adding cadmium carbonate to the sample. Nitrites can be an interference, but can be removed by adding sulfamic acid to the sample.

9.2 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10 mg/l were not found to interfere.

9.3 Fatty acids, detergents, surfactants, and other compounds may cause foaming during the distillation when they are present in high concentrations. This interference can be minimized by using a smaller sample size or by following a special extraction procedure. Check with the lab manager or supervisor for additional information.

10.0 CYANIDE STANDARDIZATION PROCEDURE

Purchased certified stock solutions do not have to be standardized weekly. Monitor stock solutions for signs of degradation and replace solutions prior to manufacturers expiration date. If stock solutions are made from dry chemicals follow the steps below.

10.1 If the cyanide stock solution has not been standardized within a week, then the stock must be standardized before proceeding further with the analysis. The standardization can be done following the procedure outlined below.

10.2 Volumetrically measure out 25.0 ml of the 1000 mg/l cyanide stock solution into a graduated plastic beaker. Add 10 to 12 drops of benzalrhodanine indicator to the solution. Place a stir bar in the beaker.

10.3 Fill a burette with 0.0192N silver nitrate solution. Titrate the sample with continuous stirring until the color changes from yellow to a brownish-pink. Approximately 25 ml of silver nitrate solution should be needed to reach this endpoint. Also analyze a method blank that has been brought to pH > 12 with KOH following this procedure.

10.4 Calculate the concentration of the stock cyanide solution using the equation shown below. If multiple standardizations of the stock are done at a given time, then the average of the standardizations should be used as the final stock concentration.

$$\text{CN, mg/l} = \frac{(A - B) \times 1000}{25 \text{ ml}}$$

Where: A = the volume of $AgNO_3$ used to titrate the sample.
B = the volume of $AgNO_3$ used to titrate the blank.

11.0 TEST FOR SULFIDES

Every sample to be distilled for cyanide should first be tested for sulfides as outlined below.

11.1 Put a drop of sample on a piece of Lead Acetate paper.

11.2 Check to see if there is darkening of the paper. The darkening indicates that sulfide is present.

11.3 If sulfide is present, take a 25-ml aliquot of the preserved sample and add powdered cadmium carbonate. Continue to add the cadmium carbonate until no more yellow precipitate is formed. Avoid a large excess of cadmium and a long contact time, which may cause the sample to lose cyanide.

11.4 Filter the sample through dry filter paper into a dry beaker to remove the yellow precipitate. Check the filtered sample with lead acetate paper to verify that no additional sulfide is present.

11.5 The filtered sample can now be distilled. Note that cadmium carbonate was added to the sample in the comments section of the distillation log. Document the sulfide results on the distillation log.

12.0 CYANIDE DISTILLATION PROCEDURE - MICRO DISTILLATION

12.1 Set the controller temperature to 120 ºC. Allow the heater block to warm up. This will take about 40 minutes.

12.2 Each sample tube should be labeled with the sample ID, the product, and the distillation date. Each QC tube should be labeled with the sample ID, the product, the distillation date, and the batch ID.

12.3 Check the pH of each sample with pH paper and record the pH on the distillation log. If the pH is less than 12, adjust the pH to $\geq$ 12 with 1.25 N NaOH and notify the supervisor and document the pH adjustment on the distillation log. Make sure to also document the sample numbers and the initial volumes in the distillation log.

12.3.1 Check the sample first with the narrow-range (app. 12.5 to 14) pH paper. If the paper changes color, then record the pH. If it does not, use the 1 to 12 pH paper to verify that the sample is at pH 12.

12.3.2 For the method blanks and spike blanks, add 50 ml of DI water which has had the pH adjusted to $\geq$12 as outlined in section 8.13.

12.4 Check samples for the presence of chlorine and/or other oxidizing reagents.

12.4.1 Put a drop of sample on a piece of Potassium iodide starch paper.

12.4.2 Check to see if there is darkening in the paper. Make a note in the distillation log if there is a color change.

12.5 Add 6.0 ml of sample or standard to the sample tube and 0.02g of sulfamic acid. For blank spike add 6 ml of 0.100 mg/L CN spike solution and for matrix spike follow the same procedure used for making the 0.100 mg/L CN spiking solution but use the sample instead of DI water and transfer 6 ml to the sample tube.

12.5.1 For soil samples :
Mix the sample well and weigh out from 0.5 to 1.0g of sample on a top-loading balance. Record the weight to nearest 0.01g. Place the sample in the sample tube, then add 5.5 ml of DI water and 0.02g of sulfamic acid solution.
Make the Matrix Spike as follows:
Place 0.5g of well-mixed sample in the sample tube and add 5.5 ml of 0.10 mg/L CN spike solution and 0.02 g of sulfamic acid solution

12.5.2 **Note**: when the calibration curve is not distilled, then a high and a low standard must be distilled with each batch. (see Sec 14.2.1)

12.6 Add 1.51 ml of 1 N NaOH (trapping solution) to level marked with **M** and place the membrane and cap.

12.7 Add 0.750 ml of MgCl2/H2SO4 to the sample tube and immediately push D end to the sample tube and press.

12.8 Place the samples and quality control into the preheated distillation block (120 ^{o}C) and set the timer for a minimum of 30 minutes not to exceed 35 min. Make sure that the samples are fully pushed down into the block to ensure complete distillation.

12.9 After 30 minutes, take out the samples one at a time following the procedure outlined below.

12.9.1 Put on heat and chemical resistant gloves. Take one sample tube from the distillation unit and quickly (within 4 seconds), pull off the bottom of the tube and discard the sample liquid into waste. Let the distillate portion cool for 10 minutes.

12.9.2 Take the distillate portion of the tube and carefully swirl all of the sample from the sides into the collection end. Tap the tube gently to bring all of the drops to the bottom. Snap off the top end (D end) and throw it away.

12.9.3 Bring the final sample volume to 6 ml with DI water and mix well. Cap and parafilm both ends of the sample and store at 4 ºC if not analyzed immediately.

12.10 Document the final volume for each sample on the distillation log. Initial and date each sample. Make sure to add any comments or observations to the distillation log.

13.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

14.0 QUALITY CONTROL

Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

14.1 Method Detection Limits (MDLs). MDLs should be established using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's ***t*** value for a 99% confidence level. MDLs should be determined semiannually and verified whenever there is a significant change in the background or instrument response.

14.2 Calibration curve. A calibration curve, consisting of a minimum of 6 points and a blank with the lowest point at or below the reporting limit, must be distilled before analyses. A distilled curve must be used for analysis of samples testing positive for sulfides. Refer to the analytical method for calibration curve acceptance criteria.

14.2.1 Note: when the calibration curve is not distilled, then a high and a low standard must be distilled with each batch to ensure that distillation technique is reliable. These results must agree within 10% with undistilled values.

14.3 Quality Control Sample (also referred to as Initial Calibration Verification Standard, (ICV)). Normally this is analyzed at the beginning of the run after the CCV and CCB checks. For EPA method 335.4 the ICV should be within 10% of the true value. For the SW846 9012B method, the ICV should be within 15% of the true value.

14.4 Method Blank. The laboratory must distill and analyze a method blank with each set of samples. A minimum of one method blank is required for every batch. The method blank must contain the analyte at <1/2RL. If the method blank is over that limit, the samples must be redigested or reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

14.5 Spike Blanks. A blank spike is required per sample batch.. For EPA 335.4 this standard must have a recovery of 90 - 110%. Since SW-846 9012B has no current information on precision and accuracy, in the absence of such data in-house criteria are to be applied. It is recommended that this standard be prepared from the same source as the calibration standards. If the blank spike does not meet the recovery limits, then the associated samples must be redistilled and reanalyzed. The exception to this rule is if the blank spike recovery is high and the results of the samples to be reported are less than the reporting limit, then the sample results can be reported with no flag.

14.6 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 10% of matrix.

14.6.1 Matrix spike recovery for EPA 335.4 must be assessed using limits of 90 - 110% recovery. Since SW-846 9012B has no current information on precision and accuracy, in the absence of such data in-house criteria or DoD 5.0 Appendix A and C are to be applied. If a matrix spike is out of control, then the laboratory performance must be assessed against Blank Spike (Sec. 14.5) and recovery problems are attributed to matrix. These data are flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the spike recovery should not be assessed against the control limits and should be footnoted to that effect.

14.6.2 The matrix spike recovery must be calculated as shown below.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} * 100 = \text{MS Recovery}$$

14.6.3 The duplicate RPD must be assessed using in house limits or DoD 5.0 App. A and C limits. Until these limits can be generated, then default limits of 20 %RPD should be applied. If a duplicate is out of control, then the results must be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± RL, then the duplicate is considered to be in control.

14.6.4 The duplicate RPD must be calculated as shown below.

$$\frac{|\text{Sample Result - Duplicate Result}| * 100}{\text{(Sample Result + Duplicate Result)/2}} = \text{\% RPD}$$

15.0 DOCUMENTATION REQUIREMENTS

A worksheet must be used to record all distillation data. Make sure that all sample ID's, initial weights or volumes, and final volumes are clearly labeled on the worksheet. Any unusual characteristics of the samples must be noted in the comment section. All reagent information such as lot numbers, vendor, date made, etc. must be recorded in the reagent log book. As each batch is completed, the distillation log must be given to the lab supervisor for review.

16.0 DATA REVIEW AND REPORTING

16.1 All samples must be updated to QC batches in the LIMS system. The analyst is responsible for reviewing all data for compliance with the QC outlined in this SOP. They are responsible for making sure that the raw data is fully documented and it is updated and entered into the LIMS system.

16.2 After the analyst review is completed, the supervisor or a designated reviewer shall review the distillation data for technical compliance to the SOP.

17.0 POLLUTION PREVENTION & WASTE MANAGEMENT

17.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 17.1.1.

17.1.1 Waste Management. Individuals performing this method must follow established waste management procedures as described in SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

18.0 ADDITIONAL REFERENCES

18.1 Lachat Micro Distillation unit reference manual
18.2 DoD QSM, Final version 5.0
18.3 SOPs GN196, QA042, QA017, SAM108

Table 1 QC Criteria

Quality Control	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration: r = coefficient of correlation	Daily	≥0.995 DoD-projects – prepared daily prior to analysis.	Rerun calibration standards, and/or prepare new calibration standards and recalibrate the instrument, or document why the data are acceptable.
Initial Calibration Verification standard (ICV)	One per calibration	EPA 335.4: 90–110% SW846 9012B: 85-115%, DoD projects 90-110%	Rerun standard, and/or prepare new standard, and/or recalibrate instrument, or document why the data are acceptable.
Continuing Calibration Verification standard (CCV), and high and low-level Standard (where applicable)	Every tenth sample	90 - 110% of the standard's true value	Rerun standard, and/or recalibrate instrument and reanalyze all samples run since the last acceptable CCV, or document why the data are acceptable.
Low-level Standard and High Standard, distilled	Every ICAL	90 - 110% of the corresponding undistilled standard signal intensity	Rerun standards, and/or recalibrate instrument and reanalyze all samples run since the last acceptable CCV, or document why the data are acceptable.
Method blank (MB) and Calibration Blank (CB)	One per batch	< ½RL; WV projects <MDL	Reanalyze, and/or stop the run and determine the source of contamination, or document why the data are acceptable.
Blank Spike (BS or LCS)	One per batch	EPA 335.4: 90-110% SW846 9012B: in-house, updated annually, DoD projects per QSM 5.0	Determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable.
MS/MSD	10% of matrix	EPA 335.4: 90-110%; %RPD <20% SW846 9012B: in-house, updated annually; DoD projects per QSM 5.0	Determine and correct the problem, reanalyze samples and MS/MSD, or document why data are acceptable

IGNITABILITY

Prepared by: Svetlana Izosimova Date: 10/26/2018

Approved by: Jonathan Miller Date: 10/26/2018

Annual Review

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TITLE: IGNITABILITY

REFERENCES: SW846 1010A, SW846 Chapter 7, ASTM D93-80

REVISED SECTIONS: sec. 3 – Reporting Ignitability in soils as 1010A MOD in LIMS; throughout the document – all samples with flashpoint <200 F must be run in duplicates, requirement for a witness is removed.

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is used to determine the flash point of various liquid samples.

1.2 Method modification without stirring is used to test solid samples.

1.3 The sample is loaded in the cup of the tester. While the sample is being stirred and slowly heated at a constant rate, a flame is directed into the cup. The flash point is defined as the lowest temperature at which the vapor generated by the sample will ignite.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation: The preservation for this analysis is not specified. Analysis requires 75 ml of sample minimum.

2.2 Holding Time: The holding time for this analysis is not specified.

3.0 REPORTING and METHOD DETECTION LIMIT

Not Applicable. Samples, which did not ignite below 200^{o} F, reported as “Not Ignitable”.

Soil samples tested by Pensky-Martens should be reported as 1010A MOD. State certifications for the method follow individual state accreditation bodies’ conventions.

4.0 INTERFERENCES

Ambient pressure, sample non-homogeneity, drafts, and operator bias can affect flash point values.

5.0 APPARATUS and MAINTENANCE

5.1 Pensky-Martens Closed Flash Tester. Maintenance includes cleaning and inspection for cracks and dents. Performance verified at least semiannually through analysis of PT samples.

5.2 Thermometer, minimum range of 30 to 200°F. Must be verified against NIST-traceable thermometer as outlined in SOPs QA002 and QA004, current revisions.

5.3 Barometer. Checked annually against National Weather Service atmospheric pressure reading. Reading must be verified within 15 mm, otherwise barometer needs to be replaced.

5.4 Propane gas tank. Inspect gas lines and hoses for leaks and crimps.

6.0 REAGENTS

Certified flashpoint reference material

Low-pressure propane gas tank.

PROCEDURE

7.1 Thoroughly clean all parts of the stove assembly and its accessories before starting the test. Soot deposits will distort the shape of the flame and turn flame orange, therefore interfering with correct identification of flashpoint. Do not use metal objects for cleaning (needles, paper clips, etc) since they can distort the shape of the test flame nozzle.

7.2 Thoroughly clean and dry all parts of the cup and its accessories before starting the test.

7.3 Bring samples to starting temperature depending on the method used for analysis. See table in Section 7.8

7.4 Fill the cup with the sample to be tested to the level indicated by the fill mark.

7.5 Place the lid on the cup and set the cup in the stove. Be sure to properly engage the locking device.

7.6 Insert the thermometer probe. Make sure the thermometer inserted to appropriate depth so it does not jam the stirrer propeller or break the thermometer stem. Jammed propeller will destroy flexible shaft assembly.

7.7 Light the pilot flame and test flame and quickly adjust them to a diameter of 4 mm each. Size reference bead located on the lid of the apparatus. Rough regulator knob located on the top of the regulator housing – looks like a pole to the right of the apparatus. Fine flame adjustment performed using test flame handle – it is also a valve knob. Flame should be pale blue, nearly colorless. Large flame (either test or pilot) will deposit soot on the assembly and will produce false positive detections due to both orange glow and possible confusion of large flame with true vapor flash.

7.8 Heating rate and stir speed are dependent of the viscosity of the sample. Viscosity of the sample can be estimated based on the origin of the sample. More viscous samples require slower heating rate and more vigorous stirring. Procedures to be used are discussed below.

Procedure	**Method A**	**Method B**
Sample Origin	distillate fuels (diesel, kerosene, heating oil, turbine fuels), new lubricating oils, and other homogeneous petroleum liquids not included in the scope of Procedure B	residual fuel oils, cutback residua, used lubricating oils, mixtures of petroleum liquids with solids, petroleum liquids that tend to form a surface film under test conditions, or are petroleum liquids of such kinematic viscosity that they are not uniformly heated under the stirring and heating conditions of Procedure A.
Heating rate	9 to 11°F per minute	2 to 3°F per minute
Stir rate	90–120 rpm	250+/- rpm
Starting temperature	30 – 50F below expected flashpoint.	60+/-10F, or 20F below expected flashpoint, whichever is lower

7.9 Attach the flexible shaft to appropriate head and turn on the stirrer unit. The stirrer should direct sample downward. Make sure the flexible shaft is not twisted, kinked, mangled, etc. and the stirrer is not impeded by thermometer probe.

7.10 Apply the test flame and test for ignitability as described below.

NOTE: All samples that show a flash point under 200°F must have the results verified by analysis in duplicate. Both results should be recorded on the data sheet.

Apply the test flame at each temperature that is a multiple of 2°F (or every 1°C) up to a temperature of 200°F.

Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is lowered into the vapor space of the cup in 0.5 sec, left in its lowered position for 1 sec., and quickly raised to its high position.

NOTE: Do not stir the sample while applying the test flame.

7.11 Record the starting temperature for each sample.

7.12 Record as the flash point, the temperature at the time the test flame application causes a distinct flash in the interior of the cup. If the flash point is greater than 200°F, then record the results as greater than the final temperature tested (200°F).

NOTE: Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame. The actual flash will have occurred when a large flame propagates itself over the surface of the sample.

8.0 CALCULATIONS

8.1 Record the temperature read in degrees Fahrenheit in the ignitability logbook. The calculation of degrees C to degrees F is as follows:

9/5 (C) + 32 = F

8.2 Correct for barometric pressure using the following equation:

Corrected Results = F + (0.06*(760-(P*760mmHg/1013mbarHg)))

Where: F = observed temperature
P = barometric pressure in mb Hg

8.3 When pressure is in mmHg, the equation simplifies to:

Corrected Results = F + (0.06*(760-P))

9.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. While the concept of Blank Spike and Matrix Spike is not applicable to ignitability test, Matrix Duplicate is used to assess method precision.

Matrix Duplicate %RPD is compared to method defined control limits. Control limits are stored in the LIMS.

10.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL QC

Upon certain circumstances data can be reported from batches with QC non-conformances. Such samples are to be qualified accordingly.

For example, %RPD between Matrix Duplicates is outside acceptance limit, the department supervisor shall review the data and determine what further corrective action is best for the batch. That may include reanalyzing the samples, reprepping and/or reanalyzing the samples, or qualifying the results as estimated. *This must be approved by the department supervisor.* If there is insufficient sample to reanalyze, the appropriate footnote and qualifiers should be added to the results. *This must be approved by the department supervisor*

11.0 DOCUMENTATION REQUIREMENTS

All data must be recorded on appropriate bench sheets, which show the results for all quality control and samples. All work must be dated and signed by the analyst. Any changes should be marked through with a single line and should be initialed and dated. Any unusual characteristics of the samples should be noted in the comment section. Data must be signed by analyst and supervisor.

12.0 CALIBRATION AND STANDARDIZATION

12.1 Thermometer calibration. The thermometers used for this test must be calibrated against an NIST certified thermometer in the temperature range of use as specified in SOP QA002, current revision. The calibration must be documented, and any correction factor applied when used. If a correction factor is used, the raw data must show the uncorrected and the corrected temperatures, the correction factor, and the date the thermometer was calibrated.

12.2 Certified Reference Material (CRM) is accompanied by Certificate of Analysis (CoA). CRM with various flash point values are commercially available. CoA is examined for accuracy and completeness of the information. For further details on standard traceability refer to SOP QA017

13.0 QC REQUIREMENTS

13.1 CRM check standard. An aliquot of CRM must be analyzed daily or with each batch of samples. The flash point of CRM is lot-dependent, recorded on CoA and uncertainty has been established at 5% of target temperature or as indicated on CoA

13.2 Alternate Certified Flash point standard should be analyzed annually.

13.3 Duplicate. A duplicate must be analyzed for every batch of 20 samples, or on analysis day, whichever is more frequent. The results obtained must be within 4F of the original result for method A and within 9F for Method B

13.4 All samples with an ignitability of less than or equal to 200°F must have results verified by analysis in duplicate. Both the original and the verification result must be recorded.. Apparatus cup must be cleaned with water, rinsed with Acetone and flame placed in the cup to remove any Acetone residue.

14.0 SAFETY

The analyst should follow normal safety procedures as outlined in the SGS Accutest Health and Safety Program. All work should be done in a hood and the analyst should wear a lab coat and safety glasses for all analytical work. Gloves and safety glasses should be worn at all times when handling samples.

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

15.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in Section 15.2.

15.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM 108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

16.0 DEFINITIONS

16.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours whichever comes first.

16.2 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

16.3 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

16.4 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

Table 1. QC Criteria

Quality Control	Frequency	Acceptance Criteria	Corrective Action
Certified Reference Material	Daily	+/-5% of declared value or as indicated on Certificate of Analysis	Determine and correct cause of the poor performance
Sample Duplicate	One per batch	Repeatability within 4F or 9F depending on method A or B being used	Determine and correct cause of the poor repeatability

PAINT FILTER LIQUIDS TEST

Prepared by: Lovelie Metzgar Date: 08/08/2016

Approved by: Luis Jimenez Date: 08/08/2016

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TITLE: PAINT FILTER LIQUIDS TEST

REFERENCES: SW846 9095B; 40 CFR, Parts 264 and 265

REVISED SECTIONS: corrected DUP frequency and added editorial for out-of-control QC (Sec. 9.0); added QC frequency to Table 1; re-branded to SGS Accutest throughout document

REPORTING LIMIT: No free liquids, or <0.5 ml/100g of sample

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is applicable to wastes, soils, or sludge. The purpose of the method is to determine if any free liquids are present in a given sample.

1.2 A specified amount of solid is placed in a paint filter. If any part of the sample passes through the filter within the 5 minute test period, the sample is considered to contain free liquids.

2.0 SAMPLE COLLECTION, PRESERVATION AND HOLDING TIME

2.1 Preservation

Samples collected into 8-oz glass jars with PTFE lined lids. No preservation is required for this test.

2.2 Holding Time

No hold time is given for this test.

3.0 REPORTING and METHOD DETECTION LIMITS

3.1 Reporting Limit. The reporting limit for this method has been established at “no free liquids”.

3.2 No MDL study possible.

4.0 INTERFERENCES

4.1 Alkaline wastes can cause the filter media to separate from the filter cone. If the sample is not disturbed, this seldom causes a problem.

4.2 Temperature can affect the results, if it is done below the freezing point of any liquid in the sample. The test must be performed above the freezing point and while not required, may exceed room temperature.

5.0 APPARATUS

The items needed for the paint filter liquids test is listed below.

5.1 Conical Paint Filter, mesh number 60 (fine meshed size). Available at local paint stores.

5.2 Support Funnel. If the paint filter, with the waste, cannot sustain its weight on the ring standard, then a funnel may be used. The funnel should either be fluted or have a mouth large enough to allow at least 1 inch of the filter mesh to protrude.

5.3 Class A graduated cylinder, 100 ml

5.4 Ring stand and ring

5.5 Laboratory timer

5.6 Top-loading balance, capable or weighing to 0.01 g. The calibration of the analytical balance should be verified each day before use with Class 1 weights. Calibrated and serviced annually by outside contractor.

5.7 Class 1 weights

Maintenance consists of checking glassware for cracks and breaks. Balances are serviced annually by a certified contractor, and any time performance changes warrant service call.

6.0 REAGENTS

No reagents are required for this test.

7.0 PROCEDURE

Below is the procedure to be followed for the analysis of samples for the paint filter liquids test.

7.1 Place a paint filter in a funnel and place the funnel in the ring stand. Place a graduated cylinder beneath the funnel.

7.2 Measure out 100 ml of liquid or 100 g of solid sample. Larger sample sizes may be used, but it should be analyzed in 100 ml or 100 g aliquots. A volumetrically measured sampled should not have any major air spaces or voids. Pour the sample into the paint filter. All of the sample must fit inside the filter.

7.2.1 Settling the sample into the filter may be done by lightly tapping the filter.

7.2.2 Materials such as sorbent pads or pillows, which do not conform to the shape of the filter, should be cut into small pieces and poured into the filter. Size reduction should be done with scissors, shears or knife to preserve as much of the original integrity of the sample as possible. Particle size should be smaller than 1 cm. Grinding the sample should be avoided as this may produce “fines particles” which would not normally be present.

7.2.3 For brittle material, like clays, silica gel and some polymers, light crushing may be used to reduce particle size when cutting the material is not practical.

7.3 Allow the sample to drain for a period of 5 minutes.

7.4 If any portion of the sample collects in the cylinder during the 5-minute period, then the sample is considered to have free liquid. Measure the liquid that is collected in the graduated cylinder. If there is nothing in the cylinder, report the sample “contains no free liquids”. If there is liquid in the cylinder, report the mls of liquid per the starting weight or volume of sample and add a footnote that states “contains free liquids”.

7.4.1 Each sample must be reported with a footnote “contains no free liquid” or “contains free liquid” depending on the results. These comments are included into LIMS standard comment list.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. While the concept of Method Blank, Blank Spike and Matrix Spike is not applicable to Paint Filter test, Matrix Duplicate is used to assess method precision.

Matrix Duplicate %RPD is compared to method defined control limits. Control limits are stored in the LIMS.

9.0 QC REQUIREMENTS

A duplicate should be prepared with each batch of 20 samples or less.

Contingencies for handling out-of-control QC.

Upon certain circumstances data can be reported from batches with QC non-conformances. Such samples are to be qualified accordingly.

For example, %RPD between Matrix Duplicates is outside acceptance limit, the department supervisor shall review the data and determine what further corrective action is best for the batch. That may include reanalyzing the samples, reprepping and/or reanalyzing the samples, or qualifying the results as estimated. *This must be approved by the department supervisor.* If there is insufficient sample to reanalyze, the appropriate footnote and qualifiers should be added to the results. *This must be approved by the department supervisor*

10.0 DOCUMENTATION REQUIREMENTS

All data must be recorded on the Paint Filter logbook, which show results for all quality control and samples. All work must be dated and signed by the analyst and reviewed by supervisor. Any changes should be marked through with a single line and initialed and dated

11.0 SAFETY

The analyst should follow normal safety procedures as outlined in the SGS Accutest Health and Safety Program. A lab coat and safety glasses should be worn at all times when working in the lab. Gloves should be worn when handling samples.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in Section 12.2.

12.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples.

13.0 DEFINITIONS

13.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours whichever comes first.

13.2 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

13.3 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

13.4 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

Table 1 QC Criteria

Quality Control	Frequency	Acceptance Criteria	Corrective Action
Sample Duplicate	One per batch	20%	Determine and correct cause of the poor reproducibility

pH BY ELECTRODE

Prepared by: Svetlana Izosimova Date: 05/19/2020

Approved by: Dave Metzgar Date: 05/20/2020

Annual Review

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TITLE: pH BY ELECTRODE

REFERENCES: SM4500H B-2000 editorial revision 2011, SW846 9045D, SW 846 9040C, DoD QSM 5-Series, TNI Standards, current revision 2016.

REVISED SECTIONS: Updated references; Removed Accutest throughout the document and replaced with Orlando; sec. 5 – added separate ATC probe and electrode with temperature compensation.

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method measures the pH of drinking, surface, and saline waters, domestic and industrial wastes, multiphase wastes, soils, sludges, and non-aqueous liquids. This method is based on SM4500H B-2000 editorial revision 2011, SW846 9040C, and SW846 method 9045D.

1.2 The pH is determined electrometrically using a combination electrode or a glass electrode with a reference potential. The pH meter is calibrated using a series of standards of known pH. For solids, sludges, and non-aqueous liquids equal amounts of the sample and DI water are mixed, and the pH of the solution is measured.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

Water samples are subject to changes when exposed to the atmosphere, therefore the sample containers should be filled completely and sealed tightly.
No special preservation is required for soil pH samples.

2.2 Holding Time

Samples should be analyzed as soon as possible, preferably in the field within 15 minutes of sample collection; or as soon as they are received by the laboratory.
Note: According to 40 CFR, part 136 and SW-846 methods, samples cannot be reported for compliance if analysis conducted after 15 min hold time has expired.

3.0 REPORTING and METHOD DETECTION LIMITS

Not applicable. Report pH values to 0.1 pH unit.

4.0 INTERFERENCES

4.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of greater than 10, the measured pH may be incorrectly low. For samples with a true pH of less than 1, the measured pH may be incorrectly high.

4.2 In addition, temperature fluctuations and dirty electrodes can cause measurement errors. (See electrode cleaning procedure below – sec. 7.1.) Monitor calibration slope as an indicator of electrode condition.

5.0 APPARATUS

The following items are needed for the analysis of pH:

5.1 pH meter with means for temperature compensation, Thermo Scientific Orion Star A Series, or equivalent

5.2 pH electrode with separate ATC probe or pH electrode combined with temperature sensor for automatic compensation

5.3 Graduated plastic beakers

5.4 Stir plate

5.5 Magnetic stir bars

6.0 REAGENTS

All reagents listed below must be made from reagent grade chemicals.

6.1 De-ionized water. This water must be monitored daily before use.

6.2 Buffer solution at pH 4, pH 7 and pH 10. Commercially available solutions that have been validated by comparison to NIST standards are recommended for routine use. All buffers must be labeled on receipt and after opening. Buffer solutions must be refreshed daily.

6.3 Additional buffers with very low pH or very high pH may also be purchased, so the full pH range of the samples can be bracketed by standards. Liquid buffers at the extremes of the pH range are typically unstable and have a short shelf life. Buffers are available in pill form at pH 2 and pH 12 and have longer shelf life. These buffers are for daily use only, and must be discarded at the end of the day.

  6.3.1 Samples outside normal calibration ranges are to be segregated into separate batches and analyzed against calibrations including both isopotential point and pH buffer bracketing the sample – i.e. 2, 4, 7 or 12, 10, 7. Use different pH meter if necessary.

7.0 PROCEDURE

Below is detailed description of the procedure that must be followed for the determination of pH. Properly document each step of the analysis.

7.1 Make sure that the pH electrode is clean. If the electrode is coated with oil or grease, then it must be washed with a 50% water-acetone solution and then rinse it well with DI water. Do not soak the electrode in the acetone solution. Place the electrode in a beaker containing pH 7 buffer and let the electrode soak for approximately 2 hours before using.

7.2 Check the pH meter daily calibration log. If the meter has been calibrated earlier in the day, proceed to step 7.5

7.3 Connect the pH electrode to the pH meter. Check that the temperature probe is plugged into the meter. Calibrate the meter using three points, pH 4, pH 7, and pH 10, stirring gently. Begin calibration with isopotential point (pH 7, see SM4500H+B, sec. 2a). Record calibration data in the pH meter daily calibration log. **NOTE I:** For details on specific calibration procedure, see the instruction manual for the meter being used.

7.4 After the calibration is complete, analyze the 3 buffer solutions to ensure that an accurate calibration was obtained. Record the results on the pH worksheet. Readings must be within 0.05 pH units of the buffer solution’s true value.

7.5 Check the average slope of the calibration and verify it is between manufacturer’s specifications. Refer to the manufacturer’s manual to determine appropriate slope. If the slope does not meet these criteria, follow the steps for cleaning the probe or replace the probe and recalibrate. Record the average slope in the pH analysis log.

7.6 For water samples and aqueous wastes, proceed to step 7.9. For soils and non-aqueous wastes continue to step 7.7

7.7 Weigh out approximately 20 grams of each sample to be analyzed into a clearly labeled beaker. Record the sample weight to the nearest 0.1 gram on the analysis log. Using Class A cylinder, add 20 ml of DI water to each sample. Record the volume added to each sample in the analysis log.

Note: If the sample forms a sludge that cannot be stirred, then more water may need to be added. Add additional water in 10 ml increments until the sample can be stirred. Record the total volume of water added in the analysis log and clearly mark any sample which required more than 20 ml of water to be added. A footnote must be added to those samples indicating the modification of this procedure.

7.8 Place the sample on a stir plate and stir for 5 minutes. Then let the sample settle for 1 hour or until most of the suspended matter has settled out of solution. Decant the supernatant into a clean beaker. Alternatively, filter or centrifuge the sample to separate the aqueous phase for measurement.

Note: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned (Sec. 7.1) if it becomes coated with oily material.

7.9 Samples should be at or close to the same temperature as the buffers. Bring the samples to room temperature. Place about 50 mL of the sample in a clean glass or plastic beaker. This sample volume does not have to be measured with Class A glassware, as long as electrode is sufficiently submerged, with room for stir bar to move freely.

7.10 Between samples and buffers, rinse the pH and ATC probes with DI water and blot dry.

7.11 Place the sample on the stir plate and stir gently to minimize CO2 entrapment. Rinse electrode with the portion of sample to be measured. Lower the pH electrode into the solution and record the pH. Also record the temperature of the sample.

7.12 After every 10 samples have been analyzed, reanalyze the buffer solutions and record the results on the pH worksheet. 2 buffers of known value must bracket the pH of the samples.

7.12.1 Samples may need to be bracketed by additional buffers outside the calibration range of 4 to 10. Reading of these extreme pH buffers is allowed to be within 0.2 pH unit of true value.

7.13 **Maintenance** generally is limited to the procedure found in 7.1. If more advanced maintenance is needed, consult owner's manual.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. While the concept of Method Blank, Blank Spike and Matrix Spike is not applicable to pH measurement, Matrix Duplicate is used to assess method precision.

Matrix Duplicate precision is compared to method defined control limits.

9.0 QUALITY CONTROL

Below are the quality control requirements for pH analyses on water samples. Refer to the SGS - Orlando quality control manual for additional information on quality control calculations and requirements.

9.1 A new calibration must be performed and documented each day that analyses are done. A pH 4, pH 7 and pH 10 solution should be analyzed immediately after the calibration is completed. The buffer checks should be within 0.05 pH units of the true values.

9.2 A calibration check standard should be analyzed after every 10 samples and after the last sample analyzed in the batch. The check standard should be within 0.05 pH units of the true value of the check standard solution. Also see 7.12.1.

9.3 A duplicate analytical sample should be analyzed for every 20 samples or one per batch, whichever comes first. The readings should be within 0.1 pH of each other. See Table 1.

9.4 The pH of a sample must be bracketed by 2 buffers of known pH.

10.0 DOCUMENTATION REQUIREMENTS

All data must be recorded in the pH logbook which shows the results for all quality control and samples. For soils and non-aqueous sludges and liquids, circle SW 846 9045D. For waters and aqueous sludges and wastes, circle SW 846 9040C and SM 4500H+B.
In addition, the pH meter that was used and the buffer solutions used should be documented. All work must be dated and signed by the analyst. Any changes should be crossed out with a single line and should be initialed and dated.

11.0 SAFETY

The analyst should follow normal safety procedures as outlined in the SGS - Orlando Health and Safety Program. Safety glasses and lab coats should be worn at all times. Gloves should be worn when handling samples.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in Section 12.2.

12.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

13.0 DEFINITIONS

13.1 **Batch:** A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours whichever comes first.

13.2 **Continuing Calibration Verification (CCV):** A check standard used to verify instrument calibration throughout an analytical run. CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

13.3 **Holding Time:** The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

13.4 **Sample Duplicate (DUP**): A replicate sample which is used to document the precision of a method in a given sample matrix.

13.5 **Preservation:** Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

Table 1 QC Criteria

Quality Control	Frequency	Acceptance Criteria	Corrective Action
Buffer reading	every 10 samples	Buffers within 4-10 calibration range ±0.05 pH units, buffers outside 4-10 calibration range within ±0.2 pH units	Clean and inspect electrode and electrical connections, recalibrate
Sample Duplicate	5% of matrix	± 0.1 pH units	Determine and correct cause of the poor reproducibility

SULFIDE

Prepared by: Jordan Cuoco Date: 09/15/2021

Approved by: Svetlana Izosimova Date: 11/11/2021

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TITLE: SULFIDE

REFERENCES: SM4500S F-2000 editorial revision 2011, SM 4020-2014, editorial revision 2020, SM1020B-2014

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is used as a measure of sulfides and is applicable to all water and wastewater. A modification of this method is used to determine water-soluble sulfides in soil samples. The iodometric titration is based on SM4500S F-2000 editorial revision 2011.

1.2 Excess iodine is added to the sample, which may have been preserved with zinc acetate and sodium hydroxide to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur when the sample is acidified with HCl. The excess iodine is back titrated with sodium thiosulfate.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

Water samples should be preserved with Zinc Acetate and Sodium Hydroxide to pH $\geq$ 9 and collected in three 250-ml bottles. Water and soil samples should be kept under refrigeration at 4°C until they are analyzed.

2.2 Holding Time

Aqueous samples should be analyzed as soon as possible, but definitely within 7 days of the date of collection. Per SM4500S-2000 editorial revision 2011, sample sediments and sludges should be analyzed within 2 weeks (1 month for frozen sample).

3.0 REPORTING LIMITS AND METHOD DETECTION LIMITS

3.1 The reporting limit for sulfide in water has been established at 1.0 mg/L and 20 mg/kg for soils.

Method Detection Limit. MDL concept is not applicable to titrimetric methods.

4.0 INTERFERENCES

Reduced sulfur compounds, which decompose in acid, such as sulfite and thiosulfate, may yield erratic results. Volatile iodine consuming substances will give high results. Samples should be taken with a minimum of aeration as oxidation may affect results.

5.0 APPPARATUS

The following items are needed for the analysis of samples following the method outlined below:

5.1 25 ml burette, class A

5.2 Graduated cylinders, class A

5.3 Volumetric flasks, Class A

5.4 Graduated beakers or flasks

5.5 Stir bars

5.6 Stir plates

5.7 Volumetric pipettes, class A

5.8 Analytical balance, verified daily with Class 1 weights. Calibrated and serviced annually by qualified contractor.

5.9 Top-loading balance, verified daily with Class 1 weights. Calibrated and serviced annually by qualified contractor.

5.10 Class 1 weights

5.11 Filtering apparatus with 934 AH Whatman glass fiber filters or equivalent

5.12 Long stem funnel

Maintenance of apparatus consists of checking glassware for cracks and breaks.

6.0 REAGENTS

Note: All chemicals listed below are ACS grade reagents unless otherwise specified. Commercially available standards and reagents should be used whenever possible. These standards and reagents must be accompanied by the Certificate of Analysis (CoA). CoA is examined for accuracy and completeness of the information, including verification of the concentration and purity. For further details on standard traceability refer to SOP QA017, current revision. DI water should be used whenever water is required. Make sure to properly label all reagents and record the reagent preparation in the reagent logbook.

6.1 Hydrochloric acid, HCl, 6N. Add 500 mL of concentrated HCl to approximately 500 mL of DI water and mix in a 1 L volumetric flask. Bring to final volume with DI water.

6.2 Standard iodine solution, 0.0250 N: Dissolve 20 to 25g of KI in a little DI water in a 1 liter volumetric. Using analytical balance, weigh 3.2g of iodine and add to the flask. Allow it to dissolve. Dilute to 1 liter and standardize against 0.025N Sodium Thiosulfate on every day of use. Discard after 3 months.

6.3 Sodium Thiosulfate solution, 0.025 N: This solution is commercially available. The solution is accompanied by Certificate of Analysis (CoA). CoA is examined for accuracy and completeness of the information, including verification of the titrant normality. Discard after 30 days from opening. For further details on standard traceability refer to SOP QA017, current revision.

6.4 Starch indicator solution: This solution is commercially available.

6.5 Zinc acetate solution: Dissolve 220 g of zinc acetate in 870 mL of DI water.

6.6 Sodium hydroxide, 6N: Weigh out 24g of sodium hydroxide and dilute to a final volume of 100 mL with DI water.

6.7 Sodium hydroxide, 1N: Dissolve 40 g of NaOH in 500 mL of DI water. Dilute to 1 liter with DI.

6.8 Sulfide Stock Solution, target value 537 mg/L: Weight approximately 1.005 +/- 0.0005g of $Na_2S\bullet 9H_2O$ on analytical balance and dissolve in approximately 245 mL DI water using volumetric flask. Adjust the pH to between 9 and 11 with 1N NaOH (6.7). Dilute to final volume of 250 ml with DI water. **NOTE:** Sodium sulfite nonahydrate is extremely hydroscopic. If crystals are large, wash briefly and blot with Kim wipes. This solution must be made weekly. Used for Blank Spike (BS / LFB / LCS) and Matrix Spikes.

6.9 Sulfide Stock Solution Verification: Volumetrically measure 5 mL of stock solution and dilute to 200 mL with DI water, quantitatively transfer into titration beaker, volumetrically add 10 mL of Iodine solution, 1 mL of 6N HCl and starch indicator and titrate slowly with the 0.025N Sodium Thiosulfate solution until the last bluish tint disappears (see also 7.6). Actual concentration of the titrated sulfide solution should be approximately 13.4 mg/L.

6.9.1 Calculate concentration of Stock as follows:

$C_{Stock} = C_{Verified} * 40$, where:

C_{Stock} - concentration of Stock (sec. 6.8)
$C_{Verified}$ – actual concentration of titrated sulfide solution (sec.6.9)

40 – dilution factor, 200 mL of final volume/5 mL of Sulfide Stock solution

6.9.2 Verify concentration of Stock every analytical day and use titrated value as actual concentration.

7.0 TITRATION PROCEDURE

7.1 Before starting on the samples, standardize the iodine solution using the following procedure.

7.1.1 Volumetrically pipette 5.0 mL of the iodine solution into the beaker. Then measure out 200 mL of DI water into the beaker. Place a stir bar in the solution and place the beaker on a stir plate. Add several drops of HCl, to bring the pH to less than 2 and then add several drops starch solution.
Note: The amount of HCl solution needed may vary with the strength of the solution. If you do not get a blue color with several drops of starch, first verify that the pH $\leq$ 2. If the pH is correct, then add addition starch solution until a blue color is obtained.

7.1.2 Fill a 25 -mL buret with 0.025 N sodium thiosulfate solution. Titrate the iodine solution with the sodium thiosulfate until the last blue color disappears. Record the volume of sodium thiosulfate used.

7.1.3 Calculate the normality of the iodine as shown below.

$$\text{N of iodine solution} = \frac{(\text{mL of Na thiosulfate}) * (0.025\ \text{N})}{(5.00\ \text{mL of iodine})}$$

7.2 For soil samples, weigh out approximately 25g on a top-loading balance and dilute to 250 mL with DI water. Record weight to nearest 0.01 g. Mix well. Let settle and filter through Whatman 934 AH filters until clear. Measure out 200 mL to use for analysis. Prepare a matrix spike, a matrix spike duplicate, a spike blank, and a method blank. For the Method Blank and Blank Spike, use 250 mL of DI water on 25 g of solid matrix. **NOTE:** The soil sample should be prepared immediately before titration.

7.3 For water samples that are **not preserved** with Zn Acetate/NaOH, mix the sample well and measure out 200 mL to use for analysis. Continue with step 7.5

7.4 For water samples that are **preserved** with Zn acetate/NaOH, mark the side of the sample bottle at the level of the sample meniscus.

7.5 Volumetrically pipette 3.0 mL of the iodine solution (6.2) into a flask and add 2 ml of 6N HCl and swirl to mix. Gently transfer the contents of the sample bottle to the flask (or the 200 mL from step 7.3) with a minimum of aeration under the surface of the iodine. Rinse the preserved sample bottle with sufficient DI to ensure a quantitative transfer to the flask. Add a stir bar and stir slowly. The solution should be yellow. If not, add additional iodine. Add a small amount of starch solution and the solution should turn blue.
Note: The amount of HCl solution needed may vary with the strength of the solution. If you do not get a blue color with several drops of starch, first verify that the pH is less than 2. If the pH is correct, then add addition starch solution until a blue color is obtained.

7.6 Titrate the sample slowly with the 0.025N sodium thiosulfate solution until the last bluish tint disappears. The solution is normally clear at the end point. Note: The sample should be gently, but continuously stirred during the titration.

7.7 For each batch of samples, set up quality control samples, consisting of a method blank, a spike blank (BS/LFB/LCS), a matrix spike and a matrix spike duplicate. The spike blank and the matrix spikes are spiked with 5.0 mL of sulfide Stock solution (6.8) when the sample is transferred to the flask. **NOTE:** For MS, MSD and LCS, use 10.0 mL of iodine, instead of 3.0 mL.

7.7.1. LCS/MS/MSD spike concentrations can be calculated as follows:

$$\text{Spike amount (mg/L)} = \frac{C_{\text{Verified Spike}} * 200}{\text{sample volume (mL)}}$$

7.8 For water samples that are preserved with Zn Acetate/NaOH, measure the volume used by filling the original bottle with water to the line marked on the side of the bottle then pour the water into a graduated cylinder. Record the volume.

7.9 If **interference is suspected**, the samples may be taken through a **pre-treatment** step as described below.

7.9.1 If the sample was not preserved, measure out 200 mL of sample. Add 0.30 mL of zinc acetate solution and 4 to 5 drops of 6N NaOH solution to bring the pH above 9. Mix gently. Let the precipitate settle for approximately 30 minutes. Continue with step 7.9.3

7.9.2 If the sample was preserved, verify the pH > 9 and mark the level of the meniscus on the side of the bottle. Additional Zn acetate may be added to ensure complete precipitation. Continue with step 7.9.3.

7.9.3 Filter the sample through glass fiber filter paper and then place the filter paper in a beaker with approximately 200 mL of DI water. Proceed with the titration starting at step 7.5.

8.0 CALCULATIONS

The calculations for both soils and waters are outlined below.

For water samples, the following calculations should be used.

$$\text{Sulfide in mg/l} = \frac{((V_iN_i) - (V_tN_t)) \times 16000}{\text{sample volume in mL}}$$

Where: V_i = volume of iodine solution in ml
N_i = normality of iodine solution
V_t = volume of sodium thiosulfate solution in ml
N_t = normality of sodium thiosulfate solution

For soil sample, the following calculation should be used.

$$\text{Sulfide in mg/kg} = \frac{((V_i N_i) - (V_t N_t)) \times 16000 \times V_f}{V_s \times (\%\text{solids}/100) \times W_i}$$

Where:
V_i = volume of iodine solution in ml
N_i = normality of iodine solution
V_t = volume of sodium thiosulfate solution in ml
N_t = normality of sodium thiosulfate solution
V_s = volume of sample titrated
V_f = final volume of sample after preparation
W_i = initial weight of sample prepared

Note: The Sulfide Calculation spreadsheet does all the required calculations. IMPORTANT: Use only verified, approved current version of internal software. When in doubt, consult QA Staff.

9.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS/LFB/LCS), matrix spikes (MS) and matrix spike duplicates (MSD). The MB and LCS are used to monitor overall method performance, while the samples, MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank Spike, Matrix Spike, and Matrix Spike Duplicates are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, Blank Spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

10.0 QUALITY CONTROL

10.1 A method blank is required at a minimum of one per batch. The concentration of sulfide in the MB must be < ½ RL.

10.2 A spike blank (LCS) is required at a minimum of one per batch. Spike Blank recovery must be within +/- 15% of true value.

10.3 A matrix spike is analyzed for every 10% of matrix. Matrix Spike recovery must be within LCS limits.

10.4 A matrix spike duplicate is analyzed for every 10% of matrix. Matrix Spike Duplicate RPD is not discussed in the reference method and must be within statistically generated control limits.

10.5 For QC criteria calculations refer to SOP QA042, current revision.

Contingencies for handling out-of-control QC. Upon certain circumstances data can be reported from batches with QC non-conformances. Such samples are to be qualified accordingly. Examples include:

- If the MB is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The negative sample results can be reported with qualification. If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination should be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. *This must be approved by the department supervisor.* Samples with hits <10 times contamination are reprepped and reanalyzed. If there is insufficient sample to reanalyze, or if the sample is re-analyzed beyond hold time, the appropriate footnote and qualifiers should be added to the results. *This must be approved by the department supervisor*
- Similarly, if the recovery of LCS is high and the associated sample is non-detect, the data may be reportable with qualification. If the recovery of LCS is below lower acceptance limit, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, reprepping and/or reanalyzing the samples, or qualifying the results as estimated. *This must be approved by the department supervisor.* If there is insufficient sample to reanalyze, or if the sample is re-analyzed beyond hold time, the appropriate footnote and qualifiers should be added to the results. *This must be approved by the department supervisor.*
- If the matrix spike recoveries are not within the established control limits, compare the recoveries to those of the LCS to assess method performance in clean QC matrix. Matrix spike recovery failures are not grounds for reanalysis but are an indication of the sample matrix effects

11.0 GLASSWARE CLEANING

All glassware should be washed with soap and tap water and then well rinsed with de-ionized water. Refer to SOP GN196, current revision.

