DRAFT
UNIFORM FEDERAL POLICY
QUALITY ASSURANCE PROJECT PLAN
REMEDIAL DESIGN

BLACKTAIL CREEK RIPARIAN ACTIONS
REMEDIAL DESIGN WORK PLAN
AND PRE-DESIGN INVESTIGATION
BUTTE PRIORITY SOILS OPERABLE UNIT OF THE
SILVER BOW CREEK/BUTTE AREA SUPERFUND SITE
SILVER BOW COUNTY, MONTANA

Prepared for:

Montana Department of Environmental Quality
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Task Order 04 under DEQ Contract No. 421042

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January 2023

TABLE OF CONTENTS

Page

LIST OF APPENDICES

LIST OF FIGURES

LIST OF ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
%D	percent difference
%R	percent recovery
%RSD	percent relative standard deviation
mg/kg	milligrams per kilogram
ARCO	Atlantic Richfield Co.
BAO	Butte Area One
bgs	below ground surface
BPSOU	Butte Priority Soils Operable Unit
BRW	Butte Reduction Works
B.S.	Bachelor of Science
BTC	Blacktail Creek
CA	corrective action
CHMM	Certified Hazardous Materials Manager
CIH	Certified Industrial Hygienist
CLP	Contract Laboratory Program
CoC	chain of custody
CQA	Certified Quality Auditor
CSP	Certified Safety Professional
CVAA	cold vapor atomic absorption
DMP	Data Management Plan
DPT	direct-push technology
DQI	data quality indicator
DQO	data quality objective
EB	equipment blank
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
FSP	Field Sampling Plan
ft	feet or foot
FTL	Field Team Leader
GIS	geographic information system
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HGL	HydroGeoLogic, Inc.
H&S	health and safety

ICP-MS	inductively coupled plasma-mass spectrometry
ID	identification
IDQTF	Intergovernmental Data Quality Task Force
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
MB	method blank
MBMG	Montana Bureau of Mines and Geology
MDEQ	Montana Department of Environmental Quality
MDL	method detection limit
M.S.	Master of Science
MS	matrix spike
MSD	matrix spike duplicate
NA	not applicable
NFG	National Functional Guideline
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
P.E.	Professional Engineer
P.G.	Professional Geologist
PM	project manager
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RA	Remedial Action
RAO	remedial action objective
RD	Remedial Design
RL	reporting limit
RPD	relative percent difference
SBC	Silver Bow Creek
SOP	Standard Operating Procedure
SOW	Statement of Work
UFP	Uniform Federal Policy
XRF	x-ray fluorescence

UFP-QAPP Revision Tracking Table
Blacktail Creek Riparian Actions Remedial Design Work Plan and Pre-Design Investigation
Silver Bow County, Montana

Revision Number	Date	Section Revised	Changes/Comments
0	August 2022	NA	Original version/draft
1	January 2023	Worksheet 21	Draft

NA = not applicable

UNIFORM FEDERAL POLICY-QUALITY ASSURANCE PROJECT PLAN
REMEDIAL DESIGN
BLACKTAIL CREEK RIPARIAN ACTIONS
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INTRODUCTION

This Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP) has been prepared by HydroGeoLogic, Inc. (HGL) for the Montana Department of Environmental Quality (MDEQ), Contract 421042. Project activities covered under this task order are to support Remedial Design (RD) efforts at the Blacktail Creek (BTC) Riparian Actions area, located in Silver Bow County, Montana.

This UFP-QAPP presents the requirements for pre-design investigation activities and for quality assurance (QA) and quality control (QC) support during these activities to be conducted by HGL.

This plan is specific to the BTC Riparian Actions area and meets the requirements and elements set forth in the U.S. Environmental Protection Agency (EPA) guidance document entitled, *Uniform Federal Policy for Quality Assurance Project Plans* (IDQTF, 2005), with the optimized worksheets developed in 2012 (IDQTF, 2012). It also includes supplemental information and requirements, as necessary, to support Site-specific objectives. The scope of the work to be performed was provided by MDEQ in the *MDEQ Statement of Work – Blacktail Creek Riparian Actions Remedial Design Work Plan and Pre-Investigation Task Order.*

WORKSHEETS #1 AND #2
TITLE AND APPROVAL PAGE

Draft, UFP-QAPP, BTC Riparian Actions RD Work Plan and Pre-Design Investigation, Silver Bow County, Montana
Document Title

MDEQ
Lead Organization

Drew Herrera, HGL
Preparer's Name and Organizational Affiliation

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August 2022
Preparation Date

MDEQ Project Manager (PM):

Signature/Date

William George
Printed Name/Organization

MDEQ QA Officer:

Signature/Date

Printed Name/Organization

Lead Contractor's PM:

Signature/Date

Drew Herrera/HGL
Printed Name/Organization

Lead Contractor's Project QC Manager:

Signature/Date

Chris Williams/HGL
Printed Name/Organization

Worksheets #1 and #2 (continued)
Title and Approval Page

Site Name/Project Name: BTC Superfund Site RD
Site Location: Silver Bow County, Montana
Contractor Name: HGL
Contract Number: 421042
Task Order Number: 04

1. Identify guidance used to prepare the UFP-QAPP: EPA Intergovernmental Data Quality Task Force (IDQTF) Workbook for UFP-QAPPs, Part 2A, 2005; optimized worksheets developed in 2012, EPA IDQTF, 2012.

2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act, Superfund Amendments and Reauthorization Act of 1986, Resource Conservation and Recovery Act, and National Oil and Hazardous Substances Pollution Contingency Plan programs.

3. Identify approval entities: See signature page 2.

4. The UFP-QAPP is: Project-specific.

5. List dates of scoping sessions that were held: Initial project kickoff/scoping meeting was held on April 12, 2022.

6. List dates and titles of UFP-QAPP documents written for previous site work, if applicable: Not applicable for this work.

7. List organizational partners (stakeholders): MDEQ, EPA Region 8.

8. List data users: MDEQ, EPA Region 8, HGL.

9. UFP-QAPP elements and required information: All UFP-QAPP worksheets are included.

WORKSHEETS #3 AND #5
PROJECT ORGANIZATION AND UFP-QAPP DISTRIBUTION

Distribution:
The following is the distribution list for the UFP-QAPP for the BTC Montana Superfund Site.

UFP-QAPP Recipients	Title	Organization	Telephone Number	Email Address
William George	PM	MDEQ	(406) 422-8870 / (406) 444-6420	william.george@mt.gov
Drew Herrera	PM	HGL	(307) 680-0026	aherrera@hgl.com
Ken Rapuano	Project Chemist	HGL	(703) 736-4546	krapuano@hgl.com
Chris Williams	QC Manager	HGL	(913) 647-2536	cwwilliams@hgl.com

Project Organization:
The roles and communication pathways for project personnel are presented in Worksheets #4, #7, and #8, and Worksheet #6, respectively. An organizational chart showing reporting relationships and communication pathways is provided as Figure 3.1.

Figure 3.1 Organizational Chart

CHMM = Certified Hazardous Materials Manager
CIH = Certified Industrial Hygienist
CQA = Certified Quality Auditor
CSP = Certified Safety Professional
H&S = health and safety
P.E. = Professional Engineer
P.G. = Professional Geologist

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

Project personnel are required to read this UFP-QAPP and sign off that they have done so before initiating activities. The qualifications of Federal and State regulatory stakeholders are under the purview of their respective agencies and are not presented in this UFP-QAPP. Personnel resumes and training/certification records are on file at HGL offices and can be provided for review upon request.

Organization: HGL

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Drew Herrera	PM	B.S., Civil Engineering: 13 years	P.E., 8-hour HAZWOPER Refresher Training	
Ken Rapuano	Project Chemist	B.S., Chemistry, M.S. Chemistry Experience: 35 years	CQA, CHMM 8-hour HAZWOPER Refresher Training	
Chris Williams	QC Manager	B.S., Geology Experience: 36 years	P.G., 8-hour HAZWOPER Refresher Training, Site Supervisor Training	

B.S. = Bachelor of Science
HAZWOPER = Hazardous Waste Operations and Emergency Response
M.S. = Master of Science

WORKSHEET #6
COMMUNICATION PATHWAYS

Communication Driver	Organization	Name	Contact Information	Procedure
Regulatory agency interface	MDEQ	William George (PM)	422-8870/406-444-6420 william.george@mt.gov	Primary point of contact for MDEQ.
Point of contact with MDEQ	HGL	Drew Herrera (Senior PM)	(307) 680-0026 aherrera@hgl.com	Project-related issues, including changes in schedule or scope, will be communicated to MDEQ by phone or email. Project information will be reported to MDEQ through monthly progress reports, email updates, teleconferences, and meetings. The HGL PM will document deviations from the UFP-QAPP and any corrective actions (CAs) and will report them to the MDEQ PM.
UFP-QAPP changes	HGL	Ken Rapuano	(703) 736-4546 krapuano@hgl.com	If errors or changed conditions require modification of the UFP-QAPP, the HGL Project Chemist will prepare revised text in collaboration with the PM and QC Manager. All changes to the UFP-QAPP will require final approval from MDEQ.
Overall project QA	HGL Project QC Manager	Chris Williams	(913) 647-2536 cwwilliams@hgl.com	Communicate program QA/QC requirements to the HGL PM and HGL project team. Determine need to develop procedural changes to address QA/QC issues.

WORKSHEET #9
PROJECT SCOPING SESSION PARTICIPANTS SHEET

Date of Planning Session: April 12, 2022

Location: Teleconference

Purpose: Project Kickoff/Scoping Meeting

Participants:

Name	Organization	Title/Role	Email
William George	MDEQ	Project Manager	william.george@mt.gov
Carolina Balliew	MDEQ	Section Supervisor	carolina.balliew@mt.gov
Drew Herrera	HGL	Senior Project Manager	aherrera@hgl.com
Don Sutton	HGL	Project Engineer	donaldsutton@hgl.com
Chris Robb	HGL	Project Engineer	crobb@hgl.com
Mark Blanchard	HGL	Denver Office Manager	mblanchard@hgl.com

Notes/Comments:

The scoping meeting clarified the work to be performed, including review of project data and schedule. HGL tasks will focus on review of background information, preparation of planning documents, a Pre-Design Investigation Work Plan, field data collection, flow and floodplain evaluations, waste volume estimates, dewatering volume estimates, geotechnical conditions evaluation, RD planning, and reporting. Field data will be collected to support the RD If project needs change, the UFP-QAPP may be revised to meet those needs.

Consensus Decisions Made: Not Applicable.

Action Items: Not Applicable.

WORKSHEET #10
CONCEPTUAL SITE MODEL

Environmental Problem

The primary goal of this project is to support the Pre-Design Investigation to address data gaps by conducting additional field investigations at the BTC Riparian Actions Area in Silver Bow County, Montana. Ultimately, this support will assist MDEQ Remedial Action (RA) Contractors perform remedial activities at the Silver Bow County Sites through technical support, design, and review of QA/QC measures.

Site Location and History

In 1983, the State of Montana filed a lawsuit in Federal District Court against the Atlantic Richfield Co. (ARCO) for injuries to the natural resources in the Upper Clark Fork River Basin, which extends from Butte to Milltown, MT. The Montana v. ARCO lawsuit, brought under Federal and State Superfund laws, sought damages from ARCO, contending that decades of mining and smelting in the Butte and Anaconda areas had greatly harmed natural resources in the basin and deprived Montanans of the use of these resources.

The State settled Montana v. ARCO through a series of settlement agreements, or consent decrees, completed and approved by the court in 1999, 2005, and 2008. One of the three injured areas in the Upper Clark Fork River Basin covered under the 2008 settlement agreement was the Butte Area One (BAO) injured groundwater and surface water site.

The BTC Riparian Actions Area will be investigated to address data gaps and satisfy design needs for the integration of restoration with the remedy of mining and mineral processing wastes in the Silver Bow Creek (SBC) and BTC Corridors. The BTC riparian corridor is within the boundaries of the Butte Priority Soils Operable Unit (BPSOU), shown on Figure 10.1. DEQ's obligations for the BTC Riparian Actions are outlined within the amended record of decision for BPSOU and the finalized Consent Decree and include the removal of tailings, wastes, and contaminated soils and sediment from BTC and SBC below the confluence with BTC wetlands as well as reconstruction of BTC and SBC below the confluence with BTC. Additionally, settlings defendants are responsible for the control of discharge of contaminated groundwater to surface water in the project area. The study area covered by this investigation work is to be performed within the approximate boundaries shown in Figure 10.2 (BTC Riparian Actions Study Area).

BTC receives the majority of its base flow contributions from Summit Valley groundwater in Butte, MT. The stream intersects both the BAO injured area restoration site and BPSOU. The BTC Riparian Actions Area, which is the focus of this data gap investigation, extends from BTC 250-feet (ft) east of Lexington Avenue, just past the confluence with Grove Gulch Creek, including its banks; the 100-year floodplain between George Street and Lexington Avenue Culverts; and the 100-year floodplain below the confluence of BTC and SBC north of George Street and East of Montana Street.

In 1879, the first large-scale mineral processing smelter (Colorado Smelter) was built on SBC, at the west end of the valley. Between 1879 and 1888, at least three more smelters of consequence (Butte Reduction Works [BRW], Parrot Smelter and Montana Ore Purchasing Company) were constructed upstream of the Colorado Smelter, which significantly altered the geomorphology and

hydrology of both SBC and the lower portion of BTC. A fifth smelter of consequence, the Bell Smelter, located west of present-day Harrison Avenue on the north bank of BTC, was constructed in 1881 and reached a peak production of approximately 30 tons per day in 1883 (primarily silver ore). Production quickly tapered and the smelter was dismantled sometime in the early 1890s. Water demands during this period increased dramatically, and the stream channels were altered significantly to keep up with the demand. At least three dams were constructed on upper SBC and the confluence area for tailings impoundment and water clarification. The dam at Montana Street was constructed for settlement of tailings from upstream smelters and resulted in significant ponding on both sides of the stream. Over time, mining and smelting waste materials aggraded in the SBC and BTC channels and floodplain, causing frequent and substantial flooding (Meinzer, 1914). In an attempt to mitigate flooding issues, berms made mostly of readily available waste were constructed throughout the confluence area. The known waste area referred to as the BTC Berm is an historic remnant of these flood control berms.

Previous Investigations and Remedial Actions

Data characterizing contaminated materials in the vicinity of the Blacktail berm are limited. In May 2013, the Montana Bureau of Mines and Geology (MBMG) conducted trenching as well as test pit and borehole investigations in known and suspected mine waste areas of the BTC and SBC confluence in Butte (MBMG, 2014a). In particular, the BTC Berm area was evaluated for contaminant concentrations and volumes of impacted sediments. This work was done to quantify the aerial extent and depth of tailings and impacted sediments for an updated characterization and volume estimate of tailings and mining impacted sediments for the State of Montana. Five soil borings were advanced in the BTC Berm to characterize the subsurface material.

The MBMG investigation showed that the BTC Berm contained tailings/impacted soils that exceeded criteria for constituents of concern above established criteria. The berm does not have thick overlying fill material, with tailings near the surface. Because the majority of soil samples collected just above the water table exceeded contaminant criteria, it was recommended that potential future removals include soils down to the water table. The majority of organic silt samples met the classification of impacted sediment. In total, the volume of tailings and impacted soils was estimated at 14,000 cubic yards.

During baseflow conditions in 2011, the MBMG conducted a continuous bromide tracer injection in the BTC and upper SBC confluence area on behalf of the Natural Resources Damage Program (MBMG, 2014b) using a sodium bromide solution. The MBMG report concluded that discharge in BTC between Oregon Avenue and George Street increased by 2.2 cubic ft per second, that is, by approximately 22 percent.

Wetlands located adjacent to BTC received the majority (99 percent) of recharge from local groundwater sources and contributed 39 percent of the flow increase observed in the studied reach of BTC (Oregon Avenue to George Street). The remaining baseflow contributions (61 percent) in BTC were groundwater inputs into the stream. The tracer study indicated that two reaches of BTC are non-gaining reaches; moreover, they may be net-losing reaches (MBMG, 2014b). Gains in streamflow were not observed in SBC from a point just downstream of Slag Wall Canyon at surface sample site SS-06 to the pumping vault on upper SBC. Metals loading assessments indicate that while there appears to be source areas for copper and zinc loading to the stream, concentrations

of contaminants of concern (arsenic, cadmium, copper, lead, and zinc) remained below Circular DEQ-7 acute and chronic life standards for dissolved concentrations throughout the study area (MBMG, 2014b). Total recoverable copper and zinc concentrations were elevated in surface water samples collected from the BTC reach from near the Lexington Avenue overpass to the confluence of BTC with SBC. Surface water samples collected from one main stem, one wetland, and two tributary samples exceeded Circular DEQ-7 acute and chronic life standards for total copper, while the two tributary samples exceeded Circular DEQ-7 acute and chronic life standards for total zinc. The sources of total recoverable copper and zinc to this area of BTC are thought to be either bed sediment loads or nearby streambank sediment (i.e., BTC Berm) or loading from historic Grove Gulch discharges. Surface water samples collected from the two wetlands, located along BTC in the BTC Berm area, exhibited water quality with elevated concentrations of copper and zinc. Both of the wetlands contributed measurable flow into BTC and are potential point sources. Concentrations of contaminants in the groundwater that recharges the wetlands near Lexington Avenue were not assessed; therefore, groundwater entering the wetlands could not be ruled out as a contaminant potential source.

TetraTech completed a Data Gap Investigation report in 2016 (TetraTech, 2016) to summarize data collected to evaluate soil, sediment, surface water, groundwater, and pore water sampling pertaining to characterization of mine wastes located at the BTC Berm area and within the historical floodplain deposits associated with the BTC and SBC riparian corridors.

Flood plain soil and mine waste samples were collected using a combination of test pits, direct-push technology (DPT) soil borings, and hand tools. X-ray fluorescence (XRF) screening of soils was also used in this investigation. Based on the magnitude and extent of contamination detected in the investigation, the revised estimated volume for the BTC Berm area after applying the additional site data, revising the assumptions, and kriging the base surface elevation was 100,185 cubic yards.

The in-stream sediment and pond sediment sampling portion of the data gap site investigation of the SBC and BTC riparian corridors consisted of collecting and sampling sediment at stream and pond stations. Sediment sample locations were co-located with surface water sampling and with pore water sampling. In general, total metals appear to concentrate in the in-stream sediments from the mouth of Grove Gulch down to the confluence with SBC and continue downstream through Slag Canyon and BRW area. In addition, metals appear to concentrate in pond sediments in two of the three wetland ponds. The increasing metals load to BTC below the mouth of Grove Gulch indicate that a possible source of metals to BTC is the Grove Gulch tributary, and the former zinc mill site located in its headwaters. Other metals trend somewhat differently, with obvious increases noted downstream of the former Bell Smelter site on BTC just downstream of Harrison Avenue as well as below the mouth of Grove Gulch. Dissolved metals transport in groundwater and precipitation on the mineral grains of the in-stream sediments and pond sediments can also not be discounted as a potential source of metals loading to the SBC and BTC riparian corridors since the gaining reaches of BTC and SBC correspond to the reaches below the Kaw/Lexington Avenue Overpass and mouth of Grove Gulch.

The surface water and pond water sampling portion of the data gap site investigation was conducted at 18 surface and 3 pond water sample stations. Based on surface water sampling results,

surface water with the highest concentration of total metals of arsenic, cadmium, copper, and lead were from wetland pond samples located immediately west of Kaw Avenue within the BTC Berm area and not from the active stream channels or tributary channels within the study area.

The in-stream sediment pore water and pond sediment pore water sampling portion of the data gaps site investigation of the SBC and BTC riparian corridors consisted of collecting and analyzing 53 natural in-stream sediment pore water samples from within the active stream channels and 4 pond sediment pore water samples from 3 wetland ponds. In general, dissolved contaminants in pore water appeared to be highest in sections of streams or wetland ponds that contain elevated contaminants in sediment, with notable exceptions such as Grove Gulch (sediment pore water did not exceed surface water quality standards) or in a few upstream reaches on BTC that are only marginally impacted with respect to streambed metals yet exceed the arsenic surface water quality standard (2 samples), the copper standard (1 sample), and the iron standard (multiple samples). Iron concentrations in pore water may not be related to mining activities, as any reducing conditions due to decay of organic material in groundwater or pore water can mobilize naturally occurring iron.

The groundwater sampling portion of the data gap site investigation included sampling 32 existing wells and 3 newly installed DPT piezometers. Based on the sampling results, groundwater with the highest concentrations of arsenic, cadmium, and/or zinc were observed in 3 primary areas: the SWC/BRW area, SBC/BTC confluence and BTC Berm area, and Northside Tailings/Diggings East areas. Groundwater east of Lexington Avenue did not exceed water quality standards for the metals analyzed for during this investigation.

Tetra Tech conducted two limited-duration, single-well pumping tests on BTC Berm Area monitoring well AMW-11. The purpose of the testing was to determine aquifer properties that would be expected to occur during construction dewatering. Based on this testing, the mean values for aquifer transmissivity and hydraulic conductivity were 591 square ft per day and 59 ft per day, respectively.

WORKSHEET #11
PROJECT/DATA QUALITY OBJECTIVES

This worksheet develops the data quality objectives (DQO) for the BTC Riparian Actions Area using a systematic planning process in accordance with EPA QA/G-4, *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA, 2006). The DQOs are developed separately below.

1. **State the Problem**. MDEQ is planning to perform remedial tasks at the BTC Riparian Actions Area. HGL's scope for this project is to develop Site-specific project plans to be approved by MDEQ and other stakeholders before work begins. Per the project Scope of Work, the plans to be developed are the Site Management Plan, a UFP-QAPP to include a Field Sampling Plan (FSP) and a Data Management Plan (DMP), and a Health and Safety Plan (HASP). The FSP is presented in this UFP-QAPP in Worksheets #17 through #22, while the DMP is presented in Worksheets #34 through #37. Other work to be performed under this task order includes fieldwork to collect additional site characterization data, review of background information, preparation of a technical memorandum summarizing mine waste disposal options, RD, and post-RD technical support during RA construction.
2. **Identify the Goals of the Project**. The overall goal of the task order is to provide technical support to MDEQ so that subsequent RAs are designed and constructed to meet project requirements in compliance with the Consent Decree and project objectives. Following acceptance of Site-specific plans, HGL will conduct field data collection activities and report findings from these activities, which will be used to support subsequent remedial designs. Data collection activities will include collection of information for flow and floodplain evaluations, tailings and contaminated soil and sediment volume estimates, estimation of excavation dewatering requirements, and geotechnical conditions within the study area. Throughout the project, HGL will provide project management support, including participating in monthly project status meeting, preparing monthly progress reports, monitoring project costs, preparing invoices, and managing the project schedule.
3. **Identify Information Inputs**. HGL will review historical background documents provided by MDEQ to provide a full understanding of the history and objectives for the project.
4. **Define the Boundaries of the Study**. The boundaries of BTC Riparian Actions Area are shown on Figure 10.2.
5. **Develop the Analytic Approach**. Sampling and analysis tasks are outlined in Worksheets #14 and #16. HGL will perform technical review and evaluation of the analytical data and prepare reports to support the project. Sample results will be evaluated against the remedial action objectives (RAOs) provided on Worksheet #15.
6. **Specify Performance or Acceptance Criteria**. Analytical QC data associated with project sample results will be compared to the measurement performance criteria of each data quality indicator (DQI), listed on Worksheet #12, to determine data quality and whether sample results are acceptable based on the established DQOs. The RAOs and sensitivity limits are specified on Worksheet #15. Analytical data will be compared to these limits. If three of the five criteria specified in Worksheet #15 are exceeded, or if any one contaminant

concentration exceeds 5,000 milligrams per kilogram (mg/kg), the material is considered tailings, waste, or contaminated soil.

7. **Develop the Detailed Plan for Obtaining Information**. The specific project tasks and schedule for data collection are located in Worksheets #14 and #16. Details on the sampling locations and field sampling procedures are presented in Worksheets #17 and #18. HGL will be responsible for all sample collection, shipment, and management. HGL also will coordinate with MDEQ for shipment of samples to the analytical laboratory, perform data validation on analytical sample results, and provide laboratory and validated data to MDEQ. Validation criteria are included in Worksheets #34, #35, and #36, and data usability assessment is discussed in Worksheet #37. Definitive data will be required for all data that will be used for comparison to RAOs.

WORKSHEET #12
MEASUREMENT PERFORMANCE CRITERIA

12.0 MEASUREMENT PERFORMANCE CRITERIA

Measurement performance criteria usually are expressed in terms of the DQI precision, accuracy, representativeness, completeness, comparability, and sensitivity, which are known collectively as precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). Of the PARCCS parameters, precision, accuracy, completeness, and sensitivity can be quantitatively measured and assessed. The parameters of comparability and representativeness are primarily qualitative in nature. The specific DQIs associated with each analytical method are presented in the method-specific tables included at the end of this worksheet.

12.1 QUANTITATIVE DATA QUALITY INDICATORS

12.1.1 Precision

Precision is the measure of variability between individual sample measurements under prescribed conditions. Precision can be assessed by replicate measurements of known laboratory standards and by analysis of duplicate environmental samples (spiked or unspiked). Precision is determined by evaluating the relative percent difference (RPD) between duplicate sample results. Replicate measurements of known standards (laboratory control sample [LCS]/laboratory control sample duplicate [LCSD] pairs), spiked samples (matrix spike [MS]/matrix spike duplicate [MSD] pairs), and laboratory duplicate analyses are routinely monitored by the laboratory by comparing the RPD with established control limits. The formula for calculating RPD is as follows:

$$RPD = \frac{|S - D|}{\frac{(S + D)}{2}} x100$$

where:
S = first sample value (original sample value); and
D = second sample value (duplicate sample value).

For this investigation, the field precision objective for discrete soil sample duplicates will be an RPD less than 50 percent. Failure of RPDs in duplicates should warrant a review of sample collection especially for soil homogenization. The precision objective for laboratory QC (MS/MSD and LCS/LCSD pairs, laboratory duplicates) will be an RPD less than 20 percent. Failure of RPDs in laboratory QC samples will be addressed in accordance with the laboratory analytical standard operating procedure (SOP).

12.1.2 Accuracy

Accuracy is the degree of agreement of a measurement to an accepted reference or true value. An evaluation of the accuracy of a measurement system provides an estimate of measurement bias. Overall analytical accuracy is assessed on a batch-specific basis by evaluating the percent recovery (%R) of known concentrations for each analyte in the LCS (and LCSD) against the QC limits. One known reference standard or LCS is analyzed for every batch (maximum of 20 samples). The accuracy of specific sample analyses is assessed by evaluating the %R of the surrogate spike

compounds (organic analyses). The %R QC criteria for MS/MSDs will be used to assess the potential for matrix interferences. The formula for calculating %R is as follows:

$$\%R = \frac{A - B}{C} \times 100$$

where:

A = the analyte concentration determined experimentally from the spiked sample;
B = the background level determined by a separate analysis of the unspiked sample (for calibration standards, LCSs, and surrogate compounds, the value of this term is zero); and
C = the amount of the spike added.

Accuracy is also measured using percent difference (%D) between a result and the expected value. The %D is usually used to evaluate accuracy when the acceptance of a QC result is dependent on another analytical result and not on a pre-defined window of acceptance. The formula for calculating %D is as follows:

$$\%D = \frac{A - B}{A} \times 100$$

where:

A = the original quantity measured, and
B = the comparison quantity measured.

The accuracy objectives for this project are presented in Table 12.1. Failure of accuracy QC elements in laboratory QC samples will be addressed in accordance with the laboratory analytical SOP.

12.1.3 Completeness

Completeness is a measure of the amount of valid data obtained compared with the amount that was expected to be obtained under correct, normal conditions. It is calculated for the aggregation of data measured for any specific sampling event or other defined set of samples (such as by site). Valid data is data which is usable in the context of the project goals and DQOs. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the dataset.

Field completeness is defined as the percentage of analytical results obtained compared with the projected number of analytical results that would be obtained from all planned sample locations. The formula for calculating sampling completeness is as follows:

$$\text{Field Completeness} = \frac{\text{Number of Data Points Obtained}}{\text{Number of Planned Data Points}} \times 100\%$$

Analytical completeness is defined as the percentage of valid (nonrejected) analytical results obtained from measurement systems compared with the total number of analytical results requested. The formula for calculating analytical completeness is as follows:

$$\text{Analytical Completeness} = \frac{\text{Number of Acceptable Laboratory Measurements}}{\text{Number of Laboratory Measurements Reported}} \times 100\%$$

The completeness objectives for this project will be field, laboratory, and overall completeness each greater than 90 percent.

12.1.4 Sensitivity

Sensitivity is defined as the capability of a method or instrument to discriminate between measurement responses representing different levels of a variable of interest. The sensitivity limits of project methods are presented in Worksheet #15.

The method detection limit (MDL) as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99 percent level of confidence. At the MDL, the false positive rate (Type I error) is 1 percent. MDLs are specific to an individual determination performed at an individual laboratory.

The reporting limit (RL) is the lowest concentration that produces a quantitative result within specified limits of precision and bias. Detected analytical results with quantitation at or above the MDL but below the RL will reported as detections by the laboratory with the qualification "J." Detected analytical results at or above the RL will be reported without qualification unless affected by a QC issue.

12.2 QUALITATIVE DATA QUALITY INDICATORS

12.2.1 Representativeness

Representativeness is the degree to which data accurately and precisely expresses a characteristic of a population, the 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 (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 A. The sample containers and preservation methods presented in Worksheet #19 and #30 will be used to ensure that samples arriving at the laboratory retain the appropriate degree of representativeness. The holding times presented in Worksheet #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. A method blank (MB) will be analyzed with every analytical or preparation batch (as appropriate to the analytical

method) to determine potential contamination introduced during routine laboratory procedures. Initial calibration blanks and continuing calibration blanks will be analyzed, as required, by analytical methods. Equipment blanks (EBs) will be collected to assess potential contamination due to field conditions (Worksheet #20). The assessment of blank samples will determine if compounds detected in the environmental samples are site-related or have been introduced through shipping, storage, field procedures, or laboratory procedures.

12.2.2 Comparability

Comparability expresses the confidence with which one dataset can be compared to another. Comparability also involves a multistep evaluation and can be related to accuracy and precision as these quantities are measures of data reliability. Data is comparable if site considerations; collection techniques; and measurement procedures, methods, and sensitivity limits are equivalent for the samples within a sample set.

For this project, comparability will be ensured through the use of the appropriate SOPs for the collection and shipment of samples. The laboratory analytical methods are definitive and use widely available technologies.

WORKSHEET #12.1
MEASUREMENT PERFORMANCE CRITERIA TABLE – METALS ANALYSES

Analytical Group	Metals (Arsenic, Cadmium, Copper, Lead, Zinc) and Mercury	
Analytical Method	EPA 6020B and 7471B	
Matrix	Soil/Sediment	
DQI	**QC Sample or Measurement Performance Activity**	**Measurement Performance Criteria**
Precision	Field Duplicate	≤ 50% RPD[1]
Accuracy	LCS, LSCD, MS, and MSD %R	LCS/LCSD – 80%-120% MS/MSD – 75%-125% MS/MSD – 80%-120% (mercury only)
Precision[1]	LCSD and MSD RPD	≤ 20% RPD
Representativeness	Equipment Rinse Blank	Not detected > RL
Representativeness	Laboratory Method Blank	No analytes detected > ½ the RL
Sensitivity	Laboratory MDL determination and verification	≤ RL
Completeness	Not applicable	≥ 90%

[1] For low-level results (detected value ≤5x RL) or when one result is a nondetection, the control limit is absolute difference ≤ RL. Nondetected values will be assigned the nominal value of the RL for making this comparison.

WORKSHEET #13
SECONDARY DATA USES AND LIMITATIONS

This worksheet includes examples of the data sources that may be used in completion of this task order. This list is representative and does not include all data sources HGL may use.

Data type	Source	Data uses relative to current project	Factors affecting the reliability of data and limitations on data use
Data Gap Investigation Technical Memorandum	TetraTech, July 2016	Provides summary of investigation results and background conditions and is to be used as a basis for the currently proposed field data collection activities.	Relevance of previous data collection methods, locations, and depths are subject to evaluation and can reveal additional data gaps to be filled.
Consent Decree, BPSOU, with Appendices	EPA, 2006	Provides project goals, including remedial actions and cleanup levels.	May need to consult with MDEQ to determine whether any cleanup levels have been updated.
Stream Characterization Report	MBMG, 2014b	Provides site background and tracer studies on adjacent water bodies.	Unknown.

WORKSHEETS #14 AND #16
PROJECT TASKS AND SCHEDULE

HGL will update the project schedule during the project as requested by the MDEQ. This UFP-QAPP will be reviewed and updated as necessary in response to changes in the initial project conditions. The field data collection tasks to be performed to support the task order RDs are described below.

Sampling Tasks:

- A summarized list of sampling tasks, broken out by locations, is provided below. For more details per task, refer to Worksheet #17, Worksheet #18, Worksheets #19 and #30, Worksheet #20, and Worksheets #26 and #27. Potential soil sampling locations are depicted on Figure 18.1. Soil cores will be collected from each of these locations. On average, boring depths will be approximately 20 feet, and soils from the cores will be screened in 5-foot intervals.

Sampling Schedule

- Field screening and sampling is scheduled to be performed in late Fall 2022, depending on when the required site access agreements are obtained.

Analysis Tasks:

The following analyses will be performed as part of this project: Metals (arsenic, cadmium, copper, lead, mercury, and zinc).

- Soil samples will be collected at the locations and from the depths shown on the table included on Worksheet #18.
- All samples collected will be screened in the field using XRF methods. No sample preparation (sieving, drying) will be performed on these samples prior to screening.
- Samples from the same locations and depths will be submitted to the analytical laboratory at a rate of 1 per 10 samples screened in the field. Samples submitted for laboratory analysis will be selected randomly. Field QA/QC samples will also be submitted for analysis, as indicated on Worksheet #20.
- All samples (XRF and laboratory) will be analyzed for arsenic, cadmium, copper, lead, mercury, and zinc.

QC Tasks:

A complete list of QC samples per matrix and analysis is provided in Worksheet #20.

- Implement field SOPs for sample collection, packaging, and transportation to the laboratory (see Appendix A, Worksheet #21 and Worksheets #26 and #27 for more details).
- The analytical laboratory will implement laboratory SOPs for sample preparation and analysis.
- Quality assurance reviews will be completed after each phase of fieldwork and on all documents.

WORKSHEETS #14 AND #16 (CONTINUED)
PROJECT TASKS AND SCHEDULE

Data Management Tasks:

- HGL will validate laboratory analytical results and results will be provided as electronic data deliverables (EDDs) in electronic laboratory reports.
- All laboratory data will be archived in the project file.

Documentation and Records: All field observations and sampling records will be entered into bound logbooks or on bound sampling data sheets. Chain of custody (CoC) forms, air bills, and field instrument calibration logs will be prepared and retained. Field forms are included in the SOPs in Appendix B or in Appendix C.

Assessment/Audit Tasks:

- Assessment/audit tasks will be completed for this project periodically.
- CAs will be performed by the Field Team Leader (FTL) for sampling tasks, and any reporting CAs will be resolved by the PM or PM designee. All CAs will be documented according to the Site Management Plan.

Data Review Tasks:

- Validated data and all related field notes, logbooks, and records will be reviewed to assess total measurement error and determine overall usability of the data for project purposes. Data limitations will be determined, and data will be compared to project DQOs and RAOs. CA will be initiated if necessary. Final data will be placed in the project database, along with any necessary qualifiers, and tables, charts, and figures generated.
- Field measurement results will be reviewed by the FTL to verify that results were obtained using properly conducted procedures.

WORKSHEET #15
REMEDIAL ACTION OBJECTIVES AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

The project-specific analytical method quantitation limits are presented in the table below. This table includes the project analyte lists for each method, the sensitivity limits achievable by the project laboratory, and the associated screening levels. The laboratory SOPs for the preparation and analytical methods associated with the limits presented in the Worksheet #15.1 table are listed in Worksheet #23 and are presented in Appendix B.

WORKSHEET #15.1
REFERENCE LIMITS AND EVALUATION TABLE – METALS IN SOIL/SEDIMENT

Analyte	Screening Level (mg/kg)	Energy Laboratories, Billings - Limits	
		MDL (mg/kg)	RL (mg/kg)
Arsenic[1]	200	0.1	0.2
Cadmium[1]	20	0.02	0.05
Copper[1]	1,000	0.1	0.5
Lead[1]	1,000	0.02	0.05
Zinc[1]	1,000	0.4	1
Mercury[2]	10	0.006	0.1

(1)EPA 6020B

(2)EPA 7141B

If three of the five criteria are exceeded, or if any one contaminant concentration exceeds 5,000 mg/kg, the material is considered tailings, waste, or contaminated soil.

WORKSHEET #17
SAMPLING DESIGN AND RATIONALE

The sampling process was designed to ensure that the sampling objectives are fulfilled for the RD. As presented in Worksheet #11, the objectives of the field investigations are as follows:

1) Characterize contaminant concentrations in soils and sediments in the specified work areas,
2) Better understand the thickness of mine waste and contaminated soil in the study area, and
3) Better delineate the areal extent of mine waste and contaminated soil in the study area.

To accomplish these objectives, HGL will implement field activities as follows:

- Perform the field inspection of the study area to gather current site conditions.
- Use the following methods or a combination of these methods to determine depths to underlying native soil: DPT rig, auger drill rig, hand auger.
- Using any or all of these same methods in addition to shallow surface material collection (hand tools) to collect soil and suspected waste and contaminated soil samples for logging, XRF screening, and/or laboratory analysis.

All field sampling activities will be conducted under the HASP and performed in accordance with HGL's SOPs and applicable laboratory SOPs, which are included in Appendices A and B, respectively.

WORKSHEET #18
SAMPLING LOCATIONS AND METHODS

Matrix	Sampling Location/ ID Number*	Depth (ft bgs)	Analytical Methods[1]	Number of Field Samples[2]	Sampling SOP References[3]	Anticipated Concentrations	Rationale for Sampling Location
Soil/Sediment	See Figure 18.1, and worksheets #26 and #27	0 to up to 20 ft below surface	Metals	Up to 228 for field screening, 10% for lab analysis	S-1 through S-12	Low to Medium	Characterize the surface and subsurface soil and sediment contamination and provide data for the estimation of mine waste and contaminated soil.

[1]See Worksheet #23
[2]Number of samples includes background samples, but does not include QC samples, which are listed in Worksheet #20.
[3]See Worksheet #21
*Sample locations will be based on accessibility and ability to perform sample collection at the proposed locations, which can vary seasonally, as illustrated on Figure 18.1. Sample IDs will be assigned as described in Worksheets #26 and #27.
bgs = below ground surface
ft = feet
ID = identification

WORKSHEET #18 (CONTINUED)
SAMPLING LOCATIONS AND METHODS

	Soil/Sediment Sampling				
Location	**Sampling Frequency/Approach**	**Proposed No. of Samples**	**No. of Field Duplicate Samples**	**No. of Samples for Lab Analysis**	**No. of Duplicate Samples for Lab Analysis**
See Figure 18.1	Samples will be collected using DPT or Vibracore borings; DPT cores will be collected continuously from ground surface to contact with native soil surface, as determined by cores and resistance. Average boring depth is estimated at 20 ft bgs. Soil cores will be collected at a rate of approximately 1 per 5 ft depth (4 samples per boring, on average) and screened using an XRF device. Select samples will be submitted for laboratory analysis at a rate of approximately 10 percent relative to the total number of soil samples screened using XRF.	Up to 276 total, from up to 69 locations	10%	23	3

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

Matrix	Parameter	Analytical and Preparation Method/ SOP Reference	Containers	Preservation Requirements	Maximum Holding Time
Soil/Sediment	Metals	EPA 6020B	4-ounce glass jar	Cool to ≤4°C	180 days
Soil/Sediment	Mercury	EPA 7471B		Cool to ≤4°C	28 days

Sample locations and ID numbers are located in Worksheet #18, along with expected concentration levels.
°C = degrees Celsius

WORKSHEET #20
FIELD QC SUMMARY

Soil samples will be screened in the field using an XRF device. Field duplicates for XRF analysis will be collected at an overall rate of 1 per 10 field samples (Worksheet #18). Samples submitted to the laboratory will be at the rate of 1 per 10 samples screened using XRF. For samples submitted for laboratory analysis, field duplicate pairs will be collected at a rate of approximately 1 per 10 field samples. MS/MSD pairs will also be collected at a rate of 1 per 20 field samples. EBs will be collected at a rate of 1 per 5 sampling days; however, if samples are collected from dedicated sampling equipment or equipment that will not be reused, EBs will not be required.

The following table summarizes the proposed number and types of samples to be collected.

Subsurface Soil and Sediment Sample Summary

Matrix	Analysis/ SOP Reference	Soil Samples	Field Duplicates	MSs	MSDs	Total # Samples Collected
Soil/Sediment	Metals/mercury by XRF (field analysis)	228	23	--	--	251
Soil/Sediment	Metals/mercury by EPA 6020B/7471B (laboratory analysis)	23	3	2	2	30

The identification of field QC samples will follow the sample nomenclature presented in Worksheets #26 and #27.

WORKSHEET #21
FIELD STANDARD OPERATING PROCEDURES

All necessary SOPs are provided in Appendices A and B and will be available for use by the field sampling team.

Reference Number	Title, Revision Date, and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
S-1	SOP 300.07 Environmental Data Base Quality Control	HGL	Excel, GIS	No	General Data Management Procedures
S-2	SOP 401.501 Field Logbook Use and Maintenance	HGL	Field logbooks, permanent markers	No	Record all fieldwork in logbook
S-3	SOP 403.01 Soil Sample Collection	HGL	Disposal gloves, scoops, sample jars	No	Use if Hydrocarbons suspected
S-4	SOP 403.02 Hand-Operated Auger Sampling	HGL	Hand auger	No	Surface soil and bank sampling
S-5	SOP 403.03 Soil or Sediment Sample Compositing	HGL	Mixing bowls and utensils	No	For collection of duplicate samples
S-6	SOP 403.04 Direct Push Technology Soil Sampling	HGL	DPT or Sonic rig	No	Subsurface soil sampling and logging,
S-7	SOP 403.06 Surface and Shallow Depth Soil Sampling	HGL	Trowel/hand auger	No	Surface soil and bank sampling
S-8	SOP 403.07 Borehole Logging	HGL	DPT rig	No	Subsurface soil logging
S-9	SOP 403.08 Sediment Sampling	HGL	Sediment sampler	No	In conjunction with surface and subsurface soil sampling, as needed
S-10	SOP 411.02: Sampling Equipment Cleaning and Decontamination	HGL	All non-disposal sampling equipment	No	Decontamination procedure
S-11	SOP 411.03 Subsurface Utility Avoidance	HGL	Location Marker (paint, flag, stake)	No	Prior to any subsurface auguring
S-12	SOP 408.511 XRF Screening Procedures	HGL	XRF Unit	Yes	Addresses Modified EPA 6200
S-13	HGL SOP 408.511.F01 XRF Usage Log	HGL	XRF Unit	No	Scan cores, Screening Samples
S-14	HGL SOP 408.511.F02 XRF Calibration Form	HGL	XRF Unit	No	Scan cores, Screening Samples

Reference Number	Title, Revision Date, and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
S-15	HGL SOP 408.511.F03 XRF Daily Log	HGL	XRF unit	No	Scan cores, Screening Samples
S-16	HGL SOP 412.501 Data Validation	HGL	Forms, Database	No	General Data Validation Procedures
S-17	ELI SOP, Field Sampling	Energy Laboratories	Forms	No	Sample chain of custody procedures
S-18	ELI SOP, Sample Receipt, Login, and Labeling.	Energy Laboratories	Forms	No	Sample tracking procedures

[1]XRF analyses will be performed in accordance with the instrument manufacturer's operator's manual, including the instrument checks described in Worksheet #22. The operator's manual will be provided to the field team once the specific device to be used has been determined. The manual will be included with field records.

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

The XRF instrument will be operated and maintained in accordance with the manufacturer's instructions. The operator's manual will be provided to the field team once the specific device to be used has been determined and is included in the SOPs presented in Appendix B. The manual will be included with field records.

Field Equipment	Activity	SOP Reference	Responsible Person	Frequency	Acceptance Criteria	Corrective Action[1]
XRF unit	Automated calibration check	S-10	Instrument operator	Instrument power-on	Per manufacturer's specifications	Remove instrument from use until serviced by a certified technician
	Energy calibration check	S-10	Instrument operator	Instrument power-on and after routine maintenance	Per manufacturer's specifications	Remove instrument from use until serviced by a certified technician
	Instrument blank check	S-10	Instrument operator	Initially and every 25 samples	No result > 2x MDL	Clean the instrument window and re-measure; if results persist, remove from use until instrument can be re-zeroed
	Calibration verification check	S-10	Instrument operator	Daily, before use, during operation, and after use	%D < 20% for target metals	Remove instrument from use until successful recalibration
	Precision measurement check	S-10	Instrument operator	Daily before use	%RSD < 20% for target metals	Remove instrument from use until serviced by a certified technician

[1]If CA does not solve the problem, the equipment will be removed from service and replaced until it has been repaired.
%RSD = percent relative standard deviation

WORKSHEET #23
ANALYTICAL STANDARD OPERATING PROCEDURES

Title, Revision Date, and/or Number	Definitive or Screening Data	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
EPA Method 6020B – Energy Laboratories ELI SOP 50-220-05	Definitive	ICP-MS	Energy Laboratories	No
EPA Method 7471B – Energy Laboratories ELI SOP 50-046-09	Definitive	CVAA Analyzer	Energy Laboratories	No

ICP-MS = inductively coupled plasma-mass spectrometry
CVAA = cold vapor atomic absorption
Y = Yes
N = No

WORKSHEET #24
ANALYTICAL INSTRUMENT CALIBRATION TABLE

Energy Laboratories will follow their internal SOPs to meet method requirements.

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

Energy Laboratories operates under a quality system that conforms to the requirements of the International Organization for Standardization 17025. The applicable equipment maintenance, testing, and inspection requirements are presented in the laboratory QA Manual (Appendix B) and in the method-specific SOPs.

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

Sample shipment procedures will include overnight shipment by commercial courier or hand delivery to Energy Laboratories. When samples are collected on a Friday, HGL will coordinate with the laboratory to ensure that the samples can be received in a timely manner.

Sample Collection, Packaging, and Shipment (Reference subsequent pages of this worksheet and field SOP[1])
Sample Collection (Personnel/Organization): Site Staff/HGL
Sample Packaging (Personnel/Organization): Site Staff/HGL
Coordination of Shipment (Personnel/Organization): FTL/HGL will coordinate sample shipment with the Energy Laboratories coordinator.
Type of Shipment/Carrier: Overnight courier or hand delivery.
Field Sample Storage (number of days from sample collection): Samples will be held in the field no longer than overnight unless prior arrangements have been made with the laboratory. Holding times must not be compromised by holding samples in the field.
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): Sample Management Staff/ Energy Laboratories
Sample Custody and Storage (Personnel/Organization): Sample Management Staff/ Energy Laboratories
Sample Preparation (Personnel/Organization): Organic Preparation Staff, Inorganic Preparation Staff, and Bench Chemists/ Energy Laboratories
Sample Determinative Analysis (Personnel/Organization): Bench Chemists/ Energy Laboratories
Sample Archiving (Reference Laboratory SOP)
Sample Extract/Digestate Storage (number of days from extraction/digestion): For 60 days from data report release or as required on a site-specific basis
Sample Disposal (Reference Laboratory SOP)
Personnel/Organization: Sample Management Staff/ Energy Laboratories
Number of Days from Analysis: 60 from data report release; unless otherwise requested

Sample Custody Requirements

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

HGL will maintain 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 their possession; (2) it is in their view after being in their possession; (3) it was in their possession and is locked up; or (4) it is in a designated secure area after being in their 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. All sample containers that leave the custody of the sampler (i.e., are shipped via common carrier) will be wrapped in bubble wrap or sealed in a plastic bag package. A custody seal will be placed on the package so that it will be broken if tampered with. Custody seals also will be placed in two locations on the shipping container (cooler or box) so that any tampering or intrusion into the contents will be evident. 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 Sample ID Procedures, below. 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,
- Sampler's initials, and
- Contract Laboratory Program (CLP) case number (if CLP is used).

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 Worksheets #19 and #30. The samples will immediately be placed on ice and will be kept chilled during the workday until packaged for shipment to the laboratory. When packaging samples for shipment, the cooler drainage plug will be closed and the cap will be sealed in place. The cooler will be lined with a heavy duty, contractor-type garbage bag. 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°C or less throughout shipment. Prior to sealing the cooler, the sampler's copy of the CoC forms will be detached and provided to the FTL 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 shipped to the laboratory as expeditiously as possible. Sample shipment will be performed in accordance with all applicable Department of Transportation regulations. The samples will be shipped to the laboratory according to the procedures identified in this worksheet. Arrangements will be made between HGL and the Energy Laboratories for samples that are to be delivered on a weekend so that sample condition and holding times are not compromised.

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal):
Laboratory custody procedures will be in accordance with Energy Laboratories SOPs.

Sample ID Procedures:
Each sample collected will be assigned a unique sample ID number and will be collected from a unique station location. Sample identifications will follow the format of **AA-LOC#-BBB-XX-YY-ZZ**, where:

- AA designates the sample type (for example SS= soil, or SD=sediment,
- LOC# is the sample location identification (such as "BR0148" for Boring 01, sample depth 48 inches),
- BBB specifies the type of analysis ("XRF" for field analysis or "LAB" for samples submitted to a laboratory), and
- XX-YY-ZZ indicates the month-day-year the sample was collected.

QC designations will be added at the end of the sample identification, as appropriate; FD stands for field duplicate and MS/MSD for matrix spike/matrix spike duplicate.

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. 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.

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;
- Custody transfer signatures and dates and times of sample transfer from the field to transporters and to the laboratory or laboratories; and
- Bill of lading or transporter tracking number, if applicable.

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. Pertinent field data, such as associated XRF screening data, will be recorded in the field logbook and on preprinted forms and retained in the project file.

WORKSHEET #28
ANALYTICAL QC AND CORRECTIVE ACTION

Energy Laboratories will be responsible for following their SOPs with regard to the general guidance for the evaluation of QC analyses and the implementation of CA for out-of-control situations.

WORKSHEET #29
PROJECT DOCUMENTS AND RECORDS

HGL will prepare and submit Site-specific documents in accordance with the Statement of Work (SOW), which can be provided upon request. These documents are to include this UFP-QAPP, the DMP and the FSP (both included in this UFP-QAPP), and a HASP. The HASP was previously submitted to MDEQ.

HGL will prepare Monthly Project Reports and will perform task order closeout procedures, as specified in the SOW. Closeout may include but is not limited to returning documents to MDEQ or other document repositories, file duplication, distribution and storage, file archiving, and preparation of a closeout report. Other documents and records to be managed under this task order are listed below.

Record	Generation	Verification
Sample Collection Documents and Records		
Access Agreements	EPA	EPA
Field notes (bound logbook)	Field staff	FTL
Sample documentation forms	Field staff	FTL
CoC records	Field staff	FTL
Airbills	Field staff	FTL
Custody seals	Field staff	FTL
CA forms	PM	QA/QC Manager
Photographs	Field staff	PM
Geographic information system (GIS) data (Per EPA SOP 2341.01A R7 Geospatial Data Deliverables)	Field staff	Database Manager
On-Site Analysis Documents and Records		
Equipment calibration logs	Field Staff	FTL
Field sampling data sheets	Field Staff	FTL
Waste disposal records	FTL	PM
Off-Site Analysis Documents and Records		
Sample receipt, custody, and tracking records	Sample Receipt Staff	Laboratory PM
Standard traceability logs	Analytical Staff	Laboratory Section Manager/QA Manager
Equipment calibration logs	Analytical Staff	Laboratory Section Manager/QA Manager
Sample preparation logs	Analytical Staff	Laboratory Section Manager/QA Manager
Analytical run logs	Analytical Staff	Laboratory Section Manager/QA Manager
Equipment maintenance, testing, and inspection logs	Analytical Staff	Laboratory Section Manager/QA Manager
Analytical discrepancy forms	Analytical Staff	Laboratory Section Manager/QA Manager

WORKSHEET #29 (CONTINUED)
PROJECT DOCUMENTS AND RECORDS

Record	Generation	Verification
Reported analytical results	Analytical Staff	Laboratory Section Manager/QA Manager
Reported results for standards, QC checks, and QC samples	Analytical Staff	Laboratory Section Manager/QA Manager
Data package completeness checklists	Analytical Staff/Section Manager	Laboratory PM/QA Manager
Sample disposal records	Assigned Laboratory Staff	Laboratory Operations Manager/QA Manager
Extraction and cleanup records	Analytical Staff	Laboratory Section Manager/QA Manager
Raw data (stored electronically)	Analytical Staff	Laboratory Database Manager/QA Manager
EDDs	Laboratory Database Manager	Database Manager
Telephone logs, emails, faxes, and correspondence	Laboratory PM	Laboratory Operations Manager
Data Assessment Documents and Records		
Data validation reports	Data Validator	Data Validation PM/Project Chemist
Automated data review reports	Data Validator	Data Validation PM/Project Chemist
Database QC spreadsheets	Project Staff	Database Manager
Data usability assessments	Project Chemist	PM
Deliverables		
Project planning documents, including UFP-QAPP and Site HASP	PM	QA/QC Manager
Project deliverables, including data evaluation reports and design reports	PM	QA/QC Manager
Site maps	Graphics Staff	PM
Design documents	Design Staff	PM
EDDs	Project Database Staff	Database Manager

WORKSHEETS #31, #32, AND #33
ASSESSMENTS AND CA

Any applicable assessments and CAs associated with the scope will be performed in accordance with the HGL Quality Manual (HGL, 2022).

Assessments:

Assessment Type	Responsible Personnel and Organization	Internal or External Assessment	Number and Frequency	Assessment Deliverable	Deliverable Due Date
Review of QAPP, SOPs, and HASP with Field Staff (a field audit will not be performed)	HGL FTL	Internal	Prior to sampling startup and with all new field staff prior to assignment	Completed acknowledgment signature pages	48 hours following review
Ongoing Review to Ensure Work is Being Performed in Accordance with QAPP	HGL FTL	Internal	Ongoing during all phases of fieldwork	None	Not applicable (NA)
Logbook and Field Form Review	HGL FTL	Internal	Daily	NA: corrections will be made directly to reviewed documents	NA
Tailgate Safety Meeting	HGL FTL	Internal	Daily	Verbal debriefing. If a safety incident occurs, a Supervisor Injury Employee Report is completed.	Any safety incidents will be reported to the PM and Corporate H&S Manager immediately
Field Sampling and CoC Form Review Against QAPP Requirements	HGL Data Manager	Internal	Daily	Corrections will be made directly to reviewed documents; communication may be in the form of email.	24 hours following assessment, if necessary

WORKSHEETS #31, #32, AND #33 (CONTINUED)
ASSESSMENTS AND CA

Assessment Response and CA:

Assessment Type	Individual(s) Notified of Findings	Assessment Response Documentation	Nature of the Deficiencies Documentation	Time Frame for Response	Responsibility for Implementing CA	Responsibility for Monitoring CA
Review of QAPP, SOPs, and HASP with Field Staff	HGL FTL	Completed acknowledgement signature pages	None	48 hours following assessment	HGL FTL	HGL FTL
Ongoing Review to Ensure That Work is Performed in Accordance with QAPPs	HGL PM	Interim CA documented pending final approval	Document in logbook	By close of same business day	HGL FTL	HGL PM and QA/QC Manager
Logbook and Field Form Review	HGL FTL	Corrections will be made directly to reviewed documents	Document in logbook	NA	HGL FTL	HGL FTL
H&S Audit	HGL Corporate H&S Officer	H&S audit report	CA Report	Within 2 weeks	HGL PM	HGL PM

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 QAPPs.

Item	Description	Data Generated Internally or Externally	Verification (completeness)	Validation (conformance to specifications)
Planning Documents/Records				
1	Approved QAPP	Internally	X	
2	Contract	Internally	X	
4	Field SOPs	Internally	X	
5	Laboratory SOPs	Internally	X	
Field Records				
6	Field logbooks	Internally	X	X
7	Equipment calibration records	Internally	X	X
8	CoC forms	Internally	X	X
9	Relevant correspondence	Internally	X	X
10	Change orders/deviations	Internally	X	X
11	Field audit reports	Internally	X	X
12	Field CA reports	Internally	X	X
Analytical Data Package				
13	Laboratory analytical data packages	Externally	X	X
14	Communication Records	Externally	X	X
15	EDD fields	Externally	X	X
16	Outputs of the electronic database	Externally	X	X
17	Data validation and audit reports, QAPP and Field Change Requests	Externally	X	X

WORKSHEET #35
DATA VERIFICATION PROCEDURES

Verification Input	Description	Responsible for Verification
CoC (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
Log review	Log reviews will be performed 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 will verify that all required information has been correctly documented in the field logbooks and sample documentation sheets.	HGL FTL
CoC (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 with the HGL PM.	Energy Laboratories Receipt Manager Laboratory PM
Analytical data package	All data used to prepare analytical data packages will be reviewed at multiple levels throughout the laboratory. The requirements for this review process are described in the laboratory's quality manual.	Energy Laboratories QA/QC Manager
Analytical data package	A review will be conducted to ensure that the appropriate analytical samples have been collected, appropriate site identifications have been used, and the correct analytical methods have been applied.	HGL Data Manager
Analytical data package[1]	Analytical reports will be reviewed to ensure that all required forms, case narratives, samples, CoC forms, logbooks, and raw data have been included.	HGL Data Validator
EDD (import)	Any EDD nonconformances from the laboratory will be reviewed and addressed before the data is processed further. The EDD also will be reviewed 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 Database Manager

[1]This verification step is performed as part of the data validation process described in Worksheet #36.

WORKSHEET #36
DATA VALIDATION PROCEDURES

Data for samples analyzed by Energy Laboratories will be validated by HGL and tabulated validated results will be provided to MDEQ. HGL will provide validated data in electronic format and in analytical reports with case narratives describing any qualifiers placed on the data.

Validation Stage	Matrix	Analytical SOP[1]	Validation Criteria	Data Validator
2B	All	Metals	HGL SOP 412.501 Data Validation, EPA/U.S. Department of Defense Stage 2A and Stage 2B	HGL personnel
2B	All	Metals	EPA National Functional Guidelines for Inorganic Methods Data[2] Review (ISM02.3) Office of Land and Emergency Management 9355.0-135 EPA SFAM01.1 2020	HGL personnel

[1]Refer to Worksheet #23.

[2]The EPA National Functional Guidelines (NFGs) include acceptance criteria specific to analyses performed in accordance with the EPA CLP Scope of Work. While the NFG validation protocols will be used to guide the data validation process and apply qualifiers, data quality performance will be evaluated against the requirements of this QAPP, the laboratory SOPs, and the method requirements, in descending order.

WORKSHEET #37
DATA USABILITY ASSESSMENT

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: Data will be received from the analytical laboratory and HGL will validate the data presented in each laboratory data report. HGL will assess the usability of the data by evaluation of DQIs as described in Worksheet #12 and evaluating if the project required quantitation limits listed in Worksheet #15 were achieved for nondetected Site contaminants of concern. In addition, data usability will be assessed as follows:

1) If no detectable results were reported and data are acceptable from the verification and validation steps, then the data are usable;
2) If detectable concentrations are reported and the verification and validation steps are acceptable, the data are usable; and
3) If verification and validation are not acceptable, the data are qualified during data validation. The data that are estimated (J), or undetected and estimated (UJ) for minor QC deviations generally do not affect the data usability. The data are rejected for major QC deviations affect data usability. The impact of rejected data will be assessed in the Data Evaluation Report, and re-sampling may be necessary.

Describe the evaluative procedures used to assess overall measurement error associated with the project:
The validation will follow the requirements of HGL's data validation SOPs to assess conformance with the requirements of the methods, SOPs, and objectives stated in this UFP-QAPP. The findings of the data validation will generate qualifiers applied to the data considered in context to assess overall usability of the data. A Data Evaluation Report will be prepared after the field sampling event by HGL that will include the results of the usability assessment review performed by the project data management team.

Identify the personnel responsible for performing the usability assessment:
HGL PM, project chemist, and database manager.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:
An overall assessment of the impact of data usability issues will be presented in the Data Evaluation Report.