12.0 DOCUMENTATION REQUIREMENTS

All data should be recorded in a sulfide analysis log. Any unusual characteristics of the samples should be noted in the comment section. In addition, all reagent information such as lot numbers, vendors, date made, etc. should be recorded in the reagent logbook. All sample ID’s and dilutions should be clearly labeled on the data.

13.0 SAFETY

The analyst should follow normal safety procedures as outlined in the SGS Accutest Health and Safety Program. Safety glasses and lab coats should be worn at all times in the lab. Gloves should be worn when handling samples.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in Section 14.2.

14.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM 108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

15.0 DEFINITIONS

15.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours whichever comes first.

15.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

15.3 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

15.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

15.5 Reagent Grade: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents, which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

15.6 Reagent Water: Water that has been generated by any process, which shall meet method specified requirements.

15.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

15.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

15.9 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

16.0 ADDITIONAL REFERENCES

SOPs GN196, QA005, QA006, QA042, QA043, QA017, SAM108
TNI Standards, 2016 revision
DoD QSM 5-series
40 CFR, Part 136, Tables IB and II
.

Table 1 QC Criteria

Quality Control	Frequency	Acceptance Criteria	Corrective Action
Method blank (MB)	One per batch	< 1/2RL	Stop the analysis, determine the source of contamination, reanalyze, or document why the data are acceptable.
Blank Spike (BS or LCS)	One per batch	within 15% of true value	Determine and correct the problem, reanalyze samples, if necessary, or document why data are acceptable.
MS/MSD	10% of matrix	Within LCS limits	Determine and correct the problem, or document why data are acceptable.
MSD	10% of matrix	Within LCS limits, %RPD within statistical in-house limits	Determine and correct cause of the poor reproducibility

APPENDIX OF SIGNIFICANT CHANGES

Revision Date	Revision Number	Affected Section(s)	Revision Description
09/15/2021	17	Entire Document	Added applicable SOP references
09/15/2021	17	References	Updated SM 4000-series revision years
09/15/2021	17	3.0	Removed MDL discussion as not applicable to titrimetric method
09/15/2021	17	6.0	Updated requirements for chemicals and added examination of CoA. Updated reagents recipes in accordance with real production demand. Added weight tolerances to reagent and standard preparations.
09/15/2021	17	15.0	Added Reagent Grade and Reagent Water to definitions
09/15/2021	17	16.0	Added Section for Additional references; Added TNI 2016 Standard, DoD QSM – 5 Series, CFR 40 Part 136 Table IB and II; Updated SOP references
09/15/2021	17	Table 1	Revised corrective action for MB
09/15/2021	17	Appendix of Significant Changes	Added
09/15/2021	08	SOP Acknowledgement Form	Added

SULFIDE

SOP Acknowledgement Form

I have read and understand this SOP. I will not knowingly deviate from this approved SOP without approval of the Department Supervisor, QA Officer, or Technical Manager. If I notice any discrepancies between this SOP and the routine procedure, I will notify the Department Supervisor so that either the SOP or procedure can be changed. Furthermore, I understand that this SOP is property of SGS North America Inc. – Orlando and may not be printed nor duplicated in any manner.

Internal SOPs referenced within this SOP: QA005, QA006, QA017, QA043, SAM108, current revisions

Print Name	Signature	Date

Print the SOP Acknowledgement Form, sign, and submit to the SGS Orlando QA department.

PERCENT SOLIDS

Prepared by: Lovelie Metzgar Date: 09/08/2015

Approved by: Luis Jimenez Date: 09/11/2015

Annual Review

Reviewed by: ______________________ Date: __________

Reviewed by: ______________________ Date: __________

Reviewed by: ______________________ Date: __________

Document Control

Issued to: QA Department Date: 09/11/2015

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TEST NAME: PERCENT SOLIDS

METHOD REFERENCE: SM2540G-1997 editorial revision 2011; SM2020-2010

DEPT: GN

REPORTING LIMIT: 2%

***Summary of Change**: added QA method reference; added apparatus detail and maintenance (Sec.5.0); Constant weight is clarified in sec. 6.7 and 6.8; provisions for larger sample size added to sec. 6.6; added use of E-doc application; added QC frequency to Table 1; listed additional references (Sec.15.0)*

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This procedure is applicable to solid matrix samples. Results of this test are used for other testing which requires calculation on dry weight basis.

1.2 An aliquot of a well-homogenized sample is weighed wet, dried at 104°±1C, and weighed when dry. The % solid is calculated from the data.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

The samples should be cooled to 4°C.

2.2 Holding Time

The samples should be analyzed within 28 days from the sampling date.

3.0 REPORTING AND METHOD DETECTION LIMITS

Reporting Limit. The reporting limit for this method has been established at 2%.

4.0 INTERFERENCE

Non-representative particulates such as leaves, sticks, and rocks should be excluded from the sample, if it is determined that their inclusion is not desired in the final results.

5.0 APPARATUS

5.1 Aluminum pans

5.2 Wooden tongue depressors

5.3 Balance: ADVPRO, verified daily with Class 1 weights.

5.4 Class 1 weights for daily verification

5.5 Drying oven: VWR Model#1326

5.6 Computer linked to balance and LIMS system.

Balances are serviced annually by a certified contractor, and any time performance changes warrant service call. For ovens' maintenance and troubleshooting consult operator's manual.

6.0 PRIMARY ANALYTICAL PROCEDURE - AUTO-SOLIDS

6.1 In the LIMS set up the work group and QC sample.

6.2 Go to the %solids.xls shortcut and on the spreadsheet click on the "Collect Data" button.

6.3 Enter Batch number (i.e. gn10352), press "ok".

6.4 Enter analyst's name and balance ID, enter user name and enter password. Enter "accufla" for the connect string, press "ok". When the system asks if the analyst wants to save the file as "GN number" of the batch, press "yes". Then press "ok" to save file.

6.5 The spreadsheet now has the sample numbers from the batch on it. Type in the sample jar's bottle number. Weigh the appropriately labeled aluminum pan and press the "print" button on the balance; the weight will appear in the spreadsheet. Repeat this step with all the empty pans for the "Tare weight".
Note: When weighing pans/samples, wait for the balance to stabilize as indicated in balance display before pressing the print button.

6.6 Carefully decant free aqueous phase, if present. Using a tongue depressor, homogenize the sample and weigh at least 5 grams of each soil sample into the appropriate pan. Using the tongue depressor, crush any large lumps in the aliquot to be weighed and dried. Weigh the full pans, pressing the "print" button to transfer the data to the spreadsheet. Save the file.

Note: For any samples where it is difficult to obtain a representative sample aliquot, increase the sample aliquot size to 25 to 50 g. Use larger size aluminum pans (Oil and Grease evaporation pans) for these samples.

6.7 Dry sample in oven at 104° C ± 1°C to a constant weight or over night. Remove from oven and cool. Samples being dried to constant weight should be placed in the oven for a minimum of one hour, allowed to cool, and then weighed. Repeat drying (1 h), cooling, weighing, and desiccating steps until weight change is less than 4% or 0.05 g, whichever is less.

6.8 Samples, which are soupy or sludgy in appearance, are to be dried overnight regardless of possible “rush” status.

6.9 Weigh the dried samples, pressing the “print” button to transfer the data to the spreadsheet. Print the spreadsheet and save the file in the “In_progress” folder and the “Review” folder.

7.0 BACK-UP PROCEDURE - MANUAL

7.1 Tare balance to zero (do not tare the aluminum pan).

7.2 Weigh aluminum pan (A) and record weight in the %solids logbook.

7.3 Carefully decant free aqueous phase, if present. Using a tongue depressor, homogenize the sample and add at least 5 grams of representative soil to be analyzed to the aluminum pan. Using the tongue depressor, crush any large lumps in the aliquot to be weighed and dried. The sample weight taken should be based on the apparent water content of the sample.

Note: The sample must be thoroughly stirred and free of rocks, sticks, etc. before weighing out the sample. See SOP QA034 on the homogenization of solid samples.

7.4 Document total weight (B) on the data sheet.

7.5 Dry sample in oven at 104° C ± 1°C to a constant weight or overnight. Samples being dried to constant weight should be placed in the oven for a minimum of one hour, allowed to cool, and then weighed. This cycle is to be repeated until constant weight is obtained.

7.6 Constant weight is defined as less than 0.05g change. Samples, which are soupy or sludgy in appearance, are to be dried overnight regardless of possible “rush” status.

7.7 Remove from oven and cool.

7.8 Obtain final weight of sample (C) + aluminum dish.

8.0 CALCULATIONS

Calculate the %solid by entering the data into the excel program designed for this procedure or by using the following equations:

8.1 Wet weight of soil in grams = (B) – (A)

Dry weight of soil in grams = (C) – (A)

$$\%\text{Solid} = \frac{(\text{Dry weight of soil})}{(\text{Wet weight of soil})} \times 100$$

8.2 Auto-Solids: %solids are calculated by the verified, approved spreadsheet using the above equations and by the LIMS when the data is transferred.

Note: The %solids E-Doc application does all the required calculations. IMPORTANT: Use only verified, approved current version of internal software. When in doubt, consult QA Staff.

9.0 QUALITY CONTROL

9.1 Verify analytical balance every day of use bracketing the range of samples against Class 1 weights. Compare with acceptance criteria written in balance calibration logbook. Do not proceed with analysis if the balance is not in compliance. For further details refer to SOP QA005, current revision.

9.2 A duplicate sample must be analyzed at a frequency of 10%.

Note: By drying samples overnight at 104°C ± 1°C it is presumed that the sample is dried completely. Therefore no re-drying is necessary. This shall be confirmed annually by re-drying 10 samples overnight and recording the weight.

10.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. While the concept of Blank Spike and Matrix Spike is not applicable to %solids test, Matrix Duplicate is used to assess method precision.

Matrix Duplicate %RPD is compared to method defined control limits. Control limits are stored in the LIMS.

11.0 DOCUMENTATION REQUIREMENTS

11.1 Oven temperature must be recorded at the start and end of the drying period.

11.2 Drying time must be recorded at the start and end of the drying period.

11.3 All data must be on the auto-solids spreadsheet (E-Doc application)

11.4 In case of using manual procedure as backup, all data must be recorded in the %solids logbook, and then transferred to the Excel spreadsheet designed for this procedure in order to calculate the %solids.

12.0 SAFETY

The analyst should follow normal safety procedures as outlined in the Laboratory Safety Manual. A lab coat and safety glasses should be worn for all lab work. Gloves should be worn when handling samples.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

13.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in Section 13.2.

13.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM 108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

14.0 DEFINITIONS

14.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours whichever comes first.

14.2 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

14.3 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

14.4 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

15.0 ADDITIONAL REFERENCES

TNI Standards, 2009 (current revision)
DoD QSM 5.0, 2013
40 CFR Part 136, Method Update Rule 2012

Table 1 QC Criteria

Quality Control	Frequency	Acceptance Criteria	Corrective Action
Sample Duplicate	10% of matrix	<5%RPD	Determine and correct cause of the poor reproducibility

COLD VAPOR ANALYSIS OF MERCURY FOR SOILS

Prepared by: David Metzgar II Date: 12/18/2020

Approved by: Svetlana Izosimova Date: 12/18/2020

Annual Review

Reviewed by: Svetlana Izosimova Date: 12/09/2021

Reviewed by: ______ Date: ______

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TITLE: COLD VAPOR ANALYSIS OF MERCURY FOR SOILS

REFERENCES: SW-846 7471B

INSTRUMENT SERIAL #: 2004 (HG4), 2019 (HG5)

WAVELENGTH: 253.7 nm

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is outlined in this SOP is based on SW846 method 7471B for soils. The types of samples that can be analyzed include soils, sediments, bottom deposits and sludge type materials. Analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW846 methods is not mandatory in response to Federal testing requirements.

1.2 The mercury is reduced to the elemental state and aerated from the solution in a closed system. The mercury vapor passes through a cell in the light path of an atomic spectrophotometer, where the absorbance is measured as a function of mercury concentration.

2.0 PRESERVATION AND BOTTLEWARE

All soils must be refrigerated at ≤ 6°C. All bottleware used by SGS - Orlando is tested for cleanliness prior to shipping to clients. Bottleware analysis results must be < ½ RL to be acceptable. Please refer to SOP SAM104 current revision for further instruction.

3.0 HOLDING TIME

All samples should be prepared and analyzed within 28 days of the date of collection.

4.0 REPORTING and METHOD DETECTION LIMIT

4.1 Reporting Limit. The reporting limit for this method has been established at 0.042mg/kg. Reporting limits (RL) are based on the extraction procedure. Reporting limits may vary depending on matrix complications and volumes. Solid matrices are reported on a dry weight basis unless otherwise noted in LIMS.

4.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria. Experimental MDLs must be determined annually for this method.

4.3 MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit annually. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level.

4.4 An MDL check standard will be analyzed at the time of the annual MDL study and on a quarterly basis. The concentration of the MDL check standard should be 2-3 times the statistical MDL. This is a qualitative check; therefore, the analyte needs to be detected only. If the analyte is not detected, the concentration of the MDL check standard must be increased until the analyte is detected. This then becomes the current MDL.

4.5 MDL's are generated for each matrix on both HG instruments. The higher of the two statistically calculated MDL's is entered into LIMS as the MDL. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

4.6 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

5.0 INTERFERENCES

Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations of sulfide as sodium sulfide as high as 20 mg/l do not interfere with mercury recoveries when following this method. Copper concentrations > 10 mg/l may also interfere with mercury recoveries. Samples that are high in chloride such as seawater, brine and industrial effluent may require as much as 12.5 ml of additional permanganate.

Note: When chloride concentrations are high, hydroxylamine sulfate and stannous sulfate should be used in place of corresponding chlorides. Finally, certain volatile organic materials will also absorb at this wavelength and can interfere. It can be determined if this type of interference is present by doing a preliminary run without reagents.

6.0 APPARATUS

6.1 A Leeman HYDRA AA II automated analyzer is used for all analysis. Currently there are two Hydra AA's in service at SGS - Orlando. Refer to the instrument manual for further details on this instrumentation.

6.2 Automatic repipettor(s).

6.3 Fisher Brand 0.45 micron (um) filter or equivalent. Filter lots are checked for cleanliness through the Method Blank process. All Method Blank analytical results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated filters must be re-filtered through acceptable filters.

6.4 Fisher Brand disposable 10 ml syringes or equivalent. Syringe lots are checked for cleanliness through the Method Blank process. All Method Blank results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated syringes must be re-filtered through acceptable syringes.

6.5 Environmental Express Hot Block or equivalent capable of maintaining a temperature of 90-95°C.

6.6 Environmental Express vessels or equivalent, 65ml capacity. Each Lot of digestion tubes comes with a Certificate of Analysis which demonstrates cleanliness as well as volume accuracy at the 50ml mark. Please refer to Digestion Tube Certificate Logbook for further information. Tube Lots are also checked through the Method Blank process. All Method Blank analytical results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Re-digestion is required for all samples prepared with the contaminated tube lot.

6.7 Class A volumetric pipette (s) and flask (s)

6.8 Thermometer(s), capable of measuring to at least 125°C, and checked against NIST traceable thermometers. Refer to SOP QA002, current revision for further information.

6.9 Fisherbrand wooden spatulas or equivalent.

6.10 Teflon boiling chips

6.11 Solid Standard Reference Material (SRM) as required per project/client specific requirements.

6.12 Top loader balance- capable of accurately weighing 0.01g. Refer to SOP QA005, current revision for balance calibration information.

6.13 Eppendorf Pipette (s) - Pipette (s) are checked daily for accuracy and to ensure they are in good working condition prior to use. Volumes are checked at 100% of maximum volume (nominal volume). Pipettes are checked within the metals department and results are stored electronically in the “Pipette Calibration Log”. Refer to SOP QA006, current revision for further information regarding pipette calibration. BIAS: mean must be within 2% of nominal volume. Precision: RSD must be $\leq$ 1% of nominal volume based on three replicates.

6.14 Data System

Microsoft Windows 7 Professional.
Instrument Software – HG4 - Leeman Labs Envoy 1.9 sp1.
Instrument Software – HG5 - Leeman Labs Envoy 2.0 sp0.

6.14.1 A computer system interfaced to the Leeman Hydraa II that allows for the continuous acquisition and storage of all data obtained throughout the duration of the analytical run sequence.

6.14.2 Data is archived to a backup server for long term storage.

7.0 REAGENTS

All chemicals listed below are trace metal grade unless otherwise specified. Refer to Acid Certificate of Analysis logbook for Certificates of Analysis and compliance with specifications of the grade listed. SGS - Orlando produces DI water to the specifications for the ASTM Type II standard designation based on the system manufacturer's performance specifications. The DI water is used exclusively for laboratory purposes. De-ionized (DI) water should be used whenever water is required. Refer to SOP QA037, current revision for more information regarding testing and monitoring. All standards and prepared reagents must be prepared every 6 months or before stock standard expiration date, whichever comes first, except as noted elsewhere in this SOP. Refer to Metals Standard Prep Logbook for further information. Some of the information included in the logbook is as follows: standard name, elements in mix, manufacturer, lot number, parent expiration date, acid matrix, stock concentration, volume of standard added, total volume, final prepared concentration, prep date, initials, MET number, and prepared standard expiration date.

7.1 Hydrochloric acid, concentrated, trace metal grade.

7.2 Nitric acid, concentrated, trace metal grade.

7.3 Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated Hydrochloric acid to one volume of concentrated Nitric acid. Be sure to make enough for the sample batch being prepared.

Caution: handle with care.

7.4 Stannous chloride, reagent grade. To 400 ml of DI water, add 50 ml of conc. Hydrochloric acid, and 50 g of stannous chloride. Dilute to 500 ml with DI water. Make sure stannous chloride solution is completely dissolved prior to use. Stannous sulfate may be used in place of stannous chloride. Stannous chloride is prepared daily. Lot number is recorded in Hg Digestion logbook.

Note: If line clogging occurs when using the automated system, a less concentrated stannous chloride solution may be used.

7.5 Sodium chloride-hydroxylamine hydrochloride, reagent grade. Add 120 g of sodium chloride and 120 g of hydroxylamine hydrochloride to 1 liter of DI water and mix well. Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.6 Potassium permanganate, reagent grade. Add 50 g of potassium permanganate to 1 liter of DI water and mix well.

Caution: Potassium permanganate is a strong oxidizing agent. Handle with care.

7.7 2 percent HCL Carrier Solution. Add 40ml concentrated HCL to a volumetric flask and dilute to 2 liters with DI water. 2 percent HCL carrier solution is prepared daily. Lot number is recorded in Hg Digestion logbook.

7.8 Mercury working standards. Mercury standard solutions are made from a purchased stock solution of 1000 ppm mercury.

7.8.1 10 ppm Hg solution. (Used to prepare 100 ppb Hg solution) Using a 10 ml class A volumetric pipette, add 10 ml of 1000 ppm stock to a 1.00 liter class A volumetric flask containing approximately 750 ml of DI water and 10 ml of concentrated nitric acid. Dilute to volume with DI water and mix well. This 10ppm standard must be prepared every six months.

7.8.2 100 ppb Hg solution. (Used to prepare calibration curve, CCV, and CRI). Using a 10 ml class A volumetric pipette, add 10 ml of 10 ppm Hg solution to a 1.00 liter class A volumetric flask containing approximately 750 ml of DI water and 10 ml of concentrated nitric acid. Dilute to volume with DI water and mix well. This 100ppb standard must be prepared every month.

7.8.3 Second source working solutions are prepared at the same concentrations as the calibration standards listed above in sections 7.9.1 and 7.9.2 except they are made from a second source. Used to prepare ICV, BSP, MS, and MSD.

7.8.4 Daily working standards used in section 8.0 are prepared and digested daily using 100ppb standard solutions.

8.0 SOIL DIGESTION AND ANALYSIS PROCEDURE

Below is a step-by-step procedure for the digestion and analysis of soil samples for mercury. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements.

8.1 Make up the standard curve as shown below. Using a wooden spatula, weigh out 0.6 g of Teflon chips on a top loader balance and place in the digestion vessel.

Clearly label each digestion vessel with a standard ID.

ml of 100 ppb Hg solution	^{A} ml of DI water	Total µg/L of Hg
0.0	50	0.0
0.10	50	0.20
0.5	50	1.0
1.5	50	3.0

2.5	50	5.0
3.0	50	6.0

[A] Denotes final digested volume

8.2 Make up the quality control samples as shown below. Using a wooden spatula, weigh out 0.6 g of Teflon chips on a top loader balance and place in the digestion vessel. Make sure to clearly label each digestion vessel.

Sample ID	ml of 100 ppb Hg solution	[B] ml of DI water	Total µg/L of Hg
*Blank Spike	1.5	50	3.0
CCV	1.5	50	3.0
Low Check (CRI)	0.10	50	0.20
Method Blank	0.0	50	0.0
*ICV	1.5	50	3.0

[B] Denotes final digested volume.

Sample ID	ml of 100 ppb Hg Solution	[C]ml of DI water	g of sample	Total µg/L of Hg
*Matrix Spike	1.5	50	0.6	3.0 +sample
*Matrix Spike Dup	1.5	50	0.6	3.0 +sample
Duplicate		50	0.6	sample

*Use second source 100 ppb solution.
[C] Denotes final digested volume

8.3 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. The sample container must have enough room to move the matrix around with the wooden spatula. Mix the sample thoroughly using the wooden spatula. Make certain the entire sample is mixed well. The wooden spatula must reach the bottom of the original container and be able to be moved through the entire sample to ensure proper mixing. If the sample is packed tightly or matrix is dense and can not be efficiently moved around in the original jar, a secondary container, such as a porcelain dish must be used. Remove sample from original container and place in the clean secondary container. While in the secondary container thoroughly mix sample around until appearing uniform in consistency. Upon completion the sample is re-packed into the original container. Refer to SOP QA034 for more information on sample homogenization. Using a wooden spatula, weigh out approximately 0.6 g on a top loader balance of a homogeneous sample and place in the digestion vessel.

8.4 Preheat the Hot Block to 90-95 degree C.

8.5 To all samples, QC and standards add the following reagents:

- 2.5 ml of DI water

- 2.5 ml of aqua regia

8.6 Swirl the samples well after each addition of reagent.

8.7 Place the vessels in the Hot Block and digest for 2 minutes at 90-95 degree C.

8.8 Cool the samples and then add 25ml of DI water and 7.5 ml of potassium permanganate. More potassium permanganate solution may be required for some samples. Enough should be added so that the purple color persists for at least 15 minutes. Ensure that equal amounts of potassium permanganate are added to the standards and blanks.

8.9 Heat the samples for 30 minutes at 90-95 degree C. Then remove and cool. Allow samples to stand quietly without manual agitation.

8.10 Begin setting up the Leeman HYDRA AA II mercury analyzer following the steps outlined below. Further details are available in the instrument manual.

8.11 Turn the Argon gas on. Check that the vent line is connected to the exhaust hood.

8.12 Inspect all pump tubing and replace if necessary. Put the tubing on the cassettes and attach to the pump head, making sure the cassette adjusters are properly adjusted to provide a smooth flow of sample and reagents. Place the fresh stannous chloride solution in the bottle. Fill the rinse bottle with 2 percent HCL carrier solution. Connect stannous chloride and HCL rinse lines.

8.13 Double click on the Envoy icon on the desktop. This will open the Envoy mercury analysis software. Click on the green and black arrow icon. This will start the pump, turn on the gas, and turn on the lamp.

8.14 Locate the “Sequence” tab at the bottom of the Envoy software page. Now click on “sequence” at the top of the page and click “new”. Type in the run sequence starting with the MB. After typing in the entire days run sequence click on the “Update” button. Click on “sequence” at the top of the page and choose “save”. Type in run sequence name as follows: Instrument-month-day-matrix-run number. (i.e. h40606s1).

8.15 Locate “Analysis” tab at bottom of Envoy page. Now click on “Analysis” at the top of the page and click “new”. Enter analysis dataset as follows: Instrument-month-day-matrix-run number (i.e. h40606s1).

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8.16 Add 3 ml of hydroxylamine hydrochloride solution to each standard and sample and swirl until the solution has been completely decolorized before analyzing. Bring samples to a final volume of 50mls using DI water. If the sample (s) contain particulate matter, it should be filtered (performed at the analytical bench), along with the method blank and blank spike through a 0.45 um syringe filter before analysis. Samples are now ready for analysis.

8.17 Calibration is performed by analyzing a series of 5 standards and a blank. All calibration curves must be determined from a linear calibration prepared in the normal manner using the established analytical procedure for the instrument. Refer to instrument manual for further detail. A correlation coefficient of ≥0.995 must be achieved, if not, analysis must be terminated, the problem corrected, and instrument re-calibrated. Calibration data is printed and included with each analytical data package. Click on the "Run sequence" icon. Instrument will start calibration. Once the calibration is complete the instrument will automatically accept the curve if the correlation coefficient ≥0.995. The instrument will then proceed to analyze the High standard, ICV, ICB, CRI, CCV, and CCB. The calibration curve and all initial QC are compared to check tables set up in the software. If any standard fails the set criteria it will be flagged on the screen as to alert the analyst. The instrument will continue the analysis of the run sequence if all QC criteria has been met.

8.18 After analysis has been completed flush the entire system with 1:1 HCL, then DI water, then allow to pump dry. Unclamp all tubing, turn off gas and lamp.

8.19 Raw data generation (PDF File)

Open the "PDF Creator" icon located on the desktop. Click on the green light, it will turn red. Go to the Envoy software. Click on the "Method" tab and then locate that days calibration curve. Click on print to PDF creator. Now go to the "Analysis" tab, click on "report" and "clear all." Click on "Load" and choose "Accutest" profile. Select all samples to be reported. Choose "report" as output. Next click on "Printer" and send to PDF creator. Leave the report title blank and click "OK". Now go back to the "PDF Creator" which should still be open on the desktop and click on "Document", "combine all." Click on the red light which should now turn green. Close the PDF creator. Go to the "Pdf shortcut" icon located on the desktop and rename .PDF to MAxxxx.pdf. Right click on MAxxxx.pdf, copy, then paste to the "LIMS Data" icon on the desktop. Open the pdf file (MAxxxx.pdf) and print to metals printer. This will generate the raw data that will be included in the run package. Now close the pdf file and archive.

8.20 LIMS data generation

Choose "CSV" as output under the "Analysis" tab. Type in analysis dataset (h40606s1), no extension. Go to the "Export" shortcut located on the desktop. Locate analysis dataset (h40606s1.csv), right click on file and open with wordpad. Change Blank, 0.2ppb, 1.0ppb, 3.0ppb, 5.0ppb, and 6.0ppb to STD1_2, STD2, STD3, STD4, STD5, and STD6. Remove all percent recoveries from the file and the save and close the file. Right click on the analysis dataset, copy/paste to "LIMS Data" icon on desktop. If the run contains any errors an "error report" will be generated to the metals printer. Correct any errors and re-send the file. Archive run sequence (.SEQ) and analysis dataset (.CSV) when done.

9.0 QC REQUIREMENTS

All QC calculations should be done as outlined in the method. QC limits are compiled by SGS - Orlando quarterly and should be used to determine if a given analysis is valid. Please refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements. Check with area supervisor or lab manager for any non-compliant quality control for further information.

9.1 Method Blank – An acceptable method blank or reagent blank must be analyzed with every batch of samples processed. The method blank must be less than one half the reporting limit. If the method blank is greater than one half the reporting limit, the samples associated with the contaminated blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-digesting and reanalyzing the samples, or qualifying the results with a “B” or “V” qualifier. All samples associated with an out of compliance method blank shall be qualified and footnoted in LIMS as well as the case narrative.

9.2 Initial Calibration Verification – An initial calibration verification (ICV) sample must be analyzed after the initial calibration has been performed. This sample must be prepared at or near the midpoint of the initial calibration from a reference material independent from the initial calibration solution. The results of the ICV must agree within 10 percent of the true value for the analysis to be valid. If the ICV fails, a new ICV or initial calibration must be performed and all samples must be reanalyzed with an acceptable ICV.

9.3 Continuing Calibration Verification –A Continuing Calibration Verification sample prepared at or near the midpoint of the initial calibration must be analyzed after every 10th sample and at the end of the analytical run. The results of the initial CCV analysis must be within 10 percent of the true value to be considered valid. All subsequent CCV’s must be within 20 percent of the true value to be considered valid. If the CCV fails, all samples analyzed after the first passing CCV must be reanalyzed.

9.4 Continuing Calibration Blank/Initial Calibration Blank – Analyze the Initial calibration blank solution at the beginning of each run and the continuing calibration blank after every tenth sample and at the end of the sample run. The ICB/CCB must be less than one half the reporting limit to be considered valid. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, or qualifying the results with a “B” or “V” qualifier, or raising the reporting limit to greater than 2 times the background concentration.

9.5 Low Level Check Standard (CRI) – A standard prepared at the low calibration concentration should be prepared and analyzed with each analytical run, at the beginning and end of each analytical run. The CRI should agree within 20 percent of the true value to be acceptable. If the initial CRI does not meet the acceptance criteria, the samples must be reanalyzed. If the closing CRI fails the criteria, the samples associated with the failing CRI shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the CRI or qualifying the results in LIMS.

9.6 Blank Spike – A blank spike (BSP) or Laboratory Control Spike (LCS) should be prepared using DI water spiked at the midpoint of the calibration curve. The blank spike must be within 20 percent of the true value for the analysis to be considered valid. If the blank spike exceeds the acceptance criteria, the samples must be re-digested and reanalyzed. A blank spike is required for every 20 field samples or for each analysis batch. Statistical control limits are generated for LCS’s for QA purposes only. Refer to section 15.0 of this SOP for further detail.

9.7 Duplicate – The laboratory must digest a duplicate for a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample must be assessed and must be $\leq$ 20 percent for sample results at or above the reporting limit. If the RPD is outside the 20 percent criteria the results must be qualified in LIMS. RPD's are also calculated in LIMS for sample results below the reporting limit. RPD's outside the 20 percent criteria are not considered failing and LIMS automatically footnotes these as "RPD acceptable due to low duplicate and sample concentrations."

9.8 Matrix Spike/Matrix Spike Duplicate/MSA – At least one Matrix Spike/Matrix Spike Duplicate pair must be prepared and analyzed with every 20 field samples. The MS/MSD recovery must agree within 20 percent of the true value. Relative standard deviation (RSD) should be $\leq$ 20 percent. If the results of the MS/MSD are outside the acceptance criteria, the data should be footnoted as possible matrix effect. In certain circumstances the Method of Standard Additions (MSA) may be needed by some project specific requirements. SGS - Orlando may perform an MSA when sample matrix is so complex that viscosity, surface tension, and components cannot be accurately matched with standards. If an MSA is not performed the results may be footnoted in LIMS. SGS - Orlando will use a single addition method as described in SW846-7000B.

9.9 When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted back into the calibration range and reanalyzed.

9.10 For each digestion batch of 20 samples, a serial dilution (performed at the analytical bench), a matrix spike (MS), a matrix spike duplicate (MSD), a duplicate (DUP), a blank spike (LCS), and a method blank should be prepared. Re-digestion is suggested for QC that does not meet the SGS - Orlando QC limits. The appropriate lab supervisor or lab manager will notify the analyst of samples that need re-digestion.

9.12 Serial Dilution - For one sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution should be prepared. For the serial dilution, a 1:5 dilution should be made on the sample. The results of the 1:5 dilution should agree within 10 percent of the true value as long as the sample and the dilution result are greater than 10 times the method detection limit and/or greater than 50 times the IDL. If the dilution is not within 10 percent then a footnote must be entered into LIMS.

10.0 DOCUMENTATION REQUIREMENTS

All digestion information should be documented in the Sample Digestion Logbook. The information required includes the sample identification (including the sample bottle number), the initial sample weight, and the final sample volume, the acids used (including lot number and manufacturer), the spiking solutions used, the digestion vessel lot number, the Teflon chips lot number, the observed temperature, corrected temperature, the thermometer ID, analyst's signature, the date of digestion, digestion start time, and digestion end time. The analyst should write additional information such as unusual sample characteristics and samples that need to be filtered (dissolved analysis) in the comment section. All raw data is printed to .PDF format and archived to a backup server for long term storage.

11.0 SAFETY

The analyst should follow normal safety procedures as outline in the SGS - Orlando Laboratory Safety Manual. Particular care should be observed in handling the strong acids and oxidizing agents. Safety glasses and lab coats should be worn at all times. Gloves should be worn when handling samples and hazardous chemicals.

12.0 CALCULATIONS

Below are the calculations, which should be used for soil samples. The concentration of the sample in µg should be obtained from the linear calibration curve.

$$\textbf{Final concentration in mg/kg} = \frac{\textbf{Concentration of sample in µg}}{\textbf{(Sample wt in g)(\%solids/100)}}$$

Matrix Spike and Matrix Spike Duplicate Recovery:

$$\frac{\textbf{(Spiked Sample Result - Sample Result)}}{\textbf{Amount Spiked}} \times 100 = \textbf{matrix spike recovery}$$

Matrix Duplicate/Matrix Spike Duplicate Relative Percent Difference:

$$\frac{(|\textbf{Sample Result - Duplicate Result}|) \times 100}{\textbf{(Sample Result + Duplicate Result)/2}} = \textbf{Duplicate RPD}$$

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

13.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids must be followed. All method users must be familiar with the waste management practices described in Section 13.2.

13.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

14.0 GENERIC DEFINITIONS

14.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours whichever comes first.

14.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

14.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. A CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

14.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

14.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

14.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

14.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the performance of a method in a given sample matrix.

14.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and performance of a method in a given sample matrix.

14.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

14.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

14.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

15.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Statistical control limits are stored in the LIMS for QA purposes only. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

16.0 GLASSWARE CLEANING

All glassware should be washed with soap and water, rinsed with 5 percent nitric acid solution, and then rinsed at least three times with DI water. Refer to SOP GN196, current revision for further information regarding glassware cleaning.

17.0 INSTRUMENT MAINTENANCE

Recommended periodic maintenance includes the items outlined below. All maintenance must be recorded in the instrument maintenance log.

17.1 Change the pump tubing weekly or as needed.

17.2 Clean the optical cell and lenses once per week or as needed.

17.3 Clean and lightly lubricate the auto sampler rails weekly or as needed.

17.4 Change the sampler tip as needed.

17.5 Inspect the liquid/gas separator, mixing coil, and all tubing connections once per week and replace as needed.

APPENDIX A

1.0 Application

Appendix A designed to supplement SOPs MET104.xx and MET105.xx for the preparation of soil samples for compliance with DoD and certain state-specific projects

2.0 Background

A theory of particulate sampling was developed by geologist Pierre Gy to improve the quality of data gathered in support of mineral exploration and mining. The MIS approach described herein is based upon Gy's theories and is applicable to environmental sampling at contaminated sites.

A large portion of sampling error is a result of compositional and distributional heterogeneity.

Compositional heterogeneity describes the variability of contaminant concentrations between the particles that make up the population in the sample. This type of heterogeneity results in fundamental error (FE).

Distributional heterogeneity occurs when particles are not randomly distributed across the population due to slight spatial variations. Spatial variability will be missed if all samples are collected from one place. This type of heterogeneity results in grouping and segregation error (GSE).

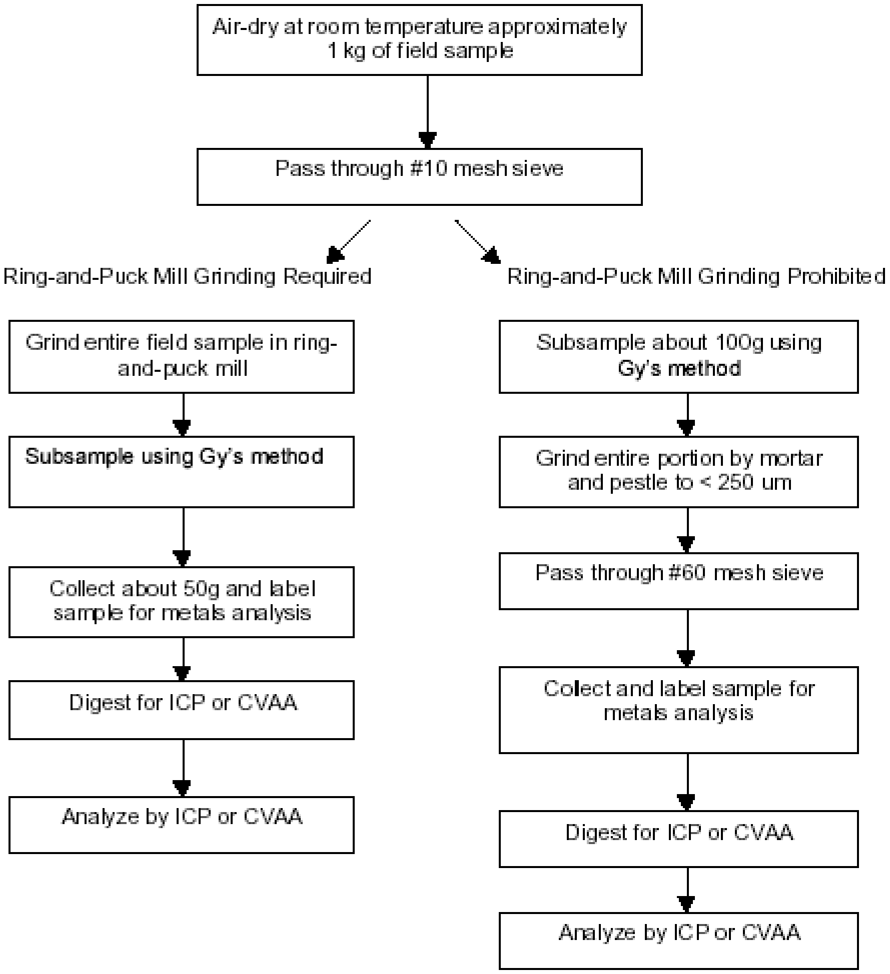
Gy found that fundamental error is directly proportionate to maximum particle size and inversely proportionate to sample size, therefore it is beneficial to collect and analyze a sample of sufficient size that consists of particulate matter where majority of contamination is present. In order to manage FE under 15%, particulate matter size must be under 2 mm and minimum sample mass above 30g.

To minimize GSE, it is imperative to collect sample increments randomly and in enough locations to capture the spatial variability, even within sample that already has been collected from the field.

3.0 Subsampling for Metals

Some projects require that metals analysis be performed on the multi incremental sample that was collected for 8330B. The technique used should be listed in the project QAPP or SOW. Consult the client if this information is not available.

See flow chart below for various subsampling techniques:



If Ring and Puck Mill grinding is required, then proceed with the grinding procedure listed in SOP OP046 for explosives. The metallic components from the Ring and Puck Mill may introduce chromium and iron into the sample.

After grinding, place a baking tray on the downdraft table. Transfer the entire sample to the tray. Shape the sample into an elongated pile with flattened top surface that it is approximately 1 cm thick. Using a rectangular scoop, collect multiple top-to-bottom cuts across the sample (see figure below). A minimum of 4 cuts should be made through each sample. Combine the cuts in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Transfer the samples to the metals department for analysis.

If Ring and Puck Mill grinding is not required then follow the procedure listed below.

Transfer the sample to a large ziplock bag after it has been air dried and sieved. Sample should be transferred over the downdraft tables to minimize dust contamination. Seal the bag and thoroughly mix the sample.

Place a baking tray on the downdraft table. Transfer the entire sample to the tray. Shape the sample into an elongated pile with flattened top surface that it is approximately 1 cm thick. Using a rectangular scoop, collect multiple top-to-bottom cuts across the sample (see figure below). A minimum of 4 cuts should be made through each sample. Combine the cuts in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Return the remaining sample to the ziplock bag or mixing bowl.

Grind each sample and MB to a particle size less than 250 um with a non-metallic mortar and pestle.

Place a baking tray on the downdraft table. Sieve each sample through a #60 sieve onto a tray.

Collect and label the samples. Transfer the samples to the metals department for analysis.

For digestion withdraw approximately 5 g of sieved material. If mortar-and-pestle grinding was specified per QAPjP, 1 g is sufficient. Follow digestion procedure outlined in the body of this SOP.

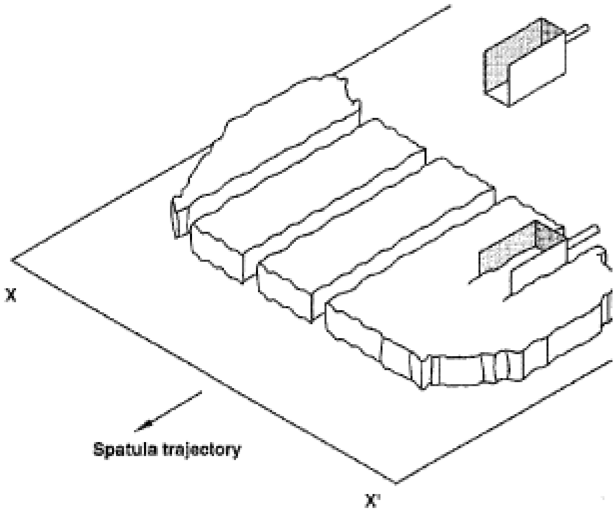


FIG. 1 Transversal Subsampling

REVISION HISTORY

Revision Date	Revision Number	Affected Section(s)	Revision Description
12/2020	13	"Revised Sections"	Added Revision History and removed Revised Sections. Sec. 7.0 removed reference to H2SO4.

COLD VAPOR ANALYSIS OF MERCURY FOR WATER SAMPLES

Prepared by: David Metzgar III Date: 12/18/2020

Approved by: Svetlana Izosimova Date: 12/18/2020

Annual Review

Reviewed by: Svetlana Izosimova Date: 12/09/2021

Reviewed by: ______ Date: ______

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Issued to: ______ Date: ______

Effective 7 days after "*" date

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TITLE: COLD VAPOR ANALYSIS OF MERCURY FOR WATER SAMPLES

REFERENCES: EPA 245.1 Rev.3, 1994, SW-846 7470A, WV 47CSR32

INSTRUMENT SERIAL #: 2004 (HG4), 2019 (HG5)

WAVELENGTH: 253.7 nm

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 The method outlined in this SOP is based on EPA method 245.1 and SW846 7470A for waters. The types of samples that can be analyzed include drinking, surface and saline waters, as well as domestic and industrial wastes.

1.2 The mercury is reduced to the elemental state and aerated from the solution in a closed system. The mercury vapor passes through a cell in the light path of an atomic spectrophotometer, where the absorbance is measured as a function of mercury concentration.

2.0 PRESERVATION AND BOTTLEWARE

All samples should be preserved with nitric acid to a pH of <2 at the time of collection. All sample pH are checked in sample receiving and within the metals department. Samples that are received with a pH >2 must be preserved to pH <2 and held for 24 hours prior to metals digestion to dissolve any metals that absorb to the container walls. Refer to SOP SAM101, current revision for further instruction. Final pH of TCLP extracts are checked and recorded in SGS - Orlando Extractions Department. Please refer to TCLP (1311) fluid determination logbook and SPLP (1312) fluid determination logbook for further information. TCLP extracts received from SGS - Orlando Extractions Department are prepared as soon as possible, no longer than 24 hours from time of receipt. If precipitation is observed during the sample preparation process the sample(s) are immediately re-prepped on dilution until no precipitation is observed. Samples received for dissolved metals analysis should be filtered and preserved to pH<2 as soon as possible and held for 24 hours prior to digestion. Refer to SGS - Orlando Sample Filtration Logbook for further information regarding sample filtration and preservation. All bottleware used by SGS - Orlando is tested for cleanliness prior to shipping to clients. Bottleware analysis results must be < ½ RL to be acceptable. Refer to SOP SAM104, current revision for further instruction.

3.0 HOLDING TIME AND STORAGE

All samples should be prepared and analyzed within 28 days of the date of collection.

Aqueous samples do not require refrigeration.

4.0 REPORTING and METHOD DETECTION LIMITS

Please refer to SOP QA020, current revision for further information regarding MDL's.

4.1 Reporting Limit. The reporting limit for this method has been established at 0.0005 mg/l.

4.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

4.3 MDLs should be established for all appropriate methods using a solution spiked at approximately 2-10 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The samples used for the MDL must be prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar days. If there are multiple instruments that will be assigned the same MDL, then the sample analyses must be distributed across all instruments. A minimum of 2 MDL's prepared and analyzed on different calendar days is required for each instrument. The same prepared extract may be analyzed on multiple instruments so long as the minimum requirement of seven preparations in at least three separate batches is maintained.

4.4 An MDL (LOD) check standard will be analyzed at the time of the MDL study and on a quarterly basis. The concentration of the MDL check standard should be 2-4 times the statistical MDL. This is a qualitative check; therefore, the analyte needs to be detected only. If the analyte is not detected, the concentration of the MDL check standard must be increased until the analyte is detected.

4.5 An LOQ check standard will be analyzed at the time of the MDL study and on a quarterly basis. The concentration of the LOQ check standard should be at the current method RL. LOQ check standard must recover within the methods blank spike requirements to be considered valid.

4.5 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I” qualifier. Some program or project specifications may require that no values below the RL be reported.

5.0 INTERFERENCES

Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations of sulfide as sodium sulfide as high as 20 mg/l do not interfere with mercury recoveries when following this method. Copper concentrations > 10 mg/l may also interfere with mercury recoveries. Samples that are high in chloride such as seawater, brine and industrial effluent may require as much as 12.5 ml of additional permanganate.

Note: When chloride concentrations are high, hydroxylamine sulfate and stannous sulfate should be used in place of corresponding chlorides.

Finally, certain volatile organic materials will also absorb at this wavelength and can interfere. It can be determined if this type of interference is present by doing a preliminary run without reagents.

6.0 APPARATUS

6.1 A Leeman HYDRA AA II automated analyzer is used for all analysis. Currently there are two Hydra AA's in service at SGS - Orlando. Refer to the instrument manual for further details on this instrumentation.

6.2 Automatic repipettor (s).

6.3 Fisher Brand 0.45 micron (um) filter or equivalent. Filter lots are checked for cleanliness through the Method Blank process. All Method Blank analytical results must be <1/2 RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated filters must be re-filtered through acceptable filters.

6.4 Fisher Brand disposable 10 ml syringes or equivalent. Syringe lots are checked for cleanliness through the Method Blank process. All Method Blank results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated syringes must be re-filtered through acceptable syringes.

6.5 Environmental Express Hot Block or equivalent capable of maintaining 90-95 ^oC.

6.6 Environmental Express digestion vessels or equivalent, 65ml capacity. Each Lot of digestion tubes comes with a Certificate of Analysis which demonstrates cleanliness as well as volume accuracy. Please refer to Digestion Tube Certificate Logbook for further information. Tube Lots are also checked through the Method Blank process. All Method Blank analytical results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Re-digestion is required for all samples prepared with the contaminated tube lot.

6.7 Class A volumetric pipette (s), flask (s), and cylinder (s)

6.8 Thermometer(s), capable of measuring a temperature of at least 125 ^oC, checked against NIST traceable thermometers. Refer to SOP QA002, current revision for further information.

6.9 Eppendorf Pipette (s) - Pipette (s) are checked daily for accuracy and to ensure they are in good working condition prior to use. Volumes are checked at 100% of maximum volume (nominal volume). Pipettes are checked within the metals department and results are stored electronically in the “Pipette Calibration Log”. Refer to SOP QA006, current revision for further information regarding pipette calibration. BIAS: mean must be within 2% of nominal volume. Precision: RSD must be $\leq$ 1% of nominal volume based on three replicates.

6.10 Top Loader Balance (used to prepare reagents) – Capable of accurately weighing 0.01 g. Refer to SOP QA005, current revision for further information.

6.11 Data System

Microsoft Windows 7 Professional
Instrument software – HG4 - Leeman Labs Envy 1.9 sp1
Instrument software – HG5 - Leeman Labs Envy 2.0 sp0

6.11.1 A computer system interfaced to the Leeman Hydraa II that allows for the continuous acquisition and storage of all data obtained throughout the duration of the analytical run sequence.