REFERENCES

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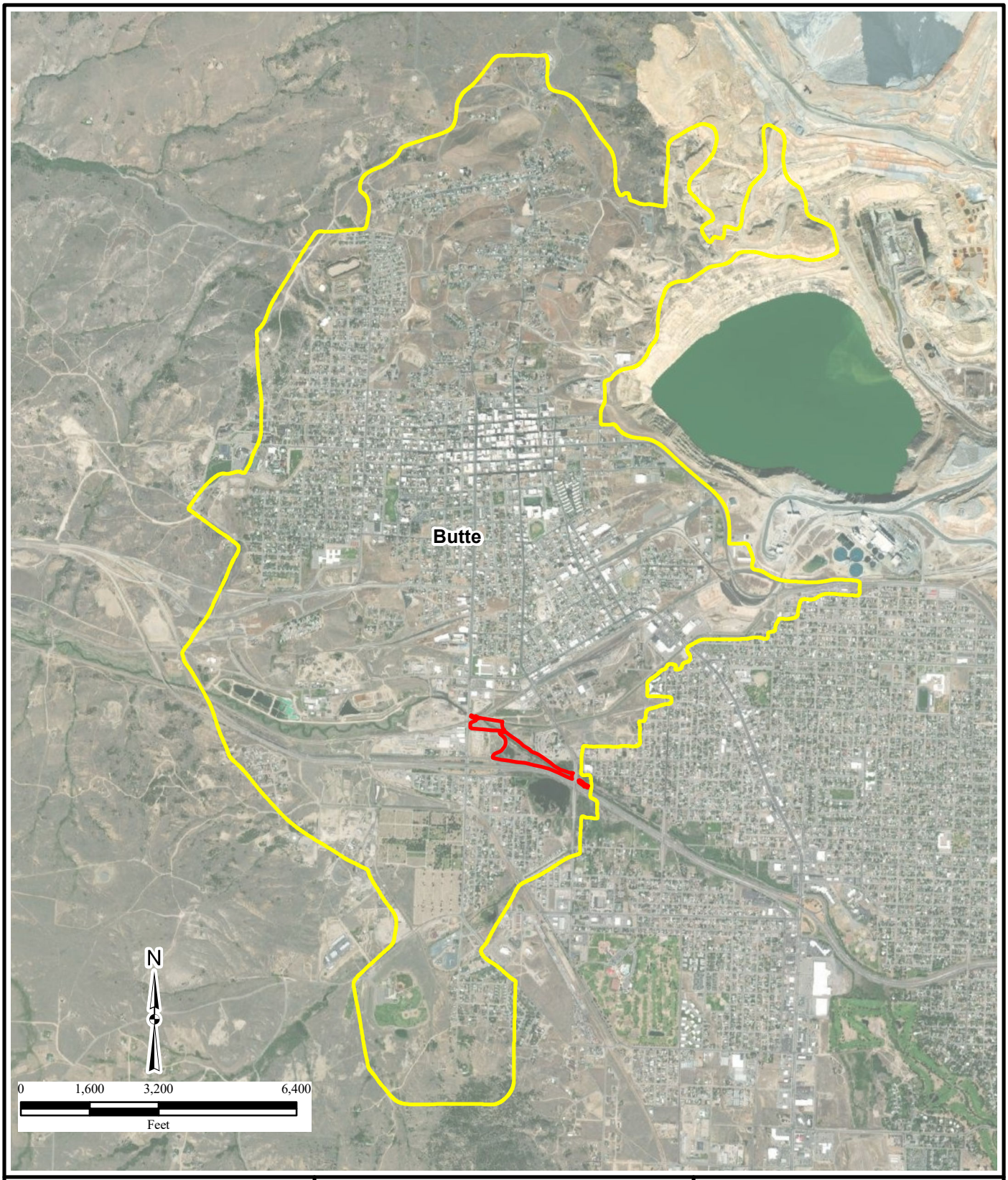
FIGURES



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Legend

BPSOU Boundary

Blacktail Creek Riparian Actions Study Area

Figure 10.1
BPSOU Boundaries

HGL HydroGeoLogic, Inc

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Source: HGL, DEQ, ArcGIS Online Imagery

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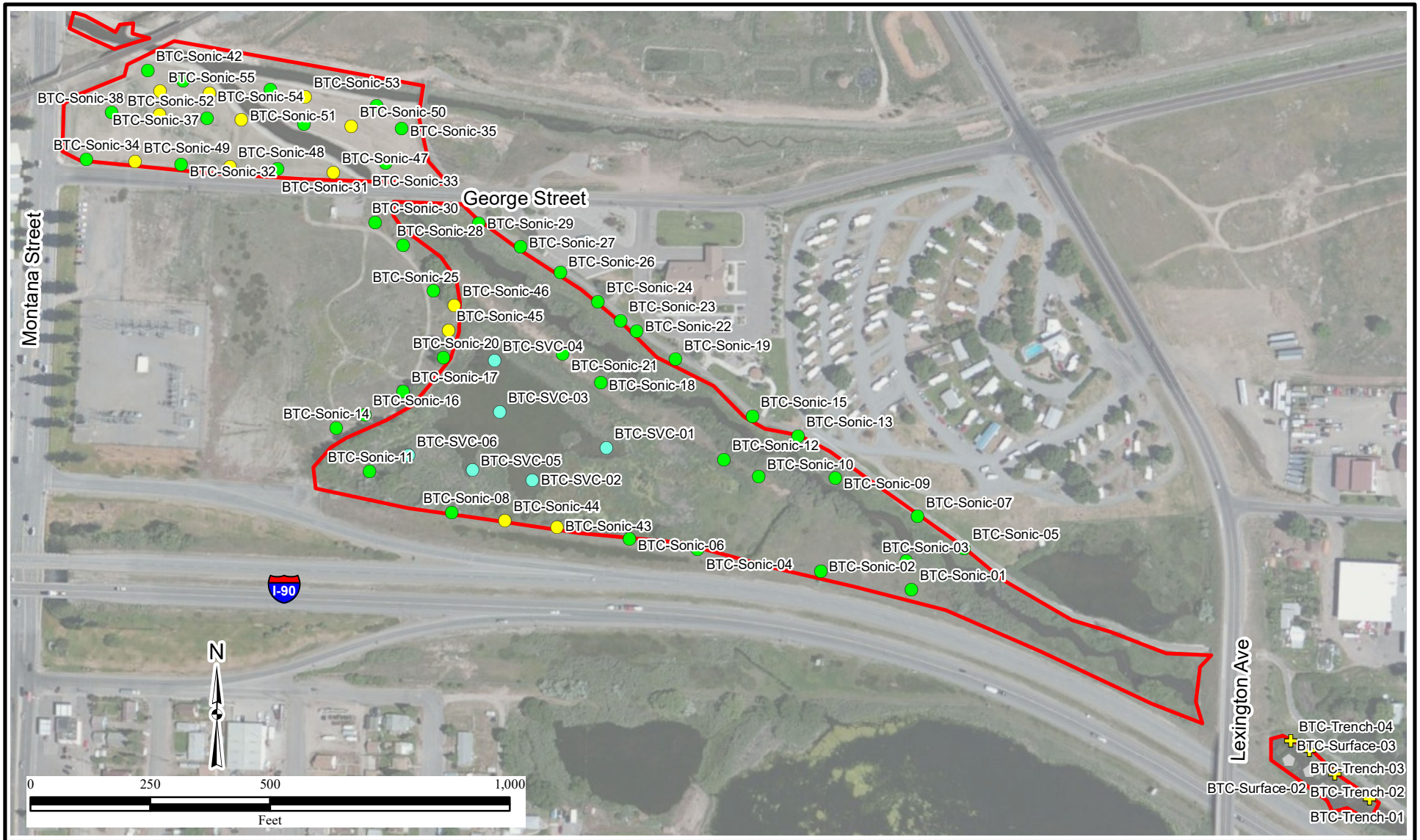
Blacktail Creek Riparian Actions Study Area

HGL HydroGeoLogic, Inc

Figure 10.2
Blacktail Creek Riparian Actions Study Area



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Source: HGL, DEQ, ArcGIS Online Imagery

HGL HydroGeoLogic, Inc

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Blacktail Creek Riparian Actions Study Area

- Sonic Priority 1
- Sonic Priority 2
- Sonic Vibracore
- Hand Dug Pit
- Hand Dug Trench

Figure 18.1 - Proposed Sampling Sites

Blacktail Creek QAPP

APPENDIX A

HGL STANDARD OPERATING PROCEDURES

- SOP 300.07 Environmental Data Base Quality Control
- SOP 401.501 Field Logbook Use and Maintenance
- SOP 403.02 Hand-Operated Auger Soil Sampling
- SOP 403.03 Soil or Sediment Sample Compositing
- SOP 403.04 Direct-Push Technology Soil and Groundwater Sampling
- SOP 403.06 Surface and Shallow Depth Soil Sampling
- SOP 403.07 Geologic Borehole Logging
- SOP 403.08 Sediment Sampling
- SOP 411.02 Sampling Equipment Cleaning and Decontamination
- SOP 411.03 Subsurface Utility Avoidance
- SOP 408.511 XRF Screening – Modified
- SOP 412.501 Data Validation

HGL HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE Approved by: 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

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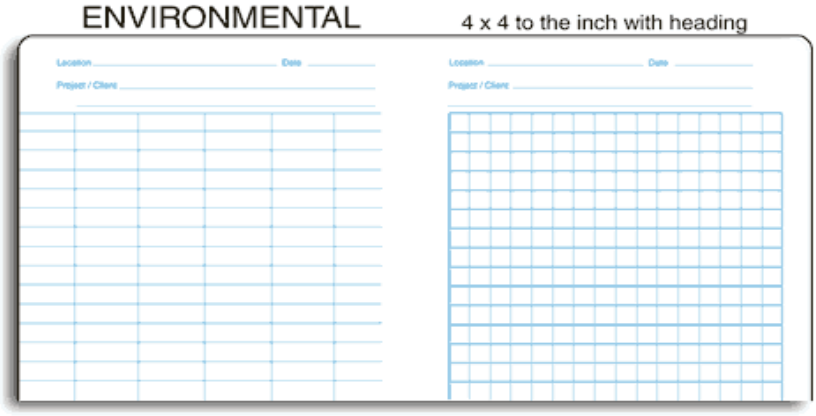
"Rite in the Rain"
ALL-WEATHER
ENVIRONMENTAL
No. 550F


ENVIRONMENTAL
4 x 4 to the inch with heading

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

Photo log:
Subject # persons - distance to subject - person taking photo - direction facing on. Time / photo # / roll #. Meteorology (wind).

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.

Anne Vogel AV 11/6/95
1

November 6, 1995, AX1015.13.00

pH Meter
Hach
Model # = 12345
Serial # = 6789

Conductivity Meter
Hach
Model # = 12345
Serial # = 6789

$c^2 = a^2 + b^2$
if $a = 3$
if $b = 4$
Then: $c^2 = 3^2 + 4^2$
$c^2 = 25$
$c = \sqrt{25}$
$c = 5$
$\pi = 3.14159$

Anne Vogel home # 123-4567
VOS Denver Office # 303/2716-9400
VOS San Francisco # 415/774-2700 (travel)

Smith Site
Badger County, Colorado
Address: 1034 W. Main Street
Manzano, Colorado 00000
Directions to Site:
West on I-70
Exit 93B.
Head South approx. 3 miles.
Site is on east side of dirt road.

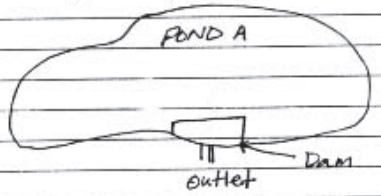
AV 11/6/95
2

November 6, 1995 Site Visit
0700 Arrive on site
Weather: 80°, sunny, slight breeze (~5 mph) from southwest.
UOS Field Team: EPA OSC:
M.R. Smith J.P. Scarton
K.W. Wagner
P.R. Lane
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 procedures:
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



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 10000 STD - Rinse probe

AV 11/6/95
4

Time	Sample	Sample#	Label #
0802	VOA	81088 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	6010

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	81088 VOA BD	106
0903	TSS	81088 TSS BD	107

0905 Decon scoop.
AV
Rinsate Samples

Time	Sample	Sample#	Label #
0920	VOA	81088 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 TSOP.
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
  - ID
  - depth
  - volume
  - containers
  - preservative
  - media
  - 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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1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the standard method and equipment used to collect soil samples at the surface or in shallow subsurface using a hand auger.

2.0 SCOPE AND APPLICATION

This procedure yields a disturbed sample and applies to a wide variety of soil types including sands, clays, and silts. A hand auger is typically a small, lightweight metal cylinder (bucket), open at both ends with a cutting bit on the bottom. Diameters typically range between 1 and 4 inches. A T-shaped handle is attached to the top of the bucket by extendable rods. The augers are rotated into the ground until the bucket is full, then lifted out of the borehole and emptied. The maximum depth of hand auger investigations is typically 10 feet below ground surface. The use of an auger is of limited value in rocky soil. This procedure is not appropriate for collecting samples at a discrete depth, but may be used to collect samples at an approximate depth.

3.0 GENERAL REQUIREMENTS

All work must be 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 must be sufficiently documented to re-create the modified process.

4.0 EQUIPMENT

The equipment required may include hand-operated, spiral-type, ship-type, open-tubular, orchard-barrel, open-spiral, closed-spiral, post-hole, clamshell, Edelman, or Iwan augers. Augers typically are used with 3- to 4-foot-long metal extension rods and T-handles (fixed or ratcheted). The use of stainless steel augers is preferred. Augers plated with chrome or coated with other materials, except Teflon®, cannot be used.

Sampling tools and equipment should be protected from contamination sources before sampling and decontaminated before and between sampling locations, as specified in SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.

5.0 PROCEDURES

1. Don clean gloves. Using a decontaminated stainless steel spoon or other approved utensil, remove surface vegetation and debris from the immediate area around the marked sampling point.
2. Do not allow sampling equipment to touch potentially contaminated surfaces.
3. Record the appropriate information and observations about the sample location in the field logbook.
4. Assemble the decontaminated auger, extension, and T-handle, if necessary, and advance the auger into the soil to the desired depth. Mark the length of the hand auger rods every 0.5 foot to determine auger head depth relative to the ground surface when advancing or tag the bottom of the borehole (if the borehole stays open) with a weighted tape measure or water level meter.
5. Withdraw the auger from the soil.
6. If a sample is not being collected, remove the soil from the auger bucket and repeat Steps 4 and 5. While removing the soil from the auger bucket, the subsurface lithology should be described as specified in SOP 403.07: *Geologic Borehole Logging*. If a sample is to be collected in the next depth interval, replace the auger bucket with a clean decontaminated bucket and repeat Steps 2 through 4. Change gloves at each sampling location, or each time a new sample is to be collected, to avoid cross-contamination.
7. Perform any field monitoring required in the project-specific planning documents.

If collecting samples for analyses other than volatile organic compound (VOC) analyses, refer to Steps 8 and 9.

8. Using a decontaminated stainless steel spoon, spatula, disposable scoop, remove soil from the auger bucket and place in a stainless steel or glass container. Food-grade disposable aluminum pans may also be used but cannot be reused. Clean nitrile gloves may be donned to remove soil from the auger bucket by hand. Discard the top 2 or 3 inches of soil in the auger as this soil may consist of borehole slough from above. Mix or composite soil as directed by the project-specific planning documents. Using a decontaminated spoon or other approved utensil, remove any large rocks or other organic material (worms, grass, leaves, roots, etc.). Clean nitrile gloves may also be donned to remove large rocks or other organic material by hand.
9. Using a decontaminated stainless steel spoon, spatula, or disposable scoop, as appropriate, place soil samples in appropriate containers. Clean nitrile gloves may be donned to place soil into appropriate containers. Place samples in containers defined according to analytical needs specified in the project-specific planning documents, label samples, and then (when appropriate) pack on ice as soon as possible.

If collecting samples for VOC analysis, refer to Steps 10 and 11.

10. Remove the hand auger from the boring when the top of the specified sampling depth has been reached. Fit a slide-hammer to the top of the appropriate number of extension rods required to reach the total depth of the hole. Attach an impact sampler to the bottom of the extension rod(s) and drive the impact sampler into the soil to a depth of at least 6 inches. Remove the sampler from the borehole.
11. Collect VOC samples in accordance with SOP 403.01.0: *VOC Soil Sample Collection*. When samples are being collected for multiple analyses, samples that can be degraded by aeration (e.g., VOCs) are collected first and with the least disturbance possible to minimize analyte loss. VOC samples must not be composited.

6.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
0	December 2010	Initial Release
1	April 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
2	August 1, 2019	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
2	June 23, 2021	Updated to incorporate client editorial comments.

HGL HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE Approved by: Jeff Dick Digitally signed by Jeff Dick DN: cn=Jeff Dick, o, ou, email=jdick@hgl.com, c=US Date: 2019.08.02 07:30:04 -04'00' Corporate Quality Manager	
Soil or Sediment Sample Compositing		**SOP No.: 403.03 (formerly 2.04)**
		SOP Category: Environmental Services
		Revision No.: 4
		Revision Date: August 1, 2019
		Review Date: August 2021

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to outline methods that may be used for field compositing soil or sediment samples before they are submitted to an analytical laboratory.

2.0 SCOPE

This procedure applies to compositing soil or sediment. This procedure does not apply to sample collection, but rather to combining samples in preparation for submittal for testing. Samples for volatile organic compound analyses must NOT be composited.

3.0 GENERAL REQUIREMENTS

All work must be performed in accordance with the site- or 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 must be sufficiently documented to re-create the modified process.

4.0 PROCEDURES

Soil or sediment that is to be sampled must be mixed as thoroughly as possible before being transferred to the sample container. Anomalous or suspected highly contaminated samples must be brought to the attention of the field manager.

- Soil or sediment that is composited must meet the following requirements:
  - Uniform collection techniques must be used to retrieve sample aliquots.
  - Aliquots must be of equal or known proportion.
  - The soil or sediment must be well mixed.
- The most common method of mixing (compositing) is referred to as quartering. The soil or sediment is placed in a pan or tray and divided into quarters. Each quarter is mixed individually, and then all quarters are mixed together to form a homogenous matrix. This procedure is repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the soil or sediment in a circular fashion and occasionally turning the soil or sediment over. Mixing bowls and

stirring devices must be stainless steel and be decontaminated prior to use. Samples are homogenized before being placed into containers, except for volatile organic analyses.

- Sampling tools, instruments, and equipment must be protected from contamination sources before use and decontaminated after use as specified in SOP 2.01: *Sampling Equipment Cleaning and Decontamination.*
- Composite samples must be packaged, labeled, and prepared for shipment in accordance with the project-specific planning documents.
- The field logbook must be completed in accordance with procedures detailed in SOP 4.07: *Field Logbook Use and Maintenance*.

5.0 RECORDS

Documentation generated as a result of this procedure must be collected and maintained in accordance with requirements specified in the project-specific planning documents.

- Complete the field logbook in accordance with procedures listed in SOP 4.07: *Field Logbook Use and Maintenance.*

6.0 REVISION HISTORY

Revision 0		Initial Release
Revision 1		Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	April 2009	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 3	April 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 4	August 1, 2019	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.

HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE
	Approved by: Dick, Jeff Digitally signed by Dick, Jeff Date: 2020.06.18 16:04:57 -04'00' Corporate QA Manager
Direct-Push Technology Soil and Groundwater Sampling	**SOP No.: 403.04 (formerly 2.05)**
	SOP Category: Environmental Services
	Revision No.: 3
	Revision Date: June 18, 2020
	Review Date: June 2022

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the standard method and equipment used to collect soil and groundwater samples using direct-push technology (DPT).

2.0 SCOPE AND APPLICATION

The DPT soil sampling method applies to a wide variety of soil types including sands, clays, and silts. Samples may be collected from discrete intervals where high sample recovery rates can be achieved such as in clays and silts. However, where sample recovery rates are low, such as may be the case in loose sand, the sample collection depth intervals may be approximate. DPT soil sampling methods are of limited value in rocky soil. Where rocky soils limit the use of DPT, a different technology, such as hollow-stem auger drilling equipment, must be used. This procedure is appropriate for collecting groundwater samples at discrete depths.

3.0 GENERAL REQUIREMENTS

All work must be performed in accordance with the project-specific planning documents. Refer to the project-specific health and safety plan and project-specific quality assurance project plan for relevant health and safety and quality control requirements, respectively.

Any deviations from specified requirements must be justified to and authorized by the project manager and/or the relevant program manager. Deviations from requirements must be sufficiently documented to re-create the modified process.

4.0 PRECAUTIONS

The following precautions should be employed during DPT sampling operations:

- Subsurface and aboveground utility lines must be identified and cleared before exploratory boring drilling activities can be performed. Procedures outlined in HGL SOP 411.03: *Subsurface Utility Avoidance*, must be followed.
- Every attempt should be made to minimize the transfer of potentially contaminated material to downhole equipment, or to any equipment and supplies stored on the site.
- Every attempt should be made to contain contaminated soil and water and to prevent further contamination of the environment.

- Sampling tools and equipment must be protected from sources of contamination before sampling and decontaminated before and between sampling, as specified in SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.

5.0 DPT SAMPLING PROCEDURES

DPT soil sampling is accomplished using a Geoprobe® or other similar truck- or track-mounted hydraulic sampler. DPT involves advancing a sampling probe using direct hydraulic pressure or a hydraulically driven rotary hammer. Boreholes are typically advanced using a 2.5- to 3-inch-diameter lead sampler attached to 1- or 2-inch-diameter probe rods, which are placed under hydraulic downward pressure. In unstable soils, a dual-tube system may be used where the lead sampler and center rods are used within larger diameter probe rods to prevent caving of material into the sample interval. Sampler sizes can vary from 1.25 to 4.5 inches in outer diameter (OD); however, 2.25- to 3.25-inch OD samplers are typical. Liner sizes can vary from 1.0 to 3.0 inches in internal diameter (ID); however, 1.125- to 1.85-inch ID are liners are typical. Borings remain open only as long as necessary to collect the soil and/or groundwater samples and log the lithology, if required by the project-specific planning documents.

Specific sampling tools could require slightly different handling methods. For example, if sampling devices and probe rod extensions do not have quick-connect fittings, adjustable or pipe wrenches could be needed to change equipment configurations. The procedures described in this SOP are for power-driven DPT methods or tube samplers, and they are consistent with ASTM International Standard Guides D6282/D6282M-14 and D6001-05(2012).

5.1.1 Soil Sampling Procedures

The soil samples obtained using DPT are collected in acetate, brass, or stainless steel sampling tubes. Acetate tubes are most commonly used. Sampling is initiated at the soil interface, unless otherwise specified in the project-specific planning documents.

- Place plastic sheeting on the ground around the sampling location to prevent cross contamination.
- Attach the direct-push sampler with liner and cutting shoe to a rod extension.
- Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). Remove the first 8 to 15 centimeters (cm) of surface soil from an approximately 15-cm radius around the drilling location to prevent near-surface soil particles from falling down the hole.
- Begin advancing the direct-push sampler, periodically removing accumulated soils. This step prevents accidentally brushing loose material back down the borehole when removing the sampler or adding probe rods.

- After reaching the desired depth, slowly and carefully remove the direct-push tool from the boring. If collecting a core sample, remove the cutting shoe and liner from the sampler and replace it with a precleaned thin-walled tube sampler. Insert a disposable acetate liner into the sampler with optional core catcher, and install the sampler and cutting shoe.
- Carefully lower the sampler down the borehole and gradually force the sampler into the soil. Care should be taken to avoid scraping the borehole sides when not using a dual-tube system. Hammering the probe rods to facilitate coring should be avoided, as the vibrations could cause the borehole walls to collapse.
- Once the sampler reaches the top of the sampling interval, drive the sampler down into the soil the length of the corer.
- Pull the probe rods and sampler out of the hole.
- Remove the sampler by twisting to prevent losing the core and unscrew the probe rods.
- Remove the cutting shoe and remove the acetate liner containing the core from the device.
- Carefully cut the acetate liner to expose the core.
- Screen the core with a field detector as described in the project-specific planning documents. If required by the project plans, collect volatile organic compound (VOC) samples immediately after opening the acetate liner. VOC samples must be collected in accordance with SOP 403.01: *VOC Soil Sample Collection*.
- Discard the top of the core (approximately 2.5 cm), as it will contain any material collected by the corer before penetration of the layer being sampled.
- Provide a lithologic description in accordance with SOP 403.07: Geologic Borehole Logging.
- If homogenization of the soil sample is appropriate for the remaining analytical parameters, or if compositing of different locations is desired, follow the procedures detailed in SOP 403.03: *Soil or Sediment Sample Compositing*. Otherwise, transfer the sample into an appropriate container with a stainless steel spoon or equivalent and secure the cap tightly.
- Label the sample bottle(s) with the appropriate sample label as described in the project-specific planning documents. Complete the label carefully and clearly, addressing all the categories or parameters.
- Place filled sample containers on ice immediately.
- Complete all chain of custody documents and record information in the field logbook in accordance with procedures listed in SOP 300.04: *Field Logbook Use and Maintenance* and on the Field Sampling Report (Attachment 1).
- Prepare the samples for shipment in accordance with the project-specific planning documents.

- Decontaminate sampling equipment after use and between sampling locations in accordance with procedures detailed in SOP 411.02: *Sampling Equipment Cleaning and Decontamination.*
- If no more cores are needed from the borehole, abandon the borehole with bentonite grout or chips and return the surface to its initial condition (e.g., topsoil, asphalt, or pavement).
- Soil generated during DPT activities that was not used for sampling should be treated as investigation-derived waste (IDW) and managed in accordance with the project-specific planning documents.

5.1.2 Groundwater Sampling Procedures

DPT groundwater samples can be collected using a hydropunch sampler. This type of groundwater sampling is best used for characterizing a site to determine the best placement of permanent wells. Procedures for collecting a water sample with a hydropunch are discussed in detail in this section. Note that the hydraulic conductivity of a formation could affect the time required to collect a sample. That is, more time could be required if groundwater recharge is slow. In those instances, the probe rods and hydropunch sampler can remain in the ground while the rig moves to another location to allow the water to recharge. After sufficient recharge, bailing or pumping can begin again.

- Place plastic sheeting on the ground around the sampling location to prevent cross contamination.
- Attach the sealed-screen sampler (hydropunch) to the probe rods.
- Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). Remove the first 8 to 15 cm of surface soil from an approximately 15-cm radius around the drilling location to prevent near-surface soil particles from falling down the hole.
- Begin advancing the hydropunch. The screen is driven to a depth such that the middle of the screen is set at the sample target depth.
- After reaching the desired depth, retract the protective outer rod of the sampler to expose the screen to groundwater. If necessary, an instrument can be lowered down through the center of the probe rods to check the water level and ensure that the sampler has sufficient water for sampling.
- Lower tubing with check valve, bailer, or peristaltic pump down through the probe rods to the screen of the hydropunch to collect the groundwater sample. Groundwater samples are collected most commonly using polyethylene or Teflon® tubing with a check valve attached to the bottom. An up/down oscillating motion on the tubing pumps the water column up in the tubing to the ground surface or until enough water volume is in the tubing for the samples. Groundwater samples are collected directly from the bottom of the tubing, after removing the check valve, and placed in sample containers according to the project-specific planning documents.

- Unless otherwise specified in the project-specific planning documents, collect the groundwater samples without purging sediment or groundwater to minimize disturbance to the sample.
- If sediment is expected in the sample, consider using sample containers without a hydrochloric acid preservative. Mixing the sediment often found in direct push groundwater samples with the hydrochloric acid causes a reaction that generates a gaseous product that creates unwanted headspace in the groundwater sample.
- If a bailer is used, retrieve the sample from the bailer and place it in an appropriate sample container.
- If a peristaltic pump is used, fill the appropriate sample container from the pump effluent tubing.
- If required, place a portion of the sample in a container to collect field parameters (temperature, pH, conductivity, dissolved oxygen, oxygen reduction potential, and turbidity).
- Label the sample bottles with the appropriate sample labels as described in the project-specific planning documents. Complete the label carefully and clearly, addressing all the categories or parameters.
- Place filled sample containers on ice immediately.
- Complete all chain of custody documents and record information in the field logbook in accordance with procedures listed in SOP 300.04: *Field Logbook Use and Maintenance* and on the Field Sampling Report (Attachment 1).
- Prepare samples for shipment in accordance with the project-specific planning documents.
- Pull the rods and hydropunch sampler from the hole.
- Decontaminate sampling equipment after each use and between sampling locations in accordance with procedures detailed in SOP 411.02: *Sampling Equipment Cleaning and Decontamination.*
- If additional samples are not needed from the borehole, abandon the borehole with bentonite chips and return the surface to its initial condition (e.g., topsoil, asphalt, or pavement).
- Manage IDW generated during hydropunch sampling in accordance with the project-specific planning documents.

6.0 RECORDS

Documentation generated as a result of this SOP must be collected and maintained in accordance with requirements specified in the project-specific panning documents.

- Document all daily field activities in the field logbook in accordance with procedures listed in SOP 300.04: *Field Logbook Use and Maintenance.*
- Complete a Field Sampling Report (Attachment 1) for each soil and groundwater sample.

7.0 REFERENCES

ASTM International (ASTM). D6282/D6282M-14: Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations.

ASTM. D6001-05(2012): Standard Guide for Direct-Push Groundwater Sampling for Environmental Site Characterization.

8.0 REVISION HISTORY

Revision 0	April 2009	Initial Release
Revision 1	April 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	February 2018	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 3	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 2.05 to 403.04.

ATTACHMENTS

Attachment 1 – Field Sampling Report

ATTACHMENT 1
FIELD SAMPLING REPORT

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FIELD SAMPLING REPORT

LOCATION: PROJECT NAME:

SITE: PROJECT NO:

SAMPLE INFORMATION

SAMPLE ID: DATE:_____ TIME:_____

MATRIX TYPE:

SAMPLE COLLECTION METHOD:

LOW-FLOW BAILER PASSIVE OTHER_____

LOT CONTROL #: ___ ___ ___ ___

(Ambient Blank # - Equipment Blank # - Trip Blank # - Cooler #)

CHAIN-OF-CUSTODY #: _____

SAMPLE BEG. DEPTH (FT):

SAMPLE END DEPTH (FT):

GRAB ( ) COMPOSITE ( )

ENTER SAMPLE NUMBERS FOR QC SAMPLES/ BLANKS ASSOCIATED WITH THIS SAMPLE:

MATRIX SPIKE (MS): _____

MATRIX SPIKE DUP (SD): _____

FIELD DUP (FD): _____

AMBIENT BLANK (AB): _____

EQUIPMENT BLANK (EB): _____

TRIP BLANK (TB): _____

CONTAINER		PRESERVATIVE/	ANALYTICAL	ANALYSIS
SIZE/TYPE	#	PREPARATION	METHOD	

NOTABLE OBSERVATIONS

PID READINGS	SAMPLE CHARACTERISTICS	MISCELLANEOUS
1st (TOC):	COLOR:	
2nd (BZ):	ODOR:	
	OTHER:	

pH_____ Temperature_____(C) Dissolved Oxygen_____(mg/L) Specific Conductivity_____(mS/cm)

Ferrous Iron_____(mg/L) Oxidation/Reduction Potential_____(mv) Turbidity_____(NTU

GENERAL INFORMATION

WEATHER: SUN/CLEAR_____OVERCAST/RAIN_____WIND DIRECTION_____AMBIENT TEMPERATURE _____

SHIPMENT VIA: FEDEX_____HAND DELIVER_____COURIER_____OTHER _____

SHIPPED TO:_____

COMMENTS: _____

SAMPLER:_____OBSERVER: _____

MATRIX TYPE CODES

DC=DRILL CUTTINGS
SL=SLUDGE
WG=GROUND WATER
SO=SOIL
LH=HAZARDOUS LIQUID WASTE
GS=SOIL GAS
SH=HAZRDOUS SOLID WASTE
WS=SURFACE WATER
SE=SEDIMENT
SW=SWAB/WIPE
W=WATER

SAMPLE COLLECTION METHOD CODES

B=BAILER
HA=HAND AUGER
BP=GAS OPERATED BLADDER PUMP
HY=HYDRASLEEVE
CS=COMPOSITE SAMPLE
NS=NON-SUBMERSIBLE PUMP
EC/TC=ENCORE/TERRA CORE SAMPLER
PP=PERISTALTIC PUMP
GB=GEOPROBE
SP=SUBMERSIBLE PUMP
H=HOLLOW STEM AUGER
SS=SPLIT SPOON
OTHER
G = GRAB
TR=TROWEL

HGL HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE Approved by: Dick, Jeff Digitally signed by Dick, Jeff Date: 2020.06.25 08:10:05 -04'00' Corporate Quality Manager
Surface and Shallow Depth Soil Sampling	**SOP No.: 403.06 (formerly 2.13)** **SOP Category: Environmental Services** **Revision No.: 3** **Revision Date: June 24, 2020** **Review Date: June 2022**

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the equipment and operations used for sampling surface and shallow depth soils. This procedure outlines the methods for soil sampling with routine field operations on environmental projects.

2.0 SCOPE AND APPLICATIONS

The objective of surface and shallow depth soil sampling is to ascertain the nature and extent of soil contamination at a site. The data can be used to identify contaminant sources, evaluate potential threats to human health or the environment, evaluate potential exposure pathways, or calculate environmental risks. For the purposes of this SOP, soil is defined as all unconsolidated materials above bedrock; surface soils are those that occur 0 to 6 inches below ground surface; and shallow depth soils are soils located above the bedrock surface and from 6 inches to 2 feet below ground surface.