6.11.2 Data is archived to a backup server for long term storage.

7.0 REAGENTS

All chemicals listed below are trace metal grade unless otherwise specified. Refer to Acid Certificate of Analysis logbook for Certificate of Analysis and compliance with the specifications of the grade listed. SGS - Orlando produces DI water to the specifications for the ASTM Type II standard designation based on the system manufacturer’s performance specifications. The DI water is used exclusively for laboratory purposes. De-ionized (DI) water should be used whenever water is required. Refer to SOP QA037, current revision for more information regarding testing and monitoring. All standards and prepared reagents must be prepared every 6 months or before stock standard expiration date, whichever comes first, except as noted elsewhere in this SOP. Refer to Metals Electronic Standard Prep Logbook for further information. Some of the information included in the logbook is as follows: standard name, elements in mix, manufacturer, lot number, parent expiration date, acid matrix, stock concentration, volume of standard added, total volume, final prepared concentration, prep date, initials, MET number, and prepared standard expiration date.

7.1 Sulfuric acid, concentrated, trace metal grade

7.2 Nitric acid, concentrated, trace metal grade

7.3 Stannous chloride, reagent grade. To 400 ml of DI water, add 50 ml of concentrated Hydrochloric acid, and 50 g of stannous chloride. Dilute to 500 ml with DI water. Stannous sulfate may be used in place of stannous chloride. Stannous chloride is prepared daily. Lot number is recorded in Hg Digestion logbook.

7.4 Sodium chloride-hydroxylamine hydrochloride, reagent grade. Add 120 g of sodium chloride and 120 g of hydroxylamine hydrochloride to 1 liter of DI water. Mix well; hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.5 Potassium permanganate, reagent grade. Add 50 g of potassium permanganate to 1 liter of DI water and mix well.

Caution: Potassium permanganate is a strong oxidizing agent. Handle with care.

7.6 Potassium persulfate, reagent grade. Add 50.0 g of potassium persulfate to 1 liter of DI water and mix well.

Caution: Potassium persulfate is a strong oxidizing agent. Handle with care.

7.7 2 percent HCl Carrier Solution. 40 mls concentrated HCl, diluted to 2 liters with DI water. 2 percent HCL carrier solution is prepared daily. Lot number is recorded in Hg Digestion logbook.

7.8 10 percent HCL rinse solution. Add 100mls of concentrated HCL to final volume of 1 liter of DI water.

7.9 Mercury working standards. Mercury standard solutions are made from a purchased stock solution of 1000 ppm mercury.

7.9.1 10 ppm Hg solution. (Used to prepare 100ppb Mercury solution) Using a 10 ml class A volumetric pipette, add 10 ml of 1000 ppm stock to a 1.00 liter class A volumetric flask containing approximately 750 ml of DI water and 10 ml of concentrated nitric acid. Dilute to volume with DI water and mix well. This 10ppm standard must be prepared every six months.

7.9.2 100 ppb Hg solution. (Used to prepare calibration curve, CCV, CRI). Using a 10 ml class A volumetric pipette, add 10 ml of 10 ppm Hg solution to a 1.00 liter class A volumetric flask containing approximately 750 ml of DI water and 10 ml of concentrated nitric acid. Dilute to volume with DI water and mix well. This 100ppb standard must be prepared every month.

7.9.3 Second source working solutions are prepared at the same concentrations as the calibration standards listed above in sections 7.8.1 and 7.8.2 except they must be from a second source.

7.9.4 Daily working standards used in section 8.0 are prepared and digested daily using 100ppb standard solutions.

8.0 WATER DIGESTION AND ANALYSIS PROCEDURE

Below is a step-by-step procedure for the digestion and analysis of water samples for mercury.

8.1 Make up the standard curve as shown below. Clearly label each digestion vessel with the standard's ID. The standard ID's should be recorded in the Mercury Digestion Logbook.

ml of 100 ppb Hg solution	ml of DI water	Total µg/L of Hg
0.0	50	0.0
0.10	50	0.20
0.50	50	1.0
1.50	50	3.0
2.50	50	5.0
3.00	50	6.0

Dilute to the 50ml mark on the digestion vessel with DI water.

8.2 Make up the quality control samples as shown below. Make sure to clearly label each digestion vessel.

Sample ID	ml of 100ppb Hg solution	ml of DI water	Total µg/L of Hg
*Spike Blank	1.5	50	3.0
CCV	1.5	50	3.0
Low Check (CRI)	0.10	50	0.20
Method Blank	0.0	50	0.0
*ICV	1.5	50	3.0

Dilute to the 50ml mark on the digestion vessel with DI water.

Sample ID	ml of 100 ppb Hg solution	ml of sample	Total µg/L of Hg
*Matrix Spike	1.5	50	3.0 + sample
*Matrix Spike Dup	1.5	50	3.0 + sample
Duplicate		50	sample

Dilute to the 50ml mark on the digestion vessel with DI water.
*Use second source 100ppb solution.

8.3 Shake sample vigorously to ensure thorough mixing. Measure out 50 ml of each sample into a labeled digestion vessel. The sample may be measured by using a Class A graduated cylinder or by using the calibrated digestion tube. If no information is available about the level of mercury in the samples to be analyzed set up a 50 ml sample size. If information is available, select a sample size that will result in an analysis value near the mid-range of the curve. For TCLP / SPLP samples, use a 5 ml initial volume. Record the volume used in the Mercury Digestion Logbook.

8.4 To all samples, QC and standards add the following reagents:

- 1.25 ml of concentrated nitric acid
- 2.5 ml of concentrated sulfuric acid
- 7.5 ml of permanganate solution
- 4.0 ml of persulfate solution

Swirl the samples well after each addition of reagent. More potassium permanganate solution may be required for some samples. Enough should be added so that the purple color persists for at least 15 minutes. Ensure that equal amounts of potassium permanganate are added to the standards and blanks.

8.5 Pre-heat the Hot Block to 90-95 degree C. Place the sample vessels in the Hot Block. Heat the samples for 2 hours, remove and cool to room temperature. Allow samples to stand quietly without manual agitation.

8.6 While the samples are digesting, begin setting up the Leeman HYDRA AA II mercury analyzer following the steps outlined below. Further details are available in the instrument manual.

8.6.1 Turn the Argon gas on. Check that the vent line is connected to the exhaust hood.

8.6.2 Inspect all pump tubing and replace if necessary. Put the tubing on the cassettes and attach to the pump head, making sure the cassette adjusters are properly adjusted to provide a smooth flow of sample and reagents. Place the fresh stannous chloride solution in the bottle. Fill the rinse bottle with 2 percent HCL carrier solution. Connect stannous chloride and HCL rinse lines.

8.6.3 Double click on the Envoy icon on the desktop. This will open the Envoy mercury analysis software. Click on the green and black arrow icon. This will start the pump, turn on the gas, and turn on the lamp.

8.6.4 Locate the “Sequence” tab at the bottom of the Envoy software page. Now click on “sequence” at the top of the page and click “new”. Type in the run sequence starting with the MB. After typing in the entire days run sequence click on the “Update” button. Click on “sequence” at the top of the page and choose “save”. Type in run sequence name as follows: Instrument-month-day-matrix-run number. (i.e. h40606w1).

8.6.5 Locate “Analysis” tab at bottom of Envoy page. Now click on “Analysis” at the top of the page and click “new”. Enter analysis dataset as follows: Instrument-month-day-matrix-run number (i.e. h40606w1).

8.6.6 Add 3 ml of hydroxylamine hydrochloride solution to each standard and sample and swirl until the solution has been completely decolorized before analyzing. Bring samples to a final volume of 50mls using DI water. If the sample (s) contain particulate matter, it should be filtered (performed at the analytical bench), along with the method blank and blank spike through a 0.45 um syringe filter before analysis. Samples are now ready for analysis.

8.6.7 Calibration is performed by analyzing a series of 5 standards and a blank. All calibration curves must be determined from a linear calibration prepared in the normal manner using the established analytical procedure for the instrument. Refer to instrument manual for further detail. A correlation coefficient of ≥0.995 must be achieved, if not, analysis must be terminated, the problem corrected, and instrument re-calibrated. Calibration data is printed and included with each analytical data package. Click on the “Run sequence” icon. Instrument will start calibration. Once the calibration is complete the instrument will automatically accept the curve if correlation coefficient >0.995. The instrument will then proceed to analyze the High standard, ICV, ICB, CRI, CCV, and CCB. The calibration curve and all initial QC are compared to check tables set up in the software. If any standard fails the set criteria it will be flagged on the screen as to alert the analyst. The instrument will continue the analysis of the run sequence if all QC criteria has been met.

8.6.8 After analysis has been completed flush the entire system with 10% HCL, then DI water, and then allow to pump dry. Unclamp all tubing, turn off gas and lamp.

8.6.9 Raw data generation (PDF File)

Open the “PDF Creator” icon located on the desktop. Click on the green light, it will turn red. Go to the Envoy software. Click on the “Method” tab and then locate that days calibration curve. Click on print to PDF creator. Now go to the “Analysis” tab, click on “report” and “clear all.” Click on “Load” and choose “Accutest” profile. Select all samples to be reported. Choose “report” as output. Next click on “Printer” and send to PDF creator. Leave the report title blank and click “OK”. Now go back to the “PDF Creator” which should still be open on the desktop and click on “Document”, “combine all.” Click on the red light which should now turn green. Close the PDF creator. Go to the “Pdf shortcut” icon located on the desktop and rename .PDF to MAxxxx.pdf. Right click on MAxxxx.pdf, copy, then paste to the “Lims Data” icon on the desktop. Open the pdf file (MAxxxx.pdf) and print to metals printer. This will generate the raw data that will be included in the run package. Now close the pdf file and archive.

8.6.10 LIMS data generation

Choose “CSV” as output under the “Analysis” tab. Type in analysis dataset (h40606w1), no extension. Go to the “Export” shortcut located on the desktop. Locate analysis dataset (h40606w1.csv), right click on file and open with wordpad. Change Blank, 0.2ppb, 1.0ppb, 3.0ppb, 5.0ppb, and 6.0ppb to STD1 (STD1_1 for 245.1, STD1_2 for 7470A), STD2, STD3, STD4, STD5, and STD6. Remove all percent recoveries from the file and the save and close the file. Right click on the analysis dataset, copy/paste to “Lims Data” icon on desktop. If the run contains any errors an “error report” will be generated to the metals printer. Correct any errors and re-send the file. Archive run sequence (.SEQ) and analysis dataset (.CSV) when done.

9.0 QUALITY CONTROL

All QC calculations should be done as outlined in the method. Please refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements. Check with area supervisor or lab manager for any non-compliant quality control for further information.

9.1 Method Blank – An acceptable method blank or reagent blank must be analyzed with every batch of samples processed. The method blank must be less than one half the reporting limit. If the method blank is greater than one half the reporting limit, the samples associated with the contaminated blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-digesting and reanalyzing the samples, or qualifying the results with a "B" or "V" qualifier. All samples associated with an out of compliance method blank shall be qualified and footnoted in LIMS as well as in the case/run narrative.

9.2 High Standard Check (for method 245.1 only) – The high calibration standard must be analyzed after the initial calibration has been performed. The results of the high standard check must agree within 5 percent of the true value for the analysis to be valid. If the high standard check fails criteria, a new HSTD or initial calibration must be performed and all samples must be re-analyzed.

9.3 Initial Calibration Verification – An initial calibration verification (ICV) sample must be analyzed after the initial calibration has been performed. This sample must be prepared at or near the midpoint of the initial calibration from a reference material independent from the initial calibration solution. The results of the ICV must agree within 10 percent of the true value for the analysis to be valid. For method 245.1 the ICV must agree within 5 percent of the true value for the analysis to be valid. If the ICV fails, a new ICV or initial calibration must be performed and all samples must be re-analyzed with an acceptable ICV.

9.4 Continuing Calibration Verification – If more than 10 samples are to be analyzed in a single day, a Continuing Calibration Verification sample prepared at or near the mid point of the initial calibration must be analyzed after every 10th sample and at the end of the analytical run. The results of the initial CCV analysis must be within 10 percent of the true value to be considered valid. All subsequent CCV's must be within 20 percent of the true value to be considered valid. For method 245.1 the results of the initial CCV must be within 5 percent of the true value for the analysis to be valid. All subsequent CCV's must be within 10 percent of the true value to be considered valid. If the CCV fails, all samples analyzed after the first passing CCV must be reanalyzed.

9.5 Continuing Calibration Blank/Initial Calibration Blank – Analyze the Initial calibration blank solution at the beginning of each run and the continuing calibration blank after every tenth sample and at the end of the sample run. The ICB/CCB must be less than one half the reporting limit to be considered valid. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, or qualifying the results with a "B" or "V" qualifier, or raising the reporting limit for all samples to greater than two times the background concentration.

9.6 Low Level Check Standard (CRI) – A standard prepared at the low calibration concentration should be prepared and analyzed at the beginning and end of each analytical run. The CRI should agree within 20 percent of the true value to be acceptable. If the initial CRI does not meet the acceptance criteria, the samples must be reanalyzed. If the closing CRI fails the criteria, the samples associated with the failing CRI shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the CRI or qualifying the results in LIMS.

9.7 Blank Spike – A blank spike (BSP) or Laboratory Control Spike (LCS) should be prepared using DI water spiked at the midpoint of the calibration curve. The blank spike must be within 20 percent of the true value for the analysis to be considered valid. For method 245.1 the results of the BSP must be within 15 percent of the true value for the analysis to be valid. If the blank spike exceeds the acceptance criteria, the samples must be re-digested and reanalyzed. A blank spike is required for every 20 field samples or for each analysis batch. Statistical control limits are generated for LCS's for QA purposes only. Refer to section 15.0 of this SOP for further detail.

9.8 Duplicate - The laboratory must digest a duplicate for a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample must be assessed and must be $\leq$ 20 percent for sample results at or above the reporting limit. If the RPD is outside the 20 percent criteria the results must be qualified in LIMS. RPD's are also calculated in LIMS for sample results below the reporting limit. RPD's outside the 20 percent criteria are not considered failing and LIMS automatically footnotes these as "RPD acceptable due to low duplicate and sample concentrations."

9.9 Matrix Spike/Matrix Spike Duplicate/MSA – At least on Matrix Spike/Matrix Spike Duplicate pair must be prepared and analyzed with every 20 field samples. The MS/MSD recovery must agree within 30 percent of the true value for method 245.1 and within 20 percent of the true value for method 7470A. Relative standard deviation (RSD) for the MSD should be $\leq$ 20 percent. If the results of the MS/MSD are outside the acceptance criteria, the data should be footnoted as possible matrix effect. In certain circumstances the Method of Standard Additions (MSA) may be needed by some project specific requirements. SGS - Orlando may perform an MSA when sample matrix is so complex that viscosity, surface tension, and components cannot be accurately matched with standards. If an MSA is not performed the results may be footnoted in LIMS. SGS - Orlando will use a single addition method as described in SW846-7000B.

9.10 When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted back into the calibration range and reanalyzed.

9.11 When preparing TCLP/SPLP samples, prepare an additional leachate blank and leachate blank spike from the extraction fluid used to extract the samples. See section 9.1 and 9.7 for acceptance criteria.

9.12 When preparing dissolved metals, an additional method blank must be prepared. The method blank must be filtered through the same filter media as the samples and then digested as usual. This is performed to ensure there is no cross contamination from the filter media into the samples. See section 9.1 for acceptance criteria.

9.13 Serial Dilution - For one sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution should be prepared. For the serial dilution, a 1:5 dilution should be made on the sample. The results of the 1:5 dilution should agree within 10 percent of the true value as long as the sample and the dilution result are greater than 10 times the method detection limit and/or greater than 50 times the IDL. If the dilution is not within 10 percent then a footnote must be entered into LIMS.

9.14 For each digestion batch of 20 samples (10 samples for method EPA 245.1), a serial dilution (performed at the analytical bench), a matrix spike (MS), a matrix spike duplicate (MSD), a duplicate (DUP), a blank spike (LCS), and a method blank should be prepared. Re-digestion is suggested for QC that does not meet the SGS - Orlando QC limits. The appropriate lab supervisor or lab manager will notify the analyst of samples that need re-digestion.

10.0 DOCUMENTATION REQUIREMENTS

All digestion information should be documented in the Sample Digestion Logbook. The information required includes the sample identification (including the sample bottle number), the initial sample volume, and the final sample volume, the acids used (including lot number and manufacturer), the spiking solutions used, the digestion vessel lot number, the observed temperature, corrected temperature, the thermometer ID, analyst's signature, the date of digestion, digestion start time, and digestion end time. The analyst should write additional information such as unusual sample characteristics and samples that need to be filtered (dissolved analysis) in the comment section. All raw data is printed to .PDF format and archived to a backup server for long term storage.

11.0 SAFETY

The analyst should follow normal safety procedures as outline in the SGS - Orlando Laboratory Safety Manual. Particular care should be observed in handling the strong acids and oxidizing agents. Safety glasses and lab coats should be worn at all times in the lab. Gloves should be worn when handling samples.

12.0 CALCULATIONS

Below are the calculations, which should be used for soil samples. The concentration of the sample in µg should be obtained from the linear calibration curve.

$$\textbf{Final concentration in mg/kg} = \frac{\textbf{Concentration of sample in } \mu\textbf{g}}{\textbf{(Sample wt in g)(\%solids/100)}}$$

Matrix Spike and Matrix Spike Duplicate Recovery:

$$\frac{\textbf{(Spiked Sample Result - Sample Result)}}{\textbf{Amount Spiked}} \times 100 = \textbf{matrix spike recovery}$$

Matrix Duplicate/Matrix Spike Duplicate Relative Percent Difference:

$$\frac{(|\text{Sample Result - Duplicate Result}|) \times 100}{(\text{Sample Result + Duplicate Result})/2} = \text{Duplicate RPD}$$

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

13.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 13.2.

13.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

14.0 GENERIC DEFINITIONS

14.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours whichever comes first.

14.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

14.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. A CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

14.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

14.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

14.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

14.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the performance of a method in a given sample matrix.

14.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and performance of a method in a given sample matrix.

14.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

14.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

14.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

15.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Statistical control limits are stored in the LIMS for QA purposes only. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors

16.0 GLASSWARE CLEANING

All glassware should be washed with soap and tap water, rinsed with 5 percent nitric acid solution, and then rinsed at least three times with DI water. Refer to SOP GN196, current revision for further information regarding glassware cleaning.

17.0 INSTRUMENT MAINTENANCE

Recommended periodic maintenance includes the items outlined below. All maintenance must be recorded in the instrument maintenance log.

17.1 Change the pump tubing weekly or as needed.

17.2 Clean the optical cell and lenses once per week or as needed.

17.3 Change the sampler tip as needed.

17.4 Inspect the liquid/gas separator, mixing coil, and all tubing connections once per week and replace as needed.

REVISION HISTORY

Revision Date	Revision Number	Affected Section(s)	Revision Description
12/2020	13	“Revised Sections”	Added Revision History and removed Revised Sections. Sec. 9.1 added detail and removed references to WV blank evaluation.

METALS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY (ICP-MS)

Prepared by: Dave Metzgar Date: 12/18/2020

Approved by: Svetlana Izosimova Date: 12/18/2020

Annual Review

Reviewed by: Svetlana Izosimova Date: 12/09/2021

Reviewed by: ______ Date: ______

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Effective 7 days after “*” date

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TITLE: METALS BY INDUCTIVELY COUPLED PLASMA – MASS SPECTROMETRY (ICP-MS)

REFERENCES: SW846 6020A, Revision 1, February 2007.
SW846 6020B, Revision 2, July 2014.
EPA200.8, Revision 5.4, 1994.

Note: Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements. Also check with metals supervisor for additional information.

Main Instrument: Agilent 7700x, serial # JP12151709
Auto-sampler: CETAC ASX500, serial # US091320A520

1.0 SCOPE AND APPLICATION

1.1 This method is applicable for the determination of total and dissolved metals in water samples and in waste extracts or in solid or aqueous digests. Please refer to table 1 for a list of reportable elements.

2.0 SUMMARY

2.1 Samples are prepared for analysis by digestion. Please refer to method specific digestion SOP’s within the metals department for more information. The prepared samples are introduced into a radiofrequency plasma by pneumatic nebulization. There the energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass to charge ratio by a quadrupole mass spectrometer. The ions transmitted through the quadrupole are detected by an electron multiplier and the ion information is processed by a data handling system.

3.0 REPORTING LIMIT(RL), Lower Limit of Quantitation (LLOQ) AND METHOD DETECTION LIMIT

3.1 Reporting limits (RL) are based on the extraction procedure. Reporting limits may vary depending on matrix complications, volumes and by client needs, but the reporting limits must always be verified with a low check which meets the criteria outlined in this SOP. Solid matrices are reported on a dry weight basis. Refer to table 1 of this SOP for SGS - Orlando typical reporting limits. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific reporting limits.

3.2 MDLs should be established for all appropriate methods using a solution spiked at approximately 2-10 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The samples used for the MDL must be prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar days. If there are multiple instruments that will be assigned the same MDL, then the sample analyses must be distributed across all instruments. A minimum of 2 MDL's prepared and analyzed on different calendar days is required for each instrument. The same prepared extract may be analyzed on multiple instruments so long as the minimum requirement of seven preparations in at least three separate batches is maintained. Please refer to SOP QA020, current revision for further information regarding MDL's.

3.3 An MDL (LOD) check standard will be analyzed at the time of the MDL study and on a quarterly basis for verification. The concentration of the MDL check standard must be 2x-4x the statistical MDL. The MDL Check Standard is carried through the entire preparation and analytical procedure. This is a qualitative check; therefore, the analyte needs to be detected only. If the analyte is not detected, the concentration of the MDL check standard must be increased to a level where the analyte is detected.

3.4 Lower limit of quantitation check sample (LOQ). The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on a quarterly basis to demonstrate the desired detection capability. The LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within 20 percent of their true value.

3.5 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

3.6 Instrument Detection Limits (IDL). IDL's should be completed upon initial instrument installation. SGS - Orlando does not report to IDL.

3.7 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

4.0 DEFINITIONS

BATCH. A group of 20 samples or less that behaves similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit within a 24 hour period. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD. (CCV) calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of 10 percent or every 2 hours during an analysis run, whichever is more frequent, and at the end of the analysis sequence. For this method, the mid-level calibration check standard criteria is ± 10 percent of the true value and the relative standard deviation for the replicates that are greater than 5 times the reporting limit (LLOQ) is less than 5 percent. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the reporting limit (LLOQ).

EXTERNAL CHECK STANDARD. (ICV) The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. It must be run after each calibration. The external check standard criteria is ± 10% of the true value and the replicates that are greater than 5 times the reporting limit (LLOQ) should have a relative standard deviation of less than 5 percent. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 sample batch. A sample batch is defined as a maximum of 20 field samples in a preparation batch over a time period of 24 hours. Assess laboratory performance against the control limits of 80 to 120 percent for method SW846-6020A and 6020B. Recovery of 85 to 115 percent for method EPA 200.8. In house limits should also be generated once sufficient data is available to support the default limits. If the lab control or spike blank is outside of the control limits for a parameter, all samples must be redigested and reanalyzed for that parameter. The exception is if the lab control or spike blank recovery is high and the results of the samples to be reported are less than the reporting limit (LLOQ). In that case, the sample results can be reported with no flag.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against method limits. If the sample and the duplicate are less than 5 times the reporting limits (LLOQ) and are within a range of ± the reporting limit (LLOQ), then the duplicate is considered to be in control.

$$\frac{(|\text{Sample Result - Duplicate Result}|) \times 100}{(\text{Sample Result + Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against default limits of 80 to 120 % recovery for method 6020A and 6020B. Recovery criteria of 70 to 130 % recovery for method 200.8. In house limits should be generated once sufficient data is available. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect.

$$\underline{(\text{Spiked Sample Result - Sample Result})} \times 100 = \text{Matrix Spike Recovery}$$

(Amount Spiked)

MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 sample batch. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less than 1/2 the reporting limit (LLOQ) for that parameter. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit. Samples associated with the contaminated blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-digesting and reanalyzing the samples, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit (LLOQ) to greater than two times the background concentration.

METHOD DETECTION LIMITS (MDLS). The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT BLANK. The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than 1/2 the reporting limit (LLOQ) for that analyte. If the reagent blank contains levels over the reporting limits (LLOQ), the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE. Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER. Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water.

STANDARD ADDITION. The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS - Orlando Health and Safety Program, which include the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

6.0 SAMPLE COLLECTION, PRESERVATION & HOLDING TIME

6.1 Aqueous samples are collected in 500ml or 1000 ml HDPP bottles. All water samples should be preserved with nitric acid to a pH of 2 or less. Solid samples are collected in glass jars (4 oz or 8 oz) with PTFE lined lid. All solid samples should be stored in a refrigerator at 4 degrees C until digestion.

6.2 All samples should be analyzed within 6 months of the date of collection.

7.0 INTERFERENCES

7.1 Several types of interferences can cause inaccuracies in trace metals determinations by ICP-MS. These interferences are discussed below.

7.2 Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. If isobaric interferences are present in the ion being analyzed, then the data must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the element of interest.

7.3 Abundance sensitivity is a property that defines the degree to which the wings of a mass peak contribute to adjacent masses and is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured next to a large one. Spectrometer resolution should be adjusted to minimize these interferences.

7.4 Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. Refer to method 200.8, 6020A and 6020B for lists of common interferences and correction equations to be applied. If these interferences cannot be avoided by the use of different isotopes, then correction equations should be applied to the data. Alternatively, collision/reaction cell technology can be applied to physically and chemically remove interferences.

7.5 Physical interferences can occur during the transfer of the solution to the nebulizer (viscosity effects).

7.6 Memory interferences can be caused by buildup on the sampler and skimmer cones, and from buildup of sample material in the torch and spray chamber. Some elements, such as mercury, can suffer from severe memory effects. In that case, gold is added to the rise solution to decrease the Hg rinse out time.

7.7 Interference correction equation procedure.

Interference correction equations are used to correct interference with target elements due to other elements or formation of polyatomic ions. Specify the elements related to the interference to be corrected. Isotope masses and isotope ratios are displayed in Mass table. Select the check boxes for the masses for which correction equations are set. Equations are displayed in the Equation table. Select the elements for which the correction equations are set. Select the masses for which the correction equations are set. Select positive or negative sign for the factor, enter masses in the Mass field and enter the factors of the correction equations in the Multiplier field. “OK” applies to settings and the specified interference correction equation is displayed in the “Select Elements on Periodic Table” dialog box.

8.0 APPARATUS

8.1 Currently in use is an Aglilent 7700x ICP-MS with collision/reaction cell capacity and HMI (High matrix interface) and the associated autosampler.

8.2 Data system

8.2.1 Microsoft Windows 7 Professional Version 2009

8.2.2 Agilent Masshunter, Version B.01.03, Build 393.17, Patch 2, 2014

8.2.3 Computer system interfaced with Agilent ICP-MS that allows continuous data acquisition and storage of all data obtained throughout the duration of the analytical run sequence. Data is backed up and archived for long-term storage

8.3 Class A volumetric glassware as needed and instrument autosampler tubes.

8.3.1 All glassware must be washed with soap and tap water and then soaked in a 10% nitric acid bath for several hours. It must then be rinsed at least 3 times with distilled, deionized water.

8.4 Polypropylene bottles for standard storage. These bottles must also be cleaned as outlined above.

9.0 REAGENTS

Please refer to electronic standard logbook for detailed information regarding standard preparation.

9.1 All chemicals listed below are reagent grade unless otherwise specified. Deionized water must be used whenever water is required. Note: All reagents can be scaled up or down proportionately if different final volumes are required.

9.2 Hydrochloric acid, trace metals grade.

9.3 Nitric acid, trace metals grade. Note – ultra trace grade may be required if lower detection limits than normal are needed.

9.4 Standard stock solutions available from Inorganic Ventures, Ultra Scientific, VHG Laboratories or equivalent. Note: All standards must be ICP-MS quality standards or must be demonstrated to be free of interferences at the levels of use. Standards should come labeled with an expiration date and certificate of concentrations from the manufacturer. If both of these items are not received, then the manufacturer should be contacted before use of the standard.

9.5 Calibration Standards: These can be made up by diluting the stock solutions to the appropriate concentrations. Fresh calibration standards should be prepared a minimum of every two weeks. They must be monitored weekly for stability.

9.5.1 Standards should be made in a low acid matrix. Concentrations of 1 to 2 percent nitric acid and 0 to 0.5 percent hydrochloric acid are suggested, although any acid concentration that provides good analytical results may be used. High chloride concentrations may cause interferences so chloride concentrations should be limited. HCl may be omitted if silver and antimony are not elements of interest.

9.5.2 Refer to the standard prep logbook for the make-up and concentrations of standards and stock solutions being used to calibrate the ICP-MS. Suggested standard levels are shown in Table 2.

9.6 Aglient P/A Factor and Tuning/Performance Check Solution. Mix 1.0 ml of PA Tuning 1 solution and 1.0 ml of PA Tuning 2 solution (available from Aglient, part number 5188-6524) and bring to 100 ml final volume with a solution of 1% nitric acid and 0.5% HCl. This final solution contains 200 ppb of As, Be, Cd, Zn; 100 ppb of Mg, Ni, and Pb; 50 ppb of Al, Ba, Bi, Co, Cr, Cu, In, Li^6, Lu, Mn, Na, Sc, Sr, Th, Tl, U, and V; and 25 ppb of Y and Yb; 100 ppb of Ge, Mo, Pd, Ru, Sb, Sn ; and 50 ppb of Ir and Ti.

9.7 Tuning Standard, Agilent ICP-MS. This solution is used to verify mass calibration and thermal stability and must contain a mix of elements representing all of the mass regions of interest. Elements include 1 ppb Ce, Co, Li, Mg, Tl, and Y.

9.8 Internal Standards. Internal standards are added to all calibration standards, quality control, and samples during analysis, normally using a second channel of the peristaltic pump and a mixing manifold. The internal standard solution is recommended to contain Sc, Y, In, Tb, and Bi.

9.8.1 For the Aglient instrument, a solution containing 1 ppm of Li, Sc, Lu, In, Tb, Bi, Te and Ge in 1 % nitric is recommended. Refer to Table 3.

9.9 Calibration /Rinse Blank. The calibration and rinse blanks are prepared by diluting acids to the same concentrations found in the standards. The calibration blank is used to establish the analytical calibration curve and the rinse blank is used to flush the instrument between samples in order to reduce memory interferences.

9.10 Continuing Calibration Verification Check (CCV). This solution is prepared by adding either mixed or single element metals solutions to a solution containing the same acid matrix as the calibration standards. The metals should be at concentrations near the middle of the calibration curve. (Note: This check is run after the calibration, after every 10 samples or every 2 hours during an analysis run, whichever is more frequent, and at the end of the sample run.) CCV should be prepared from the same source as the calibration standards. Refer to Table 2 for suggested concentrations for the CCV.

9.11 Matrix Spike and Spike Blank Solution. Suggested levels for the final concentrations of the spike are shown in Table 4. This solution is prepared by adding either mixed or single element metals solutions to a solution containing 1 percent nitric acid and 0 to 0.5 % HCl and diluting to a fixed final volume with this acid mixture.

9.12 Lab Control Solution. This solution is prepared by adding either mixed or single element metal solutions to a solution containing 1 percent nitric acid and 0 to 0.5 % HCl and diluting to a fixed final volume with this acid mixture.

9.13 Interference Element Check Solutions or spectral interference check solutions (SIC). The purpose of the ICSA and ICSAB solutions is to demonstrate the magnitude of interferences and provide an adequate test of any corrections. It is recommended that the following solutions be purchased commercially.

9.13.1 ICSA Solution. The ICSA solution contains only the interfering elements. The recommended concentrations are shown below. The ICSA solution must be made fresh weekly.

Al	100 mg/L
Ca	100 mg/L
Fe	100 mg/L
Mg	100 mg/L
Na	100 mg/L
P	100 mg/L
K	100 mg/L
S	100 mg/L
C	200 mg/L
Cl	1000 mg/L
Mo	2.00 mg/L
Ti	2.00mg/L

9.13.2 ICSAB Solution. The ICSAB solution contains both the interferents and the analytes of interest. The recommended concentrations are shown below. The ICSAB solution must be made fresh weekly.

Al	100 mg/L
Ca	100 mg/L
Fe	100 mg/L
Mg	100 mg/L
Na	100 mg/L
P	100 mg/L
K	100 mg/L
S	100 mg/L
C	200 mg/L
Cl	1000 mg/L
Mo	2.00 mg/L
Ti	2.00mg/L
As	0.020 mg/l
Cd	0.020 mg/l
Cr	0.020 mg/l
Co	0.020 mg/l
Cu	0.020 mg/l
Mn	0.020 mg/l
Ni	0.020 mg/l
Ag	0.020 mg/l
Zn	0.020 mg/l

9.14 Initial Calibration Verification (ICV) or Quality Control Sample (QCS). The metals in this solution should be at final concentrations that are at the mid-point of the calibration curve. This solution is prepared by adding either mixed or single element metals solutions to a solution containing 1 percent nitric acid and 0 to 0.5 percent hydrochloric acid and diluting to a fixed final volume with this acid mixture. Please see Table 2 for suggested levels. The ICV sample must be from an independent source from the calibration standards.

9.15 CRI Standards (also referred to as LLCCV). The CRI standard must contain the elements of interest at (or below) the reporting limit (LLOQ) for each element. The CRI level is at the reporting limit (LLOQ) as shown in Table 1. This should be prepared by diluting calibration standard(s) to the reporting limit (LLOQ) level for each element. They should be made in the same matrix as the calibration standards

9.16 Liquid Argon or Argon Gas. Argon is provided by Air Products in the large outdoor tank. No lab monitoring of the tank is normally necessary.

9.17 Helium Gas. Required for running the reaction cell on the Agilent 7700X.

10.0 INITIAL INSTRUMENT SET-UP PROCEDURE FOR THE AGILENT 7700X ICP-MS

10.1 A general procedure on how to operate the Agilent 7700X ICP-MS is given below. Refer to the operation manual for further details.

10.2 Before bringing up the instrument, make sure that the lines, the torch, the nebulizer, and the spray chamber are clean, and that there are no leaks in the torch area.

10.3 Turn the vacuum pump and the heat exchanger on and verify that the liquid argon is turned on and the helium gas is turned on.

10.4 Connect the pump tubing and engage the peristaltic pump.

10.5 Put a new solution of acid rinse into the rinse reservoir. (Note: the composition of the rinse solution may be periodically changed to minimize sample introduction problems and sample carryover.) Make sure that sufficient internal standard solution is present.

10.6 Open the ICP-MS Mass Hunter Top software. Click on the instrument and open the instrument control panel. Click the plasma on. The instrument will automatically go through the start-up cycle. Then let the instrument warm up for at least 30 minutes.

10.7 Tune the instrument on a daily basis. Tuning must always be done after moving the position of the torch or the cones. Tuning can be done either manually or by following autotune procedures. It is recommended that autotune procedures be followed initially and then manual tuning be done as a second step. The purpose of tuning is to optimize the instrument for the highest sensitivity while obtaining low levels of oxides and doubly charged species. After the tune is complete, make sure to save the optimized parameters.

10.7.1 Open the ICP-MS top software, click on the instrument, and open the ICP-MS tuning page.

10.7.2 Click file and open the 6020AB_200.8 Method .b file. Keep the internal standard line in a solution of 1% nitric acid and 0.5% hydrochloric acid. Place the carrier line into the 1 ppb tuning solution. (see 9.7). On the tuning page, click start under the tune window to see the counts and RSD values. Do not start the tune process until the count and mean have similar readings and the RSD is < 5%. The counts per second values should be > 40000 for all masses. Click stop under tune window.

10.7.3 Before starting auto tune and printing tune report, create a new batch folder from existing method 6020AB_200.8.b. Save new batch using format “xaMMDDm1”.

10.7.4 On the tuning page, click Autotune, type the date (MMDDYYM1) on the popup window and click OK. This will perform the tuning of the instrument. Verify acceptable mass calibration by monitoring the peak width measurement at 5% of peak maximum for Co_59, Y_89, and Tl_205. If the peak widths are outside of the range of 0.65 to 0.85 and the masses are off by more than 0.1 amu, then redo the mass calibration. After all criterion is met, print the report and include with raw data. The tune report is automatically stored in the batch folder.

10.8 Before calibrating, run and print out a performance test. This must include the following items.

10.8.1 Relative standard deviations of the absolute signals must be less than 5 percent for all monitored masses. This includes Li_7, Y_89, and Tl_205. If these criteria are not met, correct the problem and then repeat the stability test. Print the results of this test and store with the raw data for the run.

10.9 Before starting sample analysis, set up the internal standards. Internal standards are added to all calibration standards, quality control, and samples during analysis, normally using a second channel of the peristaltic pump and a mixing manifold. Refer to Table 3 and Section 9.8 for additional information.

10.10 To start running samples, add samples to sequence and click “Add to Queue”. Unpause once ready to start analysis.

10.11 Calibrate the instrument using a minimum of a calibration blank and three non-zero standards that bracket the desired sample concentration range. The lowest non-zero standard must be at or lower than the RL or LOQ levels for all the elements**.** (Note: The calibration standards may be included in the autosampler program or they may be run separately.) A correlation coefficient of 0.998 or better must be obtained using a first order (linear) curve fit. A minimum of three replicate integrations are required for all data acquisitions.

10.11.1 In between each analysis of a separate standard or sample, a rinse blank must be run through the lines of the sample introduction system. Each sample or standard should be aspirated for a minimum of 30 seconds prior to the acquisition of data to allow equilibrium to be established.

10.12 After the instrument is properly calibrated, begin by analyzing the ICV solution. The ICV must be run after each calibration. For the ICV, all elements to be reported must be within 10 percent of the true value and the replicates that are greater than 5 times the reporting limit (LLOQ) should have a relative standard deviation of less than 5 percent. If the ICV is outside of criteria, then the problem must be identified and corrected before samples can be run and reported for the element(s) that are outside of criteria. Correction of the problem can be verified by rerunning the check standard(s) and showing that they meet QC criteria.

10.12.1 An ICB may be run after the ICV, but is not required for this method. If it is run, then all elements must be less than ½ the reporting limit (LLOQ) for each element.

10.12.2 Run the CRI (LLCCV) solution right after the ICV and ICB, (or any other place at the beginning of the run after the ICV, ICB and before any real samples are analyzed). For the CRI, all elements of interest must be within 30% of the true value, 20% for 6020B, or within client specified limits.

10.13 Then analyze the CCV and CCB check standards. For the CCV, all elements to be reported must be within 10 percent of the true value and the replicates that are greater than 5 times the reporting limit (LLOQ) should have a relative standard deviation of less than 5 percent. For the CCB, all elements to be reported must normally be less than 1/2 the reporting limit (LLOQ). If either the CCV or CCB do not meet criteria, then elements failing this criteria must not reported in the area bracketed by this QC.

10.13.1 The internal standard levels in the CCV and CCB must also be within 30% of the internal standard level for the initial calibration. If they are outside of these levels, then no samples can be reported in the area bracketed by this QC.

10.14 After the initial QC is completed and before any samples are analyzed, the ICSA and ICSAB solutions (SIC solutions) must be analyzed. The method does not list specific criteria for the ICSA and ICSAB, but in house criteria will be applied. For all the spiked elements, the analyzed results must be within 20 percent of the true results. For unspiked elements, the interfering element solution should contain less than the absolute value of 2 times the reporting limit (LLOQ) for each element. If these criteria are not met, then samples with significant interferences cannot be reported until the instrument is optimized and the ICSA and ICSAB are within specifications.

10.14.1 If the run is longer than 12 hours, a second ICSA, ICSAB pair must be analyzed before the next 12 hours is started.

10.14.2 If mass changes are made for the analysis of an element, all QC criteria must be met for the new mass and it must be verified that appropriate correction factors are in place.

10.14.3 The Agilent 7700X includes collision/reaction cell technology. The instrument is tuned in regular (non-cell) mode and in helium (collision/reaction) cell mode. This technology is used to minimize interferences during analysis. If this technology is not applied, then correction factors for interferences must be added into the method.

10.15 After the initial analytical quality control has been analyzed, the samples and the preparation batch quality control should be analyzed. Depending on the type of digestion and the sample matrix, samples and the associated QC should normally be diluted by a factor of from 2 to 5 before analysis. This dilution factor should be indicated in the sample ID file on the instrument.

10.16 Each sample analysis must be a minimum of 3 integrations. For samples containing levels of elements greater than approximately 5 times the reporting limits (LLOQ), the relative standard deviations for the replicates should be less than 10%. If not, reanalyze the sample. If, upon reanalysis, the RSDs are acceptable, then report the data from the reanalysis. If RSD's are not acceptable on reanalysis, then the results for that element may, on the reviewer's discretion, be footnoted that there are possible analytical problems indicated by a high RSD between replicates. In some cases, an additional dilution analysis may be needed. Check with the area supervisor or manager for additional information.

10.17 The internal standard levels must be monitored for all samples and quality control. If the internal standard is not within 70%-120% of the internal standard level for the initial calibration blank, then the sample must be diluted to bring the internal standard to within the correct range. If the internal standard is still outside of the range after the initial dilution, then additional dilutions must be done until the internal standard is within the appropriate range.

If an internal standard is present in a sample, then do not use that internal standard. For example, Y is sometimes seen in real samples. If the Y recoveries are high relative to the other internal standards, then do not use the Y internal standard.

10.18 For readings that exceed the linear range for a given element, a dilution is required. For method 6020B, after calibration the laboratory may choose to analyze a standard at a higher concentration than the high standard used in the calibration curve. The standard must recover within 10 percent of the true value, and if successful, establishes the linear range. The linear range standards must be analyzed in the same instrument run as the calibration they are associated with, but may be analyzed anywhere in the run. After a high reading, the following samples must be examined for possible carryover. A verification may be necessary by rinsing the lines with an acid solution and then re-reading the sample.

10.19 Indicate dilution factors for samples using "df" followed by the dilution factor after the sample ID. There should be a space between the sample number and the df.

10.20 Between each sample, flush the nebulizer and solution uptake system with a blank rinse solution for a minimum of 30 seconds or for the required period of time to ensure that analyte memory effects are not occurring. (60 seconds is recommended for normal methods excluding Hg and B. Longer times may be needed when Hg and B are being analyzed.)

10.21 Analyze the continuing calibration verification solution and the continuing calibration blank after every ten samples and at the end of the sample run.

10.21.1 For the CCV, all elements to be reported must be within 10 percent of the true value and the replicates that are greater than 5 times the reporting limit (LLOQ) should have a relative standard deviation of less than 5 percent. If the CCV solution is not within 10 percent of the true value, no samples can be reported in the area bracketed by the failing CCV for the failing element.

10.21.2 For the CCB, all elements to be reported must be less than 1/2 the reporting limit (LLOQ).

10.22 The CRI (LLCCV) must be analyzed at the end of each calibration (analysis) batch. The acceptance criterion for the CRI check is 70 to 130% recovery, and 80 to 120% for method 6020B. If an element does not meet this criterion, then all samples for that element in the concentration range between the CRI and the CCV must be reanalyzed. Samples containing concentrations higher than the CCV may be reported as long as CCV criteria are met.

10.22.1 More frequent CRI (LLCCV) checks may be analyzed during the course of the run if system stability at the low end of the calibration is questionable or if the lab wants to ensure that fewer samples will have to be submitted for reanalysis if there is a failed CRI at the end of a run.

10.22.2 It is recommended that the CRI check be run bracketing every 4 to 8 hour period of analysis. It may be run as frequently as every 10 samples if the supervisory staff deems that this is necessary.

10.23 After the run is completed, convert the data file to a CSV format using the option on the results screen. First save the file on the local drive using the file naming system described below. Update the run in the LIMS and enter the run name into the workgroup using lower case characters. Then copy the data from the local drive to the LIMS drive.

10.23.1 The file should be named as followed- initial instrument indicator (xa), date (MMDD), year, and sequential run number for that day (M1). For example, the first run from 12/17/02 would be designated xa121702m1.csv.

10.24 Calculations are done in the LIMS using the calculations shown below.

10.24.1 Calculation for aqueous samples.

$$\text{original sample concentration of metal } (\mu\text{g/l}) =$$

$$\frac{(\text{conc. in the digestate } (\mu\text{g/l})) \times (\text{final digestate volume (ml)})}{(\text{Initial sample volume (ml)})}$$

10.24.2 Calculation for solid samples.

$$\text{original sample concentration of metal (mg/kg)} =$$

$$\frac{(\text{conc. in the digestate } (\mu\text{g/l})) \times (\text{final digestate volume (ml)})}{(\text{Initial sample weight (g)}) \times (\%\text{sol}/100)}$$

10.25 At the end of the analysis day the ICP-MS must be brought down using the following sequence.

10.21.1 Rinse the tip in a solution of 1 percent nitric acid and 0.5 percent hydrochloric acid for 10 minutes and in DI water for 20 minutes. (Note: a stronger acid solution may be needed depending on the matrix of the samples that were analyzed.)

10.21.2 Turn off the plasma using off button.

10.21.3 Release the tension on the pump tubing.

10.21.4 Turn off the cool flow and the printer.

11.0 QC REQUIREMENTS

Note: Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements. Also check with metals supervisor for additional information.

11.1 This section outlines the QA/QC requirements necessary to meet the method 6020A.

11.2 Instrument Detection Limits (IDLs). IDLs must be established for all analytes. Please refer to specific method for instructions on performing IDL studies.

11.3 Lower Limit of Quantitation (LLOQ) check standard. LLOQ is the lowest point of quantitation. The LLOQ is initially verified by the analysis of 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery should be within +/- 35 percent of the true value with an RSD $\leq$ 20 percent.

Ongoing Lower limit of quantitation (LLOQ) check sample. The lower limit of quantitation check sample should be analyzed on a quarterly basis to demonstrate the desired detection capability. The LLOQ sample is carried through the entire preparation and analytical procedure.

11.4 LLQC (Lower Limit of Quantitation Check Sample) or LOQ Verification sample. A sample must be digested and analyzed initially and on an as needed basis to verify the quantitation limits for the method. Recoveries of this check must be within 70 to 130% of the true value, 80 to 120% for method 6020B. If recoveries are outside of this level, then the reporting limit (LLOQ) must be increased to a level that can be verified.

11.5 Linear Calibration ranges. The upper limit of the linear dynamic range needs to be established for each wavelength used by determining the signal responses from a minimum of three, preferably five, different concentration standards across the linear range. The linear calibration range which may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made must be documented and kept on file. A standard at the upper limit must be prepared, analyzed and quantitated against the normal calibration curve. The calculated value should be within ±10% of the true value. Linear calibration ranges should be determined whenever there is a significant change in instrument response. They must be done at least every six months. For any readings that exceed the linear range for a given element, a dilution is required. In addition, if there are significant interferences generated from elements above the linear range, than these elements must also be diluted so that accurate interfering element corrections can be applied.

For method 6020B, after calibration the laboratory may choose to analyze a standard at a higher concentration than the high standard used in the calibration curve. The standard must recover within 10 percent of the true value, and if successful, establishes the linear range. The linear range standards must be analyzed in the same instrument run as the calibration they are associated with, but may be analyzed anywhere in the run. Normal linear range values by element are shown in Table 2.

11.6 Initial Calibration Verification (ICV) or Quality Control Sample (QCS) and Initial Calibration Blank (ICB). After every new calibration, an ICV must be analyzed. The analysis of the ICV may be followed by the analysis of the ICB, although this is not required by the method.

11.6.1 For the ICV, all elements to be reported must be within 10 percent of the true value and the replicates that exceed 5 times the reporting limit (LLOQ) should have a relative standard deviation of less than 5 percent. The ICV must be from a different source than the calibration standards and must be near the mid-point of the calibration curve. If the ICV does not meet criteria, then the problem must be identified and corrected before samples can be run and reported for the element(s) that are outside of criteria. Correction of the problem can be verified by rerunning the check standard and showing that it meets QC criteria.

11.6.2 For the ICB, all elements to be reported must be less than 1/2 the RL (LLOQ). If the ICB is outside of criteria, then the problem must be identified and corrected before samples can be run and reported for the element(s) that are outside of criteria. Correction of the problem can be verified by rerunning the check standard and showing that it meets QC criteria. Analysis of a CCB before running any reportable samples can be used to verify that the system meets calibration blank requirements.

11.7 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB). Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run.

11.7.1 For the CCV, all elements to be reported must be within 10 percent of the true value and the replicates that are greater than 5 times the reporting limit (LLOQ) should have a relative standard deviation of less than 5 percent. The CCV should be made from the same source as the calibration standards at a concentration near the mid-level of the calibration curve. If an element does not meet the recovery criteria of the CCV, than no samples can be reported for that element in the area bracketed by the CCV.

11.7.2 For the CCB, all elements to be reported must be less than ½ the RL (LLOQ). If an element does not meet this criterion, then no samples can be reported for that element in the area bracketed by the CCB.

11.8 Interference Check Standards. After the initial QC is completed and before any samples are analyzed, the ICSA and ICSAB solutions (SIC solutions) must be analyzed. The method does not give specific criteria for the ICSA and ICSAB, but in house criteria should be applied. For all the spiked elements, the analyzed results must be within 20 percent of the true results. For unspiked elements, the interfering element solution should contain less than the absolute value of 2 times the reporting limit (LLOQ) for each element. If these criteria are not met, then samples with significant interferences cannot be reported until the correction factors are optimized and the ICSA and ICSAB are within specifications.

11.8.1 If the run is longer than 12 hours, a second ICSA, ICSAB pair must be analyzed before the next 12 hours is started.

11.8.2 If mass changes are made for the analysis of an element, all QC criteria must be met for the new mass and it must be verified that appropriate correction factors are in place.

11.9 Low Level Calibration Verification (CRI or LLCCV). The CRI standard containing the elements of interest at (or below) the reporting level for each element. The CRI (LLCCV) must be analyzed at the beginning and end of each calibration (analysis) batch. The acceptance criterion for the CRI check is 70 to 130% recovery and 80-120% for method 6020B. If an element does not meet this criterion, then all bracketed samples for that element in the concentration range between the CRI and the CCV must be reanalyzed. Samples containing concentrations higher than the CCV may be reported as long as CCV criteria are met.

11.9.1 More frequent CRI (LLCCV) checks may be analyzed during the course of the run if system stability at the low end of the calibration is questionable or if the lab wants to ensure that fewer samples will have to be submitted for reanalysis if there is a failed CRI at the end of a run.

11.9.2 It is recommended that the CRI check be analyzed every 4 to 8 hour period of analysis. It may be run as frequently as every 10 samples if the supervisory staff deems that this is necessary.

11.10 Method Blank. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 sample batch.

11.10.1 The default SOP limit for the method blank is that is must be less than one half of the reporting limit (LLOQ).

11.10.2 In addition, the blank is considered acceptable if it is less than 10% of the regulatory limit, or less than 10% of the lowest sample concentration for each analyte in a given preparation batch, whichever is greater. Samples associated with the contaminated blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-digesting and reanalyzing the samples, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit (LLOQ) to greater than two times the background concentration. All samples associated with an out of compliance method blank shall be qualified and footnoted in LIMS as well as the case/run narrative.

11.11 Lab Control Sample or Spike Blank. The laboratory must digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 sample batch. The laboratory should assess laboratory performance of the lab control and spike blank against recovery limits of 80 to 120 percent for method 6020A and 6020B. Recovery must be within 85 to 115 percent for method 200.8. In house lab control and spike blank limits may also be generated to support these default limits. If the lab control or spike blank is outside of the control limits for a given element, all samples must be redigested and reanalyzed for that element.

11.11.1 If solid lab controls are used, then the manufacturer's limits should be applied.

11.12 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Recoveries should be assessed against default limits of 80 to 120 percent for method 6020A and 6020B. Recovery must be within 70 to 130 percent for 200.8. In house limits may be generated for this method for informational purposes only. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote and it is recommended that a post-digest spike be analyzed for the out of control element(s). If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: Both the matrix spike amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

((Spiked Sample Result - Sample Result) / Amount Spiked) x 100 = matrix spike recovery

11.12.1 If a post-digest spike is required, the sample should be spiked with approximately 2 times the sample level or two times the reporting limits (LLOQ), whichever is greater. Limits of 80 to 120 percent are normally applied. The serial dilution is used to confirm any matrix effects. The post-digest spike recovery must be footnoted on the matrix spike recovery or otherwise noted in the quality control summary report.

11.13 Matrix Spike Duplicate (MSD) or Matrix duplicate (DUP). The laboratory must digest a matrix spike duplicate or matrix duplicate sample for a minimum of 1 in 20 samples. The relative percent difference (rpd) between the MSD and the MS or between the DUP and the sample should be assessed. The rpd is calculated as shown below. The control limit for the duplicate rpd is method defined as 20%. If the sample and the duplicate are less than 5 times the reporting limits (LLOQ) and are within a range of ± the reporting limit (LLOQ), then the duplicate is considered to be in control. Note: Both the duplicate amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

11.13.1 If a MSD or duplicate is out of control, then the data should be checked carefully to confirm that the high rpd for a given element is not a result of an analytical problem. If an analytical problem is suspected, the MSD or duplicate must be reanalyzed for confirmation. If the initial and reanalysis are in agreement (within 20%), then the high rpd is a result of preparation or sample issues and further analysis of the initial preparation is not required. If the initial and reanalysis are not in agreement due to an analytical problem, then any affected samples in the associated batch should also be reanalyzed for that element.

11.13.2 If more than 50% of the elements in a sample (that have levels of at least 5 times the reporting limit) (LLOQ) have a high RPD, then the MSD or duplicate should be redigested for confirmation, unless the sample matrix is such that the non-homogeneity of the sample is visually apparent. If the results confirm, the results from the original MSD or duplicate should be flagged as indicative of possible sample non-homogeneity. If the results do not confirm, then the whole batch should be digested and reanalyzed.

11.13.3 If 50% or less of the elements in a sample (that have levels of at least 5 times the reporting limit)(LLOQ) have a high rpd, then the high rpd(s) should be footnoted as indicating possible sample non-homogeneity unless other problems are suspected. If problems are suspected, the reviewer will initiate redigestion and reanalysis of the batch.

11.13.4 The calculations used to calculate RPD are shown below.

$$\frac{(|\text{MS Result - MSD Result}|) \times 100}{(\text{MS Result + MSD Result})/2} = \text{MSD RPD}$$

$$\frac{(|\text{Sample Result - Duplicate Result}|) \times 100}{(\text{Sample Result + Duplicate Result})/2} = \text{Duplicate RPD}$$

11.14 Serial Dilution. A serial dilution is required on a frequency of one in 20 samples. It is normally done on the same sample as is used for the matrix spike. If the analyte concentration is within the linear dynamic range of the instrument and sufficiently high (minimally a factor of at least 100 times greater than the concentration in the reagent blank), then an analysis of a fivefold (1+4) dilution must agree to within ±10% of the original determination. If not, an interference effect must be suspected and the serial dilution result for the element with the suspected interference must be footnoted. The serial dilution is calculated as shown below.

$$\frac{100 \times ((\text{Sample result – Serial dilution result}))}{\text{Sample result}} = \text{Serial dilution percent difference}$$

11.14.1 Results of less than the IDL are treated as 0. The concentration in the reagent blank is normally < 3 times the IDL, so the factor of 100 times the concentration in the reagent blank (listed above) so the limits should be applied to sample concentrations of greater than 300 times the IDL.

12.0 DOCUMENTATION REQUIREMENTS

12.1 If samples or QC checks require reanalysis, a brief explanation of the reason must be documented on the run log. All instrument data should be exported to the LIMS system.

12.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed. The SGS - Orlando Lot Number must be cross-referenced on the standard vial.

12.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

12.4 The correction factors from each method must be printed out each time a change is made and stored in a notebook in the lab. Each time the correction factors are modified, a new printout must be obtained.

12.5 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

12.6 Supervisory (or peer) personnel must routinely review (approximately once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information should also be verified during this review.

13.0 INSTRUMENT MAINTENANCE and TROUBLESHOOTING

Recommended periodic maintenance includes the items outlined below.

13.1 Change the pump tubing weekly or as needed.

13.2 Clean the nebulizer, torch, and injector tube every two to four weeks or more often as needed.

13.3 Change the sampler tip as needed (every one to two months).

13.4 Clean the recirculating pump lines as needed.

Record all maintenance in the Maintenance logbook. Repairs by manufacturer representative and outside contractors must be documented in this logbook as well.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non hazardous aqueous wastes.

14.2.2 Hazardous aqueous wastes

14.2.3 Chlorinated organic solvents

14.2.4 Non-chlorinated organic solvents

14.2.5 Hazardous solid wastes

14.2.6 Non-hazardous solid wastes

15.0 METHOD PERFORMANCE

Method performance (accuracy and precision) is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.
Filtered method blanks and blank spikes to act as QC check of the filters. Unfiltered method blanks and blank spikes are used to monitor overall method performance.

16.0 ADDITIONAL REFERENCES

16.1 Refer to other SOP’s for ICP-MS analysis (EPA 200.8).
16.2 MUR 2007 and 2012
16.3 TNI Standards, 2009
16.4 DoD QSM ver. 5.0, 2013
16.5 WV 47CSR32

TABLE 1
ELEMENTS, MASSES, AND REPORTING LIMIT (LLOQ)FOR THE AGILENT ICP-MS

Mass and Element	Associated Tune (1 = no gas, 2= helium, 3= optimized helium) (this may vary)	CRI (LLCCV) CRI Check (ug/L)	Normal Digested Aqueous Sample Reporting (ug/l) DF 2	Normal Digested Solid Sample Reporting Limit (mg/kg) DF5	Comments
9Be	2	1.0	2.0	0.5	
11B	2	NA	NA	NA	
23Na	2	100	200	25	
24Mg	2	100	200	25	
27Al	2	100	200	25	
39K	2	100	200	25	
44Ca	2	100	200	25	
47Ti	2	1.0	2.0	0.5	
51V	2	1.0	2.0	0.5	
52Cr	2	1.0	2.0	0.5	
55Mn	2	1.0	2.0	0.5	
56Fe	2	100	200	25	
59Co	2	1.0	2.0	0.5	
60Ni	2	1.0	2.0	0.5	
63Cu	2	1.0	2.0	0.5	
66Zn	2	1.0	2.0	0.5	
75As	2	1.0	2.0	0.5	
78Se	2	1.0	2.0	0.5	
88Sr	2	1.0	2.0	0.5	
95Mo	2	1.0	2.0	0.5	
107Ag	2	1.0	2.0	0.5	
111Cd	2	1.0	2.0	0.5	
118Sn	2	1.0	2.0	0.5	
121Sb	2	1.0	2.0	0.5	
137Ba	2	1.0	2.0	0.5	
205Tl	2	1.0	2.0	0.5	
208Pb	2	1.0	2.0	0.5	

TABLE 2:
RECOMMENDED STANDARDS AND ICV AND CCV LEVELS AND NORMAL LINEAR RANGES FOR AGILENT ICP-MS (ug/L)

Mass and Element	STD A	STDB	STDC	STDD	STDE	STDF				NORMAL LINEAR RANGE	ICV	CCV
6Li	0	NA	NA	NA	NA	NA				1000	NA	NA
9Be	0	1.0	10.0	50.0	100.0	200				1000	100	100
11B	0	1.0	10.0	50.0	100.0	200				NA	100	100
23Na	0	100	1000	5000	10000	20000				100000	10000	10000
24Mg	0	100	1000	5000	10000	20000				100000	10000	10000
27Al	0	100	1000	5000	10000	20000				100000	100	100
39K	0	100	1000	5000	10000	20000				100000	10000	10000
44Ca	0	100	1000	5000	10000	20000				100000	10000	10000
47Ti	0	1.0	10.0	50.0	100.0	200				1000	100	100
51V	0	1.0	10.0	50.0	100.0	200				1000	100	100
52Cr	0	1.0	10.0	50.0	100.0	200				1000	100	100
55Mn	0	1.0	10.0	50.0	100.0	200				1000	100	100
56Fe	0	100	1000	5000	10000	20000				100000	10000	10000
59Co	0	1.0	10.0	50.0	100.0	200				1000	100	100
60Ni	0	1.0	10.0	50.0	100.0	200				1000	100	100
63Cu	0	1.0	10.0	50.0	100.0	200				1000	100	100
66Zn	0	1.0	10.0	50.0	100.0	200				1000	100	100
75As	0	1.0	10.0	50.0	100.0	200				1000	100	100
78Se	0	1.0	10.0	50.0	100.0	200				1000	100	100
88Sr	0	1.0	10.0	50.0	100.0	200				1000	100	100
95Mo	0	1.0	10.0	50.0	100.0	200				1000	100	100
107Ag	0	1.0	10.0	50.0	100.0					1000	50	50
111Cd	0	1.0	10.0	50.0	100.0	200				1000	100	100
118Sn	0	1.0	10.0	50.0	100.0	200				1000	100	100
121Sb	0	1.0	10.0	50.0	100.0	200				1000	100	100
137Ba	0	1.0	10.0	50.0	100.0	200				1000	100	100
205Tl	0	1.0	10.0	50.0	100.0	200				1000	100	100
208Pb	0	1.0	10.0	50.0	100.0	200				1000	100	100

TABLE 3
INTERNAL STANDARD MASSES AND ELEMENTS

Mass and Element (this may vary)	Associated Tune for Aglient only (1 = no gas, 2= helium, 3= optimized helium) (this may vary)	Comments
6Li	2	
45Sc	2	
72, 74 Ge	2	
115 In	2	
125 Te	2	
159 Tb	2	
175 Lu	2	
209 Bi	2	

TABLE 4
MS, MSD and BLANK SPIKE CONCENTRATIONS

Element	Soils Final Concentration in mg/kg	Aqueous Final Concentration in μg/l	
Ag	2.5	50	
Al	500	10000	
As	5	100	
B	NA	NA	
Ba	5	100	
Be	5	100	
Ca	500	10000	
Cd	5	100	
Co	5	100	
Cr	5	100	
Cu	5	100	
Fe	500	10000	
K	500	10000	
Mg	500	10000	
Mn	5	100	
Mo	5	100	
Na	500	10000	
Ni	5	100	
Pb	5	100	
Sb	5	100	
Se	5	100	
Tl	5	100	
V	5	100	
Zn	5	100	
Sn	5	100	
Sr	5	100	
Ti	5	100	
Pd	NA	NA	

REVISION HISTORY

Revision Date	Revision Number	Affected Section(s)	Revision Description
12/2020	04	“Revised Sections”	Added Revision History and removed Revised Sections. Sec. 11.10.2 added detail “All samples associated with an out of compliance method blank shall be qualified and footnoted in LIMS as well as the case/run narrative.” METHOD BLANK in Definitions – removed reference to WV blank evaluation.

METALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

Prepared by: David Metzgar III Date: 12/18/2020

Approved by: Svetlana Izosimova Date: 12/18/2020

Annual Review

Reviewed by: Svetlana Izosimova Date: 12/09/2021

Reviewed by: __________ Date: __________

Reviewed by: __________ Date: __________

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Effective 7 days after "*" date

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TITLE: METALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

REFERENCES: SW846 6010D, 2014

INSTRUMENT: THERMO 6500, SERIAL # 20100903 SSTRACE 1
INSTRUMENT: THERMO 6500, SERIAL # 20103825 SSTRACE 2
AUTOSAMPLER: CETAC 240 POSITION, SERIAL # 031038A520 SSTRACE 1
AUTOSAMPLER: CETAC 240 POSITION, SERIAL # 041048A520 SSTRACE 2

SUGGESTED WAVELENGTH (S): TABLE 2

1.0 SCOPE AND APPLICATION SUMMARY

SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

1.1 This method is applicable for the determination of metals in water, sludges, sediments, and soils. Elements that can be reported by this method include: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Titanium, Thallium, Tin, Vanadium, and Zinc.

1.2 Sample matrices are pretreated following SW846 and EPA methods for digestion of soil, sediment, sludge or water samples. Refer to specific metals department digestion SOP's for more information on digestion techniques.

1.3 This inductively coupled argon plasma optical emission spectrometer (s) (ICP-OES) uses an Echelle optical design and a Charge Injection Device (CID) solid-state detector to provide elemental analysis. Control of the spectrometer is provided by PC based iTEVA software. In the instrument, digested samples are introduced into the Thermo 6500 ICP, passed through a nebulizer and transported to a plasma torch. The element-specific emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a spectrometer, and the intensities of the emission lines are monitored with the solid state detector.

1.4 Reporting limits (RL)(LLOQ) are based on the extraction procedure. Reporting limits may vary depending on matrix complications, volumes and by client needs, but the reporting limits must always be verified with a low check which meets the criteria outlined in this SOP. Solid matrices are reported on a dry weight basis. Refer to table 1 of this SOP for

SGS - Orlando typical reporting limits. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific reporting limits.

1.5 MDLs must be established for all analytes, using a solution spiked at approximately 3 to 5 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs must be determined approximately once per year for each matrix and instrument. Please refer to SGS - Orlando QA SOP QA020, current version for further information regarding method performance criteria and experimental method detection limits.

MDLs are generated for each matrix on both ICP instruments. The higher of the two statistically calculated MDL's is entered into LIMS as the MDL. The verified MDLs are stored in the LIMS and must be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.6 LLOQ verification. LLOQ is the lowest point of quantitation. The LLOQ is initially verified by the analysis of 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery should be within +/- 35 percent of the true value with an RSD $\leq$ 20 percent.

1.7 Ongoing Lower limit of quantitation (LLOQ) check sample. The lower limit of quantitation check sample should be analyzed on a quarterly basis to demonstrate the desired detection capability. The LLOQ sample is carried through the entire preparation and analytical procedure. The mean recovery should be within +/- 35 percent of the true value with an RSD $\leq$ 20 percent.

1.8 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

1.9 Instrument Detection Limits (IDL). It is suggested that IDL's be completed upon initial instrument installation, whenever instrument conditions have significantly changed, or at a minimum annually. Instrument detection limits can be estimated as the mean of the blank results plus 3 times the standard deviation of 10 replicate analyses of the reagent blank solution. (use zero for the mean if the mean is negative) Each IDL measurement shall be performed as though it were a separate analytical sample. IDLs shall be determined and reported for each wavelength used in the analysis of the samples.

2.0 PRESERVATION AND BOTTLEWARE

All samples should be preserved with nitric acid to a pH of <2 at the time of collection. All sample pH are checked in sample receiving and within the metals department. Samples that are received with a pH >2 must be preserved to pH <2 and held for 24 hours prior to metals digestion to dissolve any metals that absorb to the container walls. Refer to SOP SAM101, current revision for further instruction. Final pH of TCLP extracts are checked and recorded in SGS - Orlando

Extractions Department. Please refer to TCLP (1311) fluid determination logbook and SPLP (1312) fluid determination logbook for further information. TCLP extracts received from SGS - Orlando Extractions Department are prepared as soon as possible, no longer than 24 hours from time of receipt. If precipitation is observed during the sample preparation process the sample(s) are immediately re-prepped on dilution until no precipitation is observed. Samples received for dissolved metals analysis should be filtered and preserved to pH<2 as soon as possible and held for 24 hours prior to digestion. Refer to SGS - Orlando Sample Filtration Logbook for further information.

All soil samples must be stored in a refrigerator at $\leq$ 6°C upon receipt. Refer to SOP SAM101, current revision for further instruction.

All bottleware used by SGS - Orlando is tested for cleanliness prior to shipping to clients. Analysis results must be less than one half the reporting limit (LLOQ) to be acceptable. Refer to SOP SAM104, current revision for further instruction.

3.0 HOLDING TIME AND BATCH SIZE

All samples must be prepared and analyzed within 6 months of the date of collection. Refer to appropriate SGS - Orlando digestion SOP, current revision for batch size criteria.

4.0 INTERFERENCES

Several types of interferences can cause inaccuracies in trace metals determinations by ICP. These interferences are discussed below.

4.1 Spectral interferences are caused by overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, and background contribution from stray light from the line emission of high concentration elements. Corrections for these interferences can be made by using interfering element corrections, by choosing an alternate analytical line, and/or by applying background correction points. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

Note: Refer to section 17.0 of this SOP for further instruction regarding interfering element correction factor generation.

4.2 Physical interferences can be caused by changes in sample viscosity or surface tension, by high acid content in a sample, or by high dissolved solids in a sample. These interferences can be reduced by making sample dilutions.

4.3 Matrix interferences in high solid samples can be overcome by using an internal standard. Yttrium/Indium mix is used for the Thermo 6500 ICP. The concentration must be sufficient for optimum precision but not so high as to alter the salt concentration of the matrix. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation.

4.4 Chemical interferences are not pronounced with ICP due to the high temperature of the plasma, however if they are present, they can be reduced by optimizing the analytical conditions (i.e. power level, torch height, etc.).

5.0 APPARATUS

5.1 Currently there are two solid state ICPs available for use in the lab. Both are Thermo 6500 ICP units. These units have been optimized to obtain lower detection limits for a wide range of elements. Since they are solid state systems, different lines may be included for elements to obtain the best analytical results. However, the lines which are normally included in the normal analysis program are shown in Table 2.

5.2 Instrument auto samplers. For random access during sample analysis.

5.3 Class A volumetric glassware and pipettes.

5.4 Polypropylene auto sampler tubes.

5.5 Eppendorf Pipette (s) - Pipette (s) are checked daily for accuracy and to ensure they are in good working condition prior to use. Volumes are checked at 100% of maximum volume (nominal volume). Pipettes are checked within the metals department and results are stored electronically in the “Pipette Calibration Log”. Refer to SOP QA006, current revision for further information regarding pipette calibration. BIAS: mean must be within 2% of nominal volume. Precision: RSD must be $\leq$ 1% of nominal volume based on three replicates.

5.6 Fisher Brand 0.45 micron (um) filter or equivalent. Filter lots are checked for cleanliness through the Method Blank process. All Method Blank analytical results must be less than one half the reporting limit(LLOQ) to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated filters must be re-filtered through acceptable filters.

5.7 Fisher Brand disposable 10 ml syringes or equivalent. Syringe lots are checked for cleanliness through the Method Blank process. All Method Blank results must be less than one half the reporting limit (LLOQ)to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated syringes must be re-filtered through acceptable syringes.

5.8 Data System

Microsoft Windows XP Professional Version 2002
Instrument software SST1 – Thermo iTEVA version 2.8.0.89
Instrument software SST2 – Thermo iTEVA version 2.7.0.87

5.8.1 A computer system interfaced to the Thermo 6500 ICP that allows for the continuous acquisition and storage of all data obtained throughout the duration of the analytical run sequence.

5.8.2 Data is archived to a backup server for long term storage.

6.0 REAGENTS

All chemicals listed below are trace metal grade unless otherwise specified. Refer to Acid Certificate of Analysis logbook for Certificates of Analysis and compliance with the specifications of the grade listed. SGS - Orlando produces DI water to the specifications for the ASTM Type II standard designation based on the system manufacturer's performance specifications. The DI water is used exclusively for laboratory purposes. De-ionized (DI) water should be used whenever water is required. Refer to SOP QA037, current revision for more information regarding testing and monitoring. Refer to the Metals Department Standard Prep Logbook for the make-up and concentrations of standards and stock solutions being used within this SOP. Some of the information included in the logbook is as follows: standard name, elements in mix, manufacturer, lot number, parent expiration date, acid matrix, stock concentration, volume of standard added, total volume, final prepared concentration, prep date, initials, MET number, and prepared standard expiration date. Standards and prepared reagents must be prepared every 6 months or before stock standard expiration date, whichever comes first**.** Refer to tables 3 through 7 of this SOP for concentration levels of standards used. Unless otherwise approved, the calibration curve must contain 3 points determined by a blank and a series of standards representing the elements of interest.

6.1 2.5 ppm Yttrium and 10 ppm Indium internal standard, made from ICP quality standard.

6.2 Hydrochloric acid, trace metals grade.

6.3 Nitric Acid, trace metals grade.

6.4 ICP quality standard stock solutions are available from Inorganic Ventures, Spex, Plasma Pure, Ultra, Environmental Express, or equivalent.

6.5 Calibration Standards. These can be made up by diluting the stock solutions to the appropriate concentrations. The calibration standards should be prepared using the same type of acid (s) and at approximately the same concentration as will result in the samples following sample preparation.

6.5.1 For calibration and quantitation an internal standard (Yttrium/Indium) is used to limit nebulization problems. If it is known that the samples contain a significantly different acid matrix, the samples must be diluted so that they are in a similar matrix to the curve. All sample results are referenced to the initial calibration blank (ICB) Internal Standard counts. The criteria is 60-125 percent of the initial calibration blank (ICB) counts. If the internal standard counts fall outside these criteria matrix effects must be suspected and the sample diluted until it meets the criteria or footnoted in LIMS as suspected matrix interference.

6.5.2 Standards must be prepared so that there is minimal spectral interference between analytes.

Note: All Ag stock and intermediate solutions must be stored away from direct sunlight.

6.6 Analytical Quality Control Solutions.

All of the solutions below are prepared by adding either mixed or single element metals solutions to a solution prepared using the same type of acid (s) and at approximately the same concentration as will result in the samples following sample preparation.

6.6.1 Blank (Calibration, ICB, CCB)

This reagent blank contains Nitric Acid at 3 percent and Hydrochloric Acid at 5 percent.

6.6.2 Initial Calibration Verification solution.

This standard solution must be made from a different source than the calibration curve. The concentrations for each element must be within the range of the calibration curve and should be approximately at the midpoint of the curve. This solution is used to verify the accuracy of the initial calibration. Levels for the ICV standard are shown in Table 4.

6.6.3 Continuing Calibration Verification solution.

The metals concentrations for this standard should be at approximately the mid-point of the calibration curve for each element. This standard should be prepared from the same source that is used for the calibration curve. Levels for the CCV standard are shown in Table 5.

6.6.4 Spectral Interference Checks (SIC). Two types of SIC checks are used. Individual element SIC are performed when the instrument is initially set up, and every six months thereafter. The mixed element SIC solution is used daily to check that the instrument is free from interference from elements typically observed in high concentration and to check that interference corrections (IEC) are still valid.

6.6.4.1 Single element interference checks – At a minimum, single element SIC checks should be performed for the following elements: Aluminum 500 mg/l; Barium 4 mg/l; Calcium 500 mg/l; Copper 4 mg/l; Iron 500 mg/l; Magnesium 500 mg/l; Manganese 4 mg/l; Molybdenum 4 mg/l; Sodium 1000 mg/l; Nickel 4 mg/l; Selenium 4 mg/l; Silicon 50 mg/l; Tin 4 mg/l; Vanadium 4 mg/l and Zn 4 mg/l.

Mixed element SIC solution – The mixed element SIC solution is used as an ongoing daily check of freedom from spectral interferences. The mixed element SIC contains the following elements: Aluminum 500 mg/l; Calcium 500 mg/l; Iron 200 mg/l; Magnesium 500 mg/l.

The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be less than 2 times the analytes LLOQ. The concentration of the SIC checks are suggested, but become the highest reportable concentration in the sample analysis and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements. The daily mixed element SIC solution is analyzed daily after calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. For spiked elements, the analyzed results must be within 20 percent of the true value for SIC check and within 10 percent for linear range check. If this criterion cannot be met then sample analysis may not proceed until the problem is corrected, or the LLOQ is raised to twice the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated and documented as contaminants in the SIC solutions. Levels for the SIC and mixed SIC can be found on tables 9 and 10.

6.7 CRIA Standard Solution (Also referred to as LLCCV)

The CRIA standard contains the elements of interest at levels equal to SGS - Orlando quantitation limits (RL). Please refer to Table 6 for list of elements of interest and concentration levels for the CRIA. If special client reporting limits are requested, then low checks corresponding to those reporting limits must also be analyzed.

6.8 Matrix Spike, Matrix Spike duplicate, and Spike Blank Solution.

This solution is prepared by adding either mixed or single element metals solutions to a solution containing 3 percent nitric acid and 5 percent hydrochloric acid and diluting to a fixed final volume with this acid mixture. Spiking solution (s) must be added to the spike blank, matrix spike, and the matrix spike duplicate prior to digestion. Levels for the MS and MSD and Spike Blank standard are shown in Table 7.

6.9 Liquid Argon or Argon Gas. (99.999% purity)

7.0 ANALYTICAL PROCEDURE

Note: Please refer to section 8 of this SOP for further detail on quality control standards. Please refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements.

7.1 General procedure on how to operate the Thermo 6500 is described below. Refer to the Thermo 6500 operation manual for further details.

7.2 Before starting up the instrument, make sure that the pump tubing is in good condition, the torch assembly, the nebulizer, and the spray chamber are clean, the dehumidifier (if used) is filled with DI water up to the level between Minimum and Maximum, and that there are no leaks in the torch area.

7.3 Turn on the recirculating cooler. Verify that the argon is turned on and there is enough for the entire days analytical run.

7.4 Tighten the pump platens and engage the peristaltic pump. Make sure sample and internal standard solutions are flowing smoothly.

7.5 Put a new solution of acid rinse into the rinse reservoir. The composition of the rinse solution may be periodically changed to minimize sample introduction problems and sample carryover. If internal standard is being used, make sure that sufficient amount of internal standard is prepared for the entire analytical run.

7.6 Start up the instrument following the sequence show below.

7.6.1 Double click the **iTEVA Control Center** Icon on the desktop. Type ***admin*** in User Name field, and then click **OK**.

7.6.2 Once the iTEVA Control Center window is opened, click on **Plasma** Icon at status bar area. Then click on **Instrument Status** to check the interlock indicators (torch compartment, purge gas supply, plasma gas supply, water flow and exhaust should be in green; drain flow and busy should be in gray) and the Optics Temperature. (It should be around 38°C.) Click on the Close box.

7.6.3 Click **Plasma On**. When the plasma is on, click close. Let the instrument warm up for 15 to 20 minutes before starting the analysis. New tubing may take an hour to stabilize.

7.7 Torch Alignment and Auto Peak

7.7.1 If the torch has been cleaned, then the torch alignment procedure must be performed.

7.7.2 Open the method and then click on **Sequence** tab, then click on **List View** Icon until you reach rack display.

7.7.3 Go to S-6 position (you can assign any position in the rack for torch alignment), then right click to select **Go** to empty sample S:6. (Now, the auto sampler tip moves from Rinse to this position).

7.7.4 Click on **Analysis** tab, then select **Torch Alignment** from Instrument drop down menu. There will be a pop up dialog box present. Click **Run**. Then there will be another dialog pop up box (This is a reminder for Torch Alignment Solution (2 ppm Zn)), click **Ok**. Now, the instrument is initializing an automated torch alignment. It takes about 7 minutes to complete this step. Progress is indicated in the progress bar.

7.7.5 After torch alignment is complete, click **Close**. Click on **Sequence** tab, then followed by **List View** Icon.

7.7.6 Go to Rinse position at rack display, right click to select Go to rinse and let it rinse for approximately 5 minutes.

7.7.7 Perform Auto Peak

7.7.8 It is recommended that the Auto Peak Adjust procedure be performed daily prior to calibration. A standard that contains all of the lines of interest is used and the system automatically makes the appropriate fine adjustments. (High standard solution should be used for this process.)

7.7.9 Click **Sequence** tab, then click on **List View** Icon until the rack is displayed.

7.7.10 Go to S-5 position (you can assign any position in the rack for auto peak adjust), then right click to select **Go** to empty sample S:5. (Now, the auto sampler tip moves from the Rinse position to this position). Click on **Analysis** tab. All elements result is shown in the display area. From Instrument drop down menu, select **Perform Auto Peak**. There will be a pop up dialog box present. Highlight “All Elements”, and then click **Run**. Then there will another pop up dialog box (This is a reminder for Auto Peak Solution), click **Ok**. Now, the instrument is performing auto peak adjust. It takes about 5 minutes to complete this process. The Auto Peak dialog box will show a green check mark in front of “All Elements”, which indicates Auto Peak is complete.

7.8 Open the method and start up the run.

7.8.1 Click on **Analyst** Icon at the workspace. Go to the method and choose Open from the drop down menu. Select the method with the latest revision number.

7.8.2 Go to **Method** tab at the bottom of left hand corner to click on **Automated Output** at the workspace area. Type a filename in Filename field in the data display area (i.e. : SA101010M1, starts with SA, then followed by MM-DD-YY, then M1; M1 indicates the first analytical run for that day, then followed by M2, M3 and so on for the second and third runs.) Click on **Apply To All Sample Types**.

7.8.3 Click on **Sequence** tab at the bottom of left hand corner. From Auto Session drop down menu bar, click on **New Auto sampler** to create a sequence. This will pop up a dialog box, then click on **New** and fill in number of samples (i.e.: 100) in the Number of Samples field and the sample I.D. (leave this field empty) in Sample Name field. Type a sequence name (i.e. : SEQ101010M1, starts with SEQ, then MM-DD-YY, then M1; M1 indicates the first analytical run for that day, then followed by M2, M3 and so on for the second and third runs) in the Sequence Name field. Click Ok, then put in “0” as settle time between sequences, and click **Ok**.

7.8.4 Right click on **Untitled** (Cetac ASX-520 Enviro 5 Named Rack is the rack that is currently used) at the workspace area, click on **Auto-Locate All** to locate all sample positions.

7.8.5 Double click on **Untitled** again, then click on the sequence name (i.e. : SEQ101010M1), on the data display area, type the sequence in Samplename column, dilution factor (if needed) in CorrFact column, check the box in front of Check column, and select an appropriate check table.

7.8.6 Once done with creating sequence, go to **Method** drop down menu and save all changes as **Save As**. There will be a Save a Method dialog box present, go to the save option to check on "Overwrite Method and bump revision number" box, and then click **Ok**.

7.8.7 Go to Sequence tab, click on List View Icon from tool bar, then click on Connect Autosampler to PC and Initialize Icon.

7.8.8 See table 8 for a typical run sequence.

7.9 Calibrate the instrument as outlined below. See table 3 for calibration standards concentrations. This calibration procedure is done a minimum of once every 24 hours. The calibration standards may be included in the auto sampler program or they may be run manually from the **Calibrate Instrument (graduated cylinder)** icon located on the Analyst tab. The instrument may be calibrated using a single point standard and a calibration blank or a multipoint calibration. If a multipoint calibration is used a minimum of three standards are required. All curves must be determined from a linear calibration prepared in the normal manner using the established analytical procedure for the instrument. Refer to instrument manual for further detail. Three exposures will be used with a percent relative standard deviation of less than 5 percent. The resulting correlation coefficient must be $\geq$0.995. If the calibration curves do not meet these criteria, analysis must be terminated, the problem corrected, and instrument re-calibrated. Correlation coefficients, slopes, and y-intercepts for each wavelength are printed and included in each analytical data package.

7.10 Initial Calibration Verification Standard (ICV).

After each calibration, a standard from a different source than the calibration standard shall be analyzed. For the ICV, all elements to be reported must be within 10 percent of the true value for 6010D. If the ICV is outside these criteria then the analysis must be terminated, problem corrected, and the instrument re-calibrated.

7.11 After analyzing the ICV, the ICB must be analyzed. The results of the ICB must be less than one half the reporting limit (LLOQ). The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a "B" or "V" qualifier, or raising the reporting limit (LLOQ) for all samples to greater than two times the background concentration.

7.12 Before analyzing any real world samples the CRIA (also referred to as LLCCV) must be analyzed. The CRIA contains elements of interest at the reporting limit. The CRIA will be analyzed at the beginning and end of each analytical run. For all elements the results must be within 20 percent of the true value. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific reporting limits (CRIA Requirement). If the initial CRIA fails no samples associated with the failing CRIA can be reported, and the CRIA should be reanalyzed for the failing elements. If the closing CRIA fails the criteria, the samples associated with the CRIA shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the CRIA, or qualifying the results in LIMS.

7.13 Before analyzing any real world samples, the Mixed element SIC solution must be analyzed. The mixed element SIC solution is used as an ongoing daily check of freedom from spectral interferences. The mixed element SIC contains the following elements: Aluminum 500 mg/l; Calcium 500 mg/l; Iron 500 mg/l; Magnesium 500 mg/l.

The daily mixed element SIC solution is analyzed daily after calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. For spiked elements, the analyzed results must be within 20 percent of the true value for SIC check and within 10 percent for linear range check. If this criterion cannot be met then sample analysis may not proceed until the problem is corrected, or the LLOQ is raised to twice the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated and documented as contaminants in the SIC solutions. Refer to section 17.0 of this SOP for Interfering Element Correction (IEC) procedure.

7.14 After the initial analytical quality control has been analyzed, the samples and the preparation batch matrix quality control shall be analyzed. Each sample analysis must be a minimum of 3 readings using at least a 5 second integration time. Between each sample, flush the nebulizer and the solution uptake system with a blank rinse solution for at least 60 seconds or for the required period of time to ensure that analyte memory effects are not occurring.

7.15 Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value for method 6010D, the CCV shall be reanalyzed to confirm the initial value. If the CCV is not within criteria after the reanalysis, no samples can be reported in the area bracketed by the failing CCV. Immediately following the analysis of the CCV the CCB shall be analyzed. The results of the CCB must be less than one half the reporting limit (LLOQ) for all elements. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit (LLOQ) for all samples to greater than two times the background concentration.

7.16 One sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution (SDL) must be prepared. For the serial dilution, a 1:5 dilution must be made on the sample. The results of the 1:5 dilution shall agree within 20 percent of the true value as long as the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 25 times greater than the LLOQ). If the results are outside these criteria then matrix interference should be suspected and the proper footnote entered into LIMS. A post digestion spike (PDS) must be performed if the SDL fails. The PDS must recover within ± 25 percent for method SW846-6010D. If the PDS is outside these limits then matrix interference must be suspected and the proper footnote entered into LIMS.

7.17 The upper limit of quantitation may exceed the highest concentration calibration point and can be defined as the “linear range”. Sample results above the linear range shall be diluted under the linear range and reanalyzed. Following calibration, the laboratory may choose to analyze a standard (or mixed standard solution) at a higher concentration than the high standard used in the calibration curve. The standard must recover within 10 percent of the true value, and if successful, establishes the linear range. The linear range standards must be analyzed in the same instrument run as the calibration they are associated with, but may be analyzed anywhere in the run. Samples following a sample with high concentrations of analyte (s) must be examined for possible carryover. Verification may be done by rinsing the lines with an acid solution and then reanalyzing the sample. A limit check table is built into the autosampler file so that samples exceeding the standardization range are flagged on the raw data.

7.18 After the instrument is optimized and all initial QC has been run, click on **Run Auto-Session** Icon to start the analytical run sequence.

7.18.1 If you need to add or delete samples once the run is started, follow the steps shown below.

7.18.2 Click on **Sequence** tab, then click on **List View** Icon at the tool bar. There is the sequence table shown on the display area.

7.18.3 Click on **Add Samples** Icon. This will pop up a dialog box, and then fill in number of samples that need to be added. Click **Ok**. By doing this, samples will be added to the end of the current sequence without a rack location.

7.18.4 On the Samplename column type in the sample I.D., correction factors, and check tables. **Click on Auto Locate All.**

7.18.5 The added samples will be analyzed at the end of the original sequence run order unless they are assigned a different run order.

7.18.6 Deleting Samples

7.18.7 Click on **Sequence** tab, and then click on **List View** Icon under the sequence display area.

7.18.8 Highlight all samples that need to be deleted and then click on the **Delete Samples** icon.

7.19 When the analysis is completed export the data to LIMS following the procedure outlined below.

7.19.1 Double click on **ePrint** Icon on desktop. There will be a **LEADTOOLS ePRINT** pop up box, click on **Finish Jobs** and **OK** boxes.

7.19.2 Double click the **PDF** Icon on the desktop; the PDF file will be present as Document_#. Right click on that file, select **rename** to change the filename to an assigned analytical run I.D. (i.e.: MA9000). This is the raw data file for MA9000.

7.19.3 Drop the raw data to the **LIMS Data Drop** icon located on the desktop.

7.19.4 By completing the above steps, the raw data (i.e.: MA9000) can be viewed and/or printed from the Raw Data Search function.

7.19.5 Go to **Analysis** tab, right click on sample header, and select export all samples. A pop up dialog box will come up, type in the analytical run I.D. (i.e.: SA101010M1) and click **Ok**. Go to **Lims Export** folder located on the desktop, right click on analytical run and change extension from .TXT to .ICP. Open the analytical file and make any necessary changes, such as deleting any samples that need to be re-run on dilution. **Save** the file. Drop the data file to the **LIMS Data Drop** icon located on the desktop. This will then send the export file to LIMS for review.

7.20 The data can be evaluated by running an automated data evaluation program, which will help to generate quality control summary pages. Each run must be evaluated as quickly as possible to make sure that all required quality control has been analyzed. With each data package include: cover sheet, copies of all prep sheets, autosampler run sequence, dilution sheets, and raw data. Label each folder with MA#, instrument run I.D., instrument used, and date.

7.21 At the end of the analysis day the ICP must be shutdown using the following sequence.

7.21.1 Place the auto sampler tip in the rinse cup and rinse in a mixed solution of approximately 5 percent nitric acid and 5 percent hydrochloric acid for 10 minutes and then in DI water for 20 minutes.

7.21.2 Turn off the plasma by clicking on the **Plasma** Icon and then by clicking **Plasma Off.**

7.21.3 Close all iTeva programs/windows.

7.21.4 Release the tension on the sample pump platens.

7.21.5 Turn off recirculating chiller.

8.0 QUALITY CONTROL

This section outlines the QA/QC operations necessary to satisfy the analytical requirements for method SW846 6010D. Please refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements. Check with the area supervisor or lab manager for any non-compliant quality control for further information.

8.1 Initial Calibration Verification Standard (ICV).

After each calibration, a standard from a different source than the calibration standard shall be analyzed. For the ICV, all elements to be reported must be within 10 percent of the true value for 6010D. If the ICV is outside these criteria then the analysis must be terminated, problem corrected, and the instrument re-calibrated.

8.2 Continuing Calibration Blank/Initial Calibration Blank.

Analyze the Initial calibration blank solution at the beginning of each run and the continuing calibration blank after every tenth sample and at the end of the sample run. The ICB/CCB must be less than one half the reporting limit (LLOQ) for each element. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit (LLOQ) for all samples to greater than two times the background concentration.

8.3 Low Standard Check (CRIA or LLCCV).

Before analyzing any real world samples the CRIA (also referred to as LLCCV) must be analyzed. The CRIA contains elements of interest at the reporting limit. The CRIA will be analyzed at the beginning and end of each analytical run. For all elements the results must be within 20 percent of the true value. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific reporting limits (CRIA Requirement). If the initial CRIA fails no samples associated with the failing CRIA can be reported, and the CRIA should be reanalyzed for the failing elements. If the closing CRIA fails the criteria, the samples associated with the CRIA shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the CRIA, or qualifying the results in LIMS.

8.4 ICSA (Mixed SIC Solution)

Before analyzing any real world samples, the Mixed element SIC solution must be analyzed. The mixed element SIC solution is used as an ongoing daily check of freedom from spectral interferences. The mixed element SIC contains the following elements: Aluminum 500 mg/l; Calcium 500 mg/l; Iron 500 mg/l; Magnesium 500 mg/l.

The daily mixed element SIC solution is analyzed daily after calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. For spiked elements, the analyzed results must be within 20 percent of the true value for SIC check and within 10 percent for linear range check. If this criterion cannot be met then sample analysis may not proceed until the problem is corrected, or the LLOQ is raised to twice the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated and documented as contaminants in the SIC solutions. Refer to section 17.0 of this SOP for Interfering Element Correction (IEC) procedure.

8.5 Continuing Calibration Verification.

Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value for method 6010D the CCV must be reanalyzed to confirm the initial value. If the CCV is not within criteria after reanalysis no samples can be reported in the area bracketed by the failing CCV.

8.6 Method Blank.

The laboratory must digest and analyze a method blank with each batch of samples. The method blank must contain elements at less than one half the reporting limit (LLOQ) for each element. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit. Samples associated with the contaminated blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-digesting and reanalyzing the samples, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit (LLOQ) to greater than two times the background concentration. All samples associated with an out of compliance method blank shall be qualified and footnoted in LIMS as well as the case narrative.

8.7 Blank Spike Sample.

The laboratory must digest and analyze a spike blank sample with each batch of samples. Blank Spikes must be within 20 percent of the true value for method SW846-6010D. If the lab control is outside of the control limits for a reportable element, all samples must be re-digested and reanalyzed for that element. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit (LLOQ). In that case, the sample results may be reported with no flag. For solid standard reference materials (SRMs) ± 20 percent accuracy may not be achievable and the manufacturer’s established acceptance criterion should be used for all soil SRMs.

8.8 Matrix Spike and Matrix Spike Duplicate Recovery.

The laboratory must digest and analyze a matrix spike and matrix spike duplicate with each batch of samples. The matrix spike recovery is calculated as shown below and must be within 20 percent of the true value for method SW846-6010D. If a matrix spike is out of control, then the results must be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and must be footnoted to that effect.

Note: Both the matrix spike amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

$$\frac{\textbf{(Spiked Sample Result - Sample Result)}}{\textbf{Amount Spiked}} \times 100 = \textbf{matrix spike recovery}$$

8.9 Matrix Duplicate/Matrix Spike Duplicate Relative Percent Difference.

The laboratory must digest a duplicate with each batch of samples. The relative percent difference (RPD) between the duplicate and the sample must be ≤ 20 percent for sample results at or above the reporting limit (LLOQ). If the RPD is outside the 20 percent criteria the results must be qualified in LIMS. RPD's are also calculated in LIMS for sample results below the reporting limit (LLOQ). RPD's outside the 20 percent criteria are not considered failing and LIMS automatically footnotes these as "RPD acceptable due to low duplicate and sample concentrations."

Note: Both the duplicate amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

$$\frac{(|\text{Sample Result - Duplicate Result}|) \times 100}{(\text{Sample Result + Duplicate Result})/2} = \text{Duplicate RPD}$$

8.10 Serial Dilution Analysis and Post Digestion Spike.

One sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution (SDL) must be prepared. For the serial dilution, a 1:5 dilution must be made on the sample. The results of the 1:5 dilution shall agree within 20 percent of the true value as long as the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 25 times greater than the LLOQ). If the results are outside these criteria then matrix interference should be suspected and the proper footnote entered into LIMS. A post digestion spike (PDS) must be performed if the SDL fails. The PDS must recover within ± 25 percent for method SW846-6010D. If the PDS is outside these limits then matrix interference must be suspected and the proper footnote entered into LIMS.

$$\frac{(\text{Sample Result - Serial Dil. Result})}{\text{Sample Result}} \times 100 = \text{Serial Dilution RPD}$$

8.11 Linear Calibration ranges.

The upper limit of quantitation may exceed the highest concentration calibration point and can be defined as the "linear range". Sample results above the linear range shall be diluted under the linear range and reanalyzed. Following calibration, the laboratory may choose to analyze a standard (or mixed standard solution) at a higher concentration than the high standard used in the calibration curve. The standard must recover within 10 percent of the true value, and if successful, establishes the linear range. The linear range standards must be analyzed in the same instrument run as the calibration they are associated with, but may be analyzed anywhere in the run. Samples following a sample with high concentrations of analyte (s) must be examined for possible carryover. Verification may be done by rinsing the lines with an acid solution and then reanalyzing the sample. A limit check table is built into the autosampler file so that samples exceeding the standardization range are flagged on the raw data.

8.12 Sample RSD

For samples containing levels of elements greater than five times the reporting limits (LLOQ), the relative standard deviation for the replicates should be less than 5%. If not, reanalyze the sample. If upon reanalysis, the RSD's are acceptable then report the data from the reanalysis. If RSD's are not acceptable upon reanalysis, then the results for that element should be footnoted that there are possible analytical problems and/or matrix interference indicated by a high RSD between replicates.