3.0 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 and discussed in the approved project plans. Deviations from requirements must be documented sufficiently to re-create the modified process.

4.0 PROCEDURES

4.1 SAMPLING EQUIPMENT

Typically, equipment required for surface and shallow depth soil should be specified in the project field sampling plan or work plan. Equipment includes the following:

- Stainless steel mixing bowl,
- Stainless steel trowels or spoons,
- Stainless steel hand auger,
- Stainless steel core sampler that uses stainless steel or Lexan® liners (optional),
- Stainless steel shovel, and
- Appropriate sample containers.

Disposable sampling equipment items, such as a sampling spoon, may be used instead of stainless steel equipment. An example of a hand auger is provided in Attachment 1.

4.2 DECONTAMINATION

Before initial use, and after each subsequent use, all nondedicated or nondisposable sampling equipment must be decontaminated using the procedures outlined in HGL SOP 411.02: *Sampling Equipment Cleaning and Decontamination.*

4.3 SAMPLING LOCATION/SITE SELECTION

Follow the sample design criteria outlined in the project plan for each sampling event. Relocate the sample sites when conditions dictate, such as when natural or artificial obstructions are present at the proposed sample location (such as boulders or asphalt). Document the actual sample locations on a topographic map or site sketch and photograph all sample locations. GPS coordinates for the new location may also need to be recorded.

4.4 GENERAL

All boreholes and pits are filled in with the material removed during sampling unless otherwise specified in the project-specific planning documents. Where a vegetative turf has been established, fill in with native soil or potting soil and replace the turf if practical in all holes or trenches when sampling is completed.

4.4.1 Homogenizing Samples

Homogenizing is the mixing of a sample to provide a uniform distribution of the contaminants. Proper homogenization ensures that the containerized samples are representative of the total soil sample collected. All samples to be composited or split should be homogenized after all aliquots have been combined. **Do not homogenize (mix or stir) samples for volatile compound analysis. Follow the procedures outlined in HGL SOP 403.01: *VOC Soil Sample Collection* for collection of such samples.**

4.4.2 Compositing Samples

Compositing is the process of physically combining and homogenizing several individual soil aliquots of the same volume or weight. Compositing samples provide an average concentration of contaminants over a certain number of sampling points. Refer to HGL SOP 403.03: *Soil or Sediment Sample Compositing*.

4.4.3 Splitting Samples

Splitting samples is performed when multiple portions of the same samples must be analyzed separately. After preparation, fill the sample containers for the same analyses one after another in

a consistent manner (parent sample for semivolatile organic compounds [SVOCs] analysis, then split sample for SVOC analysis; parent sample for total metals analysis, then split sample for total metals analysis; and so forth).

4.5 SURFACE SOIL SAMPLING

Perform the following steps for surface soil sampling:

- Before sampling, remove leaves, grass, and surface debris from the area using a decontaminated stainless steel trowel or disposable sampling spoon.
- Label the lid of the sample container with an indelible pen or affix the sample label to the side of the jar. Tape over the label to seal out dirt and water before filling the container with soil, if possible.
- Collect surface soil samples with a decontaminated stainless steel trowel, spoon, or hand auger and transfer them to a decontaminated stainless steel bowl for homogenizing. If VOC analyses are to be conducted, collect the VOC sample first following the procedures outlined in HGL SOP 403.01: *VOC Soil Sample Collection*, then transfer the appropriate aliquot of soil to the decontaminated stainless steel bowl for homogenizing.
- Collect samples in the order of volatilization sensitivity. The most common collection order is as follows:
    - VOC,
    - Purgeable organic carbon,
    - Purgeable organic halogens,
    - Total organic halogens,
    - Total organic carbon,
    - Extractable organics,
    - Total metals,
    - Phenols,
    - Cyanide, and
    - Radionuclides.
- Immediately transfer the sample into a container appropriate to the analysis being performed.
- Place the samples in a cooler with ice. The temperature in the cooler must be maintained at approximately 4ºC (if appropriate for analyses) for transport to an analytical laboratory.
- Material removed to collect the samples is returned to the boreholes and pits. Excess soil sample media should be treated as investigation-derived waste (IDW) and managed in accordance with the project-specific planning documents.
- Decontaminate all sampling equipment following HGL SOP 411.02, *Sampling Equipment Cleaning and Decontamination*.

4.6 SURFACE SOIL SAMPLING (COMPOSITE SAMPLES ONLY)

Perform the following steps for surface soil (composite) sampling:

- Before sampling, remove leaves, grass, and surface debris from the area using a decontaminated stainless steel trowel.
- Collect surface soil aliquots with a decontaminated stainless steel spoon, trowel, or hand auger and place them in a stainless steel bowl and homogenize. Homogenize the sample in accordance with HGL SOP 403.03: *Soil or Sediment Sample Compositing*. Follow the procedures outlined in HGL SOP 403.01: *VOC Soil Sample Collection*, for samples collected for VOC analysis.
- Label the sample container and place it in a cooler chilled to 4ºC . Complete the chain of custody record and pack it in the sample cooler.
- Material removed to collect the samples is returned to the boreholes and pits. Excess soil sample media IDW should be managed in accordance with the project-specific planning documents.
- Decontaminate all nondedicated sampling equipment following HGL SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.

4.7 SHALLOW DEPTH SOIL SAMPLING

Perform the following steps to collect shallow depth soil samples:

- Use a decontaminated stainless steel shovel to remove the top layer of soil and leaves, grass, and surface debris.
- Excavate soil to the pre-determined sampling depth using a decontaminated hand auger. Periodically remove the cuttings from the auger.
- When the proper sample depth is reached, remove the hand auger and all cuttings from the hole.
- Lower the decontaminated core sampler or hand auger to the bottom of the hole. When using a core sampler, it must contain a decontaminated liner appropriate for the constituents to be analyzed.
- Mark the sample interval on the hammer stem or auger.
- Operate the slide hammer on the core sampler to drive the sampler head into the soil, or advance the auger until it is flush with the interval mark at ground level.
- Record weight of hammer, length of slide, blow counts, and geologic soil data for all samples collected with a core sampler in the field logbook as outlined in HGL SOP

300.04: *Field Logbook Use and Maintenance*. This information may also be entered on Attachment 2, Surface and Shallow Soil Sampling Log.

- When the core sampler liner or auger has been advanced to the total depth of the required sample, remove it from the bottom of the hole.
- Immediately remove the liner from the core sampler and transfer the sample into a container or stainless steel bowl appropriate to the analysis being performed and then composite and homogenize it in accordance with HGL SOP 403.03: *Soil or Sediment Sample Compositing*. For VOC analysis follow the sample procedures outlined in HGL SOP 403.01: *VOC Soil Sample Collection.*
- Label the sample container and place it in a cooler chilled to 4ºC . Complete the chain of custody record and pack it in the sample cooler.
- Material removed to collect the samples is returned to the boreholes and pits. Excess soil sample media IDW should be managed in accordance with the project-specific planning documents.
- Decontaminate all sampling nondedicated equipment following HGL SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.

4.8 ABANDONMENT PROCEDURES

Abandon boreholes and fill them to grade with the material removed for sampling, if approved, or clean fill.

5.0 DOCUMENTATION

Record applicable sampling information in the field logbook as outlined in HGL SOP 300.04: *Field Logbook Use and Maintenance*. This information can also be entered on Attachment 2, Surface and Shallow Soil Sampling Log.

The project manager or an approved designee checks all field sheets and field logbooks used to record information during sampling for completeness and accuracy as soon as possible after the sampling event. Any discrepancies are noted, and the documents are returned to the originator for correction. The reviewer acknowledges that these review comments have been incorporated by signing and dating the “checked by” and “date” blanks on the field sheets and at the applicable places in the logbook.

6.0 REVISION HISTORY

Revision 0	July 2010	Initial Release
Revision 1	July 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	February 2018	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 3	June 24, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 2.13 to 403.06.

ATTACHMENTS

Attachment 1 – Example of Hand Auger and Core Sampler
Attachment 2 – Surface and Shallow Soil Sampling Log

ATTACHMENT 1
EXAMPLE OF HAND AUGER AND CORE SAMPLER

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www.ams-samplers.com

The world's finest sampling equipment.

Basic Soil Sampling Kit - 5/8" Threaded

DESCRIPTION:
Hand auger kit includes a Standard type Regular, Mud and Sand Auger plus an AMS Core Sampler* with slide hammer. Included accessories are three 4 foot (1.2m) extensions, cross handle, cleaning brush, 2 crescent wrenches and slip wrench all contained in an AMS Deluxe storage and transport case. Two sizes of kit are available, 3 1/4 inch (8.3 cm) augers with 2 inch (5.1 cm) Core Sampler and 2 1/4 inch (5.7 cm) augers with 1 1/2" Core Sampler. Quick connect is not available with this kit.

APPLICATION:
Use of the augers for accessing the sampling point at depths of up to about 12 feet (3.6 m) with the supplied extensions and AMS slide hammer. The sample may be collected within a removable retaining cylinder (liner). Plastic end caps are included.

FEATURES
AMS Soil augers are designed to rapidly remove soils of all types, using the specially designed bits on the Regular, Mud, and Sand models. The auger tips are tungsten carbide hard surfaced and heat treated before sharpening. The core sampler features a heat treated coring tip on the cylinder and a threaded end cap. All attatchment couplings are 5/8 NC threaded.

BENEFITS
For your convenience, all the items necessary for accessing a sampling point and then taking a sample are included. AMS soil buckets are the most efficient available in terms of effort required and speed. The AMS Core Sampler allows immediate core examination or a sample may be collected in a retaining cylinder for later use.

USE:
Assemble the chosen soil auger with an extension and cross handle. Place at the desired angle on the soil surface and turn three revolutions, or until full. Lift carefully from the hole and empty from the bail by tapping the cross handle on the ground. Repeat until the sampling depth is reached. Assemble core sampler to an extension(s) and slide hammer. Place in the hole and mark the extension six inches (5.1 m) above the soil surface. Use the slide hammer to drive in the the sampler to the mark and carefully remove. Disassemble, remove the liner and place the cap on each end.

HELPFUL HINTS:
Use plumbers wick on 5/8 inch male threads used with Slide Hammer to help threads stay tight. Keep all fittings and samplers clean, dry and free of dirt or Mud. You can clean tooling with soapy water. Always dry to prevent rusting. Use a wire brush on male threads. Use vegetable oil on tools to prevent fittings locking up and rusting. When using augers, use rubber O-rings on male 5/8 inch thread to help take apart.

SPECIFICATIONS:
AMS Soil Auger Kits are manufactured by AMS from all USA made materials. See separate AMS Technical Data Sheets for details on the Regular, Mud, Sand & Soil Augers, Core Sampler, Extensions, Cross Handles, Slide Hammer, and Liners. Crescent wrenches are made from chrome plated forged steel. The cleaning brush is made with nylon bristles, with a twisted wire handle. The AMS Deluxe Case is molded from glass reinforced plastic with a lid gasket and lockable hasps.

Kit Composed of the Following Items

Item	Size	Part #	Size	Part#
1- Regular Auger	3 1/4"	400.06	2 1/4"	400.08
1- Mud Auger	3 1/4"	400.18	2 1/4"	400.20
1- Sand Auger	3 1/4"	400.40	2 1/4"	400.42
1- Cross Handle		406.04		406.04
3- Thrd. Extensions	4'	408.03	4'	408.03
1- Core Sampler*	2" x 6"	404.10	1 1/2" x 6	404.38
* w/slip wrench, liner & caps				
1- Slide Hammer		400.99		400.99
1- AMS Nylon Brush	2"	430.07	1 1/2"	430.11
2- Crescent Wrenches 421.10			421.10	
1- Slip Wrench		421.29		421.29
1- AMS Deluxe Case		430.01		430.01

* Patent Pending, USA & Foreign

ANCILLARY ITEMS:
AMS Extensions, Liners, End Caps, End Cap Inserts, Sieves, Soil Color Charts, and Sample Containers.

Basic Soil Sampling Kit

Basic Soil Sampling Kit	
Size	Basic Kit Regular
2 1/4"	209.53
3 1/4"	209.51

Sampling Equipment
PowerProbe
Well Management
Pest Control
PowerCore

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ATTACHMENT 2
SURFACE AND SHALLOW SOIL SAMPLING LOG

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HydroGeoLogic, Inc
Exceeding Expectations

Surface and Shallow Soil Sampling Log

Records Management Data

Project Number	Project Name	Page
		____ of _____

General Info			
	Location		
	Surface Elevation ft.	Date Started	Date Completed
	Field Investigator		C of Cr
	Sampling Excavation Method	Sampling Method	
	Depth of Excavation ft.	Depth Water First Encountered ft.	Backfill Material

Sampling Info	Sample Number	Depth (ft)	Lithologic Description[1]	Sample Container	Analyses Requested

Plan View		Legend
		Soil Sampling Location

Recorded By:	Date	Checked By:	Date:

[1] Include such data as OVM, pH, blow counts, or other physical reading observations.

1.0 PURPOSE

This Standard Operating Procedure (SOP) defines the methodology for conducting lithologic logging of cores, cuttings, split-spoon samples, and subsurface samples collected during field operations at sites where environmental investigations are performed by HGL.

2.0 SCOPE AND APPLICATIONS

The installation of monitoring wells, piezometers, and boreholes is a standard practice at many sites requiring environmental investigations. Following the guidelines presented in this SOP will help ensure that pertinent data is collected so that all borehole logs made while installing these devices at a site can be standardized to create a consistent, uniform database from which interpretive conclusions can be made with minimal decision error. A borehole log provides lithologic descriptions to characterize the physical subsurface and the geologic and hydrologic processes operating at the site. A properly prepared borehole log serves as an essential tool for evaluating and correlating these processes.

This SOP provides guidance for routine field operations on environmental projects, and was derived from *A Compendium of Superfund Field Operations Methods*, U.S. Environmental Protection Agency (EPA) (EPA/540/P-87/001 [Office of Solid Waste and Emergency Response {OSWER Directive} 9355.0-14]); and other industry standards.

3.0 GENERAL REQUIREMENTS

All work will be 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 will 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 will be sufficiently documented to re-create the modified process.

4.0 PROCEDURES

4.1 INTRODUCTION

Boreholes should be logged by a trained geologist, or other earth scientist under the supervision of a geologist. Large-scale inferences such as vertical and horizontal extent of strata, facies changes, attitude of bedding or layering, structural features (faults, folds, fractures, dikes, etc.), location of the water table, lithologic characterizations, and the extent of subsurface contamination are made from small-scale observations recorded on the borehole log. These observations include bedding, grain

size, degree of sorting, shape of grains, color, hardness, organic vapor levels, and other observable physical characteristics including visible evidence of contamination.

Logging should document both general and specific lithologic information about the borehole. In all cases, the lithologic log should be identified with the following:

- Specific site number,
- Well/boring number,
- Drilling method,
- Location,
- Date of drilling,
- Individual logger (geologist),
- Drilling contractor,
- Significant organic vapor reading,
- Visible evidence of contamination, such as staining or odor,
- Depth to water first encountered,
- Final depth of water level,
- Well/boring elevation (if data is available),
- Total depth in feet,
- Graphic log, and,
- Lithologic description.

Lithologic descriptions for unconsolidated materials often use the Unified Soil Classification System (USCS) or standard geologic field description methods (Compton, 1962).

Lithologic descriptions of unconsolidated material should contain the following characteristics when possible:

- Soil or formation name,
- Gradation degree of sorting,
- Principal constituent,
- Specific descriptors for principal constituents (for example, plasticity, grain size, and shape),
- Firmness/hardness,
- Minor constituents,
- Moisture content,
- Color,
- Particle morphology, and

- Other descriptors (such as, visual evidence of contamination, specific monitoring equipment readings including photoionization detector [PID]/organic vapor analyzer [OVA] readings).

4.2 CLASSIFICATION SYSTEM

The following subsections describe in detail the parameters and descriptive terminology used to classify each sample for the borehole log.

4.2.1 Soil or Formation Name

The soil or formation name will include the major constituent(s) and may be preceded by a single-word modifier indicating the subordinate constituent. Percentages of each constituent will be used to classify the material without actually recording constituent percentage. The textural terms used to classify a soil are shown in Attachment 1, Triangular Diagram Showing Percentage of Sand, Silt, and Clay in Each Textural Class. If logging unconsolidated materials, a USCS symbol should be recorded. The USCS symbols are provided in Attachment 2, Unified Soil Classification System Table.

4.2.2 Gradation (Degree of Sorting)

Size sorting describes the extent to which grain size is uniform. The comparison chart listed in Attachment 3, Comparison Chart for Estimating Degree of Sorting, is used to describe coarse-grained soils being logged from a borehole. The USCS describes soils in terms of grading, which is the opposite of sorting. For example, a poorly graded sand (USCS classification SP) is well sorted and has a predominant grain size, and a well graded gravel (USCS classification GW) is poorly sorted and has a wide distribution of grain sizes.

4.2.3 Principal Constituent

Principal constituents recorded during borehole logging include an identification of the following unconsolidated material types in order of increasing grain size:

- Clay,
- Silt,
- Sand,
- Gravel,
- Cobbles, and
- Boulders.

If known, an identification of the potential source of the material should be made (such as alluvium, colluvium, artificial fill, or residual material).

4.2.4 Principal Constituent Descriptors

Additional descriptors for the principal material constituents may be added to the log to further delineate or accurately record subtle changes in the lithologic structure. Modifiers such as grain size, shape, and plasticity of materials (high, medium, and low plasticity). (Note: Plasticity is the property of permanently changing shape without movement on any visible fractures.)

4.2.5 Consistency/Density/Rock Hardness

The characteristics of unconsolidated material are often determined by hand or the Standard Penetration Test (SPT).

Hand testing of unconsolidated material involves pressing the thumb into the undisturbed material to determine its consistency based on the following descriptors:

Depth of Thumb Imprint	Cohesive Consistency (Clay)
Greater than 1 inch	Very soft
Approximately 1 inch	Soft
Approximately ¼ inch	Firm
Thumb will not indent soil but readily indented by fingernail	Hard
Thumbnail will not indent soil	Very hard

The SPT involves driving a split-spoon sampler into the material by dropping a 140-pound weight from a height of 30 inches. The resistance of the material is reported in the number of blows of the weight required to drive the spoon one foot and translates into the following descriptors:

Number of Blows/Foot	Cohesive Consistency (Clay)
0–2	Very soft
2–4	Soft
4–8	Medium
8–15	Stiff
15–30	Very stiff
30+	Hard

Number of Blows/Foot	Cohesive Consistency (Gravel)
0–4	Very loose
4–10	Loose
10–30	Medium dense
30–50	Dense
50+	Very Dense

Number of Blows/Foot	Rock Hardness
<20	Weathered
20–30	Firm
30–50	Medium Hard
50–80	Hard
80+	Very Hard

4.2.6 Minor Constituents

Constituents not previously described in the principal constituent description may be described as a percentage or by weight. Typically, modifiers for minor constituents conform to the following standards:

- No modifier < 5 percent,
- Slightly 5 to 12 percent,
- Moderately (add '-y' or '-ey' such as silty clay) 12 to 40 percent, or
- Very 40 to 50%.

4.2.7 Moisture Content

The terms used to describe the relative moisture content of a field soil sample are as follows:

- Dry – The sample is completely without moisture. Dry, silty sands, for example, will produce suspended particles when dropped by hand.
- Damp – Samples containing a very slight amount of water.
- Moist – Soils in this range are near the maximum water content for their maximum compactibility or density. Moist fine-grained soils with a water content greater than their plastic limit will form a ball when compressed in the hand.
- Wet – The soil samples are wet enough to produce free water upon shaking but still contain unoccupied air voids. Fine-grained soils close to the liquid limit would be termed wet.
- Saturated – Soils with no air voids. Samples placed in sample jars or bags will probably have standing water after a short period of time.

4.2.8 Plasticity

The plasticity of fine-grained soils is recorded on the borehole log. A fine-grained soil can be non-plastic or have low, medium, or high plasticity. The plasticity is measured by the ability to roll the material into a 1/8-inch-thick thread based on the following descriptors:

- Non-plastic – The thread cannot be rolled at any water content.
- Low plasticity – The thread can barely be rolled and a lump cannot be formed when drier than the plastic limit.
- Medium plasticity - The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
- High plasticity – It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier the plastic limit.

4.2.9 Color

The color of soil and associated materials will be recorded on the borehole log. Color descriptors should include but are not limited to the following descriptors: black, gray-black, brown, olive, mottled, and streaked. A Munsell Soil Color Chart should be used to provide general logging guidance, but specific use is not necessary for adequately describing lithology.

4.2.10 Particle Morphology

The key elements of particle morphology are roundness and sphericity. Roundness is a measure of the curvature of grain corners. Sphericity is a measure of how equal the three axial lengths (x, y, z) of an object are. Determination of both properties is facilitated by the use of a hand lens. Estimate grain roundness and sphericity in coarse-grained soils by using an American Geological Institute (AGI) data sheet (Attachment 4).

4.2.11 Other Descriptors

Field screening data collected during the drilling process may help further characterize site conditions during subsurface investigations. Readings from on-site monitoring equipment such as PIDs, flame ionization detectors (FIDs), or OVAs should be recorded at each sample interval. Other useful information includes the organic content and the presence or absence of waste material in samples.

4.2.12 Particle Size Distribution

An estimate of particle sorting by grain size is often useful for borehole logging purposes. Precise estimates of percent composition of the sample are not necessary.

USCS Grain Size Categories

Exact Size Limits	Approximate Inch Equivalents	Name of Loose Aggregate
>256 mm	>10 in.	Boulder gravel
64–256 mm	2.5–10 in.	Cobble gravel
32–64 mm	1.2–2.5 in.	Very coarse pebble gravel
16–32 mm	0.6–1.2 in.	Coarse pebble gravel
8–16 mm	0.3–0.6 in.	Medium pebble gravel
4–8 mm	0.15–0.3 in.	Fine pebble gravel
2–4 mm	0.08–0.15 in.	Granule (or very fine pebble) gravel
1–2 mm	0.04–0.08 in.	Very coarse sand
1/2–1 mm	0.02–0.04 in.	Coarse sand
1/4–1/2 mm	0.01–0.02 in.	Medium sand
1/8–1/4 mm	0.005–0.01 in.	Fine sand
1/16–1/8 mm	0.002–0.005 in.	Very fine sand
1/256–1/16 mm	0.00015–0.002 in.	Silt
<1/256 mm	<0.00015 in.	Clay (clay-size materials)

mm = millimeters
Source: Wentworth Scale; Compton 1962

The Comparison Chart for Estimating Percentage Composition (Attachment 5) can be used to estimate the percentage of various grain sizes present in a sample. However, visual estimates usually provide sufficient information for characterizing site lithology.

4.3 BOREHOLE LOGS

Record data collected during exploratory boring soil logging in the field logbook and on Attachment 6, Borehole Log. Use this log on all applicable field drilling and subsurface sampling operations.

Geologic correlation and aquifer properties prediction are dependent on good exploratory boring sample descriptions. Rotary drilling with fluids is generally unacceptable since the drilling fluids may potentially contaminate the aquifer under investigation and provide inaccurate water levels. High quality borehole data are generally acquired with a direct-push acetate-lined sampler, a split-spoon sampler, or a sonic core barrel. This method of sampling provides detailed logging because the samples collected are undisturbed. The lithofacies interpreted from air-rotary or auger cuttings logs may lack the accuracy necessary for detailed correlation. Where possible, techniques such as geophysical borehole logging will be used to supplement cuttings descriptions. Note on the log any geologic description determined from borehole cuttings. The cuttings are often mixed over the entire length of the boring.

In bedrock formations, cuttings may be acquired from a reverse circulation, air rotary, or dual-wall rotary boring. These cuttings do not provide information on the in situ properties of the materials, but do provide adequate sample description information.

In summary, close sample spacing or continuous sampling in a boring provide the best material for descriptive geology. Use traditional geologic terminology and supplement with the USCS descriptive system when appropriate. Provide sufficient data on layering and other sedimentary structures and undisturbed textures. Sample numbers, depths, and analytes should be included in each description. The applicable field methods described by Compton (1962) and AGI (1982) are recommended. These methods are fully referenced in Section 5.0.

4.4 REVIEW

Personnel conducting borehole logging of soil will record field data on Attachment 6, Borehole Log, and will record a chronological summary in the project logbook. The applicable methods outlined in this procedure shall be used to record the data on this log. The personnel conducting these operations will sign and date the "logged by" and "date" blanks on Attachment 6, Borehole Log.

The Project Manager or designee shall check all field generated data and Attachment 6, Borehole Log, for completeness and accuracy. Any discrepancies will be noted, and the logs will be returned to the originator for correction. The reviewer will acknowledge that corrections have been incorporated by signing and dating the "reviewed by" and "date" blanks on Attachment 6, Borehole Log.

5.0 REFERENCES

American Geological Institute (AGI), 1982. AGI Data Sheets. Falls Church, Virginia.

ASTM International, 2009. ASTM D2488-09a: *Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)*. West Conshohocken, Pennsylvania.

Compton, Robert R., 1962. *Manual of Field Geology*. John Wiley and Sons, Inc. New York, New York.

Munsell, 1988. Munsell Soil Color Charts. Macbeth Division, Kollmorgen Instruments Corporation, Baltimore, Maryland.

6.0 REVISION HISTORY

Revision 0	December 2010	Initial Release
Revision 1	July 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	November 20, 2019	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.

ATTACHMENTS

Attachment 1 – Triangular Diagram Showing Percentage of Sand, Silt and Clay in Each Textural Class
Attachment 2 – Unified Soil Classification System Table
Attachment 3 – Comparison Chart for Estimating Degree of Sorting
Attachment 4 – Comparison Chart for Estimating Roundness and Sphericity
Attachment 5 – Comparison Chart for Estimating Percentage Composition
Attachment 6 – Borehole Log

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ATTACHMENT 1

TRIANGULAR DIAGRAM SHOWING PERCENTAGE OF SAND, SILT AND CLAY IN EACH TEXTURAL CLASS

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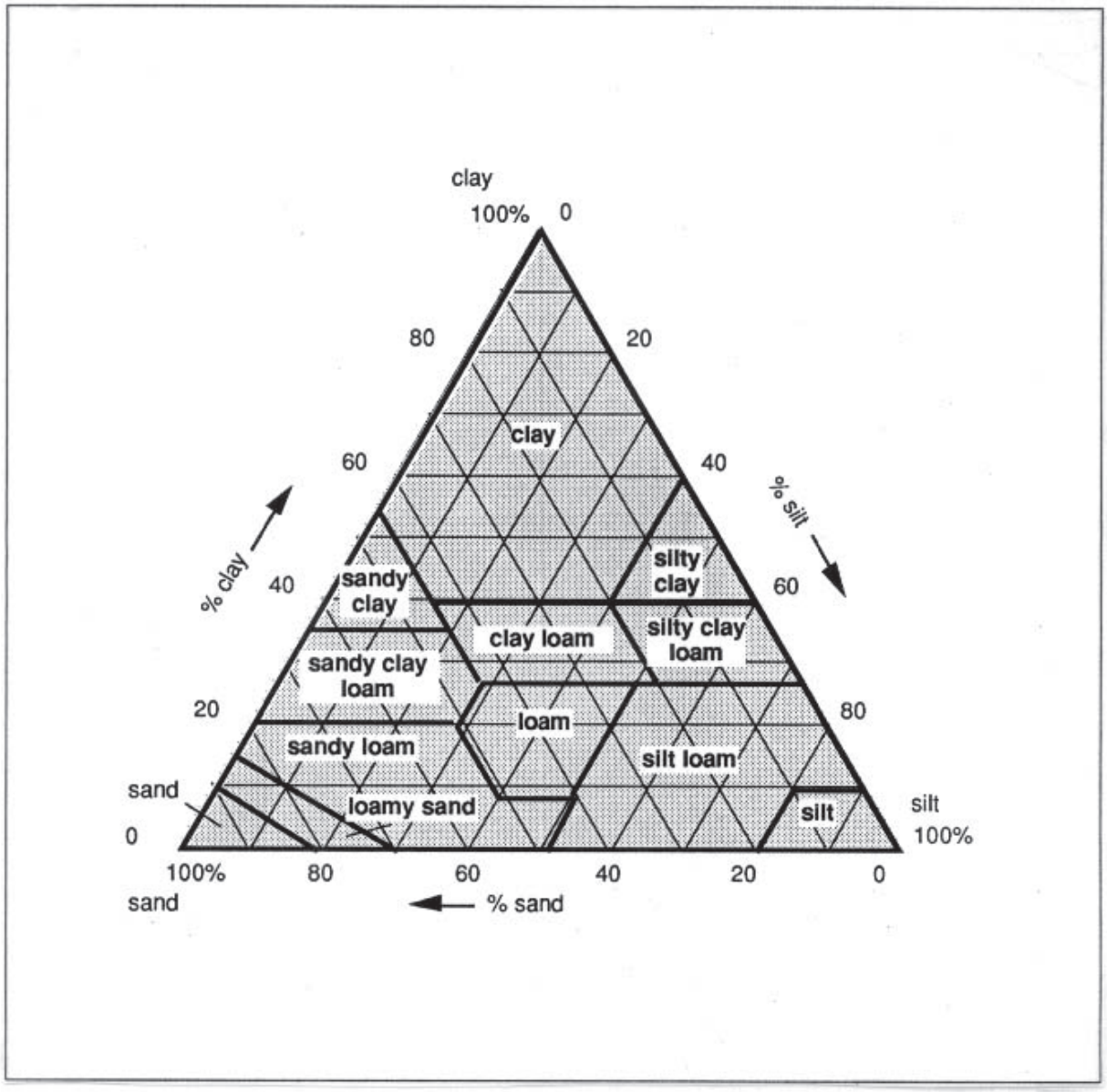
Attachment 1
Triangular Diagram Showing Percentage of Sand, Silt and Clay in Each Textural Class



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ATTACHMENT 2

UNIFIED SOIL CLASSIFICATION SYSTEM TABLE

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Attachment 2
Unified Soil Classification System Table

UNIFIED SOIL CLASSIFICATION AND SYMBOL CHART

COARSE-GRAINED SOILS (more than 50% of material is larger than No. 200 sieve size.)			
GRAVELS More than 50% of coarse fraction larger than No. 4 sieve size	Clean Gravels (Less than 5% fines)	GW	Well-graded gravels, gravel-sand mixtures, little or no fines
		GP	Poorly-graded gravels, gravel-sand mixtures, little or no fines
	Gravels with fines (More than 12% fines)	GM	Silty gravels, gravel-sand-silt mixtures
		GC	Clayey gravels, gravel-sand-clay mixtures
SANDS 50% or more of coarse fraction smaller than No. 4 sieve size	Clean Sands (Less than 5% fines)	SW	Well-graded sands, gravelly sands, little or no fines
		SP	Poorly graded sands, gravelly sands, little or no fines
	Sands with fines (More than 12% fines)	SM	Silty sands, sand-silt mixtures
		SC	Clayey sands, sand-clay mixtures
FINE-GRAINED SOILS (50% or more of material is smaller than No. 200 sieve size.)			
SILTS AND CLAYS Liquid limit less than 50%		ML	Inorganic silts and very fine sands, rock flour, silty of clayey fine sands or clayey silts with slight plasticity
		CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays
		OL	Organic silts and organic silty clays of low plasticity
SILTS AND CLAYS Liquid limit 50% or greater		MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts
		CH	Inorganic clays of high plasticity, fat clays
		OH	Organic clays of medium to high plasticity, organic silts
HIGHLY ORGANIC SOILS		PT	Peat and other highly organic soils

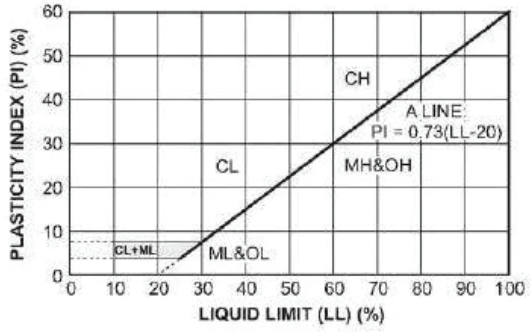
LABORATORY CLASSIFICATION CRITERIA

GW	$C_u = \frac{D_{60}}{D_{10}}$ greater than 4; $C_c = \frac{(D_{30})^2}{(D_{10} \times D_{60})}$ between 1 and 3	
GP	Not meeting all gradation requirements for GW	
GM	Atterberg limits below "A" line or P.I. less than 4	Above "A" line with P.I. between 4 and 7 are borderline cases requiring use of dual symbols
GC	Atterberg limits above "A" line with P.I. greater than 7	
SW	$C_u = \frac{D_{60}}{D_{10}}$ greater than 4; $C_c = \frac{(D_{30})^2}{(D_{10} \times D_{60})}$ between 1 and 3	
SP	Not meeting all gradation requirements for GW	
SM	Atterberg limits below "A" line or P.I. less than 4	Limits plotting in shaded zone with P.I. between 4 and 7 are borderline cases requiring use of dual symbols.
SC	Atterberg limits above "A" line with P.I. greater than 7	

Determine percentages of sand and gravel from grain-size curve. Depending on percentage of fines (fraction smaller than No. 200 sieve size), coarse-grained soils are classified as follows:

Less than 5 percent ..... GW, GP, SW, SP
More than 12 percent ..... GM, GC, SM, SC
5 to 12 percent ..... Borderline cases requiring dual symbols

PLASTICITY CHART



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ATTACHMENT 3

COMPARISON CHART FOR ESTIMATING DEGREE OF SORTING

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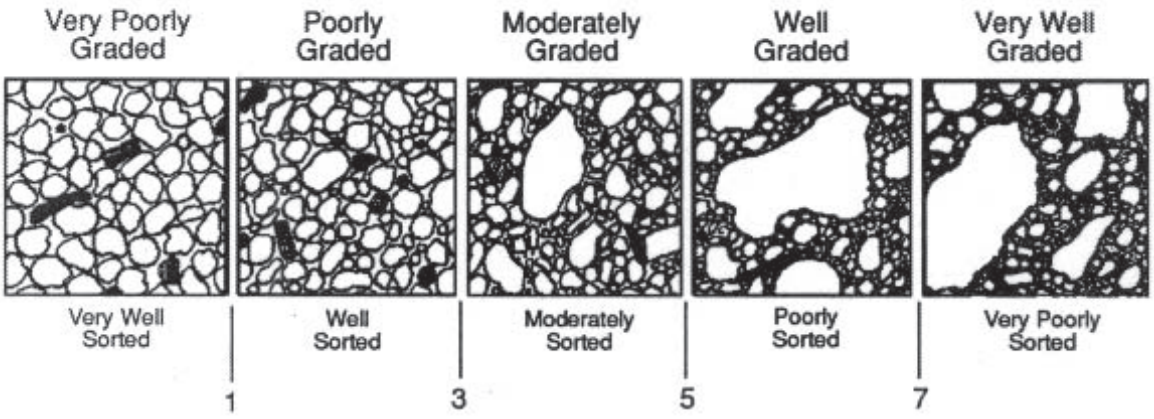
Attachment 3
Comparison Chart for Estimating Degree of Sorting



Terms for degrees of sorting. The numbers indicate the number of size-classes included by the bulk (80 percent) of the material. The drawings represent sandstones as seen with a hand lens. Silt and clay-size materials are shown diagrammatically by the fine stipple.