8.13 Interelement Spectral Interference Correction Validity

For the interelement spectral interference corrections to remain valid during sample analysis, the interferent concentration must not exceed its linear range. If the interferent concentration exceeds its linear range or its correction factor is big enough to affect the element of interest even at lower concentrations, sample dilution with reagent blank and reanalysis is required. In these circumstances, analyte dilution limits are raised by an amount equivalent to the dilution factor.

8.14 Internal Standard (Yttrium/Indium)

For any readings where the internal standard is outside of the range 60-125 percent of the internal standard level in the reference standard (Initial Calibration Blank), then the sample must be diluted until the internal standard is within range and all sample results must be footnoted in LIMS.

8.15 MSA (Method of Standard Additions)

SGS - Orlando uses the internal standard technique as an alternative to the MSA per SW846-6010D section 4.4.2. However, in certain circumstances MSA may be needed by some project specific requirements. SGS - Orlando may perform an MSA when sample matrix interference is confirmed through the post digestion spike process or may qualify the results in LIMS. SGS - Orlando will use a single addition method as described in SW846-7000B.

9.0 GLASSWARE CLEANING

All glassware must be washed with soap and tap water and then rinsed with 5 percent nitric acid. It must then be rinsed at least 3 times with DI water. Refer to SOP GN196, current revision for further information regarding glassware cleaning.

10.0 DOCUMENTATION REQUIREMENTS

Refer to the Laboratory Quality Assurance Manual for documentation requirements. All raw data is printed to .PDF format and archived to a backup server for long term storage.

11.0 SAFETY

The analyst must follow normal safety procedures as outlined in the SGS - Orlando Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor. Follow proper safety precautions when working with gas cylinders.

12.0 CALCULATIONS

For water samples, the following calculations must be used. Refer to the QC section for the calculations to be used for the QC samples.

Original sample concentration of metal (ug/l) =

$$\frac{\text{(conc. in the digestate (ug/l)) x (final digestate volume (ml))}}{\text{(initial sample volume (ml))}}$$

For soil samples, the following calculations must be used.

Concentration of the metal in the dry sample (mg/kg) =

$$\frac{\text{(conc. in the digestate (mg/l) x final digestate volume(L))}}{\text{(sample wt. (kg)) x (\% solids/100)}}$$

13.0 INSTRUMENT MAINTENANCE

Recommended periodic maintenance includes the items outlined below. All maintenance must be recorded in the instrument maintenance log.

13.1 Change the pump tubing as needed.

13.2 Clean the filter on the recirculating pump approximately once a month and dust off the power supply vents as needed.

13.3 Clean or replace the nebulizer, torch assembly, and injector tube as needed.

13.4 Change the sampler tip as needed.

13.5 Clean the recirculating pump lines and internal sock filter every 3 months or as needed.

13.6 Clean the radial view quartz surface weekly or more often if needed.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 14.2.

14.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

15.0 GENERIC DEFINITIONS

15.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours whichever comes first.

15.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

15.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. A CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

15.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

15.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the reporting level.

15.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

15.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the performance of a method in a given sample matrix.

15.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and performance of a method in a given sample matrix.

15.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

15.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

15.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

16.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Statistical control limits are stored in the LIMS for QA purposes only. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

17.0 GENERATION OF INTERFERING ELEMENT CORRECTION FACTORS

17.1 It is recommended that all IEC's be verified and updated approximately every 6 months or whenever instrument conditions change significantly. It is also recommended that elements with frequent high concentrations or with large IEC's should be checked more frequently.

17.2 Calculate the IEC correction factors and enter them into the method (refer to Thermo 6500 instrument manual). Calculate the correction factor using the equation shown below. This correction factor must be added to the correction factor already in place in the method for a given element.

$$\text{IEC} = \frac{\text{Concentration Result of the element with the interference}}{\text{Concentration result of the interfering element}}$$

17.3 Verify the new correction factors by reanalyzing the ICSA/ICSAB solutions and/or the SIC solutions or by reloading and recalculating the previously stored results. If the reanalysis is not within QC limits, make additional changes to the IEC factors and then re-verify both the individual and combined solution values.

17.4 Save and update the method.

17.5 Interfering element correction factors are saved as raw data along with the run printouts on a daily basis so that the IEC's for a given run are traceable.

TABLE 1: REPORTING LIMIT BY ELEMENT

Analyte	Water Reporting Limit(LLOQ) (ug/L)	Soil Reporting Limit(LLOQ)(mg/kg)	TCLP Reporting Limit(LLOQ) (mg/L)/MCL
Tin	50	5	
Aluminum	200	20	
Antimony	5	1	
Arsenic	10	0.5	0.10 / 5.0
Barium	200	20	10 / 100
Beryllium	4	0.5	
Cadmium	5	0.4	0.05 / 1.0
Calcium	1000	500	
Chromium	10	1	0.10 / 5.0
Cobalt	50	5	
Copper	25	2.5	
Iron	300	10	
Lead	5	1	0.5 / 5.0
Magnesium	5000	500	
Manganese	15	1.5	
Nickel	40	4.0	
Potassium	5000	500	
Selenium	10	1	0.5 / 1.0
Silver	10	1	0.10 / 5.0
Sodium	5000	500	
Thallium	10	1	
Vanadium	50	5	
Zinc	20	2	
Molybdenum	50	2.5	
Strontium	10	0.5	
Titanium	10	0.5	

TABLE 2. THERMO 6500 ANALYSIS LINES

Element	Wavelength
Al	396.1
As	189.042
Ca	317.933
Fe	259.9
Mg	279.078
Mn	257.610
Pb	220.353
Se	196.026
Tl	190.864
V	292.402
Ag	328.068
Ba	455.4
Be	313.042
Cd	226.502
Co	228.616
Cr	267.716
Cu	324.753
K	766.491
Na	589.5
Ni	231.604
Sb	206.838
Zn	206.2
Mo	202.030
Sn	189.900
Sr	407.7
Ti	334.9

TABLE 3: LOW, MID AND HIGH STANDARD LEVELS
Single Point Calibration (blank and high standard) may be used

Element	Low	Mid	High
	ug/l	**ug/l**	**ug/l**
Al	10000	40000	80000
As	500	2000	4000
Ca	10000	40000	80000
Fe	10000	40000	80000
Mg	10000	40000	80000
Mn	500	2000	4000
Pb	500	2000	4000
Se	500	2000	4000
Tl	500	2000	4000
V	500	2000	4000
Ag	62.5	250	500
Ba	500	2000	4000
Be	500	2000	4000
Cd	500	2000	4000
Co	500	2000	4000
Cr	500	2000	4000
Cu	500	2000	4000
K	10000	40000	80000
Na	10000	40000	80000
Ni	500	2000	4000
Sb	500	2000	4000
Zn	500	2000	4000
Mo	500	2000	4000
Sn	500	2000	4000
Sr	500	2000	4000
Ti	500	2000	4000

TABLE 4: ICV STANDARD LEVELS

Element	Concentration
	ug/l
Al	40000
As	2000
Ca	40000
Fe	40000
Mg	40000
Mn	2000
Pb	2000
Se	2000
Tl	2000
V	2000
Ag	250
Ba	2000
Be	2000
Cd	2000
Co	2000
Cr	2000
Cu	2000
K	40000
Na	40000
Ni	2000
Sb	2000
Zn	2000
Mo	2000
Sn	2000
Sr	2000
Ti	2000

TABLE 5: CCV STANDARD LEVELS

Element	Concentration
	ug/l
Al	40000
As	2000
Ca	40000
Fe	40000
Mg	40000
Mn	2000
Pb	2000
Se	2000
Tl	2000
V	2000
Ag	250
Ba	2000
Be	2000
Cd	2000
Co	2000
Cr	2000
Cu	2000
K	40000
Na	40000
Ni	2000
Sb	2000
Zn	2000
Mo	2000
Sn	2000
Sr	2000
Ti	2000

TABLE 6: CRIA(LLCCV) STANDARD LEVELS

Element	CRIA
	ug/l
Al	200
As	10
Ca	1000
Fe	300
Mg	5000
Mn	15
Pb	5
Se	5
Tl	10
V	50
Ag	10
Ba	200
Be	5
Cd	5
Co	50
Cr	10
Cu	25
K	5000
Na	5000
Ni	40
Sb	5
Zn	20
Mo	50
Sn	50
Sr	10
Ti	10

TABLE 7: BLANK SPIKE, MATRIX SPIKE AND MATRIX SPIKE DUPLICATE LEVELS

Element	Concentration
	ug/l
Al	27000
As	2000
Ca	25000
Fe	26000
Mg	25000
Mn	500
Pb	500
Se	2000
Tl	2000
V	500
Ag	50
Ba	2000
Be	50
Cd	50
Co	500
Cr	200
Cu	250
K	25000
Na	25000
Ni	500
Sb	500
Zn	500
Mo	500
Sn	500
Sr	500
Ti	500

TABLE 8: TYPICAL RUN SEQUENCE

BLANK
LOW
MID
HIGH
HIGH STD
ICV
ICB
CRIA
ICSA
ICSAB
CCV
CCB
MB
SB
SAMPLE1
DUPLICATE
SERIAL DILUTION
MATRIX SPIKE
MATRIX SPIKE DUPLICATE
POST DIGESTION SPIKE
SAMPLE2
SAMPLE3
CCV
CCB
SAMPLE4
SAMPLE5
SAMPLE6
SAMPLE7
SAMPLE8
SAMPLE9
SAMPLE10
SAMPLE11
SAMPLE12
SAMPLE13
CRIA CLOSING
ICSA CLOSING
ICSAB CLOSING
CCV
CCB

TABLE 9: ICSA (Mixed SIC) SOLUTION LEVELS

Element	Concentration mg/l
Al	500
As	0
Ca	500
Fe	500
Mg	500
Mn	0
Pb	0
Se	0
Tl	0
V	0
Ag	0
Ba	0
Be	0
Cd	0
Co	0
Cr	0
Cu	0
K	0
Na	0
Ni	0
Sb	0
Zn	0
Mo	0
Sn	0
Sr	0
Ti	0

TABLE 10: SINGLE ELEMENT INTERFERENCE CHECK SOLUTION (SIC) LEVELS

Element	Concentration
	mg/l
Al	500
As	0
Ca	500
Fe	500
Mg	500
Mn	4
Pb	0
Se	4
Tl	0
V	4
Ag	0
Ba	4
Be	0
Cd	0
Co	0
Cr	0
Cu	4
K	0
Na	1000
Ni	4
Sb	0
Zn	4
Mo	4
Sn	4
Si	50
Sr	0
Ti	0

REVISION HISTORY

Revision Date	Revision Number	Affected Section(s)	Revision Description
12/2020	05	“Revised Sections”	Added Revision History and removed Revised Sections.

ANALYSIS OF VOLATILE ORGANICS BY GC/MS

Prepared by: Norm Farmer Date: 12/15/2020

Approved by: Melissa Mangual Date: 12/18/2020

Annual Review

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TITLE: ANALYSIS OF VOLATILE ORGANICS BY GC/MS

REFERENCES: SW846 8260D

REVISED SECTIONS: 7.5.1.1, 7.5.1.2, 7.5.1.3, 7.5.2.5 and 11.2

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of various volatile organic compounds in water and solid matrices utilizing a gas chromatograph equipped with a mass spectrometer detector. Routine compounds can be found in Table 1.

1.1.2 The Lower Limit of Quantitation (LLOQ) or Reporting limits (RL) are based on the sample amount and the lowest calibration standard. LLOQs may vary depending on matrix complications and sample volumes. LLOQs for this method are in the range of 1.0-5.0 ug/l for aqueous samples and 5-25 ug/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.3 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the LLOQ. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported LLOQ.

1.1.4 The LLOQ for each analyte is evaluated on an annual basis for each matrix and instrument. The LLOQ verifications are prepared by spiking a clean matrix at 0.5 to 2 times the current LLOQ level. This LLOQ verification is carried through the same preparation and analytical procedures as the samples. Recovery of the analytes should be within the established limits. The DOD QSM requirements for Limit of Detection (LOD) and Limit of Quantitation (LOQ) verifications are different. See SOP QA020 for complete requirements for MDL, LOD, LOQ, and LLOQ.

1.1.5 Compounds detected at concentrations between the LLOQ and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I” qualifier. Some program or project specifications may require that no values below the LLOQ be reported.

1.1.6 For DOD projects refer to QSM 5.0, Table 4; QSM 5.1, Table B-4; or QSM 5.3, Table B-4 for additional method requirements and data qualifying guidance.

1.2 Summary

1.2.1 This method is adapted from SW846 method 8260D.

1.2.2 Samples are received, stored, and analyzed within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with SGS Orlando SOP OP020 and OP021.

1.2.4 The samples are analyzed on a gas chromatograph equipped with mass spectrometer detector.

1.2.5 The peaks detected are identified by comparison to characteristic ions and retention times specific to the known target list of compounds.

1.2.6 Additional unknown peaks with a response > 10% of the closest internal standard may be processed through a library search with comparison to a NIST database of approximately 129,000 compound spectra. An estimated concentration is quantitated by assuming a response factor of 1.

1.2.7 Manual integrations are performed in accordance with SOP QA029.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

Aqueous Samples:

2.1.1 Samples should be preserved to a pH < 2. The pH must be checked and recorded immediately after the sample analysis. If the sample is not preserved to a pH < 2, it must be noted on the report.

2.1.2 If 2-chloroethyl vinyl ether is a compound of concern, the sample should not be preserved. If acrolein and acrylonitrile are compounds of concern, the sample should be adjusted to a pH 4-5 in the field. These analytes may also be analyzed from an unpreserved sample.

2.1.3 The samples must be stored in capped vials, with minimum headspace, at ≤ 6 °C in an area free of solvent fumes. The size of any bubble caused by degassing upon cooling should not exceed 5-6mm.

Solid Samples:

2.1.4 Special 40ml vials for purge-and-trap of solid samples, as well as the collection and preservation options are described in OP020.

2.1.5 Low level soil samples are preserved by storing them in sealed VOA vials at temperatures between –10 °C to –20 °C. High level soil samples are preserved by storing them in methanol at a ratio of 1 gram of soil to 1ml of methanol.

2.2 Holding Time

2.2.1 Aqueous samples are to be analyzed within 14 days of collection, unless otherwise specified by the contract. Samples that are not preserved must be analyzed within 7 days of collection; however, the preservation deficiency must be noted in the report.

2.2.2 Samples that are preserved to pH 4-5 for the analysis of acrolein and acrylonitrile must be analyzed with 7 days of collection.

2.2.3 Solid and waste samples must be analyzed within 14 days of collection.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory blanks. The use of non-TFE tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

3.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank can serve as a check on such contamination.

3.4 Contamination by carry-over can occur whenever high level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for carry-over.

3.5 Acidification with HCl or bisulfate solution may cause the loss of 2-chloroethyl vinyl ether, acrolein, and acrylonitrile.

3.6 Soils and sediment that contain limestone ($CaCO_3$) may react with the sodium bisulfate and effervesce. The effervescing can result in significant losses of volatile organics.

3.7 Certain naturally occurring compounds (humic acids, etc.) will decompose when exposed to the bisulfate solution and form ketones, notably acetone. The amount of acetone formed is extremely matrix dependent but may be produced in significant concentrations.

3.8 The purge efficiency of select fuel oxygenates may be improved by using a heated purge. These fuel oxygenates generally include: methyl tert butyl ether (MTBE), ethyl tert butyl ether (ETBE), tert amyl methyl ether (TAME), di-isopropyl ether (DIPE), tert amyl ethyl ether (TAEE), tert amyl alcohol (TAA), tert butyl alcohol (TBA) and ethanol (ETOH).

3.8.1 Methyl tert butyl ether (MTBE) may be converted to TBA under acidic preservation and elevated purging temperatures.

3.8.2 If samples containing MTBE, TAME, ETBE or other fuel ethers have been preserved with hydrochloric acid and will be analyzed by purging at elevated temperatures, these samples must be adjusted to pH >10 with tri-sodium phosphate dodecahydrate (TSP) prior to initiation of the analysis.

3.8.3 Dibromofluoromethane (surrogate) may degrade and fail low in samples with a basic pH.

3.8.4 Tert butyl formate (TBF) may degrade at elevated purge temperatures.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or samples loaded on an instrument within the same 12-hour shift, whichever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run. For DoD QSM 5.x projects, an additional CCV must be analyzed at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Internal Standards: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Internal standards for mass spec methods are often deuterated forms of target analytes. Internal standards are used to compensate for retention time and response shifts during an analytical run.

4.6 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the reporting level.

4.7 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.12 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.13 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the purge efficiency.

4.14 Trip Blank: A sample of analyte-free matrix taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organic samples.

5.0 REAGENTS

5.1 Reagent water – distilled or deionized water free of interferences

5.2 Methanol – purge-and-trap grade or equivalent

5.3 Hydrochloric acid (HCl) – ACS reagent grade or equivalent

5.4 Sodium Bisulfate Solution – free of interferences

5.5 Tri-sodium phosphate dodecahydrate (TSP) – ACS reagent grade or equivalent

5.6 Inert Gas – UHP Helium or UHP Nitrogen

5.7 Volatile stock standards – Various mixes, traceable to Certificate of Analysis

5.8 4-Bromofluorobenzene (BFB) – instrument tuning mix

5.9 Surrogate standards –

Dibromofluoromethane	1,2-Dichloroethane-d_4
Toluene-d_8	4-Bromofluorobenzene

5.10 Internal standards –

Fluorobenzene	Chlorobenzene-d_5
1,4-Dichlorobenzene-d_4	tert-Butyl alcohol-d_{10}

6.0 APPARATUS

6.1 Gas Chromatograph – Agilent Technologies 6890 or 7890

6.1.1 Gas Chromatograph

The analytical system that is complete with a temperature programmable gas chromatograph and all required accessories, analytical columns, and gases.

6.1.2 The injection port is designed for split-splitless injection with capillary columns. The injection port must have an appropriate interface for sample introduction.

6.2 Mass Spectrometer– Agilent Technologies 5973 or 5975

The mass spectrometer must be capable of scanning from 35-300 amu every second or less utilizing 70-volt (nominal) electron energy in the electron impact ionization mode. It must also be capable of producing a mass spectrum that meets all the criteria in section 7.5.1.1 when injecting 50 ng of Bromofluorobenzene (BFB).

6.3 Purge and Trap – OI Analytical 4660 with OI Analytical 4552 or 4551A or EST Evolution with EST Centurion

6.3.1 The following autosampler models are used for purging, trapping, and desorbing the sample onto GC column.

- O.I. Model 4660 sample concentrator with 4552 Water/Soil multisampler
- O.I. Model 4660 sample concentrator with 4551A Water multisampler
- EST Evolution sample concentrator with Centurion Water/Soil multisampler

6.3.2 The sample purge vessel must be designed to accept 5 or 10ml samples with a water column at least 3 cm deep.

6.3.3 The multisampler is equipped with a heater capable of maintaining the purge chamber at 40 °C to improve purging efficiency. The heater is to be used for soil and sediment analysis.

6.3.4 The desorber must be capable of rapidly heating the trap to the manufacturer recommended desorb temperature.

6.4 Data System – Agilent Technologies MS Chemstation rev. DA 02.0x, DA 03.0x or EA02.0x.

6.4.1 A computer system interfaced to the mass spectrometer that allows for the continuous acquisition and storage of all mass spectral data obtained throughout the duration of the chromatographic program.

6.4.2 The computer utilizes software that allows searching any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP).

6.4.3 The software must allow for integrating the abundances in any EICP between specific time or scan number limits. Characteristic ions for each analyte are listed in Table 3.

6.4.4 The most recent version of the EPA/NIST mass spectral library should be available. Current NIST database contains approximately 129,000 compound spectra.

6.4.5 Data is archived to a backup server for long term storage.

6.5 Trap – OI #10 or equivalent: Tenax, Silica Gel, and Carbon Molecular Sieve.
Trap – Vocarb 3000 (K) or equivalent: Carbopack B, Carboxen 1000, Carboxen 1001

The trap should be conditioned according to the manufacturer ‘s recommendations.

6.6 Columns – RTX-624 or equivalent: 60m X 0.25mm 1.4um.
– RTX-VMS or equivalent: 40m X 0.18mm 1.0um

6.7 Gas-tight syringes and class “A” volumetric glassware for dilutions of standards and samples.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Volatile Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at temperatures between –10 °C and –20 °C, or as recommended by the manufacturer. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the MSVOA STD Summary in the Active SOP directory.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to six months (one month for gases) or the vendor's expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with methanol. The hold time for intermediate standards is one month (one week for gases) or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicates analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the volatile organics are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the LLOQ and the remaining standards define the working range of the detector.

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Instrument Conditions

Gas Chromatograph/ Mass Spectrometer

Carrier gas flow (Helium)	1.0-1.3 ml/min
Transfer line temperature	220 - 280 °C
Analyzer temperature	150 °C

Oven program – 45 °C for 2.5 minutes (RTX-VMS 40m)
10 °C/min to 80 °C for 0 minutes
15 °C/min to 185 °C for 0 minutes
30 °C/min to 240 °C for 2.5 minutes

Oven program – 35 °C for 2.5 minutes (RTX-VMS 40m)
4 °C/min to 60 °C for 0 minutes
25 °C/min to 220 °C for 0 minutes
30 °C/min to 240 °C for 1.2 minutes

Oven program – 45 °C for 2.0 minutes (RTX-624)
10 °C/min to 80 °C for 0 minutes
14 °C/min to 210 °C for 0 minutes
16 °C/min to 240 °C for 4.2 minutes

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Purge and Trap Device conditions

OI Autosampler

Purge Gas:	Helium or Nitrogen at 30-45 ml/min
Sample Temp:	Aqueous (Ambient) Soils (40°C)
Trap Temp:	<25°C
Purge Time:	6 or 11 min
Desorb:	2 min. at 190°C
Bake:	5 min. at 210°C

EST Autosampler

Purge Gas:	Helium at 35-45 ml/min
Sample Temp:	Aqueous (ambient - 35°C) Soils (40°C)
Trap Temp:	<35°C
Purge Time:	6 or 11 min
Desorb:	1-2 min. at 250°C
Bake:	6 min. at 235°C

Purge and Trap conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above

7.4 Sample Preparation

7.4.1 Water Samples

A 5ml aliquot of sample is loaded onto the purge-and-trap device and purged for either 6 or 11 minutes depending on the system. Detailed procedures are described in SOP OP021.

7.4.2 Solid Samples

A 5-gram aliquot of sample is loaded onto the purge-and-trap device. 5mls of reagent water is added along with internal standards and surrogates. Depending on the system, the sample is then purged for either 6 or 11 minutes while heated to 40ºC and mechanically agitated. Detailed procedures are described in SOP OP020.

Alternatively, a methanol aliquot from the sample is loaded onto the purge-and-trap device. 5mls of reagent water is added along with internal standards and surrogates. The sample is then purged for either 6 or 11 minutes depending on the system. Detailed procedures are described in SOP OP020 and OP021.

7.5 Gas Chromatographic Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.5.1 Initial Calibration Procedures

Before samples can be run, the GC/MS system must be tuned, the injection port inertness must be verified, and the instrument must be calibrated.

7.5.1.1 Tune Verification (BFB)

The instrument must be hardware tuned per manufacturer's instructions. Verify the instrument tune by injecting 50ng of BFB solution onto the instrument. The BFB standard may also be purged. The resulting BFB spectra must meet the criteria in the following table.

BFB KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5-9 % of mass 174
176	>95% and <101% of mass 174
177	5-9% of mass 176

Note: Criteria in this Table is tighter than that specified in SW846 8260D.

Evaluate the tune spectrum using three mass scans from the chromatographic peak and a subtraction of instrument background. This procedure is performed automatically by the MS Chemstation software by running “autofind” on the BFB peak.

Select the scans at the peak apex and one to each side of the apex. Calculate an average of the mass abundances from the three scans.

Background subtraction is required. Select a single scan in the chromatogram that is absent of any interfering compound peak and no more than 20 scans prior to the elution of BFB. The background subtraction must be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tuning compound peak.

Alternatively, the average spectra over the entire peak may be used.
All subsequent tune evaluations must use the same procedure that was used for the Initial Calibration.

If the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are met.

Analysis must not begin until the tuning criteria are met. The injection time of the acceptable tune analysis is considered the start of the 12-hour clock. The same mass spec settings must be used for the calibration standards and samples that were used for the tune evaluation standard.

7.5.1.2 Internal Standard Calibration

A minimum 5-point calibration curve is created for the volatile organic compounds and surrogates using an internal standard technique. SGS Orlando routinely performs a 7-point calibration to maximize the calibration range.

NOTE: West Virginia requires that samples preserved with sodium bisulfate be analyzed against a calibration curve that was also preserved with sodium bisulfate. In this instance, 2-chloroethyl vinyl ether and acrolein will not be reportable.

Historically, many analytical methods have relied on linear models of the calibration relationship, where the instrument response is directly proportional to the amount of a target compound. The linear model has many advantages including simplicity and ease of use. However, given the advent of new detection techniques and because many methods cannot be optimized for all the analytes to which they may be applied, the analyst is increasingly likely to encounter situations where the linear model neither applies nor is appropriate. The option of using non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to compensate for detector saturation or avoid proper instrument maintenance.

NOTE: Because of this concern, select programs including SC DHEC do not support the use of non-linear regressions.

The low point may be omitted from the calibration table for any compound with an LLOQ set at the level two standard. Additionally, the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

An entire level may be omitted provided that a minimum of 5 points remain. There must be technical justification to omit an entire level. This must be documented in the run log.

Response factors (RF) for each analyte are determined as follows:

$$RF = (A_{analyte} \times C_{istd})/(A_{istd} \times C_{analyte})$$

$A_{analyte}$ = area of the analyte
A_{istd} = area of the internal standard
$C_{analyte}$ = concentration of the analyte
C_{istd} = concentration of the internal standard.

The mean RF and standard deviation of the RF are determined for each analyte. The percent relative standard deviation (%RSD) of the response factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of RF X 100) / Mean RF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean RF can be used to quantitate target analytes in the samples. **The %RSD should be $\leq$ 15% for any DoD QSM projects.**

Alternatively, a calibration curve of response vs. amount can be plotted. This method allows for the use of average response factors, linear regressions, and non-linear regressions. Linear regressions may be unweighted or weighted as 1/x or $1/x^2$. If the correlation coefficient (r) is ≥ 0.995 ($r^2 \geq 0.990$) then the curve can be used to quantitate target analytes in the samples. Regardless of which calibration model is chosen, the laboratory should visually inspect the curve plots to see how the individual calibration points compare to the plot.

Linear Curve Fit $y = ax + b$

y = response ratio x = concentration ratio

a = linear term b = constant term

Quadratic Curve Fit $y = ax^2 + bx + c$

y = response ratio x = concentration ratio

a = quadratic term b = linear term c = constant term

Either of the two techniques described below may also be used to determine whether the calibration function meets acceptable criteria. These involve refitting the calibration data back to the model. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Use % Error to evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

% ERR = (xi-x'i) / xi * 100

x'_i = Measured amount of analyte at calibration level i, in mass or concentration units.

x_i = True amount of analyte at calibration level i, in mass or concentration units.

Percent error between the calculated and expected amounts of an analyte must be ≤ ±30% for all standards (70-130% of True Value), except the lowest point which must be ≤ ±50% for all standards (50-150% of True Value).

Alternatively, the Relative Standard Error (RSE) may be used to evaluate the difference between the measured and the true amounts or concentrations.

Calculation of Relative Standard Error (%RSE)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x'_i - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

x'_i = Measured amount of analyte at calibration level i, in mass or concentration units.

x_i = True amount of analyte at 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.

The %RSE acceptance limit criterion is ≤20% for good performing compounds and ≤30% for poor performing (PP) compounds.

Table 2 contains minimum RFs that may be used as guidance in determining whether the system is behaving properly and as a check to see if calibration standards are prepared correctly. Because the minimum RFs in Table 2 were determined using specific ions and instrument conditions that may vary, it is neither expected nor required that all analytes meet these minimum RFs. The information in this table is provided as guidance only. The signal to noise ratio at the LLOQ may be a better indication of instrument performance and sensitivity. For a target analyte whose RF <0.01 (response of peak is <1/100 the response of the IS), it is recommended to increase its concentration in relation to other analytes to make the response more comparable.

If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Adjust moisture control parameters, replace analytical trap or column, replace moisture trap or adjust desorb time, and then repeat the calibration procedure.

7.5.1.3 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV must be prepared from a second source at a mid-range concentration.

The %D for all normal analytes of interest should be ≤ 30%, the %D for all poor performing (PP) analytes of interest should be ≤ 40%. (These analytes are identified in Table 1) If the %D > 30% (>40% for PP, quantitative sample analyses should not proceed for those analytes that do not meet the ICV criteria. However, analyses may continue for those analytes that do not meet the criteria with an understanding that these results could be used for screening purposes and would be considered estimated values. Generally, if a reportable analyte is detected in a sample and the %D for that analyte was greater than 30% (>40% for PP) in the ICV, the sample will need to be reanalyzed on a system with a passing ICV for that analyte.

NOTE: For any DoD QSM project, the %D for all target compounds should be ≤ 20%. If samples must be analyzed with an analyte of interest having a %D > 20%, then the data must be qualified accordingly.

If the ICV does not meet criteria, a fresh standard must be prepared. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, analyze an ICV prepared from a third source or different lot. Determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.5.2 Continuing Calibration Verification (CCV)

7.5.2.1 8260D does not require BFB to be evaluated on a daily basis; however, select programs that SGS Orlando participates in do; therefore, this will continue to be a requirement. Inject 2ul of the tune evaluation mix at the beginning of each 12-hour shift. This may also be accomplished by purging a blank that contains BFB. Evaluate the resultant peaks against the criteria in section 7.5.1.1. The injection time of this standard starts the 12-hour window.

When the analyst is running an unattended second 12-hour window, they may opt to purge the BFB standard. This can be performed by purging an additional blank (which contains BFB) just prior to the second CCV.

7.5.2.2 Analyze a continuing calibration check standard. The CCV must be at or below the mid-point of the calibration curve.

7.5.2.3 Table 2 contains minimum RFs from 8260D and SOM 2.3 that may be used as guidance in determining whether the system is behaving properly and as a check to see if calibration standards are prepared correctly. Because the minimum RFs in Table 2 were determined using specific ions and instrument conditions that may vary, it is neither expected nor required that all analytes meet these minimum RFs.

7.5.2.4 The %D for all analytes of interest should be ≤ 20%; however, the large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of this range.

If less than 20 percent of the analytes have a %D > 20% but ≤ 50% then the analysis of samples may still proceed provided that the following criteria is met.

The CCV exceeds the upper limit (+20%) and the analyte is not expected to be present in the samples.

The CCV exceeds the lower limit (-20%) but not more than -50% and the analyte is not expected to be present in the samples. An additional check standard at the LLOQ must be analyzed and the analytes in question be detected and meet all of the qualitative identification criteria.

However, if a reportable analyte is detected in a sample and the %D for that analyte was greater than 20% in the CCV, the sample will need to be reanalyzed on a system with a passing CCV for that analyte, or the data must be qualified.

NOTE: For any DoD QSM project, the %D for all target compounds should be ≤ 20%. If samples must be reported with an analyte of interest having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

7.5.2.5 The criteria in 7.5.2.3 and 7.5.2.4 must be met for the continuing calibration to be considered valid. Only analytes that are being reported for a given sample must meet the criteria in 7.5.2.3 and 7.5.2.4.

If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated. If the second standard meets criteria then the system is considered in control and results may be reported.

Rationale for second standard such as instrument maintenance, clipped column, remade standard, etc. must be documented in the run log or maintenance log. Reanalysis of second standard without valid rationale may require the analysis of a third standard (in which case both the second and third standard would have to pass).

NOTE: For any DoD QSM project, if the second standard meets criteria, then a third standard must be analyzed. If the third standard also meets criteria then the system is considered in control and results may be reported.

If the |%D| is greater than 20%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported, i.e., the CCV failed high, the associated QC passed, and the samples were ND.

NOTE: For any DoD QSM project, if samples must be reported with a target analyte having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

NOTE: Any target analytes that are detected in the samples must have an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed, or the data must be qualified.

7.5.2.6 For DoD QSM 5.x compliance, an additional CCV must be analyzed at the end of each run. The closing CCV must be within the 12-hour Tune window.

The %D for all target compounds in this CCV should be ≤ 50%. If the %D > %50 for any target compound, then the samples must be reanalyzed at least once at the appropriate dilution. If the %D > %50 for the analytes in the reanalysis, the department supervisor shall review the data and determine what further action is necessary. This may include reanalyzing the samples at a higher dilution or qualifying the data.

NOTE: If samples are ND and an analyte in the CCV fails high, then the sample does not need to be reanalyzed.

7.5.2.7 If any of the internal standard response changes by more than a factor of two (-50% to +100%) or retention time changes by more than 10 seconds from the midpoint standard of the last initial calibration or from the daily CCV, the mass spectrometer must be inspected for malfunctions and corrections made, as appropriate. Corrective action may include re-calibration (initial Calibration) of the instrument.

7.5.3 Sample Analysis

7.5.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Tune Evaluation Mix
Initial Calibration Standards (or CCV)
QC Samples
Samples

7.5.3.2 One microliter (OI) or five microliter (EST) of internal standard/ surrogate solution is added to every 5ml of sample in the sparge vessel. Generally, 5ml of sample are transferred to the sparge vessel.

7.5.3.3 After purging, the system will automatically reverse flow and rapidly heat the trap to desorb the sample analytes onto the GC column.

7.5.3.4 Qualitative identification

The target compounds shall be identified by analysts with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. The criteria required for a positive identification are:

The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

The sample component must elute at the same relative retention time (RRT) as the daily standard. The RRT of sample component must be within ± 0.06 RRT units of the standard.

All ions present in the standard mass spectra at a relative intensity greater than 10% (major abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

The relative intensities of these ions must agree within ± 30% between the daily standard and sample spectra, (e.g., for an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%).

Structural isomers that produce very similar mass spectra must 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 25% of sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

If peak identification is prevented by the presence of interferences, the sample must be diluted so that the interference does not mask any analytes.

7.5.3.5 Quantitative analysis

When a target compound has been identified, concentration will be based on the integrated area of the quantitation ion, which is normally the base peak.

The sample matrix may produce an interference with the primary ion. This may be characterized by an excessive background signal of the same ion, which distorts the peak shape beyond a definitive integration. The interference could also, severely inhibit the response of the internal standard ion.

If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that samples be diluted so that the response falls into the middle of the calibration curve.

7.5.3.6 Library search for tentatively identified compounds

If a library search is requested, the analyst should perform a forward library search of the NIST mass spectral library to tentatively identify 10 to 20 non-target compounds.

Guidelines for making tentative identification are:

These compounds should have a response greater than 10% of the nearest internal standard. The response is obtained from the Total Ion Chromatogram.

The search is to include a spectral printout of the best library match for a particular substance. The results are to be interpreted by the analyst.

Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) must be present in the sample spectrum.

The relative intensities of the major ions must agree within ±20%.

Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum must be verified by performing further manual background subtraction to eliminate the interference created by co-eluting peaks and/or matrix interference.

Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 from the nearest internal standard and is to be tabulated on the library search summary data sheet.

7.6 Maintenance and Trouble Shooting

7.6.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.

7.6.2 All instrument maintenance must be documented in the appropriate "Instrument Repair and Maintenance" log. The log will include such items as problem, action taken, correction verification, date, and analyst.

7.6.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

7.6.4 PC and software changes must be documented in the "Instrument Repair and Maintenance" log. Software changes may require additional validation.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Internal Standards

9.1.1 Fluorobenzene, chlorobenzene-d_5, 1,4-dichlorobenzene-d_4, and tert butyl alcohol-d_{10} are used as internal standards for this method. The response of the internal standard in all subsequent runs must be within a factor of two (-50% to +100%) of the internal standard response in the opening CCV for each sequence. On days that an initial calibration is performed, the internal standard responses must be compared to the internal standard responses for the mid-point standard. The response for tert butyl alcohol-d_{10} need only be monitored if target analytes are being reported that reference that particular internal standard.

9.1.2 If the internal standard responses are not within limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, integrations, or internal standards solutions. If errors are found, recalculate the data accordingly.

9.1.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, prepare a second aliquot of sample and reanalyze the sample. If there is insufficient sample for reanalysis, footnote this on the report.

9.1.2.4 If upon reanalysis, the responses are still not within limits, the problem is considered matrix interference. The sample may need to be diluted or the results qualified.

9.1.3 Historical data has shown that when soils are preserved with sodium bisulfate the 4th internal standard (tert butyl alcohol-d_{10}) will very often fail high. If the analytes that reference tert butyl alcohol-d_{10} are non-detect, then the sample does not need to be reanalyzed; however, they must be footnoted with “Associated internal standard response outside of control limits.

9.2 Surrogates

9.2.1 Dibromofluoromethane, 1,2-dichloroethane-d_4, toluene-d_8, and 4-Bromofluorobenzene are used as the surrogate standards to monitor the efficiency of the purge-and-trap system.

A known amount of surrogate standard is added to each sample including the QC set prior to purging. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for all surrogates for the results to be acceptable.

9.2.2 If any surrogate recovery is not within the established control limits, the following are required.

9.2.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, surrogate solutions or internal standard solutions. If errors are found, recalculate the data accordingly.

9.2.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.2.2.3 If no problem is found, reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then reanalysis may not be necessary. For any DoD QSM projects the resulting data must be qualified accordingly. If there is insufficient sample for reanalysis, footnote this on the report.

9.2.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis must be reported on the final report.

9.2.2.5 Historical data has shown that samples with pH > 11 will cause dibromofluoromethane to degrade and fail low. If the remaining surrogates are within control limits, then samples with pH > 11 should be footnoted, but they do not need to be reanalyzed for dibromofluoromethane failures.

9.3 Method Blank

9.3.1 The method blank is de-ionized water or de-ionized water with 5 grams of clean sand (depending upon sample matrix) to which the surrogate standard has been added. An appropriate aliquot of methanol must also be added. The method blank is then purged along with the other samples to determine any contamination from the system or ambient sources. The method blank must be free of any analytes of interest or interferences at ½ the required LLOQ to be acceptable. Common laboratory contaminants such as methylene chloride must be below the LLOQ if present. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.3.2 If the MB is contaminated but the samples are non-detect, then the source of contamination must be investigated and documented. The sample results can be reported. **For any DoD QSM projects the resulting data must be qualified accordingly.**

9.3.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination must be investigated and documented. The samples results may be reported with the appropriate "B" or "V" qualifier. This must be approved by the department supervisor.

9.3.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination must be investigated and documented. The samples must be reanalyzed for confirmation. If there is insufficient sample to reanalyze, or if the sample is reanalyzed beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.4 Blank Spike

9.4.1 The blank spike is de-ionized water or de-ionized water with 5 grams of clean sand (depending upon sample matrix) to which the surrogate standard and spike standard have been added. An appropriate aliquot of methanol must also be added. The blank spike is then processed along with the other samples to monitor the efficiency of the purge-and-trap procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable. The large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of the established control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary.

Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A marginal exceedance in the Blank Spike is defined as a recovery being outside of 3 standard deviations but within 4 standard deviations of the mean.

The number of allowable marginal exceedances is based on the number of analytes in the Blank Spike. Marginal Exceedances must be random. If the same analyte exceeds the BS control limits repeatedly, it is an indication of a systematic problem and corrective action must be taken.

Marginal exceedances are not permitted for analytes that are deemed to be "Compounds of Interest" for a specific project. "Compounds of Concern" are different from "Target Compounds". "Target Compounds" are all analytes that are being reported for a site where "Compounds of Concern" are those analytes expected to be present at the site.

The number of allowable marginal exceedances is as follows:

1) > 90 analytes in BS, 5 analytes allowed in ME range;

2) 71-90 analytes in BS, 4 analytes allowed in ME range;

3) 51-70 analytes in BS, 3 analytes allowed in ME range;

4) 31-50 analytes in BS, 2 analytes allowed in ME range;

5) 11-30 analytes in BS, 1 analyte allowed in ME range;

6) < 11 analytes in BS, no analytes allowed in ME range.

NOTE: SC DHEC does not recognize the concept of Marginal Exceedances. Additionally, a secondary check against 70-130% limits must be performed for all analytes reported to SC DHEC.

9.4.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions or internal standard solutions. If errors are found, recalculate the data accordingly.

9.4.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.4.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary, and the sample results can be reported without qualification.

9.4.2.4 If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable. **For any DoD QSM projects the resulting data must be qualified accordingly.**

9.4.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples or qualifying the results as estimated.

9.4.2.6 If there is insufficient sample to reanalyze, or if the sample is reanalyzed beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.4.2.7 Because of their problematic nature, 2-chloroethyl-vinyl-ether and acrolein are generally not evaluated in the blank spike unless they are of specific concern for a given project.

9.5 Matrix Spike and Matrix Spike Duplicate

9.5.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then processed along with the other samples to monitor the precision and accuracy of the purge-and-trap procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.5.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.5.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions or internal standard solutions. If errors are found, recalculate the data accordingly.

9.5.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.5.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for reanalysis but are an indication of the sample matrix effects.

9.5.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = (| MS Result – MSD Result | / Average Result) X 100

The RPD for each analyte should fall within the established control limits. If more than 33% of the RPDs fall outside of the established control limits, the department supervisor shall review the data and determine if any corrective action is necessary. RPD failures are generally not grounds for batch reanalysis.

10.0 CALCULATIONS

The concentration of each target compound in the original sample is calculated as follows:

Water (ug/l) = $(\text{CONC}_{\text{inst}}) \times \text{DF}$

Soil (ug/kg) = $[(\text{CONC}_{\text{inst}}) \times (5 / W_I)] / \%\text{solids}$ (*low level soils)*

Soil (ug/kg) = $[(\text{CONC}_{\text{inst}}) \times (V_F / V_A) \times (5 / W_I) \times \text{DF}] / \%\text{solids}$ (*high level soils)*

$\text{CONC}_{\text{inst}}$ = Instrument concentration calculated from the initial calibration using mean RF or curve fit.
DF = Dilution Factor
V_F = Volume of methanol extract (ul)
V_A = Volume of methanol aliquot (ul)
W_I = Weight of sample (g)
%solids = Dry weight determination in decimal form

For high level soils, V_F is calculated as follows:

$V_F = \{\text{ml of solvent} + [(\%\text{moisture} \times W_I) / 100]\} \times 1000$ ul/ml

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Program, which includes the use of safety glasses, gloves, and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample must be treated as a potential health hazard. Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment must be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis, methanol extraction, and standards preparation are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Old stock standards are disposed of in the waste vial drum.

Dilution waste and purged aqueous samples and standards are rinsed down the drain with large amounts of water.

Samples are archived and stored for 30 days after analysis. After the storage time has elapsed, the remaining aqueous and soil samples are transferred to the appropriate drums for disposal.