Reference: Compton, R.R. 1962. *Manual of Geology*. John Wiley & Sons, Inc. New York, N p. 214

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ATTACHMENT 4

COMPARISON CHART FOR ESTIMATING ROUNDNESS AND SPHERICITY

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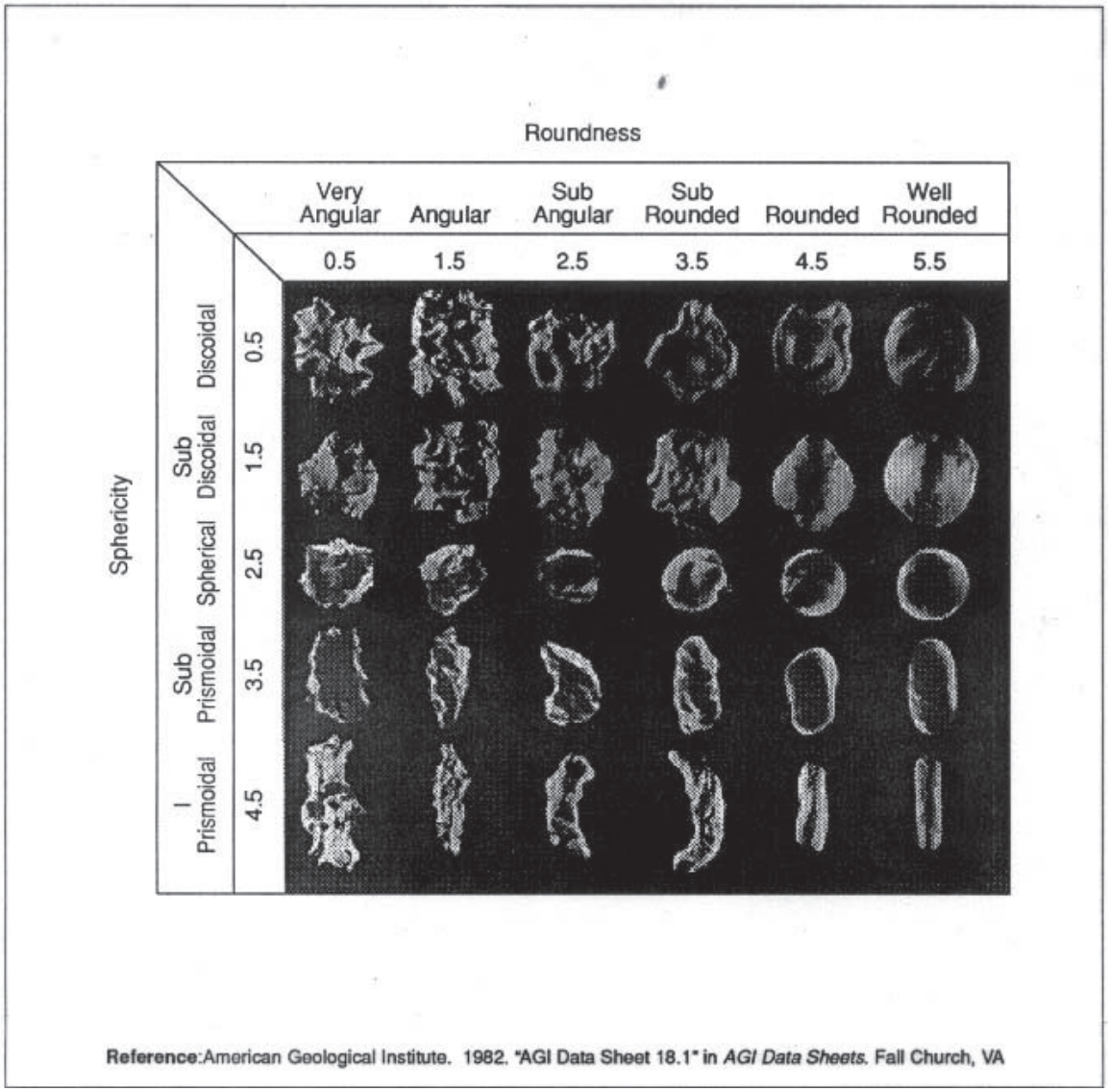
Attachment 4
Comparison Chart for Estimating Roundness and Sphericity



Reference:American Geological Institute. 1982. "AGI Data Sheet 18.1" in *AGI Data Sheets*. Fall Church, VA

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ATTACHMENT 5

COMPARISON CHART FOR ESTIMATING PERCENTAGE COMPOSITION

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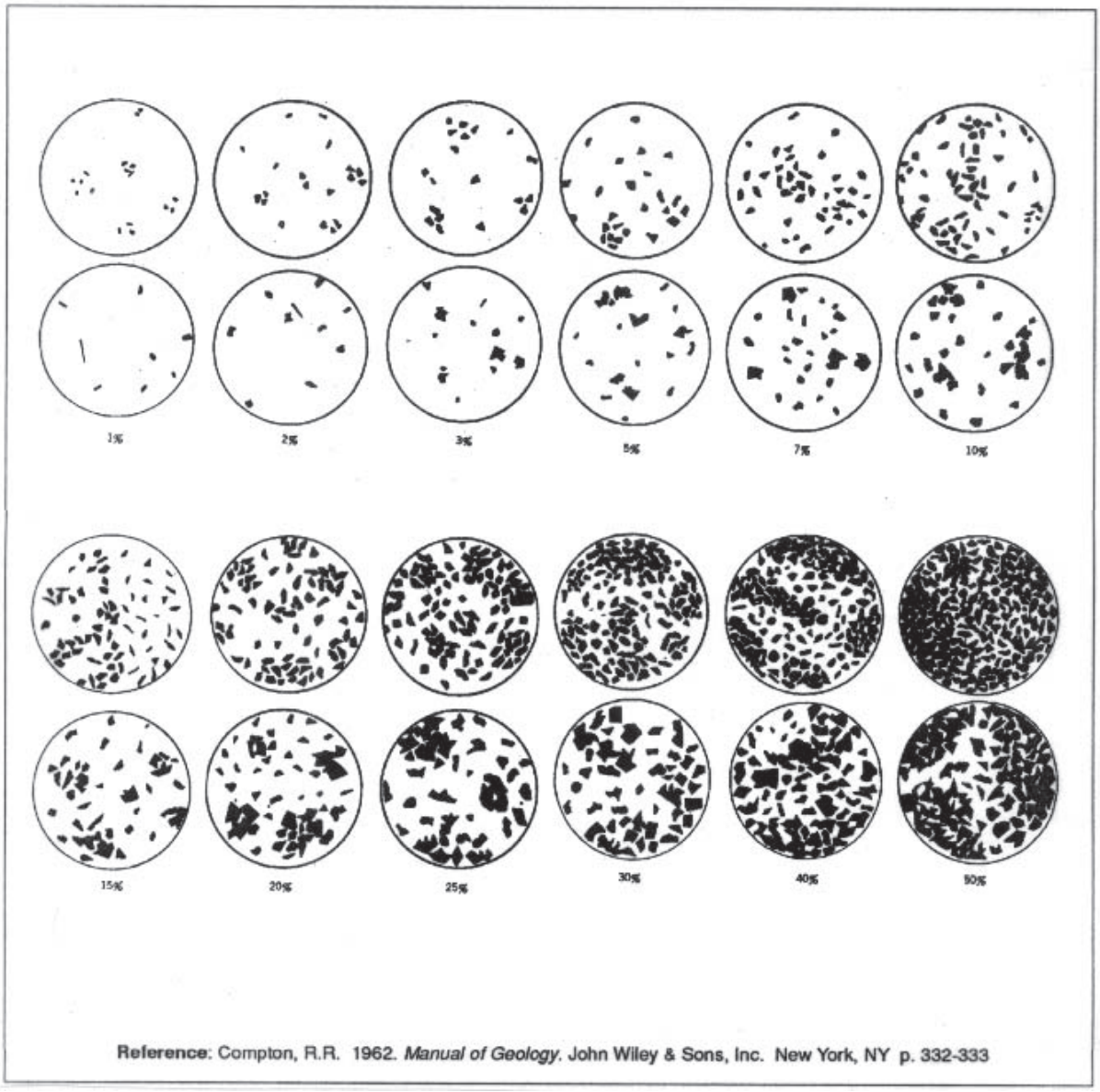
Attachment 5
Comparison Chart for Estimating Percentage Composition



Reference: Compton, R.R. 1962. *Manual of Geology*. John Wiley & Sons, Inc. New York, NY p. 332-333

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ATTACHMENT 6

BOREHOLE LOG

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Attachment 6
Borehole Log

HGL HydroGeoLogic, Inc

BORING LOG

Borehole ID: _____
Sheet _____ of ____

Project Name	Project Number	Site ID	Location
Drilling Company	Driller	Ground Elevation	Total Drilled Depth
Drilling Equipment / Drilling Method / Borehole Diameter		Date/Time Drilling Started	Date/Time Total Depth Reached
Type of Sampling Device		Water Level (bgs) First / Final	
Sample Hammer Type / Driving Wt. / Drop		Hydrogeologist	Checked by/Date
Location Description (include sketch in field logbook)			

Depth	Interval	Recovery	Blow Counts	Description (Include lithology, grain size, sorting, angularity, Munsell color name & notation, minerology, bedding, plasticity, density, consistency, etc., as applicable)	USCS Symbol	Lithology	Water Content	Remarks (Include all sample types & depth, odor, organic vapor measurements, etc.)

HydroGeoLogic, Inc

BORING LOG (cont'd)

Borehole ID: _____
Sheet _____ of ____

Project Name	Project Number	Location
Location Description (include sketch in field logbook)		

Depth	Interval	Recovery	Blow Counts	Description (Include lithology, grain size, sorting, angularity, Munsell color name & notation, minerology, bedding, plasticity, density, consistency, etc., as applicable)	USCS Symbol	Lithology	Water Content	Remarks (Include all sample types & depth, odor, organic vapor measurements, etc.)

HydroGeoLogic, Inc Exceeding Expectations	STANDARD OPERATING PROCEDURE
	Approved by: Dick, Jeff Digitally signed by Dick, Jeff Date: 2020.03.24 18:37:26 -04'00' Corporate Quality Manager

Sediment Sampling	SOP No.: 403.08 (formerly 2.15)
	SOP Category: Environmental Services
	Revision No.: 2
	Revision Date: March 25, 2020
	Review Date: March 2022

1.0 PURPOSE

This standard operating procedure (SOP) establishes the guidelines for sediment sampling using a variety of sampling devices. Methods for preventing sample and equipment cross-contamination are included. Proper sediment sampling ensures that any evaluations of sediment contamination are based on actual contaminant levels and are not based on improper sampling techniques.

This SOP provides guidance for routine field operations on environmental projects. Site-specific deviations from the methods presented herein must be approved by the HGL project manager.

2.0 SCOPE AND APPLICATIONS

Field personnel collecting sediment samples are responsible for performing the applicable tasks outlined in this procedure when conducting work related to environmental projects.

The project manager or an approved designee is responsible for checking all work performed and verifying that the work satisfies the applicable tasks required by this procedure. This verification will be accomplished by reviewing all documents and data produced during work performance.

3.0 GENERAL REQUIREMENTS

All work will be 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 will be justified to and authorized by the project manager and/or the relevant program manager and documented in the approved project plans. Deviations from requirements will be sufficiently documented to re-create the modified process.

4.0 SAMPLING EQUIPMENT AND TECHNIQUES

Sediment samples may be obtained using on-shore or off-shore techniques. Sediment sampling equipment and techniques must be designed to minimize the risk of dilution or loss of material as the sample is moved through the water column. Sediment sampling devices are described below.

4.1 DIP SAMPLERS

A dip sampler consists of a pole with a jar or scoop attached. The pole may be made of bamboo, wood, Teflon®, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is attached by a clamp.

The dip sampler is operated by submerging the jar or scoop and pulling it through the sediments to be sampled. The samples retrieved are then transferred into the appropriate sample container after decanting the liquid. Further decanting can occur while the sample is present in the sample jar. Avoid contact with sampler's gloves. Transferring the sample may require the use of a stainless steel or Teflon® spoon/spatula.

4.2 HAND-OPERATED CORE SAMPLERS

Hand-operated sediment core samplers are used to obtain sediment samples in shallow water (less than 3 feet). These samplers operate in a manner similar to soil core samplers. However, because of the saturated conditions of most sediments, provisions must be made to retain the sample within the core. Core samplers are generally constructed of a rigid metal outer tube into which a 2-inch plastic core sleeve fits with minimum clearance. The cutting edge of the core sampler has a recessed lip on which the plastic sleeve rests and that can accommodate a core retainer. This retainer is oriented such that when the sampler is pressed into the sediment, the core is free to move past the retainer. Due to construction of the retainer, the core will not fall through the retainer upon removal of the sampler from the sediment. Some core samplers are also equipped with a butterfly valve below the core barrel that helps retain the material when the sampler is removed from the sediment.

After the sampler has been removed from the sediment, the plastic sleeve is removed. The sediment is removed from the sleeve and placed in the appropriate sample container. Chlorinated organics will not be collected using core samplers because core sleeves and retainers are generally made of plastic. The hand-operated core sampler will not be useful for obtaining samples of gravelly, stony, or consolidated sediments. Examples of hand-operated core samplers are referenced in Attachment 1.

4.3 GRAVITY CORE SAMPLERS

Gravity core samplers are used to obtain sediment samples in water bodies or lagoons with depths greater than 3 to 5 feet. These types of samplers can be used for collecting 1- to 2-foot cores of surface sediments at depths of up to 100 feet beneath the water surface.

As with all core-type samplers, gravity core samplers are not suitable for obtaining samples of coarse, gravelly, stony, or consolidated deposits. They are, however, useful for fine-grained inorganic sediment sampling.

The gravity core sampler operates in a manner similar to the hand-operated core in that a 2-inch plastic sleeve fits within a metal core housing fitted with a cutting edge. Plastic nests are used to retain the core within the plastic sleeve. An opening exists above the core sleeve to allow free flow of water into and through the core as it moves vertically downward to the sediment. The sampler has a field personnel-operated, messenger-activated valve assembly that seals the opening above the plastic sleeve following sediment penetration. This valve is activated by the messenger, creating a partial vacuum to assist in sample retention during retrieval.

Samples are obtained by allowing the sampler, which is attached to approximately 100 feet of stainless steel aircraft cable, to drop to the benthic deposits. The weight of the sampler drives the core into the sediment to varying depths depending on the characteristics of the sediments. The messenger is then dropped by field personnel on the taut aircraft cable to seal the opening above the plastic sleeve. The sampler is then carefully retrieved.

Upon retrieval of the sampler, the plastic core sleeve is removed and the sample is placed in the appropriate sample container. Care should be exercised in labeling to properly identify sample orientation. Examples of gravity core samplers are referenced in Attachment 2.

4.4 DREDGES

Dredges are generally used to sample sediments that cannot easily be obtained using coring devices or when large quantities of materials are required. Various dredge designs are available for sampling in deep or turbulent waters and for obtaining samples from gravelly, stony, or dense deposits.

Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a messenger. Dredges are commonly quite heavy and may require use of a winch and crane assembly for sample retrieval.

Upon retrieval of the dredge, the sample can either be sieved or transferred directly to a sample container for labeling and storage. Examples of dredge types that could be used for sampling include Ponar, Petersen, and Ekman dredges, which are referenced in Attachment 3.

4.5 HAND AUGERS

Sediment samples may be collected using a hand auger. When using a hand auger, provisions must be made to ensure that sediment samples remain in the auger. Hand augers are best utilized when sampling non-subaqueous sediments. Additional information on hand augers can be found in SOP 403.06: *Surface and Shallow Depth Soil Sampling.*

5.0 PROCEDURES

5.1 SAMPLING SEDIMENT WITH NO OVERLYING SURFACE WATER

Sediment samples obtained from areas with no overlying surface water will be collected in accordance with the following procedures:

- Record all data in the field logbooks in accordance with SOP 300.04: *Field Logbook Use and Maintenance.*

- Insert a decontaminated Teflon® or stainless steel spoon, scoop, or trowel into the sediment to the desired depth and remove the collected sample, or rotate and push down a decontaminated hand auger into the sediment to the desired depth and remove the collected sample. A disposable scoop may be used for specified media and analytical parameters in accordance with the site-specific project plans.
- Collect samples for volatile organic compounds (VOC) analyses, if applicable, from the sampling device or from unmixed sediment placed into a stainless steel bowl in accordance with SOP 403.01: *VOC Soil Sample Collection*.
- Place the sample in a decontaminated stainless steel bowl. Stir the sample thoroughly (non-VOC samples only) with a decontaminated stainless steel spoon or spatula—or with a dedicated disposable scoop—to provide a homogeneous mixture before filling sampling containers.
- Follow the guidelines in the site-specific project plans and Quality Assurance Project Plan (QAPP) for aliquot size (mass), container type, storage conditions, and holding times. [Note: When sampling in coarse materials, such as gravel, discretion must be used to limit inclusion of large sediment particles. As the analysis of sediments performed by the laboratory is typically restricted to particles less than 2 millimeters in size, care must be taken to ensure that there is sufficient sample volume consisting of particles smaller than 2 millimeters. As a general rule, particles larger than 0.5 inch (12.7 millimeters) in size should be excluded unless a grain size analysis is planned.] Fill the appropriate sample containers as detailed in the site-specific project plans. Identify or label samples carefully and clearly, addressing all the categories or parameters.
- Label the sample containers and place the filled sample containers on ice immediately.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*, after use and between sampling if dedicated disposable scoops are not used. Don new clean gloves before beginning sampling activities and at each sampling point.
- Complete all chain of custody documents and record information in the Field Sampling Report (Attachment 4) and the field logbook (see the project-specific QAPP for sample custody procedures).

5.2 SHALLOW STREAM SEDIMENT SAMPLING

Stream sediment sampling within shallow (less than 2 feet) water will be conducted in accordance with the following procedures. Note that if co-located surface water samples are being collected, the surface water sample should be collected first.

- Collect the sample in an area of sediment accumulation, such as the inside of stream meanders, quiet shallow areas, and low-velocity zones. Avoid areas of net erosion, such as high-velocity, turbulent flow zones.

- If possible, collect the sample while remaining on the stream bank. If the sample cannot be obtained from the bank, enter the stream from a point downstream of the sediment sampling location. Consult the site health and safety plan before entering the river to avoid potential hazards. Collect the sediment sample by reaching into the stream with a decontaminated stainless steel spoon or Teflon® scoop and scooping a sample in an upstream direction. Attempt to minimize the loss of fine material. A disposable scoop may be used for specified media and analytical parameters, in accordance with the site-specific project plans.
- Collect samples for VOC analyses, if applicable, from the sampling device or from unmixed sediment placed into a stainless steel bowl in accordance with SOP 403.01: *VOC Soil Sample Collection*.
- Place sample in a stainless steel bowl and gently mix with a stainless steel spoon or dedicated disposable scoop (non-VOC samples only). Transfer the sediment samples to the appropriate sample containers using the stainless steel spoon or dedicated disposable scoop. Do not mix samples for volatile organic analyses.
- Follow the guidelines in the site-specific project plans and QAPP for aliquot size (mass), container type, storage conditions, and holding times. See note under Section 5.1 for sampling coarse materials. Fill the appropriate sample containers as detailed in the site-specific project plans. Identify or label samples carefully and clearly, addressing all the categories or parameters.
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*, after use and between sampling if dedicated disposable scoops are not used. Don new clean gloves before beginning sampling activities and at each sampling point.
- Complete all chain of custody documents and record information in the Field Sampling Report (Attachment 4) and the field logbook (see the project-specific QAPP for sample custody procedures).

5.3 SUBAQUEOUS SEDIMENT SAMPLING

Subaqueous sediment sampling from lakes, ponds, lagoons, and surface impoundments will consist of the following:

- Select the most appropriate sediment sampling device (as described in Section 4.0).
- Decontaminate all sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination*.
- If sampling from a boat equipped with an engine, attempt to collect the sample with the boat engine off or attempt to ensure that all exhaust fumes are directed away from the sample collection area until the sample has been collected.

- Lower the sampler at a controlled descent of approximately 1 foot per second until the sampler reaches the sediment surface, as indicated by a slackening of the cable. Release the weighted messenger, if applicable, to engage the closing mechanism of the dredge. Slowly retrieve the sampler and raise it at a controlled speed. When the sampler is at the water surface, attach a tag line(s) to steady and pull the sampler back into the boat. If large samplers are used, a motorized winch may be required for retrieval.
- Open and tie back any vent flaps on the sampler and carefully siphon off any overlying water, disposing of it over the side of the boat.
- Visually inspect the sample for acceptability (for example, determine if an undisturbed surface layer is evident, the overlying water is not excessively turbid, and adequate penetration is achieved). If the sample is not acceptable, discard it and collect another sample from an adjacent and upstream location.
- Carefully extrude the sediment from the sampler by slowly lifting on the winch cable and sliding the sample out the bottom of the sampler. If using core liners, remove the front face of the core liner to expose the side of the core.
- Visually inspect the side of the sample to identify any obvious stratification (such as different sediment types, sizes, or colors). If no patterns are evident, collect a sample from the surface and mid-core depth. During some investigations, it may be necessary to collect separate samples from the surface and mid-core depths. This may best be accomplished by gently scraping the side of the core with a decontaminated stainless steel scraper or knife. Scrape from the bottom to the top of the core only. If the sediment is unconsolidated, do not scrape.
- Remove the upper 2 centimeters of the sample using a decontaminated Teflon® or stainless steel scoop—or dedicated disposable scoop—and place it in the sample container. From an undisturbed area of the sample surface, scoop a 2-centimeter sample only if grain size analysis is required. After grain size analysis samples are collected, scrape off the upper sediment layer and discard it overboard. Collect samples from the mid-section of the sediment. Sediment must be removed with caution to avoid cross-contaminating the sample (that is, from exposure to engine exhaust, rust, or grease).
- Do not include nonrepresentative materials, such as twigs or debris, in the sample. Do not include sediments that have come into contact with the side of the sampler or core liner for analysis.
- Follow the guidelines in the site-specific project plans and QAPP for aliquot size (mass), container type, storage conditions, and holding times. Fill the appropriate sample containers as detailed in the site-specific project plans. Identify or label samples carefully and clearly, addressing all the categories or parameters;
- Decontaminate the sampling equipment in accordance with SOP 411.02: *Sampling Equipment Cleaning and Decontamination* after use and between sampling if dedicated

disposable scoops are not used. Don new clean gloves before beginning sampling activities and at each sampling point.

- Complete all chain of custody documents and record information in the Field Sampling Report (Attachment 4) and the field logbook (see the project-specific QAPP for sample custody procedures).

6.0 RECORDS

Documentation generated as a result of this procedure is collected and maintained in accordance with requirements detailed in the project-specific planning documents. The field logbook will be completed in accordance with procedures listed in SOP 300.04: *Field Logbook Use and Maintenance.* A Field Sampling Report will be filled out for each sediment sample collected (Attachment 4).

7.0 REVISION HISTORY

Revision 0	December 2010	Initial Release
Revision 1	August 11, 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
Revision 2	February 25, 2020	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting, which included changing the SOP number from 2.15 to 403.08.

ATTACHMENTS

Attachment 1 – Core Sampler
Attachment 2 – Gravity Core Sampler
Attachment 3 – Dredges
Attachment 4 – Field Sampling Report

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ATTACHMENT 1
CORE SAMPLER

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CORE SAMPLER

AMS Core Sampler (http://www.ams-samplers.com/hand-tooling/sludge-and-sediment-samplers/sludge-and-sediment-samplers/sludge-and-sediment-samplers.html)

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ATTACHMENT 2
GRAVITY CORE SAMPLER

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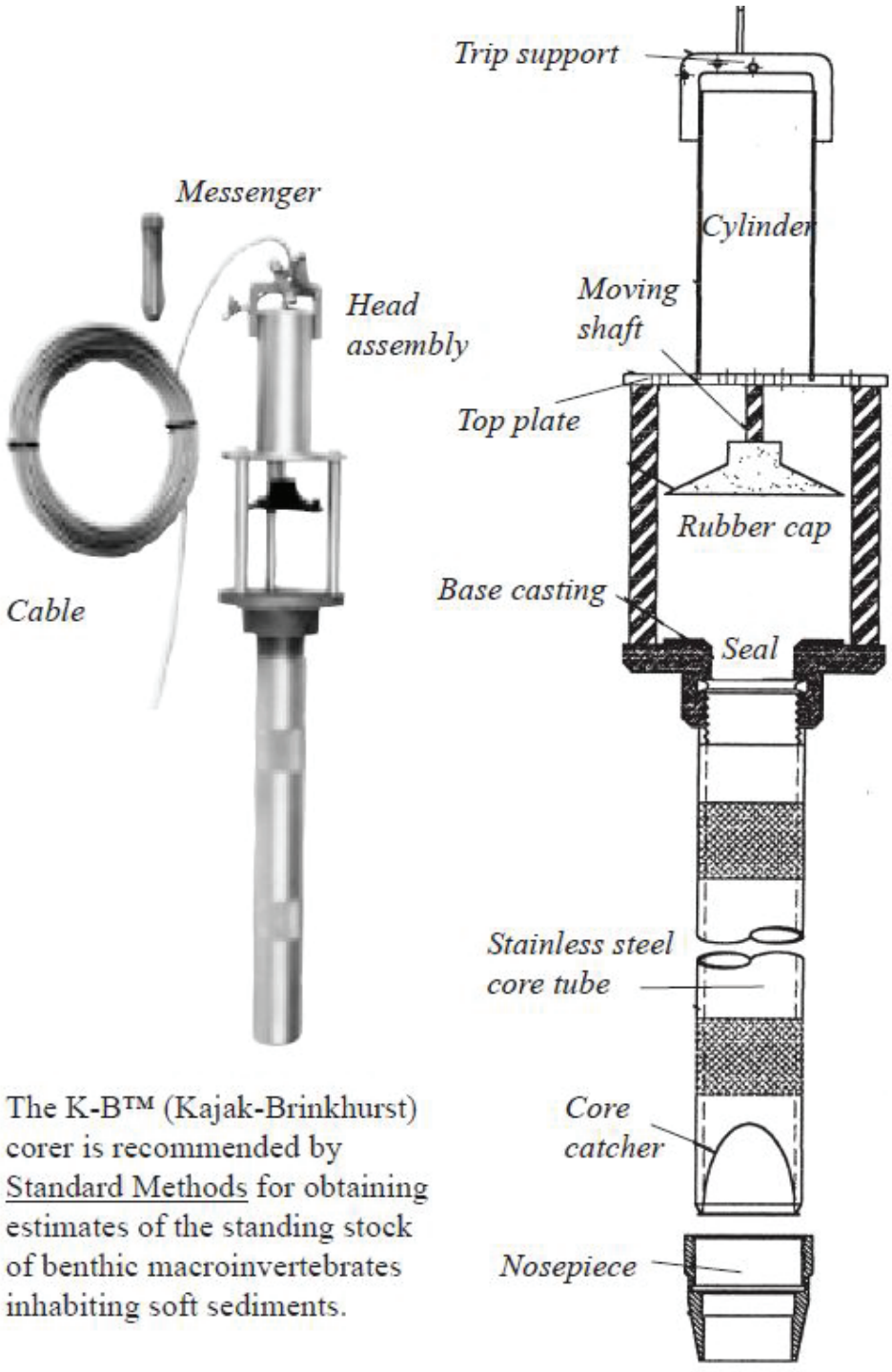
K-B GRAVITY CORER



The K-B™ (Kajak-Brinkhurst) corer is recommended by Standard Methods for obtaining estimates of the standing stock of benthic macroinvertebrates inhabiting soft sediments.