12.0 REFERENCES

SW846 Method 8000D Revision 4, July 2014

SW846 Method 8260D Revision 4, June 2018

EPA CLP SOW for Organics Superfund Methods, SOM02.3, September 2015

TABLE 1

Routine Target Analytes

Dichlorodifluoromethane	**2-Butanone**	n-Propylbenzene
Chloromethane	1,1-Dichloropropene	Bromobenzene
Vinyl Chloride	Propionitrile	1,1,2,2-Tetrachloroethane
Bromomethane	Methacrylonitrile	1,3,5-Trimethylbenzene
Chloroethane	Benzene	2-Chlorotoluene
Trichlorofluoromethane	TAME	**trans-1,4-Dichloro-2-Butene**
Ethyl Ether	1,2-Dichloroethane	1,2,3-Trichloropropane
1,2-Dichlorotrifluoroethane	Trichloroethene	**Cyclohexanone**
1,1-Dichloroethene	Methylcyclohexane	4-Chlorotoluene
Freon 113	Dibromomethane	tert-Butylbenzene
Carbon Disulfide	1,2-Dichloropropane	1,2,4-Trimethylbenzene
Iodomethane	Bromodichloromethane	**Pentachloroethane**
Acrolein	Methyl methacrylate	sec-Butylbenzene
Allyl chloride	**2-Chloroethyl vinyl ether**	4-Isopropyltoluene
Methylene Chloride	cis-1,3-Dichloropropene	1,3-Dichlorobenzene
Acetone	Toluene	1,4-Dichlorobenzene
Methyl acetate	2-Nitropropane	n-Butylbenzene
trans-1,2-Dichloroethene	4-Methyl-2-pentanone	**Benzyl Chloride**
Hexane	trans-1,3-Dichloropropene	1,2-Dichlorobenzene
Methyl Tert Butyl Ether	Tetrachloroethene	**1,2-Dibromo-3-Chloropropane**
Acetonitrile	Ethyl methacrylate	Hexachlorobutadiene
Di-isopropyl ether	1,1,2-Trichloroethane	1,2,4-Trichlorobenzene
Chloroprene	Dibromochloromethane	Naphthalene
1,1-Dichloroethane	1,3-Dichloropropane	1,2,3-Trichlorobenzene
Acrylonitrile	1,2-Dibromoethane	**Ethanol**
ETBE	**2-hexanone**	Tert Butyl Alcohol
Vinyl acetate	1-Chlorohexane	Isobutyl alcohol
cis-1,2-Dichloroethene	Ethylbenzene	Tert Amyl Alcohol
2,2-Dichloropropane	Chlorobenzene	**1,4-Dioxane**
Bromochloromethane	1,1,1,2-Tetrachloroethane	3,3-Dimethyl-1-butanol
Cyclohexane	m,p-Xylene	Tert Amy Alcohol
Chloroform	o-Xylene	**Tert Butyl Formate**
Ethyl acetate	Styrene	1,2,3-Trimethylbenzene
Tetrahydrofuran	Bromoform	
Carbon Tetrachloride	Isopropylbenzene	
1,1,1-Trichloroethane	cis-1,4-Dichloro-2-butene	

Bolded analytes are considered “Poor Performing”

TABLE 2

Recommended Minimum Response Factors

Analyte	8260D Min RF	SOM2.3 Min RF
Dichlorodifluoromethane	0.01	0.01
Chloromethane	0.01	0.01
Vinyl chloride	0.01	0.01
Bromomethane	0.01	0.01
Chloroethane	0.01	0.01
Trichlorofluoromethane	0.01	0.01
Bromochloromethane	0.1	0.1
1,1-Dichloroethene	0.06	0.02
1,1,2-Trichloro-1,2,2-trifluoroethane	0.05	0.01
Acetone	0.01	0.01
Carbon disulfide	0.1	0.01
Methyl Acetate	0.01	0.01
Methylene chloride	0.01	0.01
trans-1,2-Dichloroethene	0.1	0.07
cis-1,2-Dichloroethene	0.2	0.1
Methyl tert-Butyl Ether	0.1	0.01
1,1-Dichloroethane	0.3	0.1
2-Butanone	0.01	0.01
Chloroform	0.3	0.04
1,1,1-Trichloroethane	0.05	0.05
Cyclohexane	0.01	0.1
Carbon tetrachloride	0.1	0.02
Benzene	0.2	0.3
1,2-Dichloroethane	0.07	0.01
Trichloroethene	0.2	0.1
Methylcyclohexane	0.05	0.2

Analyte	8260D Min RF	SOM2.3 Min RF
1,2-Dichloropropane	0.2	0.1
Bromodichloromethane	0.3	0.09
cis-1,3-Dichloropropene	0.3	0.1
trans-1,3-Dichloropropene	0.3	0.01
4-Methyl-2-pentanone	0.03	0.01
Toluene	0.3	0.4
1,1,2-Trichloroethane	0.2	0.04
Tetrachloroethene	0.1	0.1
2-Hexanone	0.01	0.01
Dibromochloromethane	0.2	0.05
1,2-Dibromoethane	0.2	0.01
Chlorobenzene	0.4	0.4
Ethylbenzene	0.4	0.5
meta-/para-Xylene	0.2	0.2
ortho-Xylene	0.2	0.3
Styrene	0.2	0.2
Bromoform	0.1	0.01
Isopropylbenzene	0.4	0.7
1,1,2,2-Tetrachloroethane	0.2	0.05
1,3-Dichlorobenzene	0.5	0.5
1,4-Dichlorobenzene	0.6	0.7
1,2-Dichlorobenzene	0.6	0.4
1,2-Dibromo-3-chloropropane	0.01	0.01
1,2,3-Trichlorobenzene	0.4	0.3
1,2,4-Trichlorobenzene	0.4	0.3

TABLE 3

Characteristic Ions

Analyte	Quant. Ion	Q1	Q2	Q3
Fluorobenzene	96	70		
Dichlorodifluoromethane	85	87		
Chloromethane	50	52		
Vinyl Chloride	62	64		
Bromomethane	94	96	93	
Chloroethane	64	66	49	
Trichlorofluoromethane	101	103		
Ethyl Ether	59	45	74	
1,2-Dichlorotrifluoroethane	67	117	85	69
1,1-Dichloroethene	61	96	98	63
Freon 113	101	151	103	85
Carbon Disulfide	76	44		
Iodomethane	142	127	141	
Allyl chloride	41	39	38	76
Methylene Chloride	49	84	86	51
Acetone	58	43		
Methyl acetate	74	43	42	
trans-1,2-Dichloroethene	61	96	98	63
Hexane	56	57	43	41
Methyl Tert Butyl Ether	73	57	43	41
Acetonitrile	40	41	39	
Di-isopropyl ether	45	43	87	
Chloroprene	53	88	51	50
1,1-Dichloroethane	63	65		
Acrylonitrile	53	52	51	
ETBE	59	87	57	
Vinyl acetate	43	42		
cis-1,2-Dichloroethene	96	61	98	63
2,2-Dichloropropane	77	97		
Bromochloromethane	128	49	130	51
Cyclohexane	56	84	41	55
Chloroform	83	85	47	
Ethyl acetate	43	45		
Tetrahydrofuran	42	41	72	
Dibromofluoromethane	113	111	192	
Carbon Tetrachloride	117	119	121	82
1,1,1-Trichloroethane	97	99	61	
2-Butanone	43	72		

TABLE 3 (cont)

Characteristic Ions

Analyte	Quant. Ion	Q1	Q2	Q3
1,1-Dichloropropene	75	39	110	77
Propionitrile	54	52	55	
Methacrylonitrile	41	39	40	52
Benzene	78	51		
TAME	73	43	55	
1,2-Dichloroethane-d_4	65	67	102	
1,2-Dichloroethane	62	49	64	
Trichloroethene	95	130	97	132
Methylcyclohexane	83	55	98	42
Dibromomethane	93	95	174	172
1,2-Dichloropropane	63	62	41	76
Bromodichloromethane	83	85	47	
Methyl methacrylate	41	69	39	100
2-Chloroethyl vinyl ether	63	43	44	65
cis-1,3-Dichloropropene	75	77	39	
Chlorobenzene-d_5	117	82		
Toluene-d_8	98	100		
Toluene	91	92		
2-Nitropropane	41	43	39	
4-Methyl-2-pentanone	43	58	57	41
trans-1,3-Dichloropropene	75	77	39	49
Tetrachloroethene	166	164	129	131
Ethyl methacrylate	69	41	39	99
1,1,2-Trichloroethane	83	97	61	99
Dibromochloromethane	129	127	131	
1,3-Dichloropropane	76	78	41	39
1,2-Dibromoethane	107	109		
2-hexanone	43	58	57	
1-Chlorohexane	91	55	41	43
Ethylbenzene	91	106		
Chlorobenzene	112	77	114	51
1,1,1,2-Tetrachloroethane	131	133	119	117
m,p-Xylene	91	106	105	
o-Xylene	91	106	105	
Styrene	104	78	103	51
Bromoform	173	175	171	91
Isopropylbenzene	105	120		
1,4-Dichlorobenzene-d_4	152	151		

TABLE 3 (cont)

Characteristic Ions

Analyte	Quant. Ion	Q1	Q2	Q3
4-Bromofluorobenzene	95	174	176	
cis-1,4-Dichloro-2-butene	53	75	88	39
n-Propylbenzene	91	120		
Bromobenzene	156	158		
1,1,2,2-Tetrachloroethane	83	85		
1,3,5-Trimethylbenzene	105	120		
2-Chlorotoluene	91	126	89	
trans-1,4-Dichloro-2-Butene	53	89	75	
1,2,3-Trichloropropane	110	61	112	49
Cyclohexanone	55	98	42	69
4-Chlorotoluene	91	126		
tert-Butylbenzene	91	41	134	
1,2,4-Trimethylbenzene	105	120	119	
1,2,3-Trimethylbenzene	105	120		
Pentachloroethane	167	117	119	165
sec-Butylbenzene	105	134		
4-Isopropyltoluene	119	134		
1,3-Dichlorobenzene	146	111	148	75
1,4-Dichlorobenzene	146	111	148	75
n-Butylbenzene	92	91	134	
Benzyl Chloride	126	91	65	
1,2-Dichlorobenzene	146	111	148	75
1,2-Dibromo-3-Chloropropane	75	155	157	39
Hexachlorobutadiene	225	190	118	260
1,2,4-Trichlorobenzene	180	182	145	109
Naphthalene	128	127		
1,2,3-Trichlorobenzene	180	182	145	109
Tert Butyl Alcohol-d10	65	46		
acrolein	56	55		
Tert Butyl Alcohol	59	41	43	
tert Amyl alcohol	59	55	73	
Isobutyl alcohol	42	43	41	39
1,4-Dioxane	88	58	43	
tert-Butyl Formate	59	57	41	56
Ethanol	45	46		
3,3-Dimethyl-1-butanol	57	69	41	56

ANALYSIS OF SEMIVOLATILE ORGANICS BY GC/MS

Prepared by: Norm Farmer Date: 09/28/2020

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Annual Review

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TITLE: ANALYSIS OF SEMIVOLATILE ORGANICS BY GC/MS

REFERENCES: SW846 8270E

REVISED SECTIONS: 7.4.1.4, 7.4.2.4, 7.4.2.5 and 7.4.3.2.

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of various semivolatile organic compounds in water and solid matrices utilizing a gas chromatograph equipped with a mass spectrometer detector. Routine compounds can be found in Table 1.

1.1.2 The Lower Limits of Quantitation (LLOQ) or Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. LLOQs may vary depending on matrix complications and sample volumes. LLOQs for this method are in the range of 5.0-25.0 ug/l for aqueous samples and 170-850 ug/kg for solid samples. Solid matrices are reported on a dry weight basis.

1.1.3 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the LLOQ. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported LLOQ.

1.1.4 The LLOQ for each analyte is evaluated on an annual basis for each matrix and instrument. The LLOQ verifications are prepared by spiking a clean matrix at 0.5 to 2 times the current LLOQ level. This LLOQ verification is carried through the same preparation and analytical procedures as the samples. Recovery of the analytes should be within the established limits. The DOD QSM requirements for Limit of Detection (LOD) and Limit of Quantitation (LOQ) verifications are different. See SOP QA020 for complete requirements for MDL, LOD, LOQ, and LLOQ.

1.1.5 Compounds detected at concentrations between the LLOQ and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I” qualifier. Some program or project specifications may require that no values below the LLOQ be reported.

1.1.6 For DOD projects refer to QSM 5.0, Table 4; or QSM 5.1, Table B-4; or QSM 5.3, Table B-4 for additional method requirements and data qualifying guidance.

1.2 Summary

1.2.1 This method is adapted from SW846 Method 8270E.

1.2.2 Samples are received, stored, and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with SGS Orlando SOPs OP006 and OP007.

1.2.4 The extracts are analyzed on a gas chromatograph equipped with mass spectrometer detector.

1.2.5 The peaks detected are identified by comparison to characteristic ions and retention times specific to the known target list of compounds.

1.2.6 Additional unknown peaks with a response > 10% of the closest internal standard may be processed through a library search with comparison to an NIST database of approximately 129,000 compound spectra. An estimated concentration is quantitated by assuming a response factor of 1.

1.2.7 Manual integrations are performed in accordance with SOP QA029.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples, and 300ml jars are recommended for solid samples.

2.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be refrigerated at –10°C to –20°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. Interferences from phthalate esters can be eliminated by using plastic-free solvent containers and solvent rinsed glassware.

3.3 Other organic compounds, including chlorinated hydrocarbons, petroleum hydrocarbons, and phthalate esters may be co-extracted by this method.

3.4 Aldol condensation byproducts may form when samples are extracted using solvent mixes containing acetone. Concrete samples in particular are prone to forming aldol condensation byproducts at levels that can interfere with the analysis. Therefore, concrete samples should be extracted with 100% methylene chloride.

3.5 Benzidine and benzaldehyde are extremely reactive in the calibration mix and will quickly break down. Analytes may need to be calibrated separately.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all MS methods, a CCV must be analyzed at the beginning of each analytical run. For DoD QSM 5.x projects, an additional CCV must be analyzed at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Internal Standards: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Internal standards for mass spec methods are often deuterated forms of target analytes. Internal standards are used to compensate for retention time and response shifts during an analytical run.

4.6 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the LLOQ.

4.7 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.12 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.13 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Methylene Chloride – pesticide grade or equivalent

5.2 Semivolatile stock standards – Various mixes, traceable to Certificate of Analysis

5.3 Decafluorotriphenylphosphine mix (DFTPP) – Also contains pentachlorophenol, benzidine, and DDT.

5.4

Base/neutral surrogate standards –	Acid surrogate standards –
Nitrobenzene-d_5	Phenol-d_6
2-Fluorobiphenyl	2-Fluorophenol
p-Terphenyl-d_{14}	2,4,6-Tribromophenol

5.5 Internal standards –

1,4-Dichlorobenzene-d_4	Naphthalene-d_8
Acenaphthene-d_{10}	Phenanthrene-d_{10}
Chrysene-d_{12}	Perylene-d_{12}

6.0 APPARATUS

6.1 Gas Chromatograph – Agilent Technologies 6890 or 7890 with 7683 or 7693 Autosampler

6.1.1 Gas Chromatograph

The analytical system that is complete with a temperature programmable gas chromatograph and all required accessories, analytical columns, and gases.

6.1.2 The injection port is designed for split-splitless or on-column injection with capillary columns.

6.1.3 Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Mass Spectrometer – Agilent Technologies 5973 and 5975

The mass spectrometer must be capable of scanning from 35-500 amu every second or less utilizing a 70-volt (nominal) electron energy in the electron impact ionization mode. It must also be capable of producing a mass spectrum that meets all the criteria in section 7.4.1.1 when injecting 50 ng of Decafluorotriphenylphosphine (DFTPP).

6.3 Data System – Agilent Technologies MS Chemstation rev. DA 02.0x, DA 03.0x or EA 02.0x.

6.3.1 A computer system interfaced to the mass spectrometer that allows for the continuous acquisition and storage of all mass spectral data obtained throughout the duration of the chromatographic program.

6.3.2 The computer utilizes software that allows searching any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP).

6.3.3 The software must allow for integrating the abundances in any EICP between specific time or scan number limits. See Table 2.

6.3.4 The most recent version of the EPA/NIST mass spectral library should be available. Current NIST database contains approximately 129,000 compound spectra.

6.3.5 Data is archived to a backup server for long term storage.

6.4 Column – TG-5MS or equivalent: 30m X 0.25mm X 0.25um
– ZB-Semivolatiles: 30m X 0.25mm X 0.25um

6.5 Gas-tight syringes and class “A” volumetric glassware for dilutions of standards and extracts.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the Semivolatile Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at temperatures between –10 °C and –20 °C, or as recommended by the manufacturer. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the MS STD Summary in the Active SOP directory.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a “Certificate of Analysis” with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor’s expiration date. Once opened, the hold time is reduced to one year or the vendor’s expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with methylene chloride. The hold time for intermediate standards is six months or the vendor’s expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicates analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the semivolatile organics are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the LLOQ, and the remaining standards define the working range of the detector.

Benzidine and benzaldehyde are extremely reactive in the calibration mix and will quickly break down. Fresh standards may need to be made if these analytes are requested.

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 Gas Chromatograph Conditions

Split/Splitless Injection Port

1 or 2ul autosampler injection

Pulsed splitless or 4:1 split injection

Carrier gas – UHP Helium (7.7psi to 40psi @1.5 psi/min ramp pressure)

Injection port temperature – 280 °C   Transfer line temperature – 280 °C

Multimode Injection Port

1 or 2ul autosampler injection

Pulsed 4:1 split injection at 25psi for 0.3 min or 10:1 split injection

Carrier gas – UHP Helium (7.7psi to 38psi @2psi/min ramp pressure)

Injection port temperature – 280 °C   Transfer line temperature – 280 °C

Oven program – 40 °C for 2.0 minutes
20 °C/min to 260 °C for 0 minutes
10 °C/min to 280 °C for 0 minutes
15 °C/min to 320 °C for 1.0 minutes

Source temperature – 230 °C   Quad temperature – 150 °C

GC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Sample Preparation

7.3.1 Water Samples

A 250ml or 1000ml aliquot of sample is pH adjusted and extracted with methylene chloride utilizing separatory funnel extraction. The extract is concentrated to 1.0ml.

7.3.2 Solid Samples

A 30-gram or 15-gram aliquot of sample is extracted with methylene chloride and acetone utilizing pulse sonication or microwave extraction. The extract is concentrated to 1.0ml.

7.4 Gas Chromatographic Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, the GC/MS system must be tuned, the injection port inertness must be verified, and the instrument must be calibrated.

7.4.1.1 Tune Verification (DFTPP)

The instrument must be hardware tuned per manufacturer's instructions. Verify the instrument tune by injecting 50ng of DFTPP solution onto the instrument. The resulting DFTPP spectra must meet the criteria in the following table.

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

Note: Criteria in this Table is tighter than that specified in SW846 8270E.

Evaluate the tune spectrum using three mass scans from the chromatographic peak and a subtraction of instrument background. This procedure is performed automatically by the MS Chemstation software by running “autofind” on the DFTPP peak.

Select the scans at the peak apex and one to each side of the apex. Calculate an average of the mass abundances from the three scans.

Background subtraction is required. Select a single scan in the chromatogram that is absent of any interfering compound peak and no more than 20 scans prior to the elution of DFTPP. The background subtraction must be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tuning compound peak.

All subsequent tune evaluations must use the same procedure that was used for the Initial Calibration.

If the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are met.

Analysis must not begin until the tuning criteria are met. The injection time of the acceptable tune analysis is considered the start of the 12-hour clock. The same mass spec settings must be used for the calibration standards and samples that were used for the tune evaluation standard.

7.4.1.2 Injection Port Inertness Verification

DDT, pentachlorophenol, and benzidine must also be evaluated in the tune standard. These compounds are used to assess injection port inertness and column performance.

Pentachlorophenol and benzidine must be present at their normal responses and, no peak tailing should be visible. The tailing factor for both benzidine and pentachlorophenol must be less than 2.

DDT breakdown must not exceed 20%. Breakdown is calculated as follows:

$$\%DDT_{BREAKDOWN} = \frac{(\text{DDE Area} + \text{DDD Area}) \text{ X } 100}{(\text{DDE Area} + \text{DDD Area} + \text{DDT Area})}$$

If degradation is excessive or peak tailing is noticed, injection port maintenance is required.

This performance test must be passed before any samples or standards are analyzed.

7.4.1.3 Internal Standard Calibration

A minimum 5-point calibration curve is created for the semivolatile organic compounds and surrogates using an internal standard technique. SGS Orlando routinely performs a 6-point calibration to maximize the calibration range.

Historically, many analytical methods have relied on linear models of the calibration relationship, where the instrument response is directly proportional to the amount of a target compound. The linear model has many advantages including simplicity and ease of use. However, given the advent of new detection techniques and because many methods cannot be optimized for all the analytes to which they may be applied, the analyst is increasingly likely to encounter situations where the linear model neither applies nor is appropriate. The option of using non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to compensate for detector saturation or avoid proper instrument maintenance.

NOTE: Because of this concern, select programs including SC DHEC do not support the use of non-linear regressions.

The low point may be omitted from the calibration table for any compound with an LLOQ set at the level two standard. Additionally, the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

An entire level may be omitted provided that a minimum of 5 points remain. There must be technical justification to omit an entire level. This must be documented in the run log.

Response factors (RF) for each analyte are determined as follows:

$RF = (A_{analyte} \mathbf{X} C_{istd})/(A_{istd} \mathbf{X} C_{analyte})$

$A_{analyte}$ = area of the analyte
A_{istd} = area of the internal standard
$C_{analyte}$ = concentration of the analyte
C_{istd} = concentration of the internal standard.

The mean RF and standard deviation of the RF are determined for each analyte. The percent relative standard deviation (%RSD) of the response factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of RF X 100) / Mean RF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean RF can be used to quantitate target analytes in the samples. **The %RSD should be $\leq$ 15% for any DoD QSM projects.**

Alternatively, a calibration curve of response vs. amount can be plotted. This method allows for the use of average response factors, linear regressions, and non-linear regressions. Linear regressions may be unweighted or weighted as $1/x$ or $1/x^2$. If the correlation coefficient is $\geq$0.995 (r^2 $\geq$0.990) then the curve can be used to quantitate target analytes in the samples. Regardless of which calibration model is chosen, the laboratory should visually inspect the curve plots to see how the individual calibration points compare to the plot.

Linear Curve Fit $y = ax + b$

y = response ratio x = concentration ratio

a = linear term b = constant term

Quadratic Curve Fit $y = ax^2 + bx + c$

y = response ratio x = concentration ratio

a = quadratic term b = linear term c = constant term

Either of the two techniques described below may also be used to determine whether the calibration function meets acceptable criteria. These involve refitting the calibration data back to the model. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Use % Error to evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$\% \text{ERR} = (xi - x'i) / xi \; * \; 100$

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at calibration level i, in mass or concentration units.

Percent error between the calculated and expected amounts of an analyte must be ≤ ±30% for all standards (70-130% of True Value), except the lowest point which must be ≤ ±50% for all standards (50-150% of True Value).

Alternatively, the Relative Standard Error (RSE) may be used to evaluate the difference between the measured and the true amounts or concentrations.

Calculation of Relative Standard Error (%RSE)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

x’i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at 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.

The %RSE acceptance limit criterion is ≤20% for good performing compounds and ≤30% for poor performing (PP) compounds.

Table 2 contains minimum RFs that may be used as guidance in determining whether the system is behaving properly and as a check to see if calibration standards are prepared correctly. Because the minimum RFs in Table 2 were determined using specific ions and instrument conditions that may vary, it is neither expected nor required that all analytes meet these minimum RFs. The information in this table is provided as guidance only. The signal to noise ratio at the LLOQ may be a better indication of instrument performance and sensitivity. For a target analyte whose RF <0.01 (response of peak is <1/100 the response of the IS), it is recommended to increase its concentration in relation to other analytes to make the response more comparable.

If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Perform injection port maintenance, replace the column, or clean the source and then repeat the calibration procedure.

7.4.1.4 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV must be prepared from a second source at a mid-range concentration. Because of compound interactions, the ICV for this method is prepared as two separate standards that cover the entire list of compounds.

The %D for all analytes of interest should be ≤ 30%. If the %D > 30% quantitative sample analyses must not proceed for those analytes that do not meet the ICV criteria. However, analyses may continue for those analytes that do not meet the criteria with an understanding that these results could be used for screening purposes and would be considered estimated values. Generally, if a reportable analyte is detected in a sample and the %D for that analyte was greater than 30% in the ICV, the sample will need to be reanalyzed on a system with a passing ICV for that analyte.

NOTE: For any DoD QSM project, the %D for all target compounds should be ≤ 20%. If samples must be analyzed with an analyte of interest having a %D > 20%, then the data must be qualified accordingly.

If the ICV does not meet criteria, a fresh standard must be prepared. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, analyze an ICV prepared from a third source or different lot. Determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

7.4.2 Continuing Calibration Verification (CCV)

7.4.2.1 8270E does not require DFTPP to be evaluated on a daily basis; however, select programs that SGS Orlando participates in do; therefore, this will continue to be a requirement. Inject 1ul of the tune evaluation mix at the beginning of each 12-hour shift. Evaluate the resultant peaks against the criteria in sections 7.4.1.1 and 7.4.1.2. The injection time of this standard starts the 12-hour window.

7.4.2.2. Analyze a continuing calibration check standard. The CCV must be at or below the mid-point of the calibration curve.

7.4.2.3. Table 2 contains minimum RFs from 8270E that may be used as guidance in determining whether the system is behaving properly and as a check to see if calibration standards are prepared correctly. Because the minimum RFs in Table 2 were determined using specific ions and instrument conditions that may vary, it is neither expected nor required that all analytes meet these minimum RFs.

7.4.2.4. The %D for all analytes of interest should be ≤ 20%; however, the large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of this range.

If less than 10 percent of the analytes have a %D > 20% but ≤ 50%, then the analysis of samples may still proceed provided that the following criteria is met.

The CCV exceeds the upper limit (+20%) and the analyte is not expected to be present in the samples.

The CCV exceeds the lower limit (-20%) but not more than -50% and the analyte is not expected to be present in the samples. An additional check standard at the LLOQ must be analyzed and the analytes in question be detected and meet all of the qualitative identification criteria. Only the analytes that failed low in the initial CCV need to be evaluated in the LLOQ check.

However, if a reportable analyte is detected in a sample and the %D for that analyte was greater than 20% in the CCV, the sample will need to be reanalyzed on a system with a passing CCV for that analyte, or the data must be qualified.

NOTE: For any DoD QSM project, the %D for all target compounds should be ≤ 20%. If samples must be reported with an analyte of interest having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

7.4.2.5. The criteria in 7.4.2.3 and 7.4.2.4 must be met for the continuing calibration to be considered valid. Only analytes that are being reported for a given sample must meet the criteria in 7.4.2.3 and 7.4.2.4.

If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated. If the second standard meets criteria, then the system is considered in control and results may be reported.

Rationale for second standard such as instrument maintenance, clipped column, remade standard, etc. must be documented in the run log or maintenance log. Reanalysis of second standard without valid rationale may require the analysis of a third standard (in which case both the second and third standard would have to pass).

NOTE: For any DoD QSM project, if the second standard meets criteria, then a third standard must be analyzed. If the third standard also meets criteria, then the system is considered in control and results may be reported.

If the |%D| is greater than 20%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported, i.e., the CCV failed high, the associated QC passed, and the samples were ND.

NOTE: For any DoD QSM project, if samples must be reported with a target analyte having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

NOTE: Any target analytes that are detected in the samples must have an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed or the data must be qualified.

7.4.2.6. For DoD QSM 5.x compliance, an additional CCV must be analyzed at the end of each run. The closing CCV must be within the 12-hour tune window.

The %D for all target compounds in this CCV should be ≤ 50%. If the %D > %50 for any target compound, the samples may need to be reanalyzed. If samples must be reported with an analyte of interest having a %D >50%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

NOTE: If samples are ND and an analyte in the CCV fails high, then the sample does not need to be reanalyzed.

7.4.2.7. If any of the internal standard response changes by more than a factor of two (-50% to +100%) or retention time changes by more than 30 seconds (10 seconds for DOD QSM 5.x compliance) from the midpoint standard of the last initial calibration or the daily CCV, the mass spectrometer must be inspected for malfunctions and corrections made, as appropriate. Corrective action may include re-calibration (initial calibration) of the instrument.

7.4.3 Sample Extract Analysis

7.4.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Tune Evaluation Mix
Initial Calibration Standards (or CCV)
QC Extracts
Sample Extracts

7.4.3.2 Two microliters of internal standard solution are added to every 100ul of extract in the autosampler vial. Generally, 400ul of extract are transferred to the autosampler vial with a gas tight syringe.

7.4.3.3 One or two microliters (same amount as standards) of extract is injected into the GC by the autosampler. The data system then records the resultant peak responses and retention times.

7.4.3.4 Qualitative identification

The target compounds shall be identified by analysts with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. The criteria required for a positive identification is:

The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

The sample component must elute at the same relative retention time (RRT) as the daily standard. The RRT of sample component must be within ± 0.06 RRT units of the standard.

All ions present in the standard mass spectra at a relative intensity greater than 10% (major abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

The relative intensities of these ions must agree within ± 30% between the daily standard and sample spectra, (e.g., for an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%).

Structural isomers that produce very similar mass spectra must 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 sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

If peak identification is prevented by the presence of interferences, further cleanup may be required, or the extract must be diluted so that the interference does not mask any analytes.

7.4.3.5 Quantitative analysis

When a target compound has been identified, concentration will be based on the integrated area of the quantitation ion, which is normally the base peak.

The sample matrix may produce an interference with the primary ion. This may be characterized by an excessive background signal of the same ion, which distorts the peak shape beyond a definitive integration. The interference could also severely inhibit the response of the internal standard ion.

If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.3.6 Library search for tentatively identified compounds

If a library search is requested, the analyst should perform a forward library search of the NIST mass spectral library to tentatively identify 10 to 20 non-target compounds.

Guidelines for making tentative identification are:

These compounds should have a response greater than 12.5% of the nearest internal standard. The response is obtained from the Total Ion Chromatogram.

The search is to include a spectral printout of the best library match for a particular substance. The results are to be interpreted by the analyst.

Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) must be present in the sample spectrum.

The relative intensities the major ions must agree within ±20%.

Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum must be verified by performing further manual background subtraction to eliminate the interference created by co-eluting peaks and/or matrix interference.

Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 from the nearest internal standard and is to be tabulated on the library search summary data sheet.

7.5 Maintenance and Trouble Shooting

7.5.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.

7.5.2 All instrument maintenance must be documented in the appropriate “Instrument Repair and Maintenance” log. The log will include such items as problem, action taken, correction verification, date, and analyst.

7.5.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

7.5.4 PC and software changes must be documented in the “Instrument Repair and Maintenance” log. Software changes may require additional validation.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD).

9.1 Internal Standards

9.1.1 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12} and Perylene-d_{12} are used as internal standards for this method. The response of the internal standard in all subsequent runs must be within a factor of two (-50% to +100%) of the internal standard response in the opening CCV for each sequence. On days that an initial calibration is performed, the internal standard responses must be compared to the internal standard responses for the mid-point standard.

9.1.2 If the internal standard responses are not within limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, integrations, or internal standards solutions. If errors are found, recalculate the data accordingly.

9.1.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, prepare a second aliquot of extract and reanalyze the sample.

9.1.2.4 If upon reanalysis, the responses are still not within limits, the problem is considered matrix interference. The extract may need to be diluted or the results qualified.

9.2 Surrogates

9.2.1 Nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-d_{14} are used as the base neutral surrogate standards, and phenol-d_5, 2-fluorophenol, and 2,4,6-tribromophenol are used as the acid surrogate standards to monitor the efficiency of the extraction.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for all surrogates for the results to be acceptable.

9.2.2 If any surrogate recovery is not within the established control limits, the following are required. Note: If the samples are being analyzed for only base neutral compounds or only acid compounds, then only the relative surrogates need to be monitored.

9.2.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, surrogate solutions, or internal standard solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.2.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.2.2.3 If no problem is found, re-extract and reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary. **For any DoD QSM projects, the resulting data must be qualified accordingly.** If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.2.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis must be reported on the final report.

9.3 Method Blank

9.3.1 The method blank is either de-ionized water or a mixture of sodium sulfate and clean sand (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high-level samples. The method blank must be free of any analytes of interest or interferences at ½ the required LLOQ to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

9.3.2 If the MB is contaminated but the samples are non-detect, then the source of contamination must be investigated and documented. The sample results can be reported. **For any DoD QSM projects the resulting data must be qualified accordingly.**

9.3.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination must be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

9.3.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination must be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.4 Blank Spike

9.4.1 The blank spike is either de-ionized water or a mixture of sodium sulfate and clean sand (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable. The large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of the established control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary.

Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A marginal exceedance in the Blank Spike is defined as a recovery being outside of 3 standard deviations but within 4 standard deviations of the mean.

The number of allowable marginal exceedances is based on the number of analytes in the Blank Spike. Marginal Exceedances must be random. If the same analyte exceeds the BS control limits repeatedly, it is an indication of a systematic problem and corrective action must be taken.

Marginal exceedances are not permitted for analytes that are deemed to be “Compounds of Concern” for a specific project. “Compounds of Concern” are different from “Target Compounds”. “Target Compounds” are all analytes that are being reported for a site where “Compounds of Concern” are those analytes expected to be present at the site.

The number of allowable marginal exceedances is as follows:

1) > 90 analytes in BS, 5 analytes allowed in ME range;

2) 71-90 analytes in BS, 4 analytes allowed in ME range;

3) 51-70 analytes in BS, 3 analytes allowed in ME range;

4) 31-50 analytes in BS, 2 analytes allowed in ME range;

5) 11-30 analytes in BS, 1 analyte allowed in ME range;

6) < 11 analytes in BS, no analytes allowed in ME range

NOTE: SC DHEC does not recognize the concept of Marginal Exceedances. Additionally, a secondary check against 70-130% limits must be performed for all analytes reported to SC DHEC.

9.4.2 If the blank spike recoveries are not within the established control limits, the following are required:

9.4.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions, or internal standard solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.4.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.4.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary, and the sample results can be reported without qualification.

9.4.2.4 If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable. **For any DoD QSM projects the resulting data must be qualified accordingly.**

9.4.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample, which may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.4.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

9.4.2.7 Because of their problematic nature, benzidine, benzaldehyde, and benzoic acid are generally not evaluated in the blank spike unless they are of specific concern for a given project.

9.5 Matrix Spike and Matrix Spike Duplicate

9.5.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = ([Spike Amount – Sample Amount] / Amount Spiked) X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.5.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.5.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, spike solutions, or internal standard solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.5.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.5.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extraction but are an indication of the sample matrix effects.

9.5.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

$$\text{RPD} = (|\text{ MS Result} - \text{MSD Result }| / \text{Average Result}) \times 100$$

The RPD for each analyte should fall within the established control limits. If more than 33% of the RPDs fall outside of the established control limits, the MS and MSD must be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

10.0 CALCULATIONS

The concentration of each analyte in the original sample is calculated as follows:

$$\text{Water (ug/l)} = (\text{CONC}_{\text{inst}}) \times (V_F / V_I) \times \text{DF}$$

$$\text{Soil (ug/kg)} = ([\text{CONC}_{\text{inst}}] \times [V_F / W_I] \times \text{DF}) / \%\text{solids}$$

$\text{CONC}_{\text{inst}}$ = Instrument concentration calculated from the initial calibration using mean RF or curve fit.
DF = Dilution Factor
V_F = Volume of final extract (ul)
V_I = Volume of sample extracted (ml)
W_I = Weight of sample extracted (g)
%solids = Dry weight determination in decimal form

All soils are reported on a dry weight basis.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Program, which includes the use of safety glasses, gloves, and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample must be treated as a potential health hazard. Material Safety

Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment must be used by all analysts.

11.2 Pollution Prevention

Waste solvents from the sample analysis and standards preparation are collected in waste storage bottles and are eventually transferred to the chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 REFERENCES

SW846 Method 8000D Revision 4, July 2014

SW846 Method 8270E Revision 6, June 2018

TABLE 1: Routine Target Analytes

	Benzyl Alcohol	1,4-Dioxane	5-Nitro-o-toluidine
Benzoic Acid	1,1'-Biphenyl	Diphenylamine	N-Nitrosodiethylamine
Caprolactam	4-Bromophenyl Phenyl Ether	Diphenyl Ether	N-Nitrosodimethylamine
4-Chloro-3-methyl Phenol	Butyl Benzyl Phthalate	1,2-Diphenylhydrazine	N-Nitrosodi-n-butylamine
2-Chlorophenol	Carbazole	Disulfoton	N-Nitrosodi-n-propylamine
4-Chlorophenol	4-Chloroaniline	bis(2-Ethylhexyl)phthalate	N-Nitrosodiphenylamine
2,4-Dichlorophenol	Chlorobenzilate	Ethyl Methanesulfonate	N-Nitrosomethylethylamine
2,6-Dichlorophenol	bis(2-Chloroethoxy)methane	Famphur	N-Nitrosomorpholine
2,4-Dimethylphenol	bis(2-Chloroethyl)ether	Fluoranthene	N-Nitrosopiperidine
2,4-Dinitrophenol	bis(2-Chloroisopropyl)ether	Fluorene	N-Nitrosopyrrolidine
4,6-Dinitro-o-cresol	1-Chloronaphthalene	Hexachlorobenzene	4-Nitroquinoline 1-Oxide
Dinoseb	2-Chloronaphthalene	Hexachlorobutadiene	n-Octadecane
2-Methylphenol	4-Chlorophenyl Phenyl Ether	Hexachlorocyclopentadiene	Octamethylcyclotetrasiloxane
3&4-Methylphenol	Chrysene	Hexachloroethane	O,O,O-Triethyl Phosphorothioate
2-Nitrophenol	n-Decane	**Hexachlorophene**	Parathion
4-Nitrophenol	Decamethylcyclopentasiloxane	Hexachloropropene	Pentachlorobenzene
Pentachlorophenol	Diallate		Pentachloroethane
Phenol	Dibenz(a,h)acridine	Indeno(1,2,3-cd)pyrene	Pentachloronitrobenzene
2,3,4,6-Tetrachlorophenol	Dibenz(a,j)acridine	Isodrin	
2,4,5-Trichlorophenol	Dibenzo(a,h)anthracene	Isophorone	Phenacetin
2,4,6-Trichlorophenol	Dibenzofuran	Isosafrole	Phenanthrene
Acenaphthene	1,2-Dichlorobenzene	**Kepone**	**p-Phenylenediamine**
Acenaphthylene	1,3-Dichlorobenzene	Methapyrilene	Phorate
Acetophenone	1,4-Dichlorobenzene	3-Methylcholanthrene	2-Picoline
2-Acetylaminofluorene	3,3'-Dichlorobenzidine		Pronamide
4-Aminobiphenyl	Diethyl Phthalate	4,4'-Methylenebis(2-chloroaniline)	
	Dimethoate	Methyl Methanesulfonate	Pyrene
Aniline	p-(Dimethylamine)azobenzene	1-Methylnaphthalene	Pyridine
Anthracene	7,12-Dimethylbenz(a)anthracene	2-Methylnaphthalene	
Aramite	3,3'-Dimethylbenzidine	Methyl Parathion	Safrole
Atrazine	Dimethylnaphthalenes (total)	Naphthalene	Simazine
Azobenzene	**A,A-Dimethylphenethylamine**	1,4-Naphthoquinone	alpha-Terpineol
Benzaldehyde	Dimethyl Phthalate	1-Naphthylamine	1,2,4,5-Tetrachlorobenzene
Benzidine	Di-n-butyl Phthalate	2-Naphthylamine	Tetraethyl dithiopyrophosphate
Benzo(a)anthracene	Di-n-octyl Phthalate		Thionazin
Benzo(a)pyrene	m-Dinitrobenzene	2-Nitroaniline	Toluene-2,4-diamine
Benzo(b)fluoranthene	p-Dinitrobenzene	3-Nitroaniline	o-Toluidine
Benzo(g,h,i)perylene	2,4-Dinitrotoluene	4-Nitroaniline	1,2,4-Trichlorobenzene
Benzo(k)fluoranthene	2,6-Dinitrotoluene	Nitrobenzene	sym-Trinitrobenzene

Bolded analytes are considered "Poor Performing"

TABLE 2: Minimum Response Factors

Analyte	Min. RF
Benzaldehyde	0.01
Phenol	0.80
Bis(2-chloroethyl)ether	0.70
2-Chlorophenol	0.80
2-Methylphenol	0.70
2,2'-Oxybis-(1-chloropropane)	0.01
Acetophenone	0.01
4-Methylphenol	0.60
N-Nitroso-di-n-propylamine	0.50
Hexachloroethane	0.30
Nitrobenzene	0.20
Isophorone	0.40
2-Nitrophenol	0.10
2,4-Dimethylphenol	0.20
Bis(2-chloroethoxy)methane	0.30
2,4-Dichlorophenol	0.20
Naphthalene	0.70
4-Chloroaniline	0.01
Hexachlorobutadiene	0.01
Caprolactam	0.01
4-Chloro-3-methylphenol	0.20
2-Methylnaphthalene	0.40
Hexachlorocyclopentadiene	0.05
2,4,6-Trichlorophenol	0.20
2,4,5-Trichlorophenol	0.20
1,1'-Biphenyl	0.01
2-Chloronaphthalene	0.80
2-Nitroaniline	0.01
Dimethyl phthalate	0.01
2,6-Dinitrotoluene	0.20
Acenaphthalene	0.90
3-Nitroaniline	0.01
Acenaphthene	0.90
2,4-Dinitrophenol	0.01
4-Nitrophenol	0.01
Dibenzofuran	0.80
2,4-Dinitrotoluene	0.20
Diethyl phthalate	0.01
1,2,4,5-Tetrachlorobenzene	0.01
4-Chlorophenyl-phenyl ether	0.40
Fluorene	0.90
4-Nitroaniline	0.01
4,6-Dinitro-2-methylphenol	0.01
4-Bromophenyl-phenyl ether	0.10
N-Nitrosodiphenylamine	0.01
Hexachlorobenzene	0.10
Atrazine	0.01
Pentachlorophenol	0.05
Phenanthrene	0.70
Anthracene	0.70
Carbazole	0.01
Di-n-butyl phthalate	0.01
Fluoranthene	0.60
Pyrene	0.60
Butyl benzyl phthalate	0.01
3,3'-Dichlorobenzidine	0.01
Benzo(a)anthracene	0.80
Chrysene	0.70
Bis-(2-ethylhexyl)phthalate	0.01
Di-n-octyl phthalate	0.01
Benzo(b)fluoranthene	0.70
Benzo(k)fluoranthene	0.70
Benzo(a)pyrene	0.70
Indeno(1,2,3-cd)pyrene	0.50
Dibenz(a,h)anthracene	0.40
Benzo(g,h,I)perylene	0.50
2,3,4,6-Trichlorophenol	0.01

Note: 2,4-Dinitrophenol and 4-Nitorphenol will also need to have an average RF greater than 0.050 for any DOD QSM projects.

TABLE 3: Characteristic Ions

Analyte	Quant. Ion	Q1	Q2
Benzoic Acid	105	122	77
2-Chlorophenol	128	64	130
4-Chloro-3-methyl phenol	107	144	142
2,4-Dichlorophenol	162	164	98
2,4-Dimethylphenol	107	121	122
2,4-Dinitrophenol	184	63	154
2,6-Dichlorophenol	162	164	98
4,6-Dinitro-o-cresol	198	51	105
2-Methylphenol	108	107	77
3&4-Methylphenol	108	107	79
2-Nitrophenol	139	65	109
4-Nitrophenol	109	39	65,139
Pentachlorophenol	266	264	268
Phenol	94	65	66
2,3,4,6-Tetrachlorophenol	232	230	131
2,4,5-Trichlorophenol	196	198	200
2,4,6-Trichlorophenol	196	198	200
Acenaphthene	153	152	154
Acenaphthylene	152	151	153
Acetophenone	105	120	106
Aniline	93	66	65
Anthracene	178	179	176
Atrazine	200	58	215
Benzaldehyde	106	77	105
Benzidine	184	92	185
Benzo(a)anthracene	228	226	229
Benzo(a)pyrene	252	253	125
Benzo(b)fluoranthene	252	253	125
Benzo(g,h,i)perylene	276	138	277
Benzo(k)fluoranthene	252	253	125
4-Bromophenyl phenyl ether	248	250	141
Butyl benzyl phthalate	149	91	206
Benzyl Alcohol	108	79	77
1,1'-Biphenyl	154	153	152
2-Chloronaphthalene	162	164	127
4-Chloroaniline	127	129	65
Caprolactam	113	55	85
Carbazole	167	166	139
Chrysene	228	226	229

Analyte	Quant. Ion	Q1	Q2
bis(2-Chloroethoxy)methane	93	95	123
bis(2-Chloroethyl)ether	93	63	95
bis(2-Chloroisopropyl)ether	45	77	121
4-Chlorophenyl phenyl ether	204	206	141
1,2-Dichlorobenzene	146	148	111
1,2-Diphenylhydrazine	77	105	182
1,3-Dichlorobenzene	146	148	111
1,4-Dichlorobenzene	146	148	111
2,4-Dinitrotoluene	165	89	63
2,6-Dinitrotoluene	165	89	63
3,3'-Dichlorobenzidine	252	254	126
Dibenzo(a,h)anthracene	278	139	279
Dibenzofuran	168	139	169
m-Dinitrobenzene	168	50	76
Di-n-butyl phthalate	149	150	104
Di-n-octyl phthalate	149	150	43
Diethyl phthalate	149	177	150
Dimethyl phthalate	163	194	164
1,4-Dioxane	88	58	43
bis(2-Ethylhexyl)phthalate	149	167	279
Fluoranthene	202	101	203
Fluorene	166	165	167
Hexachlorobenzene	284	142	249
Hexachlorobutadiene	225	223	227
Hexachlorocyclopentadiene	237	235	272
Hexachloroethane	117	201	119
Indeno(1,2,3-cd)pyrene	276	138	277
Isophorone	82	138	95
1-Methylnaphthalene	142	141	115
2-Methylnaphthalene	142	141	115
2-Nitroaniline	65	92	138
3-Nitroaniline	138	108	92
4-Nitroaniline	138	92	108
Naphthalene	128	129	127
Nitrobenzene	77	123	65
N-Nitrosodimethylamine	42	74	43
N-Nitroso-di-n-propylamine	70	42	130
N-Nitrosodiphenylamine	169	168	167
Phenanthrene	178	179	176

TABLE 3: Characteristic Ions (continued)

Analyte	Quant. Ion	Q1	Q2
Pyrene	202	101	203
Pyridine	79	52	
1,2,4,5-Tetrachlorobenzene	216	214	218
1,2,4-Trichlorobenzene	180	182	145
Simazine	201	186	173
alpha-Terpineol	59	93	121
2-Acetylaminofluorene	181	223	152
4-Aminobiphenyl	169	168	141
Aramite	185	63	135
1-Chloronaphthalene	162	164	127
Chlorobenzilate	137	251	111
7,12-Dimethylbenz(a)anthracene	256	241	120
Diallate	43	86	234
Dibenz(a,j)acridine	279	139	125
Diphenyl ether	170	141	77
p-(Dimethylamine)azobenzene	120	225	77
3,3'-Dimethylbenzidine	212	213	106
Ethyl methanesulfonate	79	109	97
A,A-Dimethylphenethylamine	58	91	134
Hexachlorophene	196	198	209
Hexachloropropene	213	211	215
Isodrin	193	195	66
Isosafrole	162	131	104
Kepone	272	274	237
3-Methylcholanthrene	268	252	126
Methyl methanesulfonate	80	79	65
Methapyrilene	58	97	72
1,4-Naphthoquinone	158	102	76
1-Naphthylamine	143	115	116
2-Naphthylamine	143	115	116
5-Nitro-o-toluidine	152	77	106
4-Nitroquinoline 1-Oxide	190	160	89
N-Nitrosodi-n-butylamine	84	57	41
N-Nitrosodiethylamine	102	42	44
N-Nitrosomethylethylamine	42	88	43
N-Nitrosomorpholine	56	86	116
N-Nitrosopiperidine	42	114	55
N-Nitrosopyrrolidine	100	41	42
p-Phenylenediamine	108	80	107

Analyte	Quant. Ion	Q1	Q2
2-Picoline	93	66	92
Pentachlorobenzene	250	248	252
Pentachloroethane	167	119	117
Pentachloronitrobenzene	237	214	142
Phenacetin	108	109	179
Pronamide	173	175	145
Safrole	162	131	104
o-Toluidine	106	107	77
sym-Trinitrobenzene	75	74	213
Benzenethiol	110	66	109
Dibenz(a,h)acridine	279	139	280
Indene	116	115	63
6-Methyl Chrysene	242	241	239
Quinoline	129	102	128
Dimethoate	87	83	125
Disulfoton	88	97	142
Famphur	218	125	93
Methyl parathion	109	125	263
O,O,O-Triethyl phosphorothioate	198	121	97
Parathion	97	109	291
Phorate	75	121	97
Sulfotep	97	202	322
Thionazin	97	107	143
Resorcinol	110	81	53
Nicotine	84	133	42
2,4-Diaminotoluene	121	122	94
MOCA	231	266	140
1,4-Dichlorobenzene-d4	152	115	150
Naphthalene-d8	136	68	
Acenaphthene-d10	164	162	160
Phenanthrene-d10	188	94	80
Chrysene-d12	240	120	236
Perylene-d12	264	260	265
2-Fluorophenol	112	64	
Phenol-d5	99	42	71
Nitrobenzene-d5	82	128	54
2-Fluorobiphenyl	172	171	
2,4,6-Tribromophenol	330	332	141
Terphenyl-d14	244	122	212

Some analytes listed may not be applicable to this method due to certification requirements.

STANDARD OPERATING PROCEDURE FOR THE TOXICITY CHARACTERISTIC LEACHING OF SEMIVOLATILE ORGANICS AND METALS (TCLP)

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Annual Review

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TITLE: STANDARD OPERATING PROCEDURE FOR THE TOXICITY CHARACTERISTIC LEACHING OF SEMIVOLATILE ORGANICS AND METALS (TCLP)

REFERENCES: SW846 1311

REVISED SECTIONS: 1.1, 1.2.7, 3.1.1, 3.2.2, 3.2.6, 5.8, 6.3, 6.4, 6.9, 6.13, 7.4.1, 7.5.1, 7.5.4, 7.5.10.6, 7.5.12, 8.1.3 and 8.1.4

1.0 SUMMARY, SCOPE AND APPLICATION

1.1 Summary

For liquid and aqueous samples containing less than 0.5% solids, the sample is filtered through TCLP filter paper and the filtrate is defined as the TCLP leachate. The leachate can then be analyzed for semivolatile organics and metals.

For solid samples, the solid portion of the sample is extracted by adding extraction fluid equal to 20 times the weight of the sample and rotating the sample for 18 hours at 30 rpm. The extraction fluid used is based on the alkalinity of the solid portion of the sample. After leaching, the sample is filtered through TCLP filter paper. The leachate can then be analyzed for semivolatile organics and metals.

With several modifications the TCLP leaching can be applied to the analysis of PFAS (Perfluoroalkyl substances) in solid samples. These modifications revolve around eliminating or minimizing contact with PTFE coated surfaces or glass surfaces. PTFE may leach artifacts that interfere with the analysis and the PFAS analytes tend to adhere to glass surfaces. Modifications include using HDPE bottles instead of PTFE coated HDPE or Glass Bottles with PTFE lined caps; preparing the TCLP fluids in HDPE bottles or carboys; and using DI water from the PFAS prep lab.

1.2 Scope and Application

This procedure is applicable to samples submitted for TCLP semivolatile analysis and/or TCLP metals analysis.

1.2.1 Metals by 6010

1.2.2 Mercury by 7470

1.2.3 Semivolatiles by 8270

1.2.4 Pesticides by 8081

1.2.5 Herbicides by 8151

1.2.6 Extractable TPH by 8015

1.2.7 Perfluoroalkyl Substances (PFAS)

2.0 DISCUSSION AND COMMENTS

This procedure is adapted from SW-846 method 1311. The method utilizes an extraction bottle and rotary agitation device to evaluate the presence and mobility of semivolatile analytes and metals. It is not applicable for evaluating the mobility of volatile analytes.

3.0 PRESERVATION AND HOLDING TIMES

3.1 Preservation

3.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples. Liquid samples for the analysis of metals only may be collected in HDPE bottles.

Solid samples for TCLP PFAS analysis shall be collected in HDPE bottles. 4oz jars are recommended for solid samples.

3.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until leaching.

3.1.3 Samples for TCLP analysis should not be chemically preserved prior to leaching. After filtration, the TCLP leachate for metals should be preserved to a pH <2 with nitric acid unless the leachates will be digested immediately.