Wildco K-B Corer (http://shop.sciencefirst.com/wildco/k-b-corers/7815-k-b-corer.html)

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ATTACHMENT 3
DREDGES

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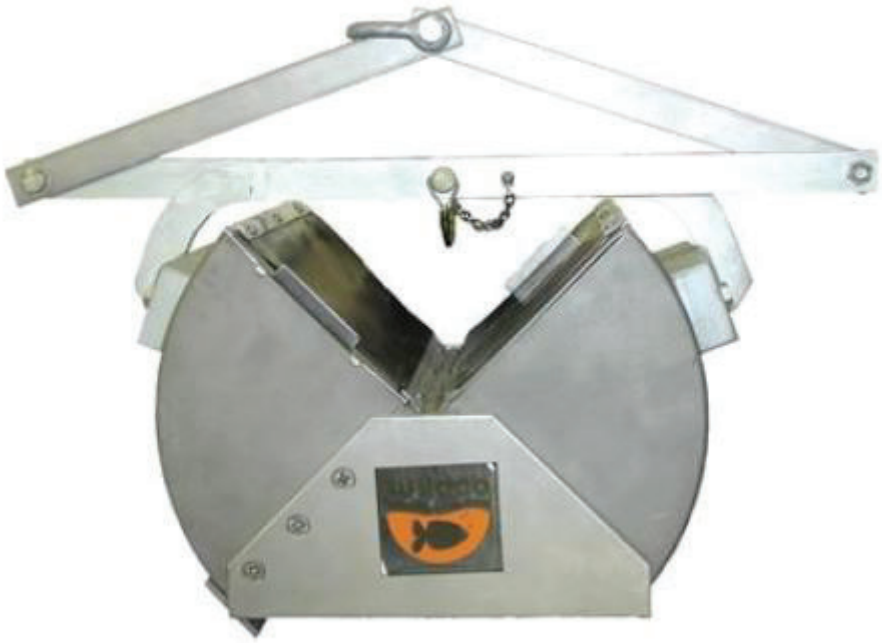
PONAR

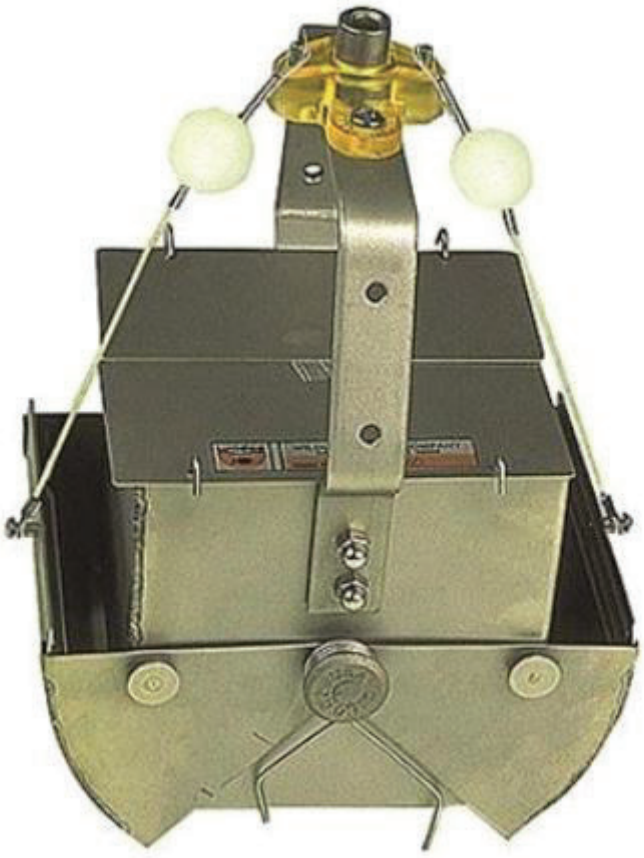


WILDCO Ponar Dredge (http://www.benmeadows.com/wildco-ponar-grabs_36816477/)

PETERSON

WILDCO Peterson Dredge (https://www.coleparmer.com/p/mn/7270)

EKMAN



EKMAN Dredge (http://www.benmeadows.com/ekman-bottom-grab-sampler_36816471/?searchterm=ekman%2bdredge)

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ATTACHMENT 4
FIELD SAMPLING REPORT

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FIELD SAMPLING REPORT

LOCATION: _____ PROJECT : _____

SITE: _____

SAMPLE INFORMATION

MATRIX _____ SAMPLE ID: _____

SAMPLING METHOD _____ DUP./REP. OF : _____

BEGINNING DEPTH _____ MATRIX SPIKE/MATRIX SPIKE DUPLICATE
YES ( ) NO ( )

END DEPTH _____

GRAB ( ) COMPOSITE ( ) DATE: _____ TIME: _____

CONTAINER SIZE/TYPE	#	PRESERVATIVE/ PREPARATION	EXTRACTION METHOD	ANALYTICAL METHOD	ANALYSIS

NOTABLE OBSERVATIONS

PID READINGS	SAMPLE CHARACTERISTICS	MISCELLANEOUS
1st	COLOR:	
2nd	ODOR:	
	OTHER:	

pH _____ Temperature _____ Dissolved oxygen _____ Specific Conductivity_____

GENERAL INFORMATION

WEATHER: SUN/CLEAR _____ OVERCAST/RAIN _____ WIND DRIECTION _____ AMBIENT TEMP _____

SHIPMENT VIA: FEDEX _____ HAND DELIVER _____ COURIER _____ OTHER _____

SHIPPED TO: _____

COMMENTS: _____

SAMPLER: _____ OBSERVER: _____

MATRIX TYPE CODES

DC=DRILL CUTTINGS
WG=GROUND WATER
LH=HAZARDOUS LIQUID WASTE
SH=HAZARDOUS SOLID WASTE
SE=SEDIMENT
SL=SLUDGE
SO=SOIL
GS=SOIL GAS
WS=SURFACE WATER
SW=SWAP\WIPE

SAMPLING METHOD CODES

B=BAILER
BR=BRASS RING
CS=COMPOSITE SAMPLE
C=CONTINUOUS FLIGHT AUGER
DT=DRIVEN TUBE
W=SWAB\WIPE
G=GRAB
HA=HAND AUGER
H=HOLLOW STEM AUGER
HP=HYDRO PUNCH
SS=SPLIT SPOON
SP=SUBMERSIBLE PUMP

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DRAFT **Blacktail Creek Riparian Actions Pre-Design Investigation** **HGL Standard Operating Procedure SOP 408.511 (Modified)** **XRF Screening Using a Field Portable XRF Analyzer**	
Adapted from: **Region 4 U.S. Environmental Protection Agency** **Laboratory Services and Applied Science Division** **Athens, Georgia**	
Title: Field X-Ray Fluorescence Measurement	**ID: LSASDPROC-107-R5**
Effective Date: February 2, 2022	**Next Review Due Date: February 2, 2026**

1. General Information

1.1. Purpose

This document describes general and specific procedures, methods, and considerations for conducting field X-ray fluorescence (XRF) measurements of soil and sediment samples for the Blacktail Creek Riparian Actions Pre-Design Investigation. This document was adapted from U.S. Environmental Protection Agency Region 4 Laboratory Services and Applied Science Division Operating Procedure for Field X-Ray Fluorescence Measurement (EPA LSASDPROC-107-R5, effective February 2, 2022).

1.2. Scope/Application

Field personnel will use the procedures in this document when measuring metals concentrations in soil, sediment, or other solids in the field. If field personnel determine in consultation with the DEQ project manager that any of the procedures described in this procedure cannot be used to obtain metals analyses of the media being sampled, and that another method or XRF instrument must be used to obtain said measurements, the variant instrument and measurement procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3. Documentation/Verification

This procedure was prepared and adapted by persons deemed technically competent based on their knowledge, skills and abilities and reviewed by a subject matter expert. The procedures have been tested in practice.

2. Precautions

2.1. Safety Precautions

Observe all applicable safety precautions when conducting field XRF measurements. Refer to the Health and Safety Plan (HASPs) and Job Hazard Assessments (JHAs) for guidelines on safety precautions. It is recommended that users take the "Radiation Safety for Handheld XRF – X-Ray Tube" or other appropriate safety courses available on the Thermo Scientific™ website. When using this procedure, minimize exposure to potential health hazards by using protective clothing, eye wear and gloves. The operator must always be aware of the instrument's orientation, the direction of its primary X-ray beam, when the primary beam is on or active, and the properties of the sample being analyzed. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

NEVER aim the primary beam at yourself or others!

2.2. Procedural Precautions

All field XRF measurements pertinent to the sampling event will be recorded in a bound field record logbook for the event. This record is created and maintained by the analyst providing the field XRF

support. After the investigation is complete, the analyst will conduct post-processing of the field measurements and will enter final measurement data into the project database and will provide the project manager with a copy of the field measurement logbook. All other records and documentation of the investigation should be recorded according to the procedures outlined in the HGL SOP 401.501 Field Logbook Use and Maintenance.

3. Limitations

The three main sources of interference in XRF analysis that may impact data quality are sample preparation error, spectral interferences, and chemical matrix interferences. Additional significant limitations that the field investigator must consider and control when conducting field analysis using XRF include soil moisture and analyte-specific sensitivity of the XRF unit.

3.1. Preparation Error

The accuracy of the analysis is strongly impacted by sample homogenization. The more homogeneous the sample, typically analyzed by the cup method, the more accurate the results. There is no control of this limitation when conducting in situ analysis. Ex-situ method samples should be sieved and dried in accordance with EPA Method 6200. Grinding of the samples will be conducted only if it is required in the project-specific plans (e.g., Sampling and Analysis Plan (SAP), Quality Assurance Project Plan (QAPP), or Work Plan).

3.2. Spectral Interference

Each element has a signature spectrum of energies and relative intensities. Many elements, however, produce X-rays of similar energy and discerning which element produced a detected X-ray is a factor of the detector's resolution capability and the software's ability to fit all of the data to the relative intensities produced by the various wavelengths.

3.3. Chemical Matrix Interference

Chemical matrix interference refers to the effect that one element has on another in producing X-rays which reach the detector. Dominant elemental components of a sample, such as silicon in soils, vary in concentration from sample to sample and therefore so does that element's influence on the other elements in the sample.

3.4. Soil moisture

Excessive soil moisture biases the results low, i.e., the higher the soil moisture in a particular matrix, the lower the reported concentration relative to the actual concentration. This limitation may be overcome by drying the sample. Without sample drying, XRF measurement results for samples with typical soil moistures within the range of 15-25% are routinely reported at values less than laboratory confirmation analysis for the same samples. The actual difference may vary significantly for all samples from a site, but the XRF results reported by the instrument are typically on the order of 70-80% of the laboratory reported value for samples in this moisture range. This factor should be taken into consideration when making decisions based on XRF results.

3.5. Instrument Analyte Sensitivity/Detection Limits

Because of peak overlaps, some analytes may have problematically high detection limits, i.e., detection limits may be higher than project action levels for certain analytes, limiting its use for rapid field screening for certain elements. One of the most common examples of this phenomenon is the lead/arsenic analyte pair. When lead and arsenic are being analyzed, the peak overlap problem results in detection limits for arsenic that are several times higher than the typical action levels published for this analyte. It commonly is necessary to perform confirmatory analysis in the laboratory to obtain analytical results for arsenic, or other analytes with high detection limits, to obtain data in the range necessary for making regulatory decisions, and to a lesser degree design decisions.

4. Operational Checks and Quality Control

4.1. Maintenance, Storage and Operation

Maintain, store, and operate all XRF instruments and equipment in accordance with the manufacturer's instructions, EPA Method 6200, and HGL SOP 408.511 XRF Screening Using an Innov-X.

4.2. System Check and Calibration

Prior to each operational period, turn on the instrument and allow the unit to perform an internal calibration. Following this calibration, conduct a performance check using the appropriate National Institute of Standards and Technology (NIST) traceable standard reference material for the analytes of concern. Verify that the value is within +/- 20% of the stated value of the standard. Following this performance check, analyze an instrument blank sample to verify the instrument is not registering false positive results for the analytes of concern. After these checks, the instrument is ready for analysis.

4.3. Operation and Quality Control Requirements

The following operational and quality control requirements also apply to operation of the XRF instrument and should be followed and documented in the field logbook maintained by the analyst:

4.3.1. Ambient Air Conditions

During operations, record the ambient air temperature for each measurement and if the ambient temperature changes by more than 10°F, recalibrate the instrument.

4.3.2. Reference Standards and Blanks

While the instrument is being used, run the reference standards and the blank at the beginning of each workday, every 4 to 5 hours of analysis time, after the instrument has been off for 1 to 2 hours or if the battery has been changed, and at the end of the period of operation, prior to turning the instrument off.

4.3.3. Duplicate Sample Analysis

For every twenty samples, or at least once per day, analyze a duplicate using the main sampling technique.

4.3.4. Replicate Sample Analysis

Once per day, check the instrument's precision by analyzing one of the site samples at least seven times in replicate.

4.3.5. Additional Guidance

EPA Method 6200 contains detailed instruction and guidance covering implementation of these procedures and any corrective actions that must be taken based on measured instrument behavior and performance. If at any time during a field investigation, it appears that the environmental conditions could jeopardize the quality of the measurement results or the instrument exhibits unusual drift, stop additional analysis until the problem is identified and corrective measures are completed. Note the stoppage and corrective measures in the in the field logbook.

5. Field X-Ray Fluorescence (XRF) Measurement Procedures

5.1. General

XRF is the property of a material to emit X-rays, with a characteristic energy, upon being irradiated by X-rays of a known source and energy. The emitted X-rays are detected by the particular XRF instrument as they impact a detector, which converts the energy of the emitted X-ray into electric current. The strength of the current is proportional to the energy of the X-ray. An onboard microprocessor counts how often an energy is detected, assigns the energy to a particular element and reports the calculated concentration for the element.

This investigation will use a Thermo Scientific™ Niton™ XL3t Multi-element XRF Spectrum Analyzer, or equivalent. This instrument uses a miniaturized X-ray tube as its source rather than a radioactive isotope for X-ray generation for analysis which reduces interferences related to the radioisotopes of the source. To the extent feasible, the same unit (i.e., the same serial number) will be used for the PDI and for potential future field confirmation sampling during remedial construcion.

6. Mode of Operation

The instrument is typically used in one of two modes, either for taking in situ measurements or ex situ (measuring sample material that has been placed in a cup or bag for analysis in an instrument tray). Both modes of operation and analysis types will be used in this investigation. The following is a brief description of these modes of operation.

6.1. In Situ Measurement

Prior to taking the in-situ measurement, clear the measurement location of any significant vegetation or obstructions, such as large clumps of grass, rocks, or debris, and scuffed or otherwise level the surface to provide a flat surface on which to place the instrument window. Place a piece of thin Mylar® film on the measurement location either directly on the core sample or ground surface to protect the instrument window and preventing it from becoming damaged or contaminated by the media being tested. After the window is pressed to the Mylar® film, the open the window for a nominal (i.e., programmed) of at least sixty seconds. Longer reading times may be employed if

recommended by the manufacturer or if field conditions and results indicate the need for longer reading times.

Because of the shallow penetration of the X-rays in typical soils, the measured concentrations are representative of the concentrations present at the very surface of the material being measured. As indicated above, excessively wet soil also affects measurements. The in-situ method will only be used for general assessment of concentrations and aid in selection of samples for ex-situ measurement and laboratory analysis. If conditions representing concentrations over a greater depth are required by the study data quality objectives (i.e., on the order of three to six inches or the planned 1-foot sample intervals), use the cup method described in Section 6.3.

6.2. Ex-Situ (Collected) Sample Preparation

Samples will be sieved, dried, and prepared for the modified ex situ field XRF analysis in accordance with the modified EPA Method 6200 for intrusive analysis, except the samples will not be ground. The prepared samples will be placed in a resealable plastic bag and labeled in accordance with the PDI Work Plan. Samples will then be analyzed using the portable XRF on the prepared and bagged sample or from an aliquot taken from the bag prepared for laboratory analysis, depending on the specific equipment included with the XRF unit.

6.3. Ex-Situ (Collected) Sample Measurement

Use this method to measure concentrations of metals in soil and sediment samples collected from a vertical interval, either as a grab or a composite sample. Collect the soil or sediment samples for routine chemical analyses in accordance the applicable SOPs included in the QAPP or PDI WP. After mixing, place the media in a clean, unused zip-closure plastic bag (or equivalent) and label the bag in accordance with the QAPP and PDI WP. Take an aliquot from the container and place it in a plastic sample analysis cup with a Mylar® covering. Load the cup containing the sample into a tray for analysis by the XRF instrument. Alternatively, measurements may be obtained by reading directly through the plastic bag if an appropriate bag sample holder is used. Window opening time considerations are the same as for the in-situ measurement procedures determined by the manufacturers recommendation for the specific instrument being used.

The concentrations reported for the samples analyzed by the cup method are representative of the interval sampled, i.e., if the sampler collected the sample from the interval of 3-4 feet below ground surface, the reported concentration, assuming thorough homogenization, will be an average of the concentrations over that interval.

7. Study Design

XRF instruments will be used for two main purposes for this investigation. First the ex-situ method will be used to rapidly assess test pit and core conditions to selection sample intervals. Secondly, it will be used to screen soil or sediment samples to minimize the number of samples that are sent to a laboratory to provide the detailed site characterization data needed to define the base of waste. These uses are summarized in the following sections.

7.1. Reconnaissance

The XRF may be used to obtain in situ measurements at many locations in a short period of time to

determine if a portion of the Site warrants further attention with respect to characterization. Conversely, the reconnaissance results may form the basis for a "no further action" decision if numerous samples in an area show very low potential COC concentration well below the project screening criteria.

7.2. Screening Support for Definitive Level Site Characterization

The XRF will be used to supplement laboratory analyses to allow for the collection of larger numbers of samples to provide a more detailed characterization of a site. A high sample density grid or sampling pattern is created to provide adequate detail to meet the data quality objectives of the study or investigation. This sampling pattern may also involve the collection of significant numbers of subsurface soil samples to characterize any contamination present in the subsurface or aid in estimate of the total COC mass present.

All samples, collected according to the procedures found in the Applicable SAP or Work Plan, will be selected by or delivered to the XRF analyst on site. The analysis of these samples is conducted according to the method described in Section 6.2 of this procedure.

7.3. Confirmatory Sampling Strategies

Based on the limiting factors described in Section 3, a confirmatory analytical scheme can be developed which minimizes the numbers of samples that must undergo laboratory analyses, yet provides definitive level data, with a high degree of confidence. Using the moisture limiting factor, there is usually a high degree of confidence that samples screened at concentrations less than 70-80% of the site action level will not exceed the action level. Of the samples that screen at or within 20-30% of the action level, most or all, can be expected with a high degree of confidence to exceed the action level.

If a reconnaissance is conducted prior to the full-scale site investigation, in addition to the in situ analysis, it is advisable to collect and analyze a small subset of the screened locations to generate site-specific moisture limiting factors. This correlation factor can be used to develop a sampling scheme with more confidence and determine the combination of in situ, ex situ, and laboratory analysis to support construcion removal confirmation sampling.

7.4. Paired Ex-Situ and Laboratory Samples

All samples analyzed via the ex-situ analysis method during the field investigation will be submitted to the laboratory for analysis. Paired XRF sample results will be compared to the laboratory results to determine if a suitable correlation can be developed to support field removal confirmatory sampling strategy and procedures.

8. References

HGL Corporate Standard Operating Procedures.

United States Environmental Protection Agency (US EPA). Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Method 6200, Revision 0, February 2007.

Thermo Scientific™ Safety Training found at: XRF Radiation Safety Training | Thermo Fisher Scientific - US

9. Revision History

Description / History	Effective Date
Document Adapted and created from EPA Sample	January 20, 2023

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.

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

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:				

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HGL HydroGeoLogic, Inc Exceeding Expectations	CORPORATE TECHNICAL PROCEDURE	
	Approved for issue by:	
	Process Owner	Jodie Johnson
	Corporate Quality Director	Rojas, Theresa Digitally signed by Rojas, Theresa Date: 2021.06.22 12:56:36 -04'00'
Data Validation, U.S. EPA/DoD Stage 2A and Stage 2B		**Document No.: HGL SOP 412.501 (formerly 4.09)**
		Process Category: Services
		Revision No.: 3
		Effective Date: June 15, 2021
		Last Review Date: June 15, 2021
		Next Review Date: June 2023

1.0 PURPOSE AND APPLICABILITY

This standard operating procedure (SOP) provides information on the methodology and protocols required to review and validate analytical data generated from the laboratory analysis of environmental media. This SOP is intended to provide general guidance for the evaluation of the quality control (QC) elements associated with analytical data. Project-specific criteria for data validation are presented in each project's Quality Assurance Project Plan (QAPP), as are the project-specific QC acceptance criteria. Users of this SOP are authors of QAPPs, preparers of electronic QAPPs (eQAPPs) supporting automated data review (ADR), data validators, and data users.

2.0 SCOPE AND APPLICATION

The U.S. Environmental Protection Agency document *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (EPA, 2009) and Department of Defense *General Data Validation Guidelines* (DoD EDQW, 2019) define five stages of data validation: Stage 1, Stage 2A, Stage 2B, Stage 3, and Stage 4. Each stage increases the level of complexity and detail in the validation process and incorporates all relevant requirements of each preceding stage. Stage 2A and Stage 2B are the two most common stages of data validation performed in support of HydroGeoLogic, Inc.'s (HGL's) environmental projects. Stage 2A validation consists of a review of sample receipt, condition, and documentation (these Stage 1 elements correspond to "data verification"); holding times; and sample-specific and batch-specific QC elements. Stage 2B validation consists of all the elements of a Stage 2A validation, with additional review of instrument and analytical system QC elements. An individual laboratory's data report format may not include a summary form for a required QC element; such cases require the examination of raw data to provide information on the affected QC element.

The appropriate stage of data validation to be performed on analytical results is determined by HGL's project scope of work (SOW) and is presented in the project QAPP. Depending on the objectives for the project dataset, the actual validation performed on any given set of results is determined on a sample- and analytical method-specific basis. Generally, Stage 2B data validation is performed on analytical results that must be considered definitive and usable for supporting final decision-making and for performing quantitative risk assessments. Stage 2A data validation is performed to provide a general assessment of sampling and laboratory performance and does not

result in data that are usable for final decision-making or risk assessment. Stage 2A validation is typically performed on data generated for natural attenuation parameters and on data generated by long-term monitoring, operations and maintenance sampling, and compliance monitoring.

Stage 3 and Stage 4 data validation involve a greater level of effort and build on the Stage 1, 2A, and 2B data validation procedures. Stage 3 validation involves recalculating sample, calibration standard, and QC analysis results; comparing instrument response to minimum response requirements; and verifying that target analytes are quantified with an appropriate internal standard. Stage 4 validation includes verifying transcription of raw data to summary forms and examination of raw instrument results, including standard preparation logs, quantitation reports, chromatograms, and mass spectra for completeness, accuracy, and technical acceptability. Performing the review components associated with Stage 3 and Stage 4 validation relies almost entirely on the validator's professional judgment and experience, and these components are not covered by this SOP. No Stage 3 or Stage 4 data validation tasks can be assigned to HGL personnel without the approval of an HGL senior chemist.

Data generated for waste characterization and data associated with QC samples generally require no validation or only a Stage 1 data verification plus evaluation of holding times unless anomalous results are noted. Federal, state, or program requirements may include performing a higher stage of validation than is normally performed on any given sample or set of samples.

The QC elements that make up data validation Stages 2A and 2B, including the Stage 1 elements on which these stages build, are provided in Attachment A. The components of Stage 3 and Stage 4 data validation are also provided for reference.

3.0 GENERAL REQUIREMENTS

3.1 PRE-REVIEW ITEMS

Prior to beginning validation of laboratory data reports, the data validator must obtain the following items and information from the project manager (or designee):

1. The correct billing code for data validation tasks;
2. The most recent version of all relevant QAPPs (including any basewide QAPP and QAPP addenda);
3. The stage of data validation to be performed on the data (multiple stages are possible depending on end use of individual samples or the results from specific analytical methods);
4. The schedule and anticipated level of effort to complete validation tasks;

5. The identity of any field duplicate or triplicate samples and the associated parent samples; and
6. The identity of any field blanks (equipment, trip, ambient, and material blanks) and the correct association protocol for each blank.

3.2 LABORATORY DATA REPORTS

The data reports produced by each laboratory typically have substantial differences in presentation, bookmarking, structure, and formatting when compared to a data report produced by another laboratory, although some similarities will be present. Each project laboratory is required to provide data packages that support the stage of review that the associated data will undergo. Summary pages that provide all the validation stage-specific information listed in Attachment A are preferred, although in some cases summary pages may need to be supplemented with information only available on instrument printouts or raw data due to limitations in laboratory report-generation software.

Before data validation, the validator should examine the laboratory data reports to ensure that all required information necessary to perform the required stage of data validation is available and presented in a format that supports the validation effort. Familiarity with the laboratory's reporting conventions improves the efficiency of the data validation process as well as the quality of the validation, as the validator will be better able to identify QC discrepancies in the reported data and judge the effect on the associated sample results.

Control limits for surrogate recoveries, laboratory control sample (LCS) and LCS duplicate (LCSD) recoveries, matrix spike (MS) and matrix spike duplicate (MSD) recoveries, LCS/LCSD precision, MS/MSD precision, and duplicate precision are usually presented in the project QAPP. If the control limits are specified in the QAPP, the validator should verify that the laboratory reports incorporate the required control limits. Failure to verify that the laboratory-reported control limits are those specified by the QAPP can cause QC discrepancies to be misidentified as conforming data points and conforming data points to be misidentified as discrepancies. In both cases, the data are not evaluated against the requirements for precision and accuracy specified in the QAPP. This scenario can result in misqualified data and in additional validation efforts to correct the laboratory-applied qualifiers. It can also result in the laboratory's failing to identify a QC discrepancy and subsequently failing to perform required corrective action. Verifying that the correct control limits are being presented prior to beginning the validation effort is the best way to ensure that the reported results meet the precision and accuracy requirements established for the project as presented in the QAPP. If discrepancies are noted, the laboratory project manager should be notified that the data reporting pages do not present the correct information and that the laboratory should ensure that all future deliverables conform to the requirements of the QAPP.

In some cases, the laboratory's internally derived control limits may be acceptable, either for entire analytical suites or individual analytes for which program limits have not been established. Where

a QAPP indicates that a set of control limits are laboratory-specific, those limits can change over time as laboratories evaluate and update their control limits. Should a laboratory data package report laboratory control limits that differ from those in the QAPP, the validator should consider the current control limits to supersede the QAPP limits and document this decision in the data validation report.

If required QC review elements or individual pages are missing from a laboratory data report, and the missing information is a result of an error in report compilation (such as a missing or illegible page), the validator should contact the laboratory project manager directly and request that the missing information be provided. If the missing information is the result of a laboratory report generation convention (that is, the lack of a required data QC element is due to report design, not to an error in report compilation), the data validator should contact the HGL project chemist. The HGL project chemist must coordinate with the laboratory project manager to ensure that any required information is provided to the data validators in alternative formats so that all QAPP-required QC elements can be reviewed.

3.3 DATA VALIDATION REPORTS

Data validation is documented in a data validation report, and each report contains a subsection for each analytical method reported in a single sample delivery group (SDG).

In cases where individual project requirements conflict with the requirements of this SOP, the project requirements take precedence and should be used throughout the data validation and evaluation process; however, the data validator or HGL senior chemist may deviate from the stated project requirements based on professional judgment. Any deviations from specified requirements must be technically appropriate, and they must be justified in the corresponding data validation report and HGL validation report review memo. Deviations in the assessment of the project dataset must also be documented in any data quality or usability evaluation associated with project report deliverables.

Example data report formats are presented in Attachment B. Note that the qualification conventions used in the example reports are based on the requirements of a specific project. The qualifiers assigned during the validation process should reflect the project's conventions.

3.4 PEER REVIEW

All data validation reports generated by HGL personnel are subject to a secondary review by either a peer or senior chemist assigned by the Chemistry Group leader. The peer reviewer evaluates the data validation report against the contents of the laboratory data report to ensure that the following applies:

1. The data validator has correctly applied the project requirements as presented in the QAPP to evaluate and qualify the reported sample results.
2. The data validator has not overlooked any QC discrepancies present in the data package.
3. The validator has correctly associated any QC discrepancies with the correct analytes and analyses.
4. The assigned data qualifiers are complete and correct.
5. The data validator has not made "boilerplate" errors (that is, the inclusion of extraneous and incorrect information in the data report as a result of using another report as a template without removing or modifying material that does not apply).

A validation report that has not been reviewed cannot be considered final.

3.5 SUBCONTRACTED DATA VALIDATION

The goal of subcontracted data validation is to generate a validated project dataset that is qualified in accordance with QAPP requirements and ready for HGL to upload into the project database. Subcontracted data validation is performed in accordance with the individual firm's internal procedures and policies; however, the overall procedure must include pre-review, validation by qualified personnel, and peer or senior review of all data validation reports (in accordance with Section 3.4) before delivery to HGL. All validation must be performed in accordance with the project QAPP and the SOW provided by HGL. In addition to a validation report, the subcontracted validator may be responsible for providing qualified data electronically in a format that allows upload into HGL's project database (see Section 6.0), usually in the form of an Excel file. The validation firm is responsible, in accordance with the project-specific data validation SOW, for any data entry, data entry QC, and removal of any residual laboratory-applied flags prior to delivery to HGL.

HGL reviews data validation reports provided by third-party contractors in accordance with the procedures presented in Attachment F. The initial data validation reports provided by the contractor must be reviewed in depth by an HGL senior chemist as soon as possible to provide the data validator with timely feedback to guide ongoing validation efforts. The primary purpose of the HGL senior chemist review is to verify that the data validators understand the QAPP and project data quality requirements and are applying these requirements correctly when reviewing each data package. Data validation involves a large amount of professional judgment, and there are multiple conventions that are technically valid. Therefore, a secondary purpose of the HGL senior chemist's review is to ensure that the conventions HGL selected are being used by the contractor to maintain consistency in evaluation and application of qualifiers from SDG to SDG within a project. When it has been established that HGL's expectations are being met, subsequent data validation reviews can be streamlined to verify that the identified QC issues discussed in each validation report led to correct qualification of the associated sample results. It should be kept in

mind, however, that many data validation firms have a pool of staff validators and there can be variability in the quality and completeness of individual data validation reports submitted from a third-party contractor.

4.0 PERSONNEL

Data validation and review must be conducted by appropriately qualified and trained personnel.

4.1 ROLES, RESPONSIBILITIES, AND QUALIFICATIONS

4.1.1 HGL Project Staff

HGL project staff are assigned in accordance with contract requirements and HGL's project management procedures. The following personnel have a wide range of responsibilities associated with their project titles; however, only the responsibilities applicable to the data validation process are discussed. It is possible for the HGL chemistry staff identified below to operate in multiple functions. For example, an HGL senior chemist can act as a project chemist for an individual project and perform the functions of both project chemist and senior chemist for that project.