3.1.4 TCLP Leachates for semivolatile organics must be protected from light and stored at ≤ 6°C from the time of filtration until extraction.

3.2 Holding Time

3.2.1 Samples submitted for the analysis of semivolatile organics including pesticides and herbicides must be leached within 14 days of collection.

3.2.2 Samples submitted for the analysis of PFAS must be leached within 14 days of collection.

3.2.3 Samples submitted for the analysis of mercury must be leached within 28 days of collection.

3.2.4 Samples submitted for the analysis of metals (except mercury) must be leached within 180 days of collection.

3.2.5 Leachates for semivolatile organics must be extracted by the appropriate procedure within 7 days of filtration.

3.2.6 Leachates for PFAS analysis must be extracted within 28 days of filtration.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Leachate Blank Spike (LBS): An aliquot of TCLP fluid spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Leachate blank spike recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.8 Leachate Blank (LB): An aliquot of TCLP fluid to which all reagents are added in the same volumes or proportions as used in sample processing. The leachate blank is processed simultaneously with the samples through all the steps of the analytical procedure. The Leachate blank is used to document contamination resulting from the analytical process.

4.9 Leachate Spike (LS): A sample leachate aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The leachate spike recoveries are used to document the bias of a method in a given sample matrix.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Reagent water – distilled or deionized - free of interferences

5.2 Hydrochloric Acid, 1.0N Fisher brand or equivalent

5.3 Sodium Hydroxide, 10.0N Fisher brand or equivalent

5.4 Glacial Acetic Acid, Reagent Grade

5.5 Extraction Fluid #1: Add 57 ml of glacial acetic acid and 64.3 ml of 10.0N sodium hydroxide to a 1000ml graduated cylinder and dilute to 1 liter with reagent water. Transfer to a PTFE lined carboy. Using a 1000ml graduated cylinder, transfer an additional 9 liters of reagent water to the carboy. Mix thoroughly. This will prepare 10 liters of Fluid #1. The preparation of all TCLP fluids must be logged in the Organics Reagent Logbook and the pH must be verified prior to use. The pH of this solution should be 4.93 ± 0.05.

NOTE: If the pH of the fluid is out of range, remake the fluid.

5.6 Extraction Fluid #2: Add 57 ml of glacial acetic acid to a 1000ml graduated cylinder and dilute to 1 liter with reagent water. Transfer to a PTFE lined carboy. Using a 1000ml graduated cylinder, transfer an additional 9 liters of reagent water to the carboy. Mix thoroughly. This will prepare 10 liters of Fluid #2. The preparation of all TCLP fluids must be logged in the Organics Reagent Logbook and the pH must be verified prior to use. The pH of this solution should be 2.88 ± 0.05.

NOTE: If the pH of the fluid is out of range, remake the fluid.

5.7 Buffer solution at pH 2, pH 4, pH 7 and pH 10. Commercially available solutions that have been validated by comparison to NIST standards are recommended for routine use. All buffers must be labeled on receipt and after opening. Buffer solutions should be refreshed weekly. **When analyzing samples from West Virginia the buffers must be refreshed on the day of use.**

5.8 Fluids for PFAS leaching: Either Fluid #1 or #2. Water should come from the PFAS prep lab and fluids must be stored in HPDE carboys. The PFTE lined carboys may leach PFAS analytes over time

6.0 GLASSWARE AND APPARATUS

6.1 Agitation apparatus – Environmental Express, Millipore Corp., or equivalent. Must be capable of rotating the extraction vessels in an end-over-end fashion at 30 ± 2 rpm.

6.2 Extraction Vessels – 2.2-liter PTFE coated HDPE bottles OR 2.2 liter Amber glass bottles. Bottles must have enough volume for the 100 gram sample and 2.0 liters of leaching fluid.

6.3 Extraction Vessels for PFAS – 2.2-liter HDPE. PTFE coated bottles cannot be used.

6.4 Filtration device - Millipore Corp.142 mm, or equivalent, capable of exerting pressures of up to 50 psi

NOTE: Older filtration vessels should be used for PFAS analysis since they are less prone to leaching contaminants that may interfere with the analysis.

6.5 Filters – Environmental Express or equivalent, 0.7um glass fiber, 142 mm diameter. Filters are acid washed by the manufacturer.

6.6 pH meter - capable of reading ± 0.05 pH units

6.7 Balance - capable of weighing ± 0.01 g

6.8 Graduated cylinders – 100ml and 1000ml

6.9 Beakers – 125ml or 250ml glass or plastic

6.10 Watch glass - appropriate to cover beaker

6.11 Magnetic Stirrer and stir bars

6.12 PTFE lined 20 liter carboy

6.13 HDPE carboy: Fluids used for PFAS analysis cannot be stored in PTFE lined carboys and should be made up fresh. Water should come from the PFAS prep lab.

6.14 Thermometer, calibrated against an NIST traceable thermometer

6.15 Water bath – adjustable temperature control

7.0 PROCEDURE

7.1 The preparation of all samples must be documented. See Section 8.1 for the various logbooks and prep sheets that are required for this method. The prep sheet will include such items as: sample ID, bottle number, initial volume, final volume, pHs, lot numbers, batch numbers, and leachate dates and times.

The extraction technician is responsible for filling out all the required information. A copy of the prep sheet will be submitted to the analyst with the leachates. The leaching start date and time are entered into LIMS.

7.2 Determination of Percent Solids

7.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration, proceed to Section 7.5.

7.2.2 If the sample is liquid or mixed phase (solid and liquid), proceed as follows. **NOTE:** If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as “totals” instead of TCLP.

7.2.3 Pre-weigh the filter and container that will receive the filtrate. Document all weights in the TCLP_SPLP Description Log.

7.2.4 Assemble the filtering apparatus as per the manufacturer's instructions.

7.2.5 Transfer a 100 gram aliquot of the sample to a beaker and record the weight. If a 100 gram aliquot is not available, inform the Department Supervisor.

7.2.6 Quantitatively transfer the sample aliquot to the filter apparatus. Slurries may be allowed to settle and the liquid portion filtered prior to transferring the solid portion of the sample. **NOTE**: If sample material has adhered to the sample container, obtain the weight of this residue and subtract from the total weight of the sample.

7.2.7 Complete the assembly of the filtration device, and gradually apply pressure until fluid is expelled or 10 psi is obtained. If no fluid is expelled, gradually increase the pressure in 10 psi increments to a maximum of 50 psi. If no fluid is expelled in any 2 minute period, stop the filtration. Shut off the pressurizing gas and vent the filtration system using the top vent.

CAUTION: DO NOT REMOVE FLANGE CLAMPS WHILE SYSTEM IS PRESSURIZED! SERIOUS INJURY MAY RESULT.

NOTE: Instantaneous application of high pressure can cause the filter to clog prematurely.

7.2.8 The material in the filtration apparatus is defined as the solid phase.

NOTE: Some high viscosity liquids (oils, paints) will not filter under these circumstances. The material remaining within the filtration device is defined as the solid phase.

NOTE: If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as "totals" instead of TCLP.

7.2.9 Remove the solid phase of the sample and the filter from the filtration apparatus. If there is a noticeable amount of filtrate entrained in the filter, then dry at 100 °C ± 20 until two successive readings yield the same value within ±1%. Record the final weight.

7.2.10 Determine the percent solids as follows:

$$\% \text{ solids} = \frac{(W - F) \times 100}{T}$$

W = Weight of sample remaining on filter
F = Weight of filter
T = Initial weight of sample used

7.2.11 If the sample contains <0.5% solids, the filtrate is defined as the sample leachate. **NOTE:** Additional aliquots may need to be filtered to generate sufficient volume for all of the analysis. Proceed to Section 7.5.10.

7.3 Determination of Particle Size

7.3.1 Evaluate the solid portion of the sample for particle size. If the solid portion of sample has a surface area equal to or greater than 3.1 cm^2/gram, then the sample does not require particle size reduction. This would apply to samples such as paper, filter material or rags.

7.3.2 Alternatively, if the solid portion of the sample is smaller than 1 cm in its narrowest dimension (i.e. is capable of passing through a 9.5 mm sieve), then the sample does not require particle size reduction.

7.3.3 If the sample does not meet the particle size criteria listed above, then prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the sample. Care should be taken to minimize any potential metals contamination.

7.3.4 Document the sample description and any particle size reduction in the TCLP_SPLP Description Log.

7.4 Determination of Extraction Fluid

7.4.1 pH Meter Calibration

Make sure that the pH electrode is clean. If the electrode is coated with oil or grease, then it must be washed with a 50% water-methanol solution and then rinse it well with DI water. Do not soak the electrode in the acetone solution. Soak the electrode in a beaker containing pH 7 buffer for approximately 2 hours before using.

Connect the pH electrode to the pH meter. Calibrate the meter using three of the following buffers: pH 2, pH 4, pH 7 or pH 10. Buffers chosen must bracket the pH range. Read back the remaining buffer. For details on specific calibration procedure, see the instruction manual for the meter being used.

Transfer fresh buffer solutions to clean 125ml beakers and place a stir bar in each. Place each beaker on the stir plate. Turn the stir plate on to get the buffer solution stirring. Buffer solutions must be stirring when calibrating the pH meter or reading the pH. Lower the pH electrode into the buffer solution. Make sure that the stir bar is not hitting the electrode.

After the calibration is complete, analyze the 3 buffer solutions to ensure that an accurate calibration was obtained. Record the results in the TCLP Fluid Determination Log. Readings must be within 0.05 pH units of the buffer solution’s true value.

7.4.2 Transfer a 5.0 gram aliquot of the solid phase of the sample to a 250 ml beaker. Record the actual weight in the fluid determination log.

NOTE: The particle size of the solid phase should be < 1mm for this step. This may require some particle size reduction.

7.4.3 Add 96.5 ml of DI water to the beaker. Place a magnetic stir bar in the beaker and cover with a watch glass. Stir vigorously for 5 minutes. Measure and record the pH in the TCLP Fluid Determination Log. If the pH is <5.0, use extraction fluid #1, and proceed to step 7.5.

7.4.4 If the pH is >5.0, add 3.5 ml of 1.0N HCl and swirl gently. Cover the beaker with a watch glass and heat to 50°C for 10 minutes.

7.4.5 Allow the solution to cool. Stir vigorously for 5 minutes. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2.

NOTE: All pH measurements must be recorded to two places after the decimal point.

7.5 Extraction of Semivolatile Organics and Metals

7.5.1 If the sample contains 100% solids, and the sample does not need particle size reduction transfer a minimum of 100g aliquot of the sample to the extraction vessel. **(Samples for PFAS analysis will need to use HDPE extraction vessels.)** Record the weight to ± 0.1 gram on the TCLP Sample Prep Sheet. Add 20 times the sample weight of the appropriate extraction fluid to the leaching vessel. Swirl gently and watch for the evolution of carbon dioxide. If no gasses are evolved, cap the container and mount on the rotary agitator. Proceed to section 7.5.4.

7.5.2 If the sample contains <0.5% solids, the filtrate obtained in step 7.2 is defined as the sample leachate. Proceed to section 7.5.10.

7.5.3 If the sample is mixed phase and/or contains >0.5% solids, transfer a 100g aliquot of the sample or more, based on % solids to the filtration device. Assemble the filtration apparatus, and gradually apply pressure to remove any free liquids. Expelled liquid is stored in a glass container and is to be recombined with sample leachate. Transfer the solid portion of the sample to the appropriate extraction vessel. Add a volume of the appropriate TCLP fluid, calculated as follows:

$$\frac{20 \times \text{sample wt (g)} \times \text{\% solids}}{100} = \text{ml extraction fluid}$$

Swirl gently and watch for evolution of carbon dioxide. Cap the extraction bottle and attach to rotary agitator. Allow the extraction to proceed 18 ± 2 hrs.

NOTE: If the sample contains <25% solids, more sample can be filtered to obtain sufficient solids for leaching such that all analyses may be performed. Consult the Department Supervisor.

7.5.4 Rotate at 30 ±2 rpm. Make sure to measure and record the rotation rate and tumbler ID on the TCLP Sample Prep Sheet. Allow the extraction to proceed for 18 ± 2 hrs.

The vessels should be vented periodically during the first hour to prevent pressure build up.

NOTE: The temperature of the extraction room must be 23 ± 2 °C during the extraction period. Record the temperature on TCLP Sample Prep Sheet. Use a Hi/Lo thermometer to monitor the room temperature throughout the extraction period.

7.5.5 After the leaching period has elapsed, remove the extraction vessels from the rotary agitator and allow them to settle.

7.5.6 Assemble the filtration device. Filter each sample into an appropriately labeled container. Multiple filters may be used. Be sure to thoroughly clean the filtration apparatus between samples.

7.5.7 If a compatible liquid was obtained in Section 7.5.3, combine the liquids at this time.

7.5.8 If the liquids are not compatible, record the total volume of the expelled liquid in the sample description logbook and submit both fractions for analysis. Notify the Department Supervisor.

NOTE: Department Supervisor will notify the Project Manager to determine if the phases are to be mathematically combined as in Section 7.5.13, or to be report separately.

7.5.9 Measure and record the pH of each leachate in the TCLP Fluid Determination Log. See Section 7.4.1 for pH meter calibration.

NOTE: All pH measurements must be recorded to two places after the decimal point.

7.5.10 Aliquot the leachate for the necessary analyses. Listed below are the minimum quantities required for analysis. Additional aliquots will be needed for QC samples.

7.5.10.1	Metals (including Mercury)	100ml
7.5.10.2	Semivolatiles by 8270	100ml
7.5.10.3	Pesticides by 8081	100ml
7.5.10.4	Herbicides by 8151	10ml
7.5.10.5	TPH by 8015	100ml
7.5.10.6	PFAS by 537 Mod	125ml

7.5.11 Leachates for metals analysis are stored in labeled plastic bottles and transferred to the metals department.

7.5.12 Leachates for PFAS analysis are stored in labeled HDPE bottles and transferred to the specialty prep lab.

7.5.13 All other aliquots (semi-volatile organics) are transferred to amber glass bottles and stored at ≤ 6°C until the subsequent extractions can be performed.

7.5.14 If individual phases are analyzed separately (see 7.5.8) conduct the appropriate analysis and combine the results mathematically by using the following formula:

Final Analyte Conc. = [(V1*C1)+(V2*C2)] / (V1+V2)

V1 = The volume of the first phase (l)
C1 = The conc. of the analyte of concern in the first phase (mg/l)
V2 = The volume of the second phase (l)
C2 = The conc. of the analyte of concern in the second phase (mg/l)

8.0 DOCUMENTATION

8.1 Documentation for this analysis is quite extensive. At minimum the following logbooks or prep sheets must be filled out completely.

8.1.1 TCLP_SPLP Description Log

8.1.2 TCLP Fluid Determination Log

8.1.3 TCLP Sample Prep Sheet

8.1.4 TCLP_SPLP Bottle Tracking Log

8.1.5 Organics Reagent Log

8.2 For every 20 extractions performed in an extraction vessel, a leachate blank must be performed using that vessel. Document this in the Extraction Bottle Tracking Log.

8.3 For each original matrix type extracted, (soil, water, sludge, etc.) a leachate spike must be performed. Various unique matrices may require their own leachate spikes.

9.0 QUALITY ASSURANCE, QUALITY CONTROL AND METHOD PERFORMANCE

9.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 6 hours; however, samples should not be added after the QC set has been completed. **NOTE:** All samples and QC samples must be leached for the required amount of time.

9.2 A leachate blank (LB) and sample duplicate (DUP) must be leached with each batch of samples. A leachate blank must be prepared for each fluid type used in a given batch.

9.3 All spiking for the matrix spike (MS) and matrix spike duplicate (MSD), leachate spike (LS), and leachate blank spike (LBS) occurs after filtration.

9.4 Method performance is monitored through the routine analysis of negative and positive control samples. Leachate blank spikes and matrix spikes are not applicable to the leaching portion of this test; however, they will be used later to assess the extraction and digestion efficiency of the specific methods performed on the leachate.

9.5 A sample duplicate is used to assess method precision. Sample duplicate %RPD is compared to method defined control limits. Control limits are stored in the LIMS.

10.0 SAFETY AND WASTE DISPOSAL

10.1 Safety

10.1.1 Safety glasses, gloves and lab coats should be worn when handling acids, samples, standards or solvents.

10.1.2 Perform all filtration in a fume hood.

10.1.3 Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS or SDS prior to using any new reagents or solvents.

10.2 Waste Disposal

10.2.1 The TCLP filter and remaining sample is placed in a waste container.

10.2.2 Extra leachate is rinsed down the drain with large amounts of water.

10.2.3 Waste soil from the homogenizing process should be placed in the “soil waste” container. **NOTE:** Waste soil from foreign soils must follow “foreign soil” disposal requirements.

11.0 REFERENCES

SW-846 Method 1311, Rev. 0, 07/92

SW-846 Method 1312, Rev. 0, 09/94

STANDARD OPERATING PROCEDURE FOR THE TOXICITY CHARACTERISTIC LEACHING OF VOLATILE ORGANICS (TCLP)

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TITLE: STANDARD OPERATING PROCEDURE FOR THE TOXICITY CHARACTERISTIC LEACHING OF VOLATILE ORGANICS (TCLP)

REFERENCES: SW846 1311

REVISED SECTIONS: 6.10, 6.11, 7.4, 7.5.1, 7.5.1.10, 7.5.3.12, 7.5.4, 7.5.6 and 8.1.2

1.0 SUMMARY, SCOPE AND APPLICATION

1.1 Summary

For liquid and aqueous samples containing less than 5.0% solids, the sample is filtered via ZHE through TCLP filter paper and the filtrate is defined as the TCLP leachate. The leachate can then be analyzed for volatile organics.

For solid samples, the solid portion of the sample is extracted via ZHE by adding extraction fluid equal to 20 times the weight of the sample and rotating the sample for 18 hours at 30 rpm. After leaching, the sample is filtered through TCLP filter paper. The leachate can then be analyzed for volatile organics.

1.2 Scope and Application

This procedure is applicable to samples submitted for TCLP volatile analysis.

1.2.1 Volatiles by 8260

1.2.2 Volatile TPH by 8015

2.0 DISCUSSION AND COMMENTS

This procedure is adapted from SW-846 method 1311. The method utilizes a zero-headspace extraction (ZHE) vessel and rotary agitation device to evaluate the presence and mobility of volatile analytes. It is not applicable for evaluating the mobility of semivolatile organics or metals analytes.

3.0 PRESERVATION AND HOLDING TIMES

3.1 Preservation

3.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples and 300ml jars are recommended for solid samples. Samples should be collected with minimal headspace.

3.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until leaching.

3.1.3 Samples for TCLP analysis should not be chemically preserved prior to leaching.

3.1.4 TCLP Leachates for volatile organics must be protected from light and stored at ≤ 6°C from the time of filtration until analysis.

3.2 Holding Time

3.2.1 Samples submitted for the analysis of volatile organics must be leached within 14 days of collection.

3.2.2 Leachates for volatile organics must be analyzed by the appropriate procedure within 14 days of filtration.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Leachate Blank Spike (LBS): An aliquot of TCLP fluid spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Leachate blank spike recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.8 Leachate Blank (LB): An aliquot of TCLP fluid to which all reagents are added in the same volumes or proportions as used in sample processing. The leachate blank is processed simultaneously with the samples through all the steps of the analytical procedure. The Leachate blank is used to document contamination resulting from the analytical process.

4.9 Leachate Spike (LS): A sample leachate aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The leachate spike recoveries are used to document the bias of a method in a given sample matrix.

4.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the purge efficiency.

5.0 REAGENTS

5.1 Reagent water – distilled or deionized - free of interferences

5.2 Sodium Hydroxide, 10.0N Fisher brand or equivalent

5.3 Glacial Acetic Acid, Reagent Grade

5.4 Extraction Fluid #1: Add 57 ml of glacial acetic acid and 64.3 ml of 10.0N sodium hydroxide to a 1000ml graduated cylinder and dilute to 1 liter with reagent water. Transfer to a PTFE lined carboy. Using a 1000ml graduated cylinder, transfer an additional 9 liters of reagent water to the carboy. Mix thoroughly. This will prepare 10 liters of Fluid #1. The preparation of all TCLP fluids must be logged in the Organics Reagent Logbook and the pH must be verified prior to use. The pH of this solution should be 4.93 $\pm$ 0.05.

NOTE: If the pH of the fluid is out of range, remake the fluid.

5.5 Buffer solution at pH 2, pH 4, pH 7 and pH 10. Commercially available solutions that have been validated by comparison to NIST standards are recommended for routine use. All buffers must be labeled on receipt and after opening. Buffer solutions must be refreshed at least weekly. **When analyzing samples from West Virginia the buffers must be refreshed on the day of use.**

6.0 GLASSWARE AND APPARATUS

6.1 Agitation apparatus – Environmental Express, Millipore Corp., or equivalent. Must be capable of rotating the extraction vessels in an end-over-end fashion at 30 ± 2 rpm.

6.2 Zero Headspace Extraction (ZHE) Vessels – Millipore Corp., or equivalent. The vessel should have an internal volume of 500 ml and be able to accommodate a 90 mm filter. The vessel must be gas tight and free of organic contaminants.

ZHE vessels must be leak tested prior to being placed into service. Additional leak testing should be performed whenever a loss of pressure during an extraction is observed. To leak test an extractor, pressurize the ZHE to 50 psi and allow it to stand unattended for one hour, and recheck the pressure.

6.2.1 If pressure is lost, check all fittings and o-rings. Replace any worn o-rings and repeat the leak test procedure.

6.2.2 If the pressure still does not hold at 50 psi, then pressurize the extractor to 10 psi and allow it to stand unattended for one hour and recheck the pressure. If it does not hold at 10 psi, the ZHE must be removed from all service until the problem is resolved.

6.2.3 If the ZHE will hold pressure at 10 psi and can maintain a filtering pressure of 50 psi when gas pressure is applied, then it can be used for extractions. Notify the Department Supervisor that further maintenance is required on this extractor.

6.3 Filtration device – Millipore Corp.142 mm, or equivalent, capable of exerting pressures of up to 50 psi (for %solids evaluation).

6.4 Fluid metering pump – Environmental Express Model TP1200, or equivalent

6.5 Filters – Environmental Express or equivalent, 0.7um glass fiber, 90 mm diameter.

6.6 VOA Vials – 40ml

6.7 Tedlar Bags

6.8 Luer Tip Syringe – 50 to 60 ml capacity.

6.9 Teflon Tubing

6.10 Beakers – 125ml or 250ml glass or plastic

6.11 Magnetic Stirrer and stir bars

6.12 pH meter - capable of reading ± 0.05 pH units

6.13 Balance - capable of weighing ± 0.01 g

6.14 Graduated cylinders – 100ml, 250ml, and 1000ml

6.15 PTFE lined 20-liter carboy

6.16 Thermometer, calibrated against an NIST traceable thermometer

7.0 PROCECDURE

7.1 The preparation of all samples must be documented. See Section 8.1 for the various logbooks and prep sheets that are required for this method. The prep sheet will include such items as: sample ID, bottle number, initial volume, final volume, pHs, lot numbers, batch numbers, and leachate dates and times.

The extraction technician is responsible for filling out all the required information. A copy of the prep sheet will be submitted to the analyst with the leachates. The leaching start date and time are entered into LIMS.

7.2 Determination of Percent Solids

7.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration, proceed to Section 7.5.

7.2.2 If the sample is liquid or mixed phase (solid and liquid), proceed as follows. **NOTE:** If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as “totals” instead of TCLP.

7.2.3 Pre-weigh the filter and container that will receive the filtrate. Document all weights in the TCLP_SPLP Description Log.

7.2.4 Assemble the filtering apparatus as per the manufacturer's instructions.

7.2.5 Transfer a 100-gram aliquot of the sample to a beaker and record the weight. If a 100-gram aliquot is not available, inform the Department Supervisor.

7.2.6 Quantitatively transfer the sample aliquot to the filter apparatus. Slurries may be allowed to settle, and the liquid portion filtered prior to transferring the solid portion of the sample. **NOTE**: If sample material has adhered to the sample container, obtain the weight of this residue and subtract from the total weight of the sample.

7.2.7 Complete the assembly of the filtration device, and gradually apply pressure until fluid is expelled or 10 psi is obtained. If no fluid is expelled, gradually increase the pressure in 10 psi increments to a maximum of 50 psi. If no fluid is expelled in any 2-minute period, stop the filtration. Shut off the pressurizing gas and vent the filtration system using the top vent.

CAUTION: Do not remove flange clamps while system is pressurized! Serious injury may result.

NOTE: Instantaneous application of high pressure can cause the filter to clog prematurely.

7.2.8 The material in the filtration apparatus is defined as the solid phase.

NOTE: Some high viscosity liquids (oils, paints) will not filter under these circumstances. The material remaining within the filtration device is defined as the solid phase.

NOTE: If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as “totals” instead of TCLP.

7.2.9 Remove the solid phase of the sample and the filter from the filtration apparatus. If there is a noticeable amount of filtrate entrained in the filter, then dry at 100 °C ± 20 until two successive readings yield the same value within ±1%. Record the final weight.

7.2.10 Determine the percent solids as follows:

$$\% \text{ solids} = \frac{(W - F) \times 100}{T}$$

W = Weight of sample remaining on filter
F = Weight of filter
T = Initial weight of sample used

7.2.11 If the sample contains <5.0% solids, the filtrate is defined as the sample leachate.

NOTE: This aliquot can NOT be used for the analysis of Volatile Organics. Proceed to Section 7.5.

7.3 Determination of Particle Size

7.3.1 Evaluate the solid portion of the sample for particle size. If the solid portion of sample has a surface area equal to or greater than 3.1 cm^2/gram, then the sample does not require particle size reduction. This would apply to samples such as paper or rags.

7.3.2 Alternatively, if the solid portion of the sample is smaller than 1 cm in its narrowest dimension (i.e. is capable of passing through a 9.5 mm sieve), then the sample does not require particle size reduction.

7.3.3 If the sample does not meet the particle size criteria listed above, then prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the sample. This should be performed as quickly as possible in order to minimize the amount of volatile analytes that may be lost.

7.3.4 Document the sample description and any particle size reduction in the TCLP_SPLP Description Log.

7.4 Determination of Extraction Fluid

Samples submitted for the analysis of volatile organics by TCLP are always extracted with Fluid #1.

pH Meter Calibration

Make sure that the pH electrode is clean. If the electrode is coated with oil or grease, then it must be washed with a 50% water-methanol solution and then rinse it well with DI water. Do not soak the electrode in the acetone solution. Soak the electrode in a beaker containing pH 7 buffer for approximately 2 hours before using.

Connect the pH electrode to the pH meter. Calibrate the meter using three of the following buffers: pH 2, pH 4, pH 7 or pH 10. Buffers chosen must bracket the pH range. Read back the remaining buffer. For details on specific calibration procedure, see the instruction manual for the meter being used.

Transfer fresh buffer solutions to clean 125ml beakers and place a stir bar in each. Place each beaker on the stir plate. Turn the stir plate on to get the buffer solution stirring. Buffer solutions must be stirring when calibrating the pH meter or reading the pH. Lower the pH electrode into the buffer solution. Make sure that the stir bar is not hitting the electrode.

After the calibration is complete, analyze the 3 buffer solutions to ensure that an accurate calibration was obtained. Record the results in the TCLP Fluid Determination Log. Readings must be within 0.05 pH units of the buffer solution's true value.

7.5 Zero Headspace Extraction

7.5.1 Percent solids is 100%.

7.5.1.1 If the sample is 100% solid, weigh out a 10 to 25 gram aliquot and quickly transfer to the ZHE. Record the weight to ± 0.1 gram on the TCLP ZHE Sample Prep Sheet.

7.5.1.2 Insert the filter and screens. Seal the vessel. Connect the gas supply to the lower fitting on the ZHE. Close the lower vent.

7.5.1.3 Apply 10 to 15 psi of pressure to the ZHE. Slowly open the top valve to expel any air from the ZHE. Slowly increase the pressure to keep the piston moving. DO NOT EXCEED 50 PSI.

7.5.1.4 Once the piston will no longer move, close the top valve.

7.5.1.5 Add 20 times the sample weight of extraction fluid to a graduated cylinder. Attach the pump and the transfer line to the ZHE.

7.5.1.6 Disconnect the gas supply from the lower ZHE fitting and open the lower vent.

7.5.1.7 Open the top valve to the transfer line and then turn on the pump. Transfer the fluid to the ZHE. After the fluid has been transferred, close the top valve and remove the transfer line.

7.5.1.8 Close the lower vent and reattach the gas supply line. Pressurize the ZHE to 5-10 psi. Disconnect the gas supply line.

7.5.1.9 Rotate the ZHE 2-3 times. Open the top valve and expel any residual headspace. Stop at the first sign of liquid. Disconnect the gas supply line.

7.5.1.10 Check the ZHE carefully for leaks and make sure that the pressure is holding to within 2 psi of the initial pressure. If not, discard the sample and set up a new aliquot in a different ZHE. Record the initial pressure on the TCLP ZHE Prep Sheet. Proceed to section 7.5.4.

7.5.2 Percent solids is <5.0%.

7.5.2.1 If the waste contains <5.0% solids, transfer at least 200ml of sample to the zero headspace extractor (ZHE).

7.5.2.2 Insert the filter and screens. Seal the vessel. Connect the gas supply to the lower fitting on the ZHE. Close the lower vent.

7.5.2.3 Apply 10 to 15 psi of pressure to the ZHE. Slowly open the top valve to expel any air from the ZHE. At the first appearance of any liquid, close the valve.

7.5.2.4 Attach a 50 ml Luer tip syringe to the ZHE. Open the valve to expel approximately 50 ml of filtrate. If the flow stops, gradually increase the gas pressure. Once you have collected 50 ml of filtrate, close the top valve.

7.5.2.5 Transfer the filtrate to a 40 ml VOA vial and cap the vial. Make sure that there is no headspace in the vial. Repeat the process to obtain three vials of filtrate. Store at ≤ 6°C until analysis. Proceed to section 7.5.11.

7.5.3 Mixed phase samples

7.5.3.1 If the sample is mixed phase (solid and liquid), proceed as follows. **NOTE:** If the sample appears to contain an organic liquid phase, notify the Department Supervisor. It is recommended that samples containing organic phases be analyzed as “totals” instead of TCLP.

7.5.3.2 Charge the ZHE with enough sample to obtain 10-25 grams of solid using the formula shown below:

$$\text{Grams of Solid} = \frac{(\text{Grams of Total Sample}) \text{ X } (\%\text{ solids})}{100}$$

7.5.3.3 Insert the filter and screens. Seal the vessel. Connect the gas supply to the lower fitting on the ZHE. Close the lower vent.

7.5.3.4 Apply 10 to 15 psi of pressure to the ZHE. Slowly open the top valve to expel any air from the ZHE. Slowly increase the pressure to keep the piston moving. At the first appearance of any liquid, close the valve.

7.5.3.5 Attach a 50 ml Luer tip syringe to the ZHE. Open the valve to expel the liquid phase. If the flow stops, gradually increase the gas pressure to a maximum of 50 psi. Once you have collected the liquid phase, close the top valve.

7.5.3.6 Transfer the liquid phase to a Tedlar bag or a 40 ml VOA vial. Make sure that there is no headspace in the vial.

7.5.3.7 Add 20 times the sample weight of extraction fluid into a graduated cylinder. Attach the pump and the transfer line to the ZHE.

7.5.3.8 Disconnect the gas supply from the lower ZHE fitting and open the lower vent.

7.5.3.9 Open the top valve to the transfer line and then turn on the pump. Transfer the fluid to the ZHE. After the fluid has been transferred, close the top valve and remove the transfer line.

7.5.3.10 Close the lower vent and reattach the gas supply line. Pressurize the ZHE to 5-10 psi. Disconnect the gas supply line.

7.5.3.11 Rotate the ZHE 2-3 times. Open the top valve and expel any residual headspace. Stop at the first sign of liquid. Disconnect the gas supply line.

7.5.3.12 Check the ZHE carefully for leaks and make sure that the pressure is holding to within 2 psi of the initial pressure. If not, discard the sample and set up a new aliquot in a different ZHE. Record the initial pressure on the TCLP ZHE Prep Sheet. Proceed to section 7.5.4.

7.5.4 Rotate at 30 ±2 rpm. Make sure to measure and record the rotation rate and tumbler ID on the TCLP ZHE Sample Prep Sheet. Allow the extraction to proceed for 18 ± 2 hrs.

NOTE: The temperature of the extraction room must be 23 ± 2 °C during the extraction period. Record the temperature on TCLP ZHE Sample Prep Sheet. Use a Hi/Lo thermometer to monitor the room temperature throughout the extraction period.

7.5.5 After the leaching period has elapsed, remove the extraction vessels from the rotary agitator and allow them to settle.

7.5.6 Check and record the pressure of each ZHE on the TCLP ZHE Prep Sheet. If the pressure has dropped by more than 10 psi, the samples should be discarded and reprepped.

7.5.7 Attach a 50 ml Luer tip syringe to the ZHE. Connect the gas supply to the lower fitting on the ZHE. Open the top valve to expel approximately 50 ml of filtrate. If the flow stops, gradually increase the gas pressure (DO NOT EXCEED 50 PSI). Once you have collected 50 ml of filtrate, close the top valve.

Transfer the filtrate to a 40 ml VOA vial and cap the vial. Make sure that there is no headspace in the vial. Repeat the process to obtain three vials of filtrate. Store at ≤ 6°C until analysis.

7.5.8 Alternatively, connect one end of the Teflon tubing to the ZHE. Place the other end in a 40 ml VOA vial. Connect the gas supply to the lower fitting on the ZHE. Open the top valve to transfer the filtrate to the VOA vial. If the flow stops, gradually increase the gas pressure (DO NOT EXCEED 50 PSI). Once you have completely filled the VOA vial, close the top valve.

Cap the vial. Make sure that there is no headspace in the vial. Repeat the process to obtain three vials of filtrate. Store at ≤ 6°C until analysis.

7.5.9 If a compatible liquid was obtained in Section 7.5.3.6, combine the liquids at this time. It may be easier to transfer the initial filtrate and the final leachate to a Tedlar bag for mixing.

7.5.10 If the liquids are not compatible, record the total volume of the expelled liquid in the sample description logbook and submit both fractions for analysis. Notify the Department Supervisor.

NOTE: Department Supervisor will notify the Project Manager to determine if the phases are to be mathematically combined as in Section 7.5.13, or to be report separately.

7.5.11 Aliquot the leachate for the necessary analyses. Listed below are the minimum quantities required for analysis. Additional aliquots will be needed for QC samples.

7.5.11.1	Volatiles by 8260	3 x 40ml
7.5.11.2	Volatile TPH by 8015	3 x 40ml

7.5.12 All aliquots for volatile organics are transferred to 40 ml VOA vials and stored at ≤ 6°C until analysis.

7.5.13 If individual phases are analyzed separately (see 7.5.10) conduct the appropriate analysis and combine the results mathematically by using the following formula:

$$\text{Final Analyte Conc.} = [(V1*C1)+(V2*C2)] / (V1+V2)$$

V1 = The volume of the first phase (l)
C1 = The conc. of the analyte of concern in the first phase (mg/l)
V2 = The volume of the second phase (l)
C2 = The conc. of the analyte of concern in the second phase (mg/l)

8.0 DOCUMENTATION

8.1 Documentation for this analysis is quite extensive. At minimum the following logbooks or prep sheets must be filled out completely.

8.1.1 TCLP_SPLP Description Log

8.1.2 TCLP ZHE Sample Prep Sheet

8.1.3 ZHE Tracking Log

8.1.4 Organics Reagent Log

8.2 For every 20 extractions performed in an extraction vessel, a leachate blank must be performed using that vessel. Document this in the ZHE Tracking Log.

8.3 For each original matrix type extracted, (soil, water, sludge, etc.) a leachate spike must be performed. Various unique matrices may require their own leachate spikes.

9.0 QUALITY ASSURANCE, QUALITY CONTROL AND METHOD PERFORMANCE

9.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 6 hours; however, samples should not be added after the QC set has been completed. **NOTE:** All samples and QC samples must be leached for the required amount of time.

9.2 A leachate blank (LB) and sample duplicate (DUP) must be leached with each batch of samples.

9.3 All spiking for the matrix spike (MS) and matrix spike duplicate (MSD), leachate spike (LS), and leachate blank spike (LBS) occurs after filtration.

9.4 Method performance is monitored through the routine analysis of negative and positive control samples. Leachate blank spikes and matrix spikes are not applicable to the leaching portion of this test; however, they will be used later to assess the purge efficiency of the specific methods performed on the leachate.

9.5 A sample duplicate is used to assess method precision. Sample duplicate %RPD is compared to method defined control limits. Control limits are stored in the LIMS.

10.0 SAFETY AND WASTE DISPOSAL

10.1 Safety

10.1.1 Safety glasses, gloves and lab coats should be worn when handling acids, samples, standards or solvents.

10.1.2 Perform all filtration in a fume hood.

10.1.3 Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS or SDS prior to using any new reagents or solvents.

10.2 Waste Disposal

10.2.1 The TCLP filter and remaining sample is placed in a waste container.

10.2.2 Extra leachate is rinsed down the drain with large amounts of water.

10.2.3 Waste soil from the homogenizing process should be place in the “soil waste” container. **NOTE:** Waste soil from foreign soils must follow “foreign soil” disposal requirements.

11.0 REFERENCES

SW-846 Method 1311, Rev. 0, 07/92

SW-846 Method 1312, Rev. 0, 09/94

CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

SGS North America Inc. - Orlando
4405 Vineland Road, Suite C-15
Orlando, FL 32811

Fulfills the requirements of

ISO/IEC 17025:2017

and

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V 5.3)

In the field of

TESTING

This certificate is valid only when accompanied by a current scope of accreditation document. The current scope of accreditation can be verified at www.anab.org.

R. Douglas Leonard Jr., VP, PILR SBU

Expiry Date: 15 December 2024
Certificate Number: L2229

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

AND

U.S. DEPARTMENT OF DEFENSE (DOD) QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES (DOD QSM V 5.3)

SGS North America Inc. - Orlando
4405 Vineland Road, Suite C-15
Orlando, FL 32811
Svetlana Izosimova, Ph. D., QA Officer
407-425-6700

TESTING

Valid to: **December 15, 2024**

Certificate Number: **L2229**

Environmental

Drinking Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537 rev. 1.1	Perfluorohexanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluoroheptanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorooctanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorononanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorodecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluoroundecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorododecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorotridecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorotetradecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorobutanesulfonic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorohexanesulfonic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorooctanesulfonic Acid
LC/MS/MS	EPA 537 rev. 1.1	N-Methyl perfluorooctanesulfonamidoacetic acid
LC/MS/MS	EPA 537 rev. 1.1	N-Ethyl perfluorooctanesulfonamidoacetic acid

Drinking Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537.1	Perfluorohexanoic Acid
LC/MS/MS	EPA 537.1	Perfluoroheptanoic Acid
LC/MS/MS	EPA 537.1	Perfluorooctanoic Acid
LC/MS/MS	EPA 537.1	Perfluorononanoic Acid
LC/MS/MS	EPA 537.1	Perfluorodecanoic Acid
LC/MS/MS	EPA 537.1	Perfluoroundecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorododecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorotridecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorotetradecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorobutanesulfonic Acid
LC/MS/MS	EPA 537.1	Perfluorohexanesulfonic Acid
LC/MS/MS	EPA 537.1	Perfluorooctanesulfonic Acid
LC/MS/MS	EPA 537.1	N-Methyl perfluorooctanesulfonamidoacetic acid
LC/MS/MS	EPA 537.1	N-Ethyl perfluorooctanesulfonamidoacetic acid
LC/MS/MS	EPA 537.1	ADONA
LC/MS/MS	EPA 537.1	2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA; GenX)
LC/MS/MS	EPA 537.1	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS; F53B minor)
LC/MS/MS	EPA 537.1	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS; F53B major)
LC/MS/MS	EPA 533	Perfluorobutanoic acid
LC/MS/MS	EPA 533	Perfluoropentanoic acid
LC/MS/MS	EPA 533	Perfluorohexanoic acid
LC/MS/MS	EPA 533	Perfluoroheptanoic acid
LC/MS/MS	EPA 533	Perfluorooctanoic acid
LC/MS/MS	EPA 533	Perfluorononanoic acid

Drinking Water

Technology	Method	Analyte
LC/MS/MS	EPA 533	Perfluorodecanoic acid
LC/MS/MS	EPA 533	Perfluoroundecanoic acid
LC/MS/MS	EPA 533	Perfluorododecanoic acid
LC/MS/MS	EPA 533	Perfluorobutanesulfonic acid
LC/MS/MS	EPA 533	Perfluoropentanesulfonic acid
LC/MS/MS	EPA 533	Perfluorohexanesulfonic acid
LC/MS/MS	EPA 533	Perfluoroheptanesulfonic acid
LC/MS/MS	EPA 533	Perfluorooctanesulfonic acid
LC/MS/MS	EPA 533	4:2 Fluorotelomer sulfonate
LC/MS/MS	EPA 533	6:2 Fluorotelomer sulfonate
LC/MS/MS	EPA 533	8:2 Fluorotelomer sulfonate
LC/MS/MS	EPA 533	Perfluoro-3-methoxypropanoic acid
LC/MS/MS	EPA 533	Perfluoro-4-methoxybutanoic acid
LC/MS/MS	EPA 533	Nonafluoro-3,6-dioxaheptanoic acid
LC/MS/MS	EPA 533	Perfluoro(2-ethoxyethane)sulfonic acid
LC/MS/MS	EPA 533	Hexafluoropropylene oxide dimer acid
LC/MS/MS	EPA 533	4,8-Dioxa-3H-perfluorononanoic acid
LC/MS/MS	EPA 533	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid
LC/MS/MS	EPA 533	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid

Non-Potable Water

Technology	Method	Analyte
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-Chloropropane (DBCP)
GC/ECD	EPA 504.1	1,2-Dibromoethane (EDB)
GC/ECD	EPA 504.1	1,2-Dibromo-3-Chloropropane (DBCP)
GC/ECD	EPA 504.1	1,2,3-Trichloropropane (1,2,3-TCP)
GC/FID	EPA 8015C/D	Diesel range organics (DRO)

Non-Potable Water

Technology	Method	Analyte
GC/FID	EPA 8015C/D	Oil Range Organics (ORO)
GC/FID	EPA 8015C/D	Gasoline range organics (GRO)
GC/ECD	EPA 608.3; EPA 8081B	4,4`-DDD
GC/ECD	EPA 608.3; EPA 8081B	4,4`-DDE
GC/ECD	EPA 608.3; EPA 8081B	4,4`-DDT
GC/ECD	EPA 608.3; EPA 8081B	Aldrin
GC/ECD	EPA 608.3; EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 608.3; EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 608.3; EPA 8081B	delta-BHC
GC/ECD	EPA 608.3; EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 608.3; EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 608.3; EPA 8081B	alpha-Chlordane
GC/ECD	EPA 608.3; EPA 8081B	gamma-Chlordane
GC/ECD	EPA 608.3; EPA 8081B	Dieldrin
GC/ECD	EPA 608.3; EPA 8081B	Endosulfan I
GC/ECD	EPA 608.3; EPA 8081B	Endosulfan II
GC/ECD	EPA 608.3; EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 608.3; EPA 8081B	Endrin
GC/ECD	EPA 608.3; EPA 8081B	Endrin aldehyde
GC/ECD	EPA 608.3; EPA 8081B	Endrin ketone
GC/ECD	EPA 608.3; EPA 8081B	Heptachlor
GC/ECD	EPA 608.3; EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 608.3; EPA 8081B	Methoxychlor
GC/ECD	EPA 608.3; EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 608.3; EPA 8081B	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 608.3; EPA 8081B	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 608.3; EPA 8081B	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 608.3; EPA 8081B	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 608.3; EPA 8081B	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 608.3; EPA 8081B	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 608.3; EPA 8081B	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	Total PCB

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/FPD	EPA 8141B	Azinphos-methyl (Guthion)
GC/FPD	EPA 8141B	Bolstar (Sulprofos)
GC/FPD	EPA 8141B	Carbophenothion
GC/FPD	EPA 8141B	Chlorpyrifos
GC/FPD	EPA 8141B	Coumaphos
GC/FPD	EPA 8141B	Demeton-o
GC/FPD	EPA 8141B	Demeton-s
GC/FPD	EPA 8141B	Demeton
GC/FPD	EPA 8141B	Diazinon
GC/FPD	EPA 8141B	Dichlorovos (DDVP Dichlorvos)
GC/FPD	EPA 8141B	Dimethoate
GC/FPD	EPA 8141B	Disulfoton
GC/FPD	EPA 8141B	EPN
GC/FPD	EPA 8141B	Ethion
GC/FPD	EPA 8141B	Ethoprop
GC/FPD	EPA 8141B	Famphur
GC/FPD	EPA 8141B	Fensulfothion
GC/FPD	EPA 8141B	Fenthion
GC/FPD	EPA 8141B	Malathion
GC/FPD	EPA 8141B	Merphos
GC/FPD	EPA 8141B	Methyl parathion (Parathion methyl)
GC/FPD	EPA 8141B	Mevinphos
GC/FPD	EPA 8141B	Monocrotophos
GC/FPD	EPA 8141B	Naled
GC/FPD	EPA 8141B	Parathion ethyl
GC/FPD	EPA 8141B	Phorate
GC/FPD	EPA 8141B	Ronnel
GC/FPD	EPA 8141B	Stirofos
GC/FPD	EPA 8141B	Sulfotepp
GC/FPD	EPA 8141B	Tetraethyl pyrophosphate (TEPP)
GC/FPD	EPA 8141B	Thionazin (Zinophos)
GC/FPD	EPA 8141B	Tokuthion (Prothiophos)
GC/FPD	EPA 8141B	Trichloronate
GC/FPD	EPA 8141B	O,O,O-Triethyl phosphorothioate
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4-D

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop (Dichlorprop)
GC/ECD	EPA 8151A	Dinoseb (2-sec-butyl-4,6-dinitrophenol DNBP)
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2,4,5-TP)
GC/FID	RSK-175	Acetylene
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/FID	RSK-175	Propane
GC/FID	FL-PRO	Total Petroleum Hydrocarbons (TPH)
GC/FID	MA-VPH	Volatile petroleum range organics (VPH)
GC/FID	MA-EPH	Extractable petroleum range organics (EPH)
GC/FID	IA-OA1	Gasoline range organics (GRO)
GC/FID	IA-OA2	Diesel range organics (DRO)
GC/FID	TN-GRO	Gasoline range organics (GRO)
GC/FID	TN-EPH	Extractable petroleum range organics (EPH)
GC/FID	WI-DRO	Diesel range organics (DRO)
GC/FID	KS LRH	Low-Range Hydrocarbons (LRH)
GC/FID	KS MRH	Mid-Range Hydrocarbons (MRH)
GC/FID	KS HRH	High-Range Hydrocarbons (HRH)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,1,1-Trichloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,1,2-Trichloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,1-Dichloroethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,1-Dichloroethylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,1-Dichloropropene
GC/MS	EPA 624.1; EPA 8260B/C/D	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,2,3-Trichlorobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,2,3-Trichloropropane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,2,4-Trichlorobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,2,4-Trimethylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,2-Dibromoethane (EDB Ethylene dibromide)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,2-Dichloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,2-Dichloroethene (total)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,2-Dichloropropane
GC/MS	EPA 8260B/C/D	1,2-Dichlorotrifluoroethane (Freon 123)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,3,5-Trimethylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,3-Dichloropropane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8260B/C	1-Chlorohexane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	2,2-Dichloropropane

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	2-Butanone (Methyl ethyl ketone MEK)
GC/MS	EPA 624.1; EPA 8260B/C/D	2-Chloroethyl vinyl ether
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	2-Chlorotoluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	2-Hexanone
GC/MS	EPA 8260B/C	2-Nitropropane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	4-Chlorotoluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Acetone
GC/MS	EPA 8260B/C/D	Acetonitrile
GC/MS	EPA 624.1; EPA 8260B/C/D	Acrolein (Propenal)
GC/MS	EPA 624.1; EPA 8260B/C/D	Acrylonitrile
GC/MS	EPA 8260B/C/D	Allyl chloride (3-Chloropropene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Benzene
GC/MS	EPA 8260B/C/D	Benzyl Chloride
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Bromobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Bromochloromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Bromodichloromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Bromoform
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	n-Butylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	sec-Butylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	tert-Butylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Carbon disulfide
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Carbon tetrachloride

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Chlorobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Chloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Chloroform
GC/MS	EPA 8260B/C/D	Chloroprene
GC/MS	EPA 624.1; EPA 8260B/C/D	Cyclohexane
GC/MS	EPA 8260B/C/D	Cyclohexanone
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	cis-1,2-Dichloroethylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	trans-1,2-Dichloroethylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	cis-1,3-Dichloropropene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	trans-1,3-Dichloropropylene
GC/MS	EPA 8260B/C/D	cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260B/C/D	trans-1,4-Dichloro-2-butene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Di-isopropylether (DIPE)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Dibromochloromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Dibromomethane (Methylene Bromide)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Dichlorodifluoromethane
GC/MS	EPA 8260B/C/D	Diethyl ether
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D SIM	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Ethanol (Ethyl Alcohol)
GC/MS	EPA 8260B/C/D	Ethyl acetate
GC/MS	EPA 8260B/C/D	Ethyl methacrylate
GC/MS	EPA 8260B/C	Ethyl tert-butyl alcohol (ETBA)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Ethyl tert-butyl ether (ETBE)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Ethylbenzene

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Hexachlorobutadiene
GC/MS	EPA 8260B/C/D	Hexane
GC/MS	EPA 8260B/C/D	Iodomethane (Methyl iodide)
GC/MS	EPA 8260B/C/D	Isobutyl alcohol (2-Methyl-1-propanol)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	p-Isopropyltoluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Isopropylbenzene
GC/MS	EPA 8260B/C/D	Methacrylonitrile
GC/MS	EPA 624.1; EPA 8260B/C/D	Methyl Acetate
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Methyl bromide (Bromomethane)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Methyl chloride (Chloromethane)
GC/MS	EPA 624.1; EPA 8260B/C/D	Methylcyclohexane
GC/MS	EPA 8260B/C/D	Methyl methacrylate
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Methylene chloride
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Naphthalene
GC/MS	EPA 8260B/C/D	Pentachloroethane
GC/MS	EPA 8260B/C/D	Propionitrile (Ethyl cyanide)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	n-Propylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Styrene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	tert-Amyl alcohol (TAA)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	tert-Amyl methyl ether (TAME)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	tert-Butyl alcohol (TBA)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	tert-Butyl formate (TBF)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Tetrachloroethylene (Perchloroethylene)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C/D	Tetrahydrofuran
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Toluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Trichloroethene (Trichloroethylene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Trichlorofluoromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Vinyl acetate
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Vinyl chloride
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	Xylene (total)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	m,p-Xylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260B/C/D	o-Xylene
GC/MS	EPA 625.1; EPA 8270D/E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 625.1; EPA 8270D/E	1,2,4-Trichlorobenzene
GC/MS	EPA 625.1; EPA 8270D/E	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 625.1; EPA 8270D/E	1,2-Diphenylhydrazine
GC/MS	EPA 8270D/E	1,3,5-Trinitrobenzene (1,3,5-TNB)
GC/MS	EPA 625.1; EPA 8270D/E	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 8270D/E	1,3-Dinitrobenzene (1,3-DNB)
GC/MS	EPA 625.1; EPA 8270D/E	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8270D/E	1,4-Naphthoquinone
GC/MS	EPA 8270D/E	1,4-Phenylenediamine
GC/MS	EPA 8270D/E	1-Chloronaphthalene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	1-Methylnaphthalene
GC/MS	EPA 8270D/E	1-Naphthylamine
GC/MS	EPA 625.1; EPA 8270D/E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4,5-Trichlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4,6-Trichlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4-Dichlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4-Dimethylphenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4-Dinitrophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4-Dinitrotoluene (2,4-DNT)

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8270D/E	2,6-Dichlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,6-Dinitrotoluene (2,6-DNT)
GC/MS	EPA 8270D/E	2-Acetylaminofluorene
GC/MS	EPA 625.1; EPA 8270D/E	2-Chloronaphthalene
GC/MS	EPA 625.1; EPA 8270D/E	2-Chlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-o-cresol)
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	2-Methylnaphthalene
GC/MS	EPA 625.1; EPA 8270D/E	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270D/E	2-Naphthylamine
GC/MS	EPA 625.1; EPA 8270D/E	2-Nitroaniline
GC/MS	EPA 625.1; EPA 8270D/E	2-Nitrophenol
GC/MS	EPA 8270D/E	2-Picoline (2-Methylpyridine)
GC/MS	EPA 625.1; EPA 8270D/E	3,3`-Dichlorobenzidine
GC/MS	EPA 8270D/E	3,3`-Dimethylbenzidine
GC/MS	EPA 8270D/E	3-Methylcholanthrene
GC/MS	EPA 625.1; EPA 8270D/E	3&4-Methylphenol (m,p-Cresol)
GC/MS	EPA 625.1; EPA 8270D/E	3-Nitroaniline
GC/MS	EPA 8270D/E	4-Aminobiphenyl
GC/MS	EPA 625.1; EPA 8270D/E	4-Bromophenyl phenyl ether
GC/MS	EPA 625.1; EPA 8270D/E	4-Chloro-3-methylphenol
GC/MS	EPA 625.1; EPA 8270D/E	4-Chloroaniline
GC/MS	EPA 625.1; EPA 8270D/E	4-Chlorophenyl phenylether
GC/MS	EPA 8270D/E	4-Dimethyl aminoazobenzene
GC/MS	EPA 625.1; EPA 8270D/E	4-Nitroaniline
GC/MS	EPA 625.1; EPA 8270D/E	4-Nitrophenol
GC/MS	EPA 8270D/E	5-Nitro-o-toluidine
GC/MS	EPA 8270D/E	7,12-Dimethylbenz(a) anthracene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Acenaphthene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Acenaphthylene
GC/MS	EPA 625.1; EPA 8270D/E	Acetophenone
GC/MS	EPA 625.1; EPA 8270D/E	Aniline
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Anthracene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D/E	Aramite
GC/MS	EPA 625.1; EPA 8270D/E	Atrazine
GC/MS	EPA 625.1; EPA 8270D/E	Benzaldehyde
GC/MS	EPA 625.1; EPA 8270D/E	Benzidine
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Benzo(a)anthracene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Benzo(a)pyrene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Benzo(b)fluoranthene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Benzo(g,h,i)perylene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Benzo(k)fluoranthene
GC/MS	EPA 625.1; EPA 8270D/E	Benzoic acid
GC/MS	EPA 625.1; EPA 8270D/E	Benzyl alcohol
GC/MS	EPA 625.1; EPA 8270D/E	Biphenyl (1,1'-Biphenyl)
GC/MS	EPA 625.1; EPA 8270D/E	bis(2-Chloroethoxy)methane
GC/MS	EPA 625.1; EPA 8270D/E	bis(2-Chloroethyl) ether
GC/MS	EPA 625.1; EPA 8270D/E	bis(2-Chloroisopropyl) ether (2,2`-Oxybis(1-chloropropane))
GC/MS	EPA 625.1; EPA 8270D/E	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 625.1; EPA 8270D/E	Butyl benzyl phthalate
GC/MS	EPA 625.1; EPA 8270D/E	Carbazole
GC/MS	EPA 625.1; EPA 8270D/E	Caprolactam
GC/MS	EPA 8270D/E	Chlorobenzilate
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Chrysene
GC/MS	EPA 8270D/E	Diallate
GC/MS	EPA 625.1; EPA 8270D/E	Di-n-butyl phthalate
GC/MS	EPA 625.1; EPA 8270D/E	Di-n-octyl phthalate
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270D/E	Dibenz(a,j)acridine
GC/MS	EPA 625.1; EPA 8270D/E	Dibenzofuran
GC/MS	EPA 625.1; EPA 8270D/E	Diethyl phthalate
GC/MS	EPA 625.1; EPA 8270D/E	Dimethyl phthalate

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8270D/E	a,a-Dimethylphenethylamine
GC/MS	EPA 8270D/E	Diphenyl Ether
GC/MS	EPA 8270D/E	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 8270D/E	Ethyl methanesulfonate
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Fluoranthene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Fluorene
GC/MS	EPA 625.1; EPA 8270D/E	Hexachlorobenzene
GC/MS	EPA 625.1; EPA 8270D/E	Hexachlorobutadiene
GC/MS	EPA 625.1; EPA 8270D/E	Hexachlorocyclopentadiene
GC/MS	EPA 625.1; EPA 8270D/E	Hexachloroethane
GC/MS	EPA 8270D/E	Hexachlorophene
GC/MS	EPA 8270D/E	Hexachloropropene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D	Isodrin
GC/MS	EPA 625.1; EPA 8270D/E	Isophorone
GC/MS	EPA 8270D/E	Isosafrole
GC/MS	EPA 8270D/E	Kepone
GC/MS	EPA 8270D/E	Methapyrilene
GC/MS	EPA 8270D/E	Methyl methanesulfonate
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Naphthalene
GC/MS	EPA 625.1; EPA 8270D/E	Nitrobenzene
GC/MS	EPA 8270D/E	Nitroquinoline-1-oxide
GC/MS	EPA 8270D/E	n-Nitroso-di-n-butylamine
GC/MS	EPA 625.1; EPA 8270D/E	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270D/E	n-Nitrosodiethylamine
GC/MS	EPA 625.1; EPA 8270D/E	n-Nitrosodimethylamine
GC/MS	EPA 625.1; EPA 8270D/E	n-Nitrosodiphenylamine
GC/MS	EPA 8270D/E	n-Nitrosodiphenylamine/Diphenylamine (analyte pair)
GC/MS	EPA 8270D/E	n-Nitrosomethylethylamine
GC/MS	EPA 8270D/E	n-Nitrosomorpholine
GC/MS	EPA 8270D/E	n-Nitrosopiperidine
GC/MS	EPA 8270D/E	n-Nitrosopyrrolidine

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8270D/E	Pentachlorobenzene
GC/MS	EPA 8270D/E	Pentachloroethane
GC/MS	EPA 8270D/E	Pentachloronitrobenzene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Pentachlorophenol
GC/MS	EPA 8270D/E	Phenacetin
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Phenanthrene
GC/MS	EPA 625.1; EPA 8270D/E	Phenol
GC/MS	EPA 8270D/E	Pronamide (Kerb)
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Pyrene
GC/MS	EPA 625.1; EPA 8270D/E	Pyridine
GC/MS	EPA 8270D/E	Safrole
GC/MS	EPA 8270D/E	Simazine
GC/MS	EPA 8270D/E	Thionazin (Zinophos)
GC/MS	EPA 8270D/E	o-Toluidine
GC/MS	EPA 8270D/E	Dimethoate
GC/MS	EPA 8270D/E	Disulfoton
GC/MS	EPA 8270D/E	Famphur
GC/MS	EPA 8270D/E	Methyl parathion (Parathion methyl)
GC/MS	EPA 8270D/E	Parathion ethyl
GC/MS	EPA 8270D/E	Phorate
GC/MS	EPA 8270D/E	O,O,O-Triethyl phosphorothioate
HPLC	EPA 8330A/B	1,3,5-Trinitrobenzene (1,3,5-TNB)
HPLC	EPA 8330A/B	1,3-Dinitrobenzene (1,3-DNB)
HPLC	EPA 8330A/B	2,4,6-Trinitrotoluene (2,4,6-TNT)
HPLC	EPA 8330A/B	2,4-Dinitrotoluene (2,4-DNT)
HPLC	EPA 8330A/B	2,6-Dinitrotoluene (2,6-DNT)
HPLC	EPA 8330A/B	2-Amino-4,6-dinitrotoluene (2-am-dnt)
HPLC	EPA 8330A/B	2-Nitrotoluene
HPLC	EPA 8330A/B	3,5-Dinitroaniline
HPLC	EPA 8330A/B	3-Nitrotoluene
HPLC	EPA 8330A/B	4-Amino-2,6-dinitrotoluene (4-am-dnt)
HPLC	EPA 8330A/B	4-Nitrotoluene
HPLC	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)

Non-Potable Water		
Technology	**Method**	**Analyte**
HPLC	EPA 8330A/B	Nitrobenzene
HPLC	EPA 8330A/B	Nitroglycerin
HPLC	EPA 8330A/B	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
HPLC	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC	EPA 8330A/B	Pentaerythritoltetranitrate (PETN)
HPLC	EPA 8330A/B	2,4-diamino-6-Nitrotoluene
HPLC	EPA 8330A/B	2,6-diamino-4-Nitrotoluene
HPLC	EPA 8330A/B	DNX
HPLC	EPA 8330A/B	MNX
HPLC	EPA 8330A/B	TNX
LC/MS/MS	EPA 6850	Perchlorate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorobutanoic Acid (PFBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoropentanoic Acid (PFPeA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexanoic Acid (PFHxA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroheptanoic Acid (PFHpA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanoic Acid (PFOA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorononanoic Acid (PFNA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorodecanoic Acid (PFDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroundecanoic Acid (PFUnA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorododecanoic Acid(PFDoA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorotridecanoic Acid (PFTrDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorotetradecanoic Acid (PFTA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorobutanesulfonic Acid (PFBS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexanesulfonic Acid(PFHxS)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanesulfonic Acid(PFOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorononanesulfonic Acid(PFNS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorodecanesulfonic Acid(PFDS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroheptanesulfonic acid(PFHpS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoropentanesulfonic Acid(PFPeS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctane sulfonamide (PFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Methyl perfluorooctanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	4:2 Fluorotelomer Sulfonate (FTS 4:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	6:2 Fluorotelomer Sulfonate(FTS 6:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	8:2 Fluorotelomer Sulfonate (FTS 8:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	ADONA
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA; GenX)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS; F53B minor)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS; F53B major)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	3:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	5:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	7:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	10:2 Fluorotelomer sulfonate

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorododecanesulfonic acid
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-4-methoxybutanoic acid (PFMBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexadecanoic acid (PFHxDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctadecanoic acid (PFOcDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	4-PFecHS (Perfluoro-4-ethylcyclohexanesulfonate)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Methyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Ethyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Methyl perfluorooctane sulfonamidoethanol
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Ethyl perfluorooctane sulfonamidoethanol
ICP	EPA 200.7; EPA 6010C/D	Aluminum
ICP	EPA 200.7; EPA 6010C/D	Antimony
ICP	EPA 200.7; EPA 6010C/D	Arsenic
ICP	EPA 200.7; EPA 6010C/D	Barium
ICP	EPA 200.7; EPA 6010C/D	Beryllium
ICP	EPA 200.7; EPA 6010C/D	Cadmium
ICP	EPA 200.7; EPA 6010C/D	Calcium
ICP	EPA 200.7; EPA 6010C/D	Chromium
ICP	EPA 200.7; EPA 6010C/D	Cobalt
ICP	EPA 200.7; EPA 6010C/D	Copper
ICP	EPA 200.7; EPA 6010C/D	Iron
ICP	EPA 200.7; EPA 6010C/D	Lead
ICP	EPA 200.7; EPA 6010C/D	Magnesium
ICP	EPA 200.7; EPA 6010C/D	Manganese
ICP	EPA 200.7; EPA 6010C/D	Molybdenum

Non-Potable Water

Technology	Method	Analyte
ICP	EPA 200.7; EPA 6010C/D	Nickel
ICP	EPA 200.7; EPA 6010C/D	Potassium
ICP	EPA 200.7; EPA 6010C/D	Selenium
ICP	EPA 200.7; EPA 6010C/D	Silver
ICP	EPA 200.7; EPA 6010C/D	Sodium
ICP	EPA 200.7; EPA 6010C/D	Strontium
ICP	EPA 200.7; EPA 6010C/D	Thallium
ICP	EPA 200.7; EPA 6010C/D	Tin
ICP	EPA 200.7; EPA 6010C/D	Titanium
ICP	EPA 200.7; EPA 6010C/D	Vanadium
ICP	EPA 200.7; EPA 6010C/D	Zinc
ICP/MS	EPA 200.8; EPA 6020A/B	Aluminum
ICP/MS	EPA 200.8; EPA 6020A/B	Antimony
ICP/MS	EPA 200.8; EPA 6020A/B	Arsenic
ICP/MS	EPA 200.8; EPA 6020A/B	Barium
ICP/MS	EPA 200.8; EPA 6020A/B	Beryllium
ICP/MS	EPA 200.8; EPA 6020A/B	Cadmium
ICP/MS	EPA 200.8; EPA 6020A/B	Calcium
ICP/MS	EPA 200.8; EPA 6020A/B	Chromium
ICP/MS	EPA 200.8; EPA 6020A/B	Cobalt
ICP/MS	EPA 200.8; EPA 6020A/B	Copper
ICP/MS	EPA 200.8; EPA 6020A/B	Iron
ICP/MS	EPA 200.8; EPA 6020A/B	Lead
ICP/MS	EPA 200.8; EPA 6020A/B	Magnesium
ICP/MS	EPA 200.8; EPA 6020A/B	Manganese
ICP/MS	EPA 200.8; EPA 6020A/B	Molybdenum
ICP/MS	EPA 200.8; EPA 6020A/B	Nickel
ICP/MS	EPA 200.8; EPA 6020A/B	Potassium
ICP/MS	EPA 200.8; EPA 6020A/B	Selenium
ICP/MS	EPA 200.8; EPA 6020A/B	Silver
ICP/MS	EPA 200.8; EPA 6020A/B	Sodium
ICP/MS	EPA 200.8; EPA 6020A/B	Strontium
ICP/MS	EPA 200.8; EPA 6020A/B	Thallium
ICP/MS	EPA 200.8; EPA 6020A/B	Tin
ICP/MS	EPA 200.8; EPA 6020A/B	Titanium
ICP/MS	EPA 200.8; EPA 6020A/B	Vanadium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 200.8; EPA 6020A/B	Zinc
CVAA	EPA 7470A	Mercury
CVAA	EPA 245.1	Mercury
UV/VIS	EPA 7196A	Hexavalent Chromium (Cr6+)
UV/VIS	EPA 9012B	Cyanide (Total)
IC	EPA 300; EPA 9056A	Bromide
IC	EPA 300; EPA 9056A	Chloride
IC	EPA 300; EPA 9056A	Fluoride
IC	EPA 300; EPA 9056A	Nitrate
IC	EPA 300; EPA 9056A	Nitrite
IC	EPA 300; EPA 9056A	Sulfate
IC	EPA 300; EPA 9056A	Total nitrate-nitrite
IC	EPA 300; EPA 9056A	Orthophosphate
Automated Colorimetry	EPA 350.1	Ammonia
Automated Colorimetry	EPA 350.1	Ammonia, Gas Diffusion Option
Automated Colorimetry	EPA 351.2	Total Kjeldahl Nitrogen
Automated Colorimetry	EPA 420.4	Total Phenolics
Manual Colorimetry	EPA 365.3	Orthophosphate
Automated Colorimetry	EPA 365.1	Orthophosphate
Automated Colorimetry	EPA 365.1	Total Phosphorus
Manual Colorimetry	EPA 365.3	Total Phosphorus
Titrimetric	SM 2320B-11	Alkalinity, Total
Titrimetric	SM 4500-S2 F-11	Sulfide, Iodometric
Gravimetric Methods	EPA 1664A; EPA 1664B; EPA 9070A	Oil and Grease
Gravimetric Methods	SM 2540B-11	Total Residue (Total Solids)
Gravimetric Methods	SM 2540C-11	Filterable Residue (Total Dissolved Solids)
Gravimetric Methods	SM 2540D-11	Non-Filterable Residue (Total Suspended Solids)
Electrometric Methods	SM 4500H+B-11; EPA 9040C	Hydrogen Ion (Ph)
Electrometric Methods	EPA 120.1	Specific conductivity
Combustion	EPA 9060A	Total Organic Carbon
Combustion	SM 5310B-11	Total Organic Carbon
Ignitability	EPA 1010A	Flash Point

Non-Potable Water		
Technology	**Method**	**Analyte**
Ignitability	EPA 1020B/ASTM D3278-78	Flash Point
Waste Characterization	EPA Ch.7	Reactive Cyanide and Reactive Sulfide
Waste Characterization	EPA Section 7.3	Reactive Cyanide
Waste Characterization	EPA Section 7.3	Reactive Sulfide
Preparation	**Method**	**Type**
Organic Preparation	EPA 3510C	Separatory Funnel Liquid-Liquid Extraction
Organic Preparation	EPA 3511	Micro-extraction
Organic Preparation	EPA 3535A; EPA 3535A MOD	Solid Phase Extraction
Organic Preparation	EPA 8015C/D	Non-Halogenated Organics (Alcohols), direct injection
Organic Preparation	EPA 8151A	Chlorinated Herbicides, Liquid-Liquid Extraction
Organic Preparation	EPA 608; EPA 625	Separatory Funnel Liquid-Liquid Extraction
Volatile Organic Preparation	SW836 5030B	Closed System Purge and Trap
Volatile Organic Preparation	EPA 624	Closed System Purge and Trap
Volatile Organic Preparation	SM 6200B-11	Closed System Purge and Trap
Lachat MicroDistillation	EPA 9012B	Cyanide MicroDistillation; proprietary method
Inorganic Preparation	EPA 3010A	Metals Acid Digestion by Hotblock
Inorganic Preparation	EPA 7470A	CVAA Digestion by Hotblock
Organics Cleanup	EPA 3660B	Sulfur Cleanup
Organics Cleanup	EPA 3665A	Sulfuric Acid Cleanup

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-Chloropropane (DBCP)
GC/FID	EPA 8015C/D	Diesel range organics (DRO)
GC/FID	EPA 8015C/D	Oil Range Organics (ORO)
GC/FID	EPA 8015C/D	Gasoline range organics (GRO)
GC/FID	EPA 8015C/D	Ethanol
GC/FID	EPA 8015C/D	2-Ethoxyethanol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/FID	EPA 8015C/D	Isobutyl alcohol (2-Methyl-1-propanol)
GC/FID	EPA 8015C/D	Isopropyl alcohol (2-Propanol)
GC/FID	EPA 8015C/D	Methanol
GC/FID	EPA 8015C/D	n-Butyl alcohol
GC/FID	EPA 8015C/D	n-Propanol
GC/ECD	EPA 8081B	4,4`-DDD
GC/ECD	EPA 8081B	4,4`-DDE
GC/ECD	EPA 8081B	4,4`-DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 8081B	alpha-Chlordane
GC/ECD	EPA 8081B	gamma-Chlordane
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 (Chlorinated camphene)
GC/ECD	EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 8082A	Aroclor-1260 (PCB-1260)

Solid and Chemical Materials

Technology	Method	Analyte
GC/ECD	EPA 8082A	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	Total PCB
GC/FPD	EPA 8141B	Azinphos-methyl (Guthion)
GC/FPD	EPA 8141B	Bolstar (Sulprofos)
GC/FPD	EPA 8141B	Carbophenothion
GC/FPD	EPA 8141B	Chlorpyrifos
GC/FPD	EPA 8141B	Coumaphos
GC/FPD	EPA 8141B	Demeton-o
GC/FPD	EPA 8141B	Demeton-s
GC/FPD	EPA 8141B	Demeton
GC/FPD	EPA 8141B	Diazinon
GC/FPD	EPA 8141B	Dichlorovos (DDVP Dichlorvos)
GC/FPD	EPA 8141B	Dimethoate
GC/FPD	EPA 8141B	Disulfoton
GC/FPD	EPA 8141B	EPN
GC/FPD	EPA 8141B	Ethion
GC/FPD	EPA 8141B	Ethoprop
GC/FPD	EPA 8141B	Famphur
GC/FPD	EPA 8141B	Fensulfothion
GC/FPD	EPA 8141B	Fenthion
GC/FPD	EPA 8141B	Malathion
GC/FPD	EPA 8141B	Merphos
GC/FPD	EPA 8141B	Methyl parathion (Parathion methyl)
GC/FPD	EPA 8141B	Mevinphos
GC/FPD	EPA 8141B	Monocrotophos
GC/FPD	EPA 8141B	Naled
GC/FPD	EPA 8141B	Parathion ethyl
GC/FPD	EPA 8141B	Phorate
GC/FPD	EPA 8141B	Ronnel
GC/FPD	EPA 8141B	Stirofos
GC/FPD	EPA 8141B	Sulfotepp
GC/FPD	EPA 8141B	Tetraethyl pyrophosphate (TEPP)
GC/FPD	EPA 8141B	Thionazin (Zinophos)
GC/FPD	EPA 8141B	Tokuthion (Prothiophos)
GC/FPD	EPA 8141B	Trichloronate

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/FPD	EPA 8141B	O,O,O-Triethyl phosphorothioate
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop (Dichlorprop)
GC/ECD	EPA 8151A	Dinoseb (2-sec-butyl-4,6-dinitrophenol DNBP)
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2,4,5-TP)
GC/FID	FL-PRO	Total Petroleum Hydrocarbons (TPH)
GC/FID	MA-VPH	Volatile petroleum range organics (VPH)
GC/FID	MA-EPH	Extractable petroleum range organics (EPH)
GC/FID	IA-OA1	Gasoline range organics (GRO)
GC/FID	IA-OA2	Diesel range organics (DRO)
GC/FID	TN-GRO	Gasoline range organics (GRO)
GC/FID	TN-EPH	Extractable petroleum range organics (EPH)
GC/FID	KS LRH	Low-range Hydrocarbons (LRH)
GC/FID	KS MRH	Mid-Range Hydrocarbons (MRH)
GC/FID	KS HRH	High-Range Hydrocarbons (HRH)
GC/MS	EPA 8260B/C/D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/C/D	1,1,1-Trichloroethane
GC/MS	EPA 8260B/C/D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/C/D	1,1,2-Trichloroethane
GC/MS	EPA 8260B/C/D	1,1-Dichloroethane
GC/MS	EPA 8260B/C/D	1,1-Dichloroethylene
GC/MS	EPA 8260B/C/D	1,1-Dichloropropene
GC/MS	EPA 8260B/C/D	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260B/C/D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/C/D	1,2,3-Trichloropropane
GC/MS	EPA 8260B/C/D	1,2,4-Trichlorobenzene

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	EPA 8260B/C/D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/C/D	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B/C/D	1,2-Dibromoethane (EDB Ethylene dibromide)
GC/MS	EPA 8260B/C/D	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 8260B/C/D	1,2-Dichloroethane
GC/MS	EPA 8260B/C/D	1,2-Dichloroethene (total)
GC/MS	EPA 8260B/C/D	1,2-Dichloropropane
GC/MS	EPA 8260B/C/D	1,2-Dichlorotrifluoroethane (Freon 123)
GC/MS	EPA 8260B/C/D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/C/D	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 8260B/C/D	1,3-Dichloropropane
GC/MS	EPA 8260B/C/D	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8260B/C/D	1-Chlorohexane
GC/MS	EPA 8260B/C/D	2,2-Dichloropropane
GC/MS	EPA 8260B/C/D	2-Butanone (Methyl ethyl ketone MEK)
GC/MS	EPA 8260B/C/D	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/C/D	2-Chlorotoluene
GC/MS	EPA 8260B/C/D	2-Hexanone
GC/MS	EPA 8260B/C/D	2-Nitropropane
GC/MS	EPA 8260B/C/D	4-Chlorotoluene
GC/MS	EPA 8260B/C/D	4-Methyl-2-pentanone (MBK)
GC/MS	EPA 8260B/C/D	Acetone
GC/MS	EPA 8260B/C/D	Acetonitrile
GC/MS	EPA 8260B/C/D	Acrolein (Propenal)
GC/MS	EPA 8260B/C/D	Acrylonitrile
GC/MS	EPA 8260B/C/D	Allyl chloride (3-Chloropropene)
GC/MS	EPA 8260B/C/D	Benzene
GC/MS	EPA 8260B/C/D	Benzyl Chloride
GC/MS	EPA 8260B/C/D	Bromobenzene
GC/MS	EPA 8260B/C/D	Bromochloromethane
GC/MS	EPA 8260B/C/D	Bromodichloromethane
GC/MS	EPA 8260B/C/D	Bromoform
GC/MS	EPA 8260B/C/D	n-Butylbenzene
GC/MS	EPA 8260B/C/D	sec-Butylbenzene
GC/MS	EPA 8260B/C/D	tert-Butylbenzene

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	EPA 8260B/C/D	Carbon disulfide
GC/MS	EPA 8260B/C/D	Carbon tetrachloride
GC/MS	EPA 8260B/C/D	Chlorobenzene
GC/MS	EPA 8260B/C/D	Chloroethane
GC/MS	EPA 8260B/C/D	Chloroform
GC/MS	EPA 8260B/C/D	Chloroprene
GC/MS	EPA 8260B/C/D	Cyclohexane
GC/MS	EPA 8260B/C/D	Cyclohexanone
GC/MS	EPA 8260B/C/D	cis-1,2-Dichloroethylene
GC/MS	EPA 8260B/C/D	trans-1,2-Dichloroethylene
GC/MS	EPA 8260B/C/D	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/C/D	trans-1,3-Dichloropropylene
GC/MS	EPA 8260B/C/D	cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260B/C/D	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260B/C/D	Di-isopropylether (DIPE)
GC/MS	EPA 8260B/C/D	Dibromochloromethane
GC/MS	EPA 8260B/C/D	Dibromomethane (Methylene Bromide)
GC/MS	EPA 8260B/C/D	Dichlorodifluoromethane
GC/MS	EPA 8260B/C/D	Diethyl ether
GC/MS	EPA 8260B/C/D; EPA 8260B/C/D SIM	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 8260B/C/D	Ethanol (Ethyl Alcohol)
GC/MS	EPA 8260B/C/D	Ethyl acetate
GC/MS	EPA 8260B/C/D	Ethyl methacrylate
GC/MS	EPA 8260B/C/D	Ethyl tert-butyl alcohol (ETBA)
GC/MS	EPA 8260B/C/D	Ethyl tert-butyl ether (ETBE)
GC/MS	EPA 8260B/C/D	Ethylbenzene
GC/MS	EPA 8260B/C/D	Ethylene Oxide
GC/MS	EPA 8260B/C/D	Hexachlorobutadiene
GC/MS	EPA 8260B/C/D	Hexane
GC/MS	EPA 8260B/C/D	Iodomethane (Methyl iodide)
GC/MS	EPA 8260B/C/D	Isobutyl alcohol (2-Methyl-1-propanol)
GC/MS	EPA 8260B/C/D	p-Isopropyltoluene
GC/MS	EPA 8260B/C/D	Isopropylbenzene
GC/MS	EPA 8260B/C/D	Methacrylonitrile
GC/MS	EPA 8260B/C/D	Methyl Acetate
GC/MS	EPA 8260B/C/D	Methyl bromide (Bromomethane)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C/D	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B/C/D	Methylcyclohexane
GC/MS	EPA 8260B/C/D	Methyl methacrylate
GC/MS	EPA 8260B/C/D	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260B/C/D	Methylene chloride
GC/MS	EPA 8260B/C/D	Naphthalene
GC/MS	EPA 8260B/C/D	Pentachloroethane
GC/MS	EPA 8260B/C/D	Propionitrile (Ethyl cyanide)
GC/MS	EPA 8260B/C/D	n-Propylbenzene
GC/MS	EPA 8260B/C/D	Styrene
GC/MS	EPA 8260B/C/D	tert-Amyl alcohol (TAA)
GC/MS	EPA 8260B/C/D	tert-Amyl methyl ether (TAME)
GC/MS	EPA 8260B/C/D	tert-Butyl alcohol (TBA)
GC/MS	EPA 8260B/C/D	tert-Butyl formate (TBF)
GC/MS	EPA 8260B/C/D	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260B/C/D	Tetrahydrofuran
GC/MS	EPA 8260B/C/D	Toluene
GC/MS	EPA 8260B/C/D	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260B/C/D	Trichlorofluoromethane
GC/MS	EPA 8260B/C/D	Vinyl acetate
GC/MS	EPA 8260B/C/D	Vinyl chloride
GC/MS	EPA 8260B/C/D	Xylene (total)
GC/MS	EPA 8260B/C/D	m,p-Xylene
GC/MS	EPA 8260B/C/D	o-Xylene
GC/MS	EPA 8270D/E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270D/E	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D/E	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 8270D/E	1,2-Diphenylhydrazine
GC/MS	EPA 8270D/E	1,3,5-Trinitrobenzene (1,3,5-TNB)
GC/MS	EPA 8270D/E	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 8270D/E	1,3-Dinitrobenzene (1,3-DNB)
GC/MS	EPA 8270D/E	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8270D/E	1,4-Naphthoquinone
GC/MS	EPA 8270D/E	1,4-Phenylenediamine
GC/MS	EPA 8270D/E	1-Chloronaphthalene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	1-Methylnaphthalene

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	EPA 8270D/E	1-Naphthylamine
GC/MS	EPA 8270D/E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270D/E	2,4,5-Trichlorophenol
GC/MS	EPA 8270D/E	2,4,6-Trichlorophenol
GC/MS	EPA 8270D/E	2,4-Dichlorophenol
GC/MS	EPA 8270D/E	2,4-Dimethylphenol
GC/MS	EPA 8270D/E	2,4-Dinitrophenol
GC/MS	EPA 8270D/E	2,4-Dinitrotoluene (2,4-DNT)
GC/MS	EPA 8270D/E	2,6-Dichlorophenol
GC/MS	EPA 8270D/E	2,6-Dinitrotoluene (2,6-DNT)
GC/MS	EPA 8270D/E	2-Acetylaminofluorene
GC/MS	EPA 8270D/E	2-Chloronaphthalene
GC/MS	EPA 8270D/E	2-Chlorophenol
GC/MS	EPA 8270D/E	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-o-cresol)
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	2-Methylnaphthalene
GC/MS	EPA 8270D/E	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270D/E	2-Naphthylamine
GC/MS	EPA 8270D/E	2-Nitroaniline
GC/MS	EPA 8270D/E	2-Nitrophenol
GC/MS	EPA 8270D/E	2-Picoline (2-Methylpyridine)
GC/MS	EPA 8270D/E	3,3`-Dichlorobenzidine
GC/MS	EPA 8270D/E	3,3`-Dimethylbenzidine
GC/MS	EPA 8270D/E	3-Methylcholanthrene
GC/MS	EPA 8270D/E	3&4-Methylphenol (m,p-Cresol)
GC/MS	EPA 8270D/E	3-Nitroaniline
GC/MS	EPA 8270D/E	4-Aminobiphenyl
GC/MS	EPA 8270D/E	4-Bromophenyl phenyl ether
GC/MS	EPA 8270D/E	4-Chloro-3-methylphenol
GC/MS	EPA 8270D/E	4-Chloroaniline
GC/MS	EPA 8270D/E	4-Chlorophenyl phenylether
GC/MS	EPA 8270D/E	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270D/E	4-Nitroaniline
GC/MS	EPA 8270D/E	4-Nitrophenol
GC/MS	EPA 8270D/E	5-Nitro-o-toluidine
GC/MS	EPA 8270D/E	7,12-Dimethylbenz(a) anthracene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Acenaphthene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Acenaphthylene
GC/MS	EPA 8270D/E	Acetophenone
GC/MS	EPA 8270D/E	Aniline
GC/MS	EPA 8270D; EPA 8270D SIM	Anthracene
GC/MS	EPA 8270D/E	Aramite
GC/MS	EPA 8270D/E	Atrazine
GC/MS	EPA 8270D/E	Benzaldehyde
GC/MS	EPA 8270D/E	Benzidine
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Benzo(a)anthracene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Benzo(a)pyrene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270D/E	Benzoic acid
GC/MS	EPA 8270D/E	Benzyl alcohol
GC/MS	EPA 8270D/E	Biphenyl (1,1'-Biphenyl)
GC/MS	EPA 8270D/E	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270D/E	bis(2-Chloroethyl) ether
GC/MS	EPA 8270D/E	bis(2-Chloroisopropyl) ether (2,2`-Oxybis(1-chloropropane))
GC/MS	EPA 8270D/E	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 8270D/E	Butyl benzyl phthalate
GC/MS	EPA 8270D/E	Carbazole
GC/MS	EPA 8270D/E	Caprolactam
GC/MS	EPA 8270D/E	Chlorobenzilate
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Chrysene
GC/MS	EPA 8270D/E	Diallate
GC/MS	EPA 8270D/E	Di-n-butyl phthalate
GC/MS	EPA 8270D/E	Di-n-octyl phthalate
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270D/E	Dibenz(a,j)acridine
GC/MS	EPA 8270D/E	Dibenzofuran
GC/MS	EPA 8270D/E	Diethyl phthalate
GC/MS	EPA 8270D/E	Dimethyl phthalate
GC/MS	EPA 8270D/E	a,a-Dimethylphenethylamine

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	EPA 8270D/E	Diphenyl Ether
GC/MS	EPA 8270D/E	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 8270D/E	Ethyl methanesulfonate
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Fluoranthene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Fluorene
GC/MS	EPA 8270D/E	Hexachlorobenzene
GC/MS	EPA 8270D/E	Hexachlorobutadiene
GC/MS	EPA 8270D/E	Hexachlorocyclopentadiene
GC/MS	EPA 8270D/E	Hexachloroethane
GC/MS	EPA 8270D/E	Hexachlorophene
GC/MS	EPA 8270D/E	Hexachloropropene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D/E	Isodrin
GC/MS	EPA 8270D/E	Isophorone
GC/MS	EPA 8270D/E	Isosafrole
GC/MS	EPA 8270D/E	Kepone
GC/MS	EPA 8270D/E	Methapyrilene
GC/MS	EPA 8270D/E	Methyl methanesulfonate
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Naphthalene
GC/MS	EPA 8270D/E	Nitrobenzene
GC/MS	EPA 8270D/E	Nitroquinoline-1-oxide
GC/MS	EPA 8270D/E	n-Nitroso-di-n-butylamine
GC/MS	EPA 8270D/E	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270D/E	n-Nitrosodiethylamine
GC/MS	EPA 8270D/E	n-Nitrosodimethylamine
GC/MS	EPA 8270D/E	n-Nitrosodiphenylamine
GC/MS	EPA 8270D/E	n-Nitrosodiphenylamine/Diphenylamine (analyte pair)
GC/MS	EPA 8270D/E	n-Nitrosomethylethylamine
GC/MS	EPA 8270D/E	n-Nitrosomorpholine
GC/MS	EPA 8270D/E	n-Nitrosopiperidine
GC/MS	EPA 8270D/E	n-Nitrosopyrrolidine
GC/MS	EPA 8270D/E	Pentachlorobenzene
GC/MS	EPA 8270D/E	Pentachloroethane
GC/MS	EPA 8270D/E	Pentachloronitrobenzene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Pentachlorophenol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270D/E	Phenacetin
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Phenanthrene
GC/MS	EPA 8270D/E	Phenol
GC/MS	EPA 8270D/E	Pronamide (Kerb)
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Pyrene
GC/MS	EPA 8270D/E	Pyridine
GC/MS	EPA 8270D/E	Safrole
GC/MS	EPA 8270D/E	Simazine
GC/MS	EPA 8270D/E	o-Toluidine
GC/MS	EPA 8270D/E	Dimethoate
GC/MS	EPA 8270D/E	Disulfoton
GC/MS	EPA 8270D/E	Famphur
GC/MS	EPA 8270D/E	Methyl parathion (Parathion methyl)
GC/MS	EPA 8270D/E	Parathion ethyl
GC/MS	EPA 8270D/E	Phorate
GC/MS	EPA 8270D/E	Sulfotepp
GC/MS	EPA 8270D/E	Thionazin (Zinophos)
GC/MS	EPA 8270D/E	O,O,O-Triethyl phosphorothioate
HPLC	EPA 8330A/B	1,3,5-Trinitrobenzene (1,3,5-TNB)
HPLC	EPA 8330A/B	1,3-Dinitrobenzene (1,3-DNB)
HPLC	EPA 8330A/B	2,4,6-Trinitrotoluene (2,4,6-TNT)
HPLC	EPA 8330A/B	2,4-Dinitrotoluene (2,4-DNT)
HPLC	EPA 8330A/B	2,6-Dinitrotoluene (2,6-DNT)
HPLC	EPA 8330A/B	2-Amino-4,6-dinitrotoluene (2-am-dnt)
HPLC	EPA 8330A/B	2-Nitrotoluene
HPLC	EPA 8330A/B	3,5-Dinitroaniline
HPLC	EPA 8330A/B	3-Nitrotoluene
HPLC	EPA 8330A/B	4-Amino-2,6-dinitrotoluene (4-am-dnt)
HPLC	EPA 8330A/B	4-Nitrotoluene
HPLC	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC	EPA 8330A/B	Nitrobenzene
HPLC	EPA 8330A/B	Nitroglycerin
HPLC	EPA 8330A/B	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
HPLC	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC	EPA 8330A/B	Pentaerythritoltetranitrate (PETN)
HPLC	EPA 8330A/B	DNX
HPLC	EPA 8330A/B	MNX
HPLC	EPA 8330A/B	TNX
LC/MS/MS	EPA 6850	Perchlorate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorobutanoic Acid (PFBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoropentanoic Acid (PFPeA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexanoic Acid (PFHxA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroheptanoic Acid (PFHpA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanoic Acid (PFOA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorononanoic Acid (PFNA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorodecanoic Acid (PFDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroundecanoic Acid (PFUnA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorododecanoic Acid(PFDoA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorotridecanoic Acid (PFTrDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorotetradecanoic Acid (PFTA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorobutanesulfonic Acid (PFBS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexanesulfonic Acid(PFHxS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctanesulfonic Acid(PFOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorononanesulfonic Acid(PFNS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorodecanesulfonic Acid(PFDS)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoroheptanesulfonic acid(PFHpS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoropentanesulfonic Acid(PFPeS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctane sulfonamide (PFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Methyl perfluorooctanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	4:2 Fluorotelomer Sulfonate (FTS 4:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	6:2 Fluorotelomer Sulfonate(FTS 6:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	8:2 Fluorotelomer Sulfonate (FTS 8:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	ADONA
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA; GenX)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS; F53B minor)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS; F53B major)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	3:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	5:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	7:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	10:2 Fluorotelomer sulfonate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorododecanesulfonic acid
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro-4-methoxybutanoic acid (PFMBA)

Solid and Chemical Materials

Technology	Method	Analyte
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorohexadecanoic acid (PFHxDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	Perfluorooctadecanoic acid (PFOcDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	4-PFecHS (Perfluoro-4-ethylcyclohexanesulfonate)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Methyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Ethyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Methyl perfluorooctane sulfonamidoethanol
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	N-Ethyl perfluorooctane sulfonamidoethanol
ICP	EPA 6010C/D	Aluminum
ICP	EPA 6010C/D	Antimony
ICP	EPA 6010C/D	Arsenic
ICP	EPA 6010C/D	Barium
ICP	EPA 6010C/D	Beryllium
ICP	EPA 6010C/D	Cadmium
ICP	EPA 6010C/D	Calcium
ICP	EPA 6010C/D	Chromium
ICP	EPA 6010C/D	Cobalt
ICP	EPA 6010C/D	Copper
ICP	EPA 6010C/D	Iron
ICP	EPA 6010C/D	Lead
ICP	EPA 6010C/D	Magnesium
ICP	EPA 6010C/D	Manganese
ICP	EPA 6010C/D	Molybdenum
ICP	EPA 6010C/D	Nickel
ICP	EPA 6010C/D	Potassium
ICP	EPA 6010C/D	Selenium
ICP	EPA 6010C/D	Silver
ICP	EPA 6010C/D	Sodium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	EPA 6010C/D	Strontium
ICP	EPA 6010C/D	Thallium
ICP	EPA 6010C/D	Tin
ICP	EPA 6010C/D	Titanium
ICP	EPA 6010C/D	Vanadium
ICP	EPA 6010C/D	Zinc
ICP/MS	EPA 6020A/B	Aluminum
ICP/MS	EPA 6020A/B	Antimony
ICP/MS	EPA 6020A/B	Arsenic
ICP/MS	EPA 6020A/B	Barium
ICP/MS	EPA 6020A/B	Beryllium
ICP/MS	EPA 6020A/B	Cadmium
ICP/MS	EPA 6020A/B	Calcium
ICP/MS	EPA 6020A/B	Chromium
ICP/MS	EPA 6020A/B	Cobalt
ICP/MS	EPA 6020A/B	Copper
ICP/MS	EPA 6020A/B	Iron
ICP/MS	EPA 6020A/B	Lead
ICP/MS	EPA 6020A/B	Magnesium
ICP/MS	EPA 6020A/B	Manganese
ICP/MS	EPA 6020A/B	Molybdenum
ICP/MS	EPA 6020A/B	Nickel
ICP/MS	EPA 6020A/B	Potassium
ICP/MS	EPA 6020A/B	Selenium
ICP/MS	EPA 6020A/B	Silver
ICP/MS	EPA 6020A/B	Sodium
ICP/MS	EPA 6020A/B	Strontium
ICP/MS	EPA 6020A/B	Thallium
ICP/MS	EPA 6020A/B	Tin
ICP/MS	EPA 6020A/B	Titanium
ICP/MS	EPA 6020A/B	Vanadium
ICP/MS	EPA 6020A/B	Zinc
CVAA	EPA 7471B	Mercury
UV/VIS	EPA 7196A	Hexavalent Chromium (Cr6+)

Solid and Chemical Materials

Technology	Method	Analyte
UV/VIS	EPA 9012B	Cyanide (Total)
IC	EPA 9056A	Bromide
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 9056A	Sulfate
IC	EPA 9056A	Total nitrate-nitrite
Gravimetric Methods	SM 2540G	% solids
Electrometric Methods	EPA 9045D	Hydrogen Ion (pH)
Ignitability	EPA 1010A MOD	Flash Point
Ignitability	EPA 1020B MOD	Flash Point
Waste Characterization	EPA Ch.7	Reactive Cyanide and Reactive Sulfide
Waste Characterization	EPA Section 7.3	Reactive Cyanide
Waste Characterization	EPA Section 7.3	Reactive Sulfide
Preparation	**Method**	**Type**
Organics Preparation	EPA 3510C	Separatory Funnel Liquid-Liquid Extraction; Leachates
TCLP Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
SPLP Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Organics Preparation	EPA 8011	Microextraction
Organics Preparation	EPA 3546	Microwave Extraction
Organics Preparation	EPA 3550C	Ultrasonic Extraction
Organics Preparation	EPA 3580A	Waste Dilution for Extractable Organics
Organics Preparation	EPA 8330A; EPA 8332	Ultrasonic Extraction
Organics Preparation	EPA 8330B	Shaker Table Extraction
Volatile Organics Preparation	EPA 3585	Waste Dilution for Volatile Organics
Volatile Organics Preparation	EPA 5030A	Closed System Purge and Trap; Bulk Soils
Volatile Organics Preparation	EPA 5030B	Closed System Purge and Trap; Leachates and Methanol Extracts
Volatile Organics Preparation	EPA 5035; EPA 5035A	Closed System Purge and Trap
Organics Cleanup	EPA 3660B	Sulfur Cleanup
Organics Cleanup	EPA 3665A	Sulfuric Acid Cleanup

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Lachat MicroDistillation	EPA 9012B	Cyanide MicroDistillation; proprietary method
Inorganic Preparation	EPA 3010A	Metals Acid Digestion by Hotblock; Leachates
Inorganic Preparation	EPA 3050B	Metals Acid Digestion by Hotblock
Inorganic Preparation	EPA 3060A	Alkaline Digestion, Cr6+
Inorganic Preparation	EPA 7470A	CVAA Digestion by Hotblock; Leachates
Inorganic Preparation	EPA 7471B	CVAA Digestion by Hotblock

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2229.

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