HGL Project Manager – Provides the data validation team with the information listed in Section 3.1, either directly or through a designee (such as a task manager). Ensures that all required project personnel, including sample collection, laboratory, and data validation subcontractors, are provided with the current project QAPP as well as any QAPP revisions in a timely fashion.

HGL Project Chemist – Provides guidance on analytical method requirements for sampling, preservation, and holding time requirements to field sampling teams. Resolves issues not covered by the QAPP or other guidance documents. Ensures that laboratory performance is in accordance with HGL's project technical requirements. For projects with subcontracted data validation, reviews data validation reports to verify that the data validation contractor is performing in accordance with the contract SOW and the QAPP (see Appendix F). After ensuring that the laboratory and validation contractors, if applicable, have performed in accordance with HGL's project technical requirements, provides approval of invoices for payment.

HGL Senior Chemist – For some projects, this role may be identified as "program chemist" based on client organizational designating conventions. Assists senior program chemist in implementing the data validation program and provides technical input to support the program. Assists the project chemist in resolving issues not covered by the QAPP or other guidance documents. Assists the project chemist in ensuring that laboratory and validation contractor, if applicable, is performing in accordance with HGL's project technical requirements. Assists project manager in communicating data quality issues to the client and addressing client or stakeholder concerns. Assists senior program chemist in identifying and resolving deficiencies in project laboratory or

subcontracted validator performance. Trains junior project staff in data validation and monitors performance.

HGL Senior Program Chemist – Provides overall direction to HGL's data validation program. Works with senior HGL management to resolve deficiencies in project laboratory or subcontracted validator performance.

4.1.2 Data Validation Staff

Data validation staff includes data validators and peer reviewers who are assigned on an as-needed basis. Data validation staff can consist of qualified HGL personnel including chemists, geologists, environmental scientists, or other technical staff who have been trained in data validation by an HGL senior chemist or are judged by an HGL senior chemist to have sufficient experience in data validation. The qualifications and roles of data validation staff are described below.

HGL Data Validator – Must have at least a bachelor's degree in chemistry or other scientific discipline. The HGL data validator performs data validation, communicates with the laboratory to resolve issues, and writes the data validation reports. Data validation reports generated by an HGL validator with less than 1 year of experience must be reviewed by an HGL senior chemist.

HGL Peer Reviewer – Must have at least a bachelor's degree in chemistry or other scientific discipline and at least 2 years of data validation experience. Peer reviewers perform a complete review of the findings of each data validation report against the associated laboratory data deliverable and determine if the validator has (1) addressed all QC issues affecting project data in accordance with the requirements of the project QAPP, (2) assigned the correct qualifiers to the reported data, (3) complied with project validation conventions, and (4) presented a clear description of the data quality issues affecting the reported data. Peer reviewers with less than 1 year of peer review experience are subject to approval by an HGL senior chemist before assignment.

Depending on the size of the project and staffing requirements, multiple data validators and peer reviewers may be assigned to a project; a data validator assigned to one laboratory deliverable may be a peer reviewer for another laboratory deliverable validation report. It is recommended, but not required, that each project's project chemist be one of the HGL personnel assigned to perform data validation and peer review tasks for that project.

4.2 TRAINING REQUIREMENTS

HGL data validation staff must be trained directly by an HGL senior chemist. This training preferably takes place in person to allow for greater efficiency in instruction, evaluation, and feedback. Training includes validation of laboratory data reports followed by feedback and revision.

5.0 PROCEDURES

Data will be reviewed and qualified in accordance with the project QAPP and validator judgment. The qualification guidelines presented in each QAPP are based on the project data quality objectives (DQOs) and must specify the stage of data validation required to meet those DQOs. Stage 2A and Stage 2B are the most common stages of validation specified by project QAPPs. These stages of data validation usually include only the examination of the information presented on laboratory-generated summary forms. This approach is generally sufficient to determine that the laboratory is following analytical method, programmatic, and project-specific requirements.

On occasion, a review of specific raw data elements is necessary to supplement the information presented on the summary reporting forms. Stage 4 data validation, which includes a detailed review of instrument raw data and laboratory records and provides the most rigorous evaluation of data quality, is occasionally specified by a project contract. Where required, Stage 3 or Stage 4 validation is commonly performed on a specified subset of project data, such as 10 percent. Unless otherwise specified in the project QAPP, the checks and recalculations associated with Stage 3 and Stage 4 validation should be performed at the frequencies presented in Section 4.7 of the *General Data Validation Guidelines* (DoD EDQW, 2019b). Stage 4 validation is highly dependent on the professional expertise and experience of the validator and is specific to individual analytical methods and instrumentation. Consequently, the procedures required to complete this stage of data validation are not included in this SOP.

The specific procedures required to perform data validation vary greatly among data reports. The sources of variation include method QC requirements, client and regulatory requirements, laboratory-specific reporting conventions, and sample matrix. General guidelines for the evaluation of Stage 2A QC elements and method-specific Stage 2B QC elements are presented in Attachment C.

Stage 2A validation can be supported by ADR, such as the web-based ADR functionalities provided by Environmental Synectics, Inc. (Synectics) and the FUDSChem ADR program developed by the Department of Defense, as part of its scope of data management services. A description of the ADR process and its integration into the data validation process is presented in Attachment D. When ADR is incorporated into a project that requires Stage 2B validation, the data are validated to Stage 2A by ADR followed by manual verification of the ADR results and additional manual validation to complete the Stage 2B validation.

6.0 DATABASE QUALIFICATION

After the method-specific data validation reports for an SDG have been generated in accordance with Section 3.3 and reviewed in accordance with Section 3.4, the data qualifiers assigned by the validator are applied to electronic database output files. The procedures for data entry, review, and

upload are presented in HGL SOP 300.07 (formerly 303.01): *Environmental Data Management*.[1] During what is referred to as the "100 percent QC stage" of this process, all residual laboratory-generated information flags not retained as the final qualification must be removed from each result. The only laboratory-generated flags that are retained are those that have been accepted as the final qualifier by the data validator. When data validation has been subcontracted, the contractor is responsible for removing residual laboratory flags before delivering the qualified data files to HGL.

In some cases, projects require the application of a reason code as well as a qualifier to validated results. In such cases, the HGL project chemist develops a list of reason codes, and the HGL database manager uploads these reason codes to the database. Common reason codes are included in Attachment E. If HGL has not mandated a specific reason code protocol for a project, data validation subcontractors may use their internally developed reason codes.

7.0 SENIOR DATA RE-EVALUATION

When severe QC discrepancies are encountered, it may become necessary to reject associated data points. Rejected data points cause data gaps in the resulting dataset and can prevent that dataset from being used to achieve project DQOs; however, not all data gaps attributable to rejected results have an equal impact. Of special concern are (1) rejected results that affect a contaminant that could be present at the subject site or (2) rejection of a large number of analytes in individual samples because of sample-specific or batch-specific QC issues.

If results are rejected in the initial data validation, the issue must be evaluated for referral to an HGL senior chemist for supplemental senior review. This review includes discussions with laboratory quality assurance personnel, examination of raw data, and evaluation of the end use of the affected data. The review evaluates the feasibility of applying a less severe qualifier. In some cases, a less severe qualifier will not be technically justified, and an R qualifier will be applied to the affected results. In others, it may be determined that the affected results can be used to support decision-making, and the application of a less severe qualifier is technically appropriate. In all cases where HGL determines that rejection is not required, in contradiction to the requirements of the QAPP, an HGL senior chemist documents this judgment. This documentation must be made available to the client for review and approval, either in the form of technical memoranda or discussion in the associated project report (see Section 3.3).

[1] When updated, SOP 300.07 will be renumbered as HGL SOP 411.501.

8.0 REFERENCES

U.S. Department of Defense (DoD) Environmental Data Quality Workgroup (EDQW) and the U.S. Department of Energy (DOE) Consolidated Audit Program (DOECAP) Data Quality Workgroup (DOE-DQW), 2019. *General Data Validation Guidelines*. November.

U.S. Environmental Protection Agency (EPA), 2009. *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use.* OSWER 9200.1-85; EPA-540-R-08-005. January.

9.0 REVISION HISTORY

Revision Number	Revision Date	Reasons for Revision
0	November 2012	Initial Release
1	April 2017	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
2	February 2018	Updated to incorporate lessons learned on the process and to reflect changes in SOP formatting.
3	June 15, 2021	Updated to incorporate lessons learned on the process and changes in DoD programmatic requirements and to reflect changes in SOP formatting, which included changing the SOP number from 4.09 to HGL SOP 412.501.

ATTACHMENTS

Attachment A Components of the Stages of Data Review
Attachment B Example Data Validation Reports
Attachment C General Validation Guidelines
Attachment D Automated Data Review
Attachment E HGL Data Qualification Reason Codes
Attachment F Review of Subcontracted Data Validation Reports

ATTACHMENT A
Components of the Stages of Data Review

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ATTACHMENT A
Components of the Stages of Data Review

All Analytical Fractions	Stage 1	Stage 2A	Stage 2B	Stage 3	Stage 4
Case narrative	X	X	X	X	X
Chain of custody	X	X	X	X	X
Sample receipt and log-in forms	X	X	X	X	X
Sample identification (ID) cross reference (HydroGeoLogic, Inc. sample ID to laboratory sample ID)	X	X	X	X	X
Sample discrepancy reports, corrective action, and client communications	X	X	X	X	X
Holding times (preparation and analysis)		X	X	X	X
LCS/LCSD(1) recoveries and precision		X	X	X	X
MS/MSD(2) recoveries and precision		X	X	X	X
Method blanks		X	X	X	X
Field blanks (trip, ambient, equipment, and material blanks)		X	X	X	X
Field duplicate precision		X	X	X	X
GC/MS, LC/MS, and LC/MS/MS Organic Analytical Fractions	**Stage 1**	**Stage 2A**	**Stage 2B**	**Stage 3**	**Stage 4**
Surrogate recoveries		X	X	X	X
Instrument tuning			X	X	X
Instrument initial calibration (including minimum relative response factors [RRFs])			X	X	X
Second source calibration verification			X	X	X
Instrument continuing calibration verification (including minimum RRFs)			X	X	X
Internal standards or labeled standards			X	X	X
Calculations				X	X
Chromatograms					X
Quantitation reports					X
Mass spectra					X
Transcription					X
GC and HPLC Organic Fractions(3)	**Stage 1**	**Stage 2A**	**Stage 2B**	**Stage 3**	**Stage 4**
Surrogate recoveries		X	X	X	X
Instrument initial calibration			X	X	X
Second source calibration verification			X	X	X
Instrument continuing calibration verification			X	X	X
Degradation summary (organochlorine pesticides only)			X	X	X
Retention times			X	X	X
Confirmation			X	X	X
Calculations				X	X
Chromatograms					X
Quantitation reports					X
Transcription					X

ATTACHMENT A (continued)
Components of the Stages of Data Review

Metals Fractions	Stage 1	Stage 2A	Stage 2B	Stage 3	Stage 4
Laboratory duplicate(2) precision		X	X	X	X
Serial dilution results		X	X	X	X
Post-digestion spike recoveries		X	X	X	X
Initial and continuing calibration blanks			X	X	X
Instrument tuning (ICP-MS methods only)			X	X	X
Internal standards (ICP-MS methods only)			X	X	X
Initial multipoint calibration(4)			X	X	X
Low-level calibration verification			X	X	X
High-level calibration verification			X	X	X
Initial and continuing calibration verification			X	X	X
Interference check sample results			X	X	X
Recovery test recoveries (GFAA methods only)			X	X	X
Method of standard addition results			X	X	X
Calculations				X	X
Interelement correction factors					X
Instrument raw data					X
General Chemistry Fractions	**Stage 1**	**Stage 2A**	**Stage 2B**	**Stage 3**	**Stage 4**
Laboratory duplicate(2) precision		X	X	X	X
Method-specific QC checks(5)		X	X	X	X
Initial and continuing calibration blanks			X	X	X
Initial multipoint calibration			X	X	X
Initial and continuing calibration verification			X	X	X
Method-specific instrument QC			X	X	X
Calculations				X	X
Instrument raw data					X

(1) LCSDs are not a requirement for any method or project; however, they are often provided by the laboratory. They are reviewed when available.
(2) The analytical methods allow for metals and general chemistry precision to be evaluated either using MS/MSDs or laboratory duplicates at the laboratory's discretion. Often laboratories provide both. The data validator reviews all available QC data provided by the laboratory.
(3) These methods use a second column or detector to confirm detected results. QC elements for both columns/detectors should be reviewed during the validation process.
(4) Initial multipoint calibration is optional for ICP methods; if performed, the validator reviews the associated results.
(5) An example of method-specific QC checks is distillation checks for cyanide analysis.

Notes:
GC/MS = gas chromatography/mass spectrometry
GFAA = graphite furnace atomic absorption
HPLC = high-performance liquid chromatography
ICP = inductively coupled plasma
ICP-MS = inductively coupled plasma-mass spectrometry
LC/MS = liquid chromatography/mass spectrometry
LC/MS/MS = liquid chromatography/tandem mass spectrometry
LCS = laboratory control sample
LCSD = laboratory control sample duplicate
MS = matrix spike
MSD = matrix spike duplicate
QC = quality control

ATTACHMENT B
Example Data Validation Report

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B.1
Example Data Validation Report

USEPA Stage 2B Validation Report

Section 1 – General Information

Site: Hero Air Force Base	SDG #: ABC-1234
Laboratory: TestGood Labs	Date: 08/31/2020
HydroGeoLogic, Inc. Reviewer: Justin Hersh HGL Senior Reviewer: Denise Rivers (09/09/20)	Project: AF0055.001.02.03

Client Sample ID	Laboratory Sample ID	Laboratory Receipt Date	Sampling Date and Time	Matrix
HAFB-MW01	ABC-1234-01	08/01/2020	07/31/20 10:10	Water
HAFB-DUP01	ABC-1234-02	08/01/2020	07/31/20 10:10	Water
TB-08122020	ABC-1234-03	08/01/2020	07/31/20 08:00	Water QC
HAFB-MW02	ABC-1234-04	08/01/2020	07/31/20 12:05	Water
HAFB-EB01	ABC-1234-05	08/01/2020	07/31/20 14:00	Water QC

1a. Narrative and Completeness Review – The case narrative and data package were checked for completeness. It was noted that the laboratory reported its internally derived control limits instead of the QAPP control limits for PCBs and TRPH. The QAPP control limits were used to evaluate the data. No other discrepancies were noted.

Qualification: None required.

1b. Sample Delivery and Condition – All samples arrived intact at the laboratory in acceptable condition and temperature and were properly preserved, as applicable. Proper custody was documented, with one exception. Field duplicate HAFB-DUP01 was incorrectly associated with sample HAFB-MW02 while in the field; the correct parent sample is HAFB-MW01, which will be amended in all field paperwork and the data validation report for this SDG.

Qualification: None required.

1c. Equipment Blanks – One equipment blank, identified as HAFB-EB01, was associated with all samples analyzed for PCBs in this SDG and was free from contamination.

Qualification: None required.

1d. Field Duplicate – Sample HAFB-DUP01 is a field duplicate of sample HAFB-MW01. Detections for the duplicate pair and the calculated RPD or absolute difference, as applicable, are listed in the table below.

ANALYTE	HAFB-MW01		HAFB-DUP01		RPD or |Diff|
	Conc.	LOQ	Conc.	LOQ	
VOCs					
Isopropylbenzene	11	1.0	13	1.0	16.7%
Total Metals					
Antimony	0.5	1.0	0.75	1.0	0.25

ANALYTE	HAFB-MW01		HAFB-DUP01		RPD or |Diff|
	Conc.	LOQ	Conc.	LOQ	
Pesticides					
Dieldrin	23	1.0	24	1.0	4.3%
Wet Chemistry					
Sulfate	5.87	0.5	5.93	0.5	1.0%

Qualification: None required.

Section 2 – Volatile Organic Compounds (SW-846 Method 8260B)

Client Sample ID	Laboratory Sample ID	Analysis Batch
HAFB-MW01	ABC-1234-01	690453
HAFB-DUP01	ABC-1234-02	690453
TB-08122020	ABC-1234-03	690193

2a. Holding Times – All samples were analyzed within the 14-day holding time required by the QAPP for preserved aqueous samples.

Qualification: None required.

2b. Initial Calibration – One initial calibration (ICAL) was associated with all samples in this SDG. The ICAL performed for instrument MSV11 on 08/14/20 (associated with batches 690193 and 690453) had acceptable mean RRFs for all SPCCs and acceptable %RSDs for all CCCs. All target analytes had acceptable RRFs and %RSDs. The second source ICV associated with this initial calibration met the control criteria established by the QAPP for all target analytes.

Qualification: None required.

2c. Continuing Calibration – Two continuing calibration verification (CCV) and two closing CCV standards were associated with the samples in this SDG. The CCV and closing CCV standards analyzed on 08/17/20 for batch 690193 had acceptable CCRFs for all SPCCs and acceptable %Ds for all CCCs. The %Ds for all target analytes met the control limits established by the QAPP.

The CCV and closing CCV standards analyzed on 08/20/20 for batch 690453 had acceptable CCRFs for all SPCCs and acceptable %Ds for all CCCs. The %Ds for all target analytes met the control limits established by the QAPP.

Qualification: None required.

2d. GC/MS Tuning – The sample analytical sequences were all performed within 12 hours of an acceptable GC/MS tune.

Qualification: None required.

2e. Internal Standards – All internal standards met the peak area and retention time criteria.

Qualification: None required.

2f. Surrogates – All surrogate recoveries were within the control limits specified in the QAPP for aqueous samples.

Qualification: None required.

2g. Laboratory Control Sample – Two LCS/LCSD pairs were associated with the samples in this SDG. Both LCS/LCSDs for batches 690193 and 690453 met all %R and RPD control limits established by the QAPP.

Qualification: None required.

2h. MS/MSD – MS/MSD analyses were performed for all target analytes on sample HAFB-MW01 from this SDG. The %R and RPD results were within the QAPP control limits with the exception of 1 high recovery (135%) for the MS. The isopropylbenzene result for parent sample HAFB-MW01 was a detection above the LOQ and should be qualified J.

***Qualification:* The isopropylbenzene result for sample HAFB-MW01 was qualified J.**

2i. Method Blank – Two method blanks were associated with the samples in this SDG. The blanks analyzed on 08/17/20 and 08/20/20 for batches 690193 and 690453, respectively, were free from contamination.

Qualification: None required.

2j. Trip Blanks – One trip blank, identified as TB-08122020, was submitted with the samples in this SDG and was free from contamination.

Qualification: None required.

Section 3 – Total Metals (ICP-MS; SW-846 Method 6020B)

Client Sample ID	Laboratory Sample ID	Preparation Batch	Analysis Batch(1)
HAFB-MW01	ABC-1234-01	695011	695628
HAFB-DUP01	ABC-1234-02	695010	695628
HAFB-MW02	ABC-1234-04	695011	695628

(1) Samples analyzed for total antimony, iron, and lead only.

3a. Holding Times – All samples were analyzed within the 6-month holding time required by the QAPP for preserved aqueous samples.

Qualification: None required.

3b. Calibration – All %R results for the ICV, bracketing CCV, and LDR standards, met the 90-110% recovery criterion for both target metals. The %R results for the low-level CCV standards met the 80-120% QAPP criteria.

Qualification: None required.

3c. Calibration Blanks – The ICBs and CCBs associated with the sample analyses were free from contamination, with one exception. The CCB analyzed on 11/06/20 at 1347 for analysis batch

695628 was contaminated with total antimony (0.73 µg/L), yielding an action level of 3.65 µg/L. The dissolved antimony result for sample HAFB-MW02 was a detection below the action level and should be qualified U.

Qualification: **The total antimony result for sample HAFB-MW02 was qualified U.**

3d. Interference Check Samples – Two ICSA and ICSAB sets were analyzed with the samples in this SDG. All non-spiked target metals results were less than the LOD in the ICSAs. All spiked metals met the 80-120% QAPP control criteria for the ICSAB standards.

Qualification: None Required.

3e. ICP Serial Dilutions/Post Digestion Spike Samples – A serial dilution and/or post digestion spike (PDS) were performed for total metals antimony, iron, and lead on sample HAFB-MW01 from this SDG. All PDS %R results were within the QAPP control limits. All metals were less than 50x the respective LOD, and the serial dilution %D results were not calculated or applicable.

Qualification: None Required.

3f. Laboratory Control Sample – Two LCS standards were associated with the samples in this SDG. The LCS standards for preparation batches 695011 and 695010 met all %R control limits established by the QAPP.

Qualification: None required.

3g. MS/MSD – MS/MSD analyses were performed for total metals antimony, iron, and lead on sample HAFB-MW01 from this SDG. All %R and RPD results were within the QAPP control criteria.

Qualification: none required.

3h. Laboratory Duplicate Sample – A laboratory duplicate analysis was not performed on a sample from this SDG.

Qualification: None required.

3i. Method Blank – Two method blanks were associated with the samples in this SDG. The method blanks for preparation batches 695011 and 695010 were free from contamination.

Qualification: None required.

Section 4 – Polychlorinated Biphenyls (SW-846 Method 8082A)

Client Sample ID	Laboratory Sample ID	Preparation Batch	Analysis Batch
HAFB-MW01	ABC-1234-01	232943	232958
HAFB-DUP01	ABC-1234-02	232943	232958
HAFB-MW02	ABC-1234-04	232943	232958

4a. Holding Times – All samples were extracted and analyzed within the 1 year holding time specified in the QAPP for aqueous samples.

Qualification: None required.

4b. Initial Calibration – All target analytes in the primary and secondary column ICALs had %RSDs less than the method maximum of 20% or r^2 values greater than 0.99. All second source ICV %Ds were less than the method maximum of 20%.

Qualification: None required.

4c. Continuing Calibration – In the instance of PCBs, single peaks are not qualified if the average %D was within the QAPP control limit. All %Ds for CCVs bracketing the samples were less than the 20% method maximum stated in the QAPP.

Qualification: None required.

4d. Internal Standards – All internal standards met the peak area and retention time criteria.

Qualification: None required.

4e. Surrogates – All surrogate recoveries were within the QAPP acceptance limits.

Qualification: None required.

4f. Laboratory Control Sample – One LCS was associated with all samples in this SDG. The LCS for preparation batch 232943 met the %R control limits established in the QAPP.

Qualification: None required.

4g. MS/MSD – Matrix spike/matrix spike duplicate analyses were not requested or performed on a sample from this SDG.

Qualification: None required.

4h. Method Blank – One method blank was associated with all samples in this SDG. The method blank prepared on 01/12/21 for batch 232943 was free from contamination.

Qualification: None required.

4i. Detection Confirmation – All results for the samples in this SDG were non-detect.

Qualification: None required.

Section 5 – Petroleum Range Organics (TRPH; Method FL-PRO)

Client Sample ID	Laboratory Sample ID	Preparation Batch	Analysis Batch
HAFB-MW01	ABC-1234-01	231795	231789
HAFB-DUP01	ABC-1234-02	231795	231789
HAFB-MW02	ABC-1234-04	231795	231789

5a. Holding Times – All samples were extracted within the 7-day holding period required for aqueous samples and analyzed within 40-days of preparation.

Qualification: None required.

5b. Initial Calibration – One initial calibration was associated with the samples in this SDG. The target analyte in the ICAL had a %RSD less than the method maximum of 20% or an r^2 value greater than 0.99. No second source ICV was presented.

Qualification: None required.

5c. Continuing Calibration – Two continuing calibration verification (CCV) standards were associated with the samples in this SDG. All CCV %Ds were less than the 25% method maximum stated in the QAPP.

Qualification: None required.

5d. Surrogates – All surrogate recoveries were within the QAPP acceptance limits.

Qualification: None required.

5e. Retention Times – All retention times met the QAPP criteria.

Qualification: None required.

5f. Laboratory Control Sample – One LCS was associated with the samples in this SDG. The LCS for preparation batch 231795 met the %R control limit established in the QAPP.

Qualification: None required.

5g. MS/MSD – Matrix spike/matrix spike duplicate analyses were performed for TRPH on sample HAFB-MW01 from this SDG. All %R and RPD results met the QAPP control criteria.

Qualification: None required.

5h. Method Blank – One method blank was associated with the samples in this SDG. The method blank prepared on 12/11/20 for batch 231795 was free from contamination.

Qualification: None required.

Section 6 – Polynuclear Aromatic Hydrocarbons (SW-846 Method 8270D-SIM)

Client Sample ID	Laboratory Sample ID	Preparation Batch	Analysis Batch
HAFB-MW01	ABC-1234-01	340410	340438
HAFB-DUP01	ABC-1234-02	340410	340438
HAFB-MW02	ABC-1234-04	340410	340438

6a. Holding Times – All samples were prepared within the 7-day holding time required by the QAPP for aqueous samples and analyzed within 40-days of extraction.

Qualification: None required.

6b. Surrogates – The surrogate recoveries were within the control limits specified in the QAPP for

aqueous samples, with two exceptions. The recoveries for surrogate 2-methylnaphthalene-d10 were below the lower QAPP criteria for samples HAFB-MW01 (38%) and HAFB-DUP01 (24%). All results for both samples were non-detections and should be qualified UJ.

Qualification: **All results for samples HAFB-MW01 and HAFB-DUP01 were qualified UJ.**

6c. Initial Calibration – One initial calibration was associated with the samples in this SDG. For the initial calibration run on 05/13/20, all target analytes had %RSDs less than the method maximum of 20% or r^2 values greater than 0.99. All second source ICV %Ds were within the 80%-120% criteria.

Qualification: None required.

6d. Continuing Calibration – One continuing calibration verification (CCV) and one closing CCV standards were associated with the samples in this SDG. The CCV standards that were associated with the samples in this SDG had %Ds within the QAPP acceptance limits.

Qualification: None required.

6e. GC/MS Tuning – The sample analytical sequences were all performed within 12 hours of an acceptable GC/MS tune.

Qualification: None required.

6f. Internal Standards – All internal standards met the peak area and retention time criteria.

Qualification: None required.

6g. Laboratory Control Sample – One LCS/LCSD pair was associated with the samples in this SDG. The LCS/LCSD for preparation batch 340410 met all %R and RPD control limits established in the QAPP.

Qualification: None required.

6h. MS/MSD – Matrix spike and matrix spike duplicate analyses were performed for all target PAHs on sample HAFB-MW01 from this SDG. The table below lists all MS/MSD recoveries and RPDs that were outside of the QAPP control limits and the appropriate qualification, as necessary.

Parent Sample	Prep Batch	Compound	%R / %R / RPD	Qualifier	Affected Samples
HAFB-MW01	340410	1-Methylnaphthalene	34% / OK / 52%	UJ	1
		2-Methylnaphthalene	29% / OK / 49%	UJ	1
		Naphthalene	18% / 37% / 69%	UJ	1

Qualification: **Please refer to the table above.**

6i. Method Blank – One method blank was associated with the samples in this SDG. The blank prepared on 10/08/20 for batch 340410 was free from contamination.

Qualification: None required.

Section 7 – Organochlorine Pesticides (SW-846 Method 8081B)

Client Sample ID	Laboratory Sample ID	Prep Batch	Analysis Batch
HAFB-MW01	ABC-1234-01	592177	592626
HAFB-DUP01	ABC-1234-02	592177	592664
HAFB-MW02	ABC-1234-04	592177	592626
HAFB-EB01	ABC-1234-05	592177	592626

7a. Holding Times – All samples were prepared within the required 7-day holding period for aqueous samples and analyzed within 40-days of extraction.

Qualification: None required.

7b. Surrogates – All surrogate recoveries were within the QAPP acceptance limits.

Qualification: None required.

7c. Second-Column Confirmation – Pesticide detections require secondary column confirmation. The RPD calculated from corresponding primary and secondary column heptachlor epoxide results for sample HAFB-MW02 was less than the 40% QAPP criteria.

Qualification: None required.

7d. Initial Calibration – One initial calibration was associated with the samples in this SDG. The target analyte had a %RSD less than the method maximum of 20% or an r^2 value greater than 0.99 for both standards. The second source ICV %Ds were less than the method maximum of 20%.

Qualification: None required.

7e. Continuing Calibration – Two continuing calibration verification (CCV) standards were associated with the samples in this SDG. All CCV %Ds for the target analyte were less than the 20% method maximum stated in the QAPP.

Qualification: None required.

7f. Breakdown Check – The degradation of endrin and 4,4'-DDT was $\leq$15% as specified in the QAPP.

Qualification: None required.

7g. Retention Time Window – All target analytes met the retention time criteria established in the QAPP.

Qualification: None required.

7h. Laboratory Control Sample – One LCS/LCSD pair was associated with all samples in this SDG. The LCS/LCSD for preparation batch 592177 met all %R and RPD control limits established in the QAPP.

Qualification: None required.

7i. MS/MSD – Matrix spike and matrix spike duplicate analyses were performed for all target analytes on sample HAFB-MW01 from this SDG. All %R and RPD results met the criteria established by the QAPP.

Qualification: None required.

7j. Method Blank – One method blank was associated with the samples in this SDG. The method blank prepared on 08/08/16 for batch 592177 was free from contamination.

Qualification: None required.

Section 8 – Sulfate (SW-846 Method 9056A)

Client Sample ID	Laboratory Sample ID	Analysis Batch
HAFB-MW01	ABC-1234-01	654604
HAFB-DUP01	ABC-1234-02	654604
HAFB-MW02	ABC-1234-04	654604

8a. Holding Times – All samples were analyzed within the 28-day holding time required by the QAPP for aqueous samples.

Qualification: None required.

8b. Calibrations – The initial calibration performed on 07/11/20 met the criteria established by the QAPP. All %R results for the bracketing CCV standards met the 90-110% recovery criterion for sulfate.

Qualification: None required.

8c. Calibration Blanks – All CCBs associated with the sample analyses were free from contamination.

Qualification: None required.

8d. Method Blanks – One method blank was associated with all samples in this SDG. The method blank analyzed on 08/23/20 for batch 654604 was free from contamination.

Qualification: None Required.

8e. Laboratory Control Sample – One LCS sample was associated with all samples in this SDG. The LCS result for batch 654604 met the %R requirements established by the QAPP.

Qualification: None required.

8f. MS/MSD – Matrix spike and matrix spike duplicate analyses were performed for sulfate on sample HAFB-MW01 from this SDG. All %R and RPD results met the QAPP criteria.

Qualification: None required.

8g. Laboratory Duplicate Sample – A laboratory duplicate analysis was not performed on a sample from this SDG.

Qualification: none required.

Section 9 – Compound Quantitation

Analyte non-detections are reported as the LOD and qualified U. These U qualifiers are retained unless superseded by a more severe qualifier. Analytes detected between the LOQ and DL are reported as either J- or I-qualified results by the laboratory. The I-qualifiers are changed to J flags per the QAPP requirements and these J qualifiers are retained unless superseded by a more severe qualifier. The non-standard M-qualifiers applied by the laboratory to indicate the manual integration of results should be removed from all samples.

Qualification: **All non-standard I-qualifiers applied by the laboratory were changed to J flags. The non-standard M-qualifiers applied by the laboratory were removed from all samples.**

Qualification Summary Table (all concentrations in mg/L or μg/L depending on the method):

Sample	Analyte	Lab Value	Lab Qualifier	HGL Value	HGL Qualifier
HAFB-MW01	Isopropylbenzene	21.4	--	21.4	J
	All PAH results	Varies	U / UM / UJ1 / UMJ1	Varies	UJ
HAFB-DUP01	All PAH results	Varies	U / UM	Varies	UJ
HAFB-MW02	Antimony, total	0.73	I	0.73	U
	Iron, total	83.7	I	83.7	J
	All PAH results	Varies	UM	Varies	U

Only environmental samples and field duplicates are included in the above table. Field blanks are used to evaluate the sample data but are not qualified during the review process.

PFAS Stage 2A Data Validation Checklist

Method: LC/MS/MS and Isotope Dilution Compliant with Table B-15 of DoD QSM 5.3
Project Name: Off-Base Drinking Water Site Inspection, USAF Installations, Multiple Sites
Sample Delivery Group: FA82510

Laboratory ID	Sample ID	Received	Collected		Matrix	Sample Type
ABC-1234-01	HAFB-MW01	1/21/2021	1/20/2021	13:45	Water	Normal
ABC-1234-02	HAFB-DUP01	1/21/2021	1/20/2021	13:22	Water	Field Duplicate
ABC-1234-04	HAFB-MW02	1/21/2021	1/20/2021	13:23	Water	Normal

	Yes	No	NA	Comments
I. Case Narrative/Sample Receipt/Holding Times				
Were all samples listed on the COC reported with the correct sample IDs?	●	○	○	
Did the case narrative include any issues that impact the data validation?	○	●	○	
Were samples received in proper containers and properly preserved?	●	○	○	
Were there any discrepancies noted at sample receipt?	○	●	○	
Were all samples listed on the COC analyzed?	●	○	○	
Were all holding times met?	●	○	○	
II. DoD QSM Specified Ion Transitions				
Were the ion transitions those specified in QSM Table B-15 (below)? PFOA: 413 → 369 PFOS: 499 → 80 PFHxS: 399 → 80 PFBS: 299 → 80 4:2 FTS: 327 → 307 6:2 FTS: 427 → 407 8:2 FTS: 527 → 507 NEtFOSAA: 584 → 419 NMeFOSAA: 570 → 419	●	○	○	
III. Extracted Internal Standard (EIS) Recoveries				
Were EIS recoveries within the control limits specified in the QAPP or 50-150%, if QSM limits used)?	●	○	○	
Were EIS retention times within 0.40 minutes of retention time of midpoint std in ICAL or initial CCV?	●	○	○	
IV. Laboratory Blanks				
Was a laboratory blank associated with every sample in this SDG?	●	○	○	
Were the laboratory blanks free of contamination?	○	●	○	The MB was contaminated with 2.4 ng/L PFOS. All three PFOS detections were greater than the action level, and no qualification was required.
V. Field blanks				
Were field blanks included in this SDG?	●	○	○	
Were target compounds detected in the field blanks?	○	●	○	

VI. Equipment blanks				
Were equipment blanks included in this SDG?	○	◉	○	
Were target compounds detected in the equipment blanks?	○	○	◉	
VII. Matrix spike/Matrix spike duplicates				
Were matrix spike (MS) and matrix spike duplicate (MSD) analyzed in this SDG?	○	◉	○	An MS and laboratory duplicate from another site were reported with the samples in this SDG.
Were the MS/MSD percent recoveries (%R) and the relative percent differences (RPD) within the QC limits?	○	○	◉	All recoveries were within control for the batch MS. For the laboratory duplicate, the absolute difference of the PFHxS results met the criteria.
VIII. Laboratory control samples				
Was an LCS/LCSD analyzed per extraction batch for this SDG?	◉	○	○	No LCSD.
Were the LCS percent recoveries (%R) and relative percent difference (RPD) within the QC limits?	◉	○	○	
IX. Field duplicates				
Were field duplicate pairs identified in this SDG?	◉	○	○	
Did the field duplicate meet the criteria specified in the QAPP?	◉	○	○	
X. Compound quantitation				
Did the reported list of analytes include all those specified in the QAPP?	◉	○	○	
Did the laboratory reporting limits (i.e. DL, LOD, LOQ) meet the QAPP?	◉	○	○	
Did reported results include both branched and linear isomers?	◉	○	○	
XI. Overall assessment of Data				
Overall assessment of data was found to be acceptable.	◉	○	○	

Reviewer: John Powell	**Date: 02-07-2021**	**Second Reviewer: Denise Rivers**	**Date: 02-08-2021**

Table 1: Qualification Summary (all concentrations in ng/L):

Sample ID	Analyte	Lab Concentration	Lab Qualifier	HGL Concentration	HGL Qualifier

The following provides a brief explanation of the data validation qualifiers assigned to results during the data review process by the 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 quality control 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 project chemist), but exclusion of the data is recommended.

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ATTACHMENT C
General Data Validation Conventions

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ATTACHMENT C
General Data Validation Conventions

1.0 INTRODUCTION

The general conventions presented below describe the evaluation and qualification process applied to project data undergoing a Stage 2A or Stage 2B data validation. The data validator should always use the Quality Assurance Project Plan (QAPP) as the primary source for project-specific validation requirements. Where the general conventions presented below conflict with the requirements presented in the QAPP, the QAPP requirements should take precedence. Situations that are not covered by the project QAPP or by the general conventions should be referred to a HydroGeoLogic, Inc. (HGL) senior chemist for resolution.

Note that the guidance presented in this attachment assumes that the project QAPP presents validation and qualification criteria based on the quality control (QC) requirements of the U.S. Department of Defense (DoD)/Department of Energy (DOE) Consolidated Quality Systems Manual (QSM), version 5.3. Laboratory certification under the DoD Environmental Laboratory Accreditation Program is performed under the requirements of the QSM version current at the time of certification. This recertification process is on an approximately 18-month cycle. As a result, some project QAPPs will cite the version of the QSM that was in effect at the time of the project laboratory's accreditation; also, there are still QAPPs in use that have data qualification protocols based on the QC requirements of older versions of the QSM. If the guidance presented in this attachment conflicts with the project QAPP qualification protocols, the requirements of the project QAPP should take precedence unless alternative direction is received from the client project manager. As additional versions of the DoD QSM are issued, new project QAPPs will incorporate the most up-to-date DoD requirements consistent with project laboratory certification status.

2.0 SENSITIVITY LIMITS

The principal reasons for assigning data qualifiers are the magnitude of detected results relative to the associated sensitivity limits and the conventions for reporting nondetected results. There are two principal conventions for establishing sensitivity limits, the conventions originally established by the U.S. Environmental Protection Agency (EPA) to support the Contract Laboratory Program (CLP) and the conventions established by DoD. Both are in common use and are described below. Table C.1 presents the DoD terms, their definitions, and the corresponding EPA terms that are also in common usage.

Table C.1
Sensitivity Limit Definitions[(1)]

Sensitivity Limit Term	Definition	Corresponding EPA Terms
Detection limit (DL)	The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.	Method detection limit (MDL)
Limit of detection (LOD)	The smallest amount or concentration of a substance that must be present in a sample to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. An LOD may be used as the lowest concentration for reliably reporting a nondetect of a specific analyte in a specific matrix with a specific method at 99% confidence.	--
Limit of quantitation (LOQ)	The lowest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ is set at or above the concentration of the lowest initial calibration standard and within the calibrated range.	Reporting limit Quantitation limit Practical quantitation limit Method quantitation limit Contract-required detection limit Contract-required quantitation limit

(1) Terms and definitions are from Section 3.1 of the QSM, version 5.3 (May 2019).

2.1 EPA SENSITIVITY LIMIT CONVENTIONS

The EPA convention involves setting a concentration limit above which analytical results are considered to be of sufficient quantitative significance to be reported without qualification (unless affected by QC issues). In practice, this limit is established at or above the low point on the calibration curve for each target analyte. A variety of terms has been applied to this limit, including reporting limit (RL), practical quantitation limit, and method quantitation limit. EPA's CLP uses the term contract-required quantitation limit, although historical data may include the term contract required detection limit (CRDL) applied to inorganic results. Results between the MDL and RL are reported as detections qualified as estimated due to being below the calibrated range. Results below the MDL are considered nondetected results and are reported as the numerical value of the MDL or the RL (depending on project-specific requirements) qualified U.

For many of HGL's DoD projects, the EPA sensitivity limit conventions have been superseded by the DoD conventions described in Section 2.2; however, most projects performed for non-DoD clients will still use the EPA conventions. Older DoD projects with existing basewide QAPPs also may retain the use of EPA conventions to maintain comparability with the existing project dataset or to comply with state or permit data reporting requirements.

2.2 DOD SENSITIVITY LIMIT CONVENTIONS

The current DoD sensitivity limit conventions were introduced in version 4 of the QSM in April 2009 and have remained in use in subsequent versions of the QSM. QSM version 4 established a three-tiered system of DL, LOD, and LOQ. The QSM provides definitions for all these terms; however, in practical applications, the DL and LOQ are used in an analogous fashion as the MDL and RL, respectively, are used in the EPA sensitivity conventions. Results between the DL and LOQ are reported as detections qualified as estimated due to being below the calibrated range. The LOD term was introduced in QSM version 4 and corresponds to the lowest level that can be present in a sample and have a 99 percent probability of being detected in that sample. In the DoD conventions, results below the DL are considered nondetected results and are reported as the numerical value of the LOD qualified U.

3.0 DATA QUALIFIERS

Each validated result consists of three components: (1) a numerical value that corresponds to a concentration, (2) a data qualifier, and (3) the concentration units. The concentration can correspond to a detected value or to a proxy value used for nondetected results in that is assigned accordance with the conventions presented in the project QAPP. The data validation process generally focuses on the application of the appropriate data qualifier on each result. Some projects will require a change to the numerical concentration presented under specific circumstances (see Section 3.2.4).

Data qualification indicates that an analytical result falls into one of three broad categories: (1) usable; (2) usable but estimated; and (3) unusable. The validation conventions presented below do not present specific qualification requirements. The qualifiers to be used for a project will be defined in that project's QAPP. The allowed final data qualifiers will be defined depending on the client and the regulatory body that will be the final recipients of the data. Descriptions of commonly applied data qualifiers are presented below, but the data validator must use the qualification requirements specified in the QAPP for each project.

The most used data qualification conventions for DoD projects will be based on those qualifiers listed and defined in the DoD General Data Validation Guidelines.

3.1 LABORATORY-APPLIED FLAGS

In some cases, data points may be reported by the laboratory with one or more informational flags, such as an alphanumeric code or a symbol. These flags are not considered valid qualifiers and should be automatically removed from all affected data points, with the exceptions noted in Sections 3.2.2, 3.2.4, and 3.3.1 below. In some cases, the laboratory-applied informational flag will mimic a valid final qualifier but may or may not be applicable as the final qualifier. In such cases, the validator's discussion of the effect of a QC discrepancy on the associated results should

also include a discussion of whether laboratory-applied flags that mimic a valid qualifier should be retained, deleted, or altered. All residual laboratory-applied flags that are not accepted as the final qualifier by the data validator must be removed from the electronic data at what is referred to as the "100 percent QC stage" of data upload and incorporation into the project database (see Section 6.0 of the standard operating procedure [SOP]).

3.2 QUALIFICATION OF DETECTED RESULTS

3.2.1 Detected Results Not Requiring Qualification

Results that are detected within the calibrated range of the instrument and that are not associated with a QC discrepancy will be accepted by the validation process as the numerical value of the concentration (with appropriate units) and without any data qualifier.

3.2.2 Detected Results below the Calibrated Range

Detected results with concentrations equal to or greater than the DL but below the LOQ (corresponding to the lower limit of the calibrated range of the instrument) are considered to be estimated results by default. Laboratories report such results with an informational flag to indicate that the result is below the calibrated range. This informational flag is most often a "J," "B" (CLP convention for inorganic results), or "I" (Florida Department of Environmental Protection convention). In some cases, these flags correspond to commonly used final qualifiers that are applied to such results. When the laboratory assigns a flag that corresponds to the project qualification convention, the assigned flag can be accepted as the final qualifier by the validator if no other qualification is required for a QC issue. In other cases, the validator will need to specify that, absent any other qualification on specific results, the laboratory's default flag for a detected result below the LOQ is globally changed to the project-specific qualifier.

3.2.3 Detected Results Requiring Qualification as Estimates

Detected results affected by QC issues will be qualified as estimated values as required by the project validation guidelines. The most common qualifier used to indicate an estimated result is "J," although it is common for projects to use alternative qualifiers if the overall direction of bias can be determined. These alternative qualifiers can include the DoD qualifiers "J+" if the bias is high, or "J–" if the bias is low.

3.2.4 Detected Results Requiring Qualification as Artifacts

One of the goals of data validation is to determine if detected concentrations of analytes reported in samples are representative of site conditions. Detected concentrations reported by the laboratory that are artifacts of the sampling, shipping, storage, preparation, and analytical processes that the sample undergoes are not representative of the site and must be identified by the validator. The most common procedure to identify results as artifacts is to apply the qualification of "U."

In addition to being used to identify artifacts under some conventions, the U qualifier is almost universally used to identify nondetected results (see Section 3.3.1). When the U qualifier is used both as a laboratory qualifier for identifying nondetects and as a validator qualifier for identifying artifacts, the final qualifier will not allow the data user to determine whether the analyte in question is a nondetection or was determined to be an artifact. However, artifacts are treated in the same fashion as nondetections for most end uses of analytical data, so in practice this convention does not introduce unacceptable ambiguity into interpreting the qualified result. The quantitated value associated with the U qualifier assigned to an artifact can be the originally reported detected value, the LOD, or the LOQ (or equivalent), depending on the data reporting conventions presented in the project QAPP. For projects using the DoD sensitivity limit conventions, results qualified U as artifacts that have a concentration that exceeds the DL but are lower than the associated LOD will have the reported concentration changed at a minimum to the value of the LOD or to a higher value as directed by the data validation protocols.

3.3 QUALIFICATION OF NONDETECTED RESULTS

3.3.1 Nondetected Results Not Requiring Qualification

Nondetected results receive a final qualifier of U in almost every data qualification convention. Depending on the requirements of the QAPP, the quantitated value associated with the U qualifier can either be the DL (or equivalent), the LOD, or the LOQ (or equivalent). The reporting conventions to be used for each project should be included in the project QAPP and should be confirmed with the laboratory prior to generating project results. For most projects, a large majority of the reported laboratory results will be nondetections. Ensuring that the laboratory will report nondetected data flagged U using the same protocols as are required for the final U qualification will allow the data validator to retain the laboratory flags unchanged.

Some laboratories report nondetected results as "ND" or as "<#," where # represents a number that can be the DL (or equivalent), LOD, or LOQ (or equivalent). The data validation report should indicate that such results are considered to be the equivalent of results qualified U according to the project conventions, unless superseded by a more severe qualifier.

3.3.2 Nondetected Results Requiring Qualification as Estimated

Nondetected results affected by QC issues will be qualified as estimated values as required by the project validation guidelines. The most common qualifier used to indicate an estimated result is the combination qualifier "UJ." Nondetected results are not considered to be affected by high bias or precision discrepancies (except when reported as part of a duplicate or triplicate set of analyses that also includes detections of the affected analyte). As with nondetected results not requiring qualification, the quantitated value associated with the qualified result can be the DL (or

equivalent), the LOD, or the LOQ (or equivalent), depending on the project conventions for reporting nondetected results.

3.4 REJECTED RESULTS

Data points affected by severe QC discrepancies are potentially unusable for their intended purposes as described in the project data quality objectives. The data qualification guidelines presented in the QAPP establish the circumstances under which data is rejected or otherwise noted as suspect by the validator. Any data rejected or identified as suspect in the data validation process should be evaluated by the HGL project chemist and the project team to determine if a final qualifier of R should be applied or if a less severe qualifier can be justified. If a less severe qualifier is selected for the affected results, the technical rationale must be included in the HGL data validation report (internal data validation) or the HGL data validation report review memo (subcontracted data validation). The technical rationale must also be included in any data quality evaluation provided as part of the project deliverables (see Section 3.3 of the main body of this SOP).

A result that receives a final qualifier of R should have the "Report Usability" field in the associated electronic file populated with Y. The Report Usability field should only be populated with N if the result is superseded by another result (see Section 3.5 below).

3.4.1 Rejection of Detected Results

Most data qualification conventions will not require rejection of detected results unless severe instrumental or systematic deficiencies are identified. Detected results with extreme high or low bias that are compromised by severe discrepancies in sample collection or shipment or that were generated while the analytical system was unacceptably compromised will not be of sufficient quality to be incorporated into a quantitative risk assessment. In some cases, however, data points rejected in accordance with the validation protocols may have limited usability.

> *Example*: A detected result is associated with a severe low bias, but the result is greater than the screening level for the site. Although the validation protocols indicate this result should be rejected, the affected result could be used to determine if that compound were a contaminant of concern at the site if it was above the associated screening value. However, the numerical value could be too compromised to be incorporated into the quantitative determination of risk at the site.

Rejected detected results are qualified R; quantitated values should not be reported in association with a result qualified R.

3.4.2 Rejection of Nondetected Results

Nondetected results are generally rejected under more circumstances than detected results. This is because most projects consider a Type II (false negative) error to be a more severe error than a Type I (false positive) error. Rejected nondetected results are qualified R; quantitated values should not be reported in association with a result qualified R.

3.4.3 DoD Data Rejection Conventions

The most recent DoD data qualification conventions (DoD EMDQ, 2019) include an X flag. The X flag is intended to be used as an interim qualifier that replaces the R qualifier at the data validation stage and is replaced by the R qualifier or a less severe qualifier at the data usability stage. HGL's multiple stages of data validation review and the data usability assessment procedures included in project QAPPs are analogous to the intended use of the DoD X flag. HGL's procedures ensure that data qualified R during the validation process are subject to additional technical evaluation to determine if the R qualifier is an appropriated final qualifier. While many current HGL QAPPs indicate that the data validator should apply R qualifiers pending further review, new QAPPs for DoD clients should incorporate the most recent DoD data qualifiers, including the use of the X flag as an initial qualifier at the validation stage.

3.5 QUALIFICATION OF EXCLUDED RESULTS

In cases where multiple analysis results are reported for a sample due to dilution or reanalysis, all analyses are to be reviewed. Based on the body of QC data, the validator should select one definitive result for each analyte in each sample, and all other results for that analyte in that sample are denoted as superseded by applying an # qualifier.[2] Clearly indicating results that are not to be used with an # assists in managing data for report preparation and database submittal. Results that receive an # qualifier do not need to be further validated or qualified; however, the validation narrative should include the rationale for selecting the definitive result. Results receiving an # qualifier should be included in the data qualification table in each validation report, with the analysis receiving the qualification clearly differentiated from the other analyses performed on the same sample. Where large categories of results in a sample analysis receive an # qualifier, this qualifier may be noted for the class of results (for example, "All nondetections") instead of as an analyte-by-analyte listing. Applying an # qualifier may result in the data for the full analyte list for a particular sample being composed of results from multiple analyses. For example, in an original analysis/diluted analysis pair, all analytes in the original analysis are considered definitive except for those analytes that exceeded the calibrated range, which are reported from the diluted analysis.

[2] HGL previously applied an X qualifier. In the most recent DoD data validation guidance (DoD EMDQ, 2019), X is an interim data flag to be applied instead of R at the validation stage.

3.6 RESULTS WITH MULTIPLE APPLICABLE QUALIFIERS

Some results may be affected by more than one QC discrepancy. In such cases, the final qualifier applied to each result is the highest priority qualifier as defined by the project QAPP.

When "U" is used the qualifier to denote an artifact, the validator should treat the associated result as a detection when evaluating additional qualification for other QC issues.

> *Example*: A result is determined to be an artifact and the conventions call for that result to be qualified U. Another QC issue also affects that result, and the qualification conventions call for a detected result to be qualified J and a nondetected result to be qualified R or X. The validator should apply UJ as the final qualifier instead of R or X to any affected results that were originally reported as detections but have been qualified U as a result of being considered an artifact. However, once the data validation stage is complete, the Detected field in the electronic data deliverable should be populated with N in accordance with Section 3.3.2 above.

4.0 STAGE 2A QC ELEMENTS

The following are general guidelines for reviewing the QC elements identified as Stage 2A QC elements in Attachment A. Final qualification will be applied in accordance with the QAPP. As Stage 2A data validation includes the components of a Stage 1 data review, the Stage 1 components are included in the requirements for Stage 2A validation.

4.1 CASE NARRATIVE

Qualification is usually not required based on the results of the case narrative; however, the validator should review the narrative prior to beginning validating the data package. The narrative can assist in identifying QC issues, describe corrective action or causes for QC discrepancies, describe sample receipt discrepancies, and indicate any special client instructions for the sample analyses. In the data validation report, the validator should include any items of note that were in the narrative, as well as indicate if there were any errors or omissions in the laboratory narrative.

4.2 CHAIN OF CUSTODY

Review the chain of custody (CoC) form and verify that there are no discrepancies. Some general issues can include difficult-to-read sample IDs, crossed-out items, incorrect analyses requested, incorrect or missing time of collection, and missing or incorrect preservative information. The laboratory also may indicate additional information on the CoC form such as special client requests, sample receipt temperature, and samples added or deleted from those requested on the chain. Generally, results are not qualified based on the CoC form alone; however, this information can be useful to the validator.

4.3 SAMPLE RECEIPT AND LOG-IN FORMS

This form should be checked for discrepancies in sample temperature and sample preservation; discrepancies between the sample labels and the CoC forms; missing, broken, or damaged bottles; and bubbles in containers that should have zero headspace. Results may be qualified based on sample receipt and condition.

Some methods, such as metals and volatile organic compounds (VOC), allow for alternatives if preservation requirements are not met. Aqueous VOC samples must be submitted with zero headspace; however, samples may arrive at the laboratory with some headspace. A VOCs sample with headspace is considered to be acceptable if the bubble in the vial is less than "pea-sized" (defined as approximately ¼ inch or 6 millimeters). If larger bubbles or headspace is observed in VOC samples, this may be an indication of a reaction of the acid preservative with the sample matrix causing effervescence. The HGL project manager should be alerted as soon as possible so that corrective action can be implemented, including resampling or eliminating preservative in future VOC samples collected from the affected locations.

Although it is good practice to ship all samples iced, temperature discrepancies are less likely to affect persistent organic compounds like polynuclear aromatic hydrocarbons, pesticides, and polychlorinated biphenyls (PCBs); temperature discrepancies should have minimal to no effect on metals samples. If the samples were delivered to the laboratory by courier on the same day they were collected, the samples may not have had enough time to chill to the acceptance range (0 to 6 degrees Celsius [°C]). In such cases, the sample temperature is considered to be compliant if the samples arrived at the laboratory iced and were refrigerated on arrival.

Current EPA guidance (EPA, 2014) allows for acid-preserved aqueous metals samples to be shipped and stored at ambient temperature. Soil samples collected by incremental sampling methodology are dried at ambient temperatures over a period of days at the laboratory. Although individual QAPPs may specify temperature requirements for these samples, the impact the samples arriving at the laboratory >6°C is negligible and this should be considered by the validators when evaluating the effect on the analytical results.

4.4 SAMPLE ID CROSS REFERENCE

Review the laboratory listing of HGL sample identifications (IDs) against the CoC form. Common errors involving letter/numeral substitutions include "0" and "O" or "D"; "5" and "S"; "6" and "G"; and "8" and "B." Another common error is inconsistencies in incorporating dashes or spaces in sample IDs.

Errors can occur at sample login when the parent sample and the requested matrix spike (MS) and matrix spike duplicate (MSD) samples are submitted in using an ID format that inserts "MS" and "MSD" into a long string of alphanumeric characters: "PARENTSAMPLEID,"

"PARENTMSSAMPLEID," and "PARENTMSDSAMPLEID." When there is no clear indication that a sample is an MS or an MSD sample, the laboratory log-in department may not notice that the sample IDs are indicating an MS or MSD, causing these samples to be logged in as "normal" samples. The result is that instead of results for parent sample and an MS/MSD pair, the samples are analyzed as a sample triplicate. In such cases, the laboratory log-in department should be notified to be alert for such sample IDs, and the HGL project manager should be alerted that more explicit instructions should be provided to the laboratory when submitting MS/MSDs.

4.5 HOLDING TIMES

The holding times for preparation and analysis for each analytical method should be presented in the project QAPP. Holding times expressed in hours are evaluated based on time of collection to time of preparation or analysis, as measured in hours and holding times expressed in days are evaluated based on calendar days elapsed, with the sampling date considered day "0."

The validator should be aware that time zone difference and daylight savings time need to be accounted for when evaluating holding time to the hour. Also, some sampling teams assign a "dummy" sample collection time (such as "1200") to field duplicate samples. Before qualifying field duplicate sample results for a holding time exceedance of less than a day, the validator should verify the actual sample collection time with the field team.

The validator has some discretion to consider a holding time exceedance to be nominal and determine that qualification is not necessary.

4.6 LCS/LCSD RECOVERIES AND PRECISION

As discussed in Section 3.2 of the SOP, the validator should verify that the control limits reported by the laboratory match those required in the project QAPP. Note that laboratory control sample duplicates (LCSD) are not a QC element required by any analytical methods; however, reporting an LCSD in association with a laboratory control sample (LCS) is a common laboratory practice. When LCSDs are reported, the accuracy performance should be evaluated in the same manner as the associated LCS, and discrepancies in either the LCS or LCSD should be considered grounds for qualifying associated data. In some cases, however, the validator can consider acceptable performance in the LCS or LCSD as a mitigating factor and reduce the severity of the data qualifier applied to associated results for a discrepancy in the other member of the LCS/LCSD pair. The decision to reduce the severity of the data qualifier in this instance should be discussed in the data validation report.

LCSs (and LCSDs) should be spiked with the full list of target analytes unless the QAPP specifically allows for the use of a shorter list. The exception is in the analysis of PCBs. Because there are multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs LCSs are spiked with a standard containing only PCB-1016 and PCB-1260. Generally, discrepancies

shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

LCS/LCSD recoveries that are above the acceptance limits are usually considered not to affect nondetected results. In cases of extremely high recoveries (approaching 200 percent or greater) the validator should consider whether an analytical system problem has occurred. If the cause for abnormally high recoveries is not noted in the case narrative, the validator should contact the laboratory and request an explanation for such anomalies. In some cases, such discrepancies can be traced to accidental double-spiking and the recoveries will meet acceptance criteria when calculated using the actual spiked concentration. However, the validator should consider the qualification of nondetected results associated with unusually high recoveries if the underlying cause indicates a problem in the analytical system.

When LCS/LCSD precision (the reported relative percent difference [RPD]) does not meet the requirements for an analyte, detected results for the affected analyte should be qualified in the associated samples. Nondetected results generally do not require qualification for LCS/LCSD precision discrepancies.

4.7 MS/MSD RECOVERIES AND PRECISION

The evaluation of MS/MSDs is generally the same as the evaluation performed on LCSs and (if performed) LCSDs. Given that MS/MSDs are intended as verification that the laboratory can detect target analytes in the project-specific sample matrix, only MS/MSD analyses performed on HGL-collected samples from the same site (or installation) are considered applicable to the associated sample results. Laboratories often report MS/MSD results from a different sample delivery group (SDG) as batch control without the client sample ID. When a batch control MS/MSD is reported, the validator should use the laboratory sample ID to confirm whether the MS/MSD is actually from a site sample reported in a different SDG or from a non-site sample. If the MS/MSD is from a site sample, it will be considered applicable to associated results. If the MS/MSD cannot be associated with a site sample, it is sufficient to indicate that that one or more reported MS/MSDs were performed on non-project samples and were not used to evaluate the data. No qualification should be applied based on discrepancies in non-project MS/MSDs unless the underlying cause of the discrepancy is suspected to be a problem with the analytical system.

MS/MSD recovery discrepancies in samples that have concentrations of the affected target analytes greater than 4 times the spiked concentration are not considered applicable; this is commonly referred to as the "4 times rule." However, in many cases, the RPD for such MS/MSDs can still be evaluated and used to qualify associated results.

Some laboratories compare the concentrations detected in the MS and the MSD to calculate precision rather than compare the percent recoveries. This convention can cause RPDs to be an incorrect representation of the analyte-specific precision if the spiked concentration in the MS

differs substantially from the spiked concentration in the MSD. The validator should examine the MS and MSD spike concentrations to determine if the reported RPD, calculated using a direct comparison of the detected concentrations, is not relevant. The validator should verify that the RPDs reported for MS/MSD results are calculated using the percent recoveries or that the expected concentration in the MS is the same as in the MSD. If the RPDs are calculated using noncomparable spike concentrations, the validator should use alternative means, such as comparing the reported MS and MSD percent recoveries, to determine if precision criteria were met.

Dilution should reduce or eliminate matrix effects and MS/MSD discrepancies in cases where the MS and/or MSD were diluted require some interpretation on the part of the reviewer to determine whether there is actually a matrix effect or whether some other factor is contributing to the discrepancy. In cases where MS/MSD recoveries are calculated from spike recoveries that are above the calibrated range, the reviewer should evaluate whether any discrepancies are a result of matrix effects or are a result of the inherent unreliability of such results.

MSs (and MSDs) should be spiked with the full list of target analytes unless the QAPP specifically allows for the use of a shorter list. The exception is in the analysis of PCBs. Because of the existence of multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs MS/MSDs are spiked with a mixture of PCB-1016 and PCB-1260. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

For some methods, it is permissible to analyze a single MS as a check for accuracy and use a laboratory duplicate as the check for precision. Laboratory duplicate evaluation is discussed under field duplicates (Section 4.11). If the laboratory performs both an MSD and a laboratory duplicate, both should be evaluated and used to qualify associated results. As with MSs and MSDs, laboratory duplicate results may be from a site sample reported in another SDG or from a non-site sample, and the validator should determine the applicability of laboratory duplicate results reported from other SDGs.

The qualification of results for MS/MSD discrepancies is project- and method-specific. Generally, inorganic and wet chemistry MS/MSD results are considered to be associated with all environmental samples in the same preparation batch and organic MS/MSD results are considered to be associated only with the parent sample.

The QAPP should include additional instructions for evaluating and qualifying results based on MS/MSD discrepancies. Nondetected results generally do not require qualification for MS/MSD precision discrepancies. MS/MSD recoveries that are above the acceptance limits are usually considered not to affect nondetected results. In cases of extremely high recoveries (approaching 200 percent or greater) that are not attributable to native analyte concentration or matrix effects, the validator should consider whether an analytical system problem is occurring. If the cause for

abnormally high recoveries is not noted in the case narrative, the validator should contact the laboratory and request an explanation for such anomalies. In some cases, such discrepancies can be traced to accidental double-spiking and the recoveries will meet acceptance criteria when calculated using the actual spiked concentration. However, the validator should consider the qualification of nondetected results associated with unusually high recoveries if the underlying cause indicates a problem in the analytical system.

4.8 SERIAL DILUTIONS AND POST-DIGESTION SPIKES

For DoD projects, serial dilution and post-digestion spike (PDS) analyses are only required for metals analyses and only if the MS/MSD shows discrepancies. Data are not qualified based on serial dilution or PDS results alone; they are used to supplement the overall evaluation of matrix effects if the MS/MSD shows discrepancies or is not applicable due to an elevated target analyte concentration in the parent sample (greater than 4 times the spike concentration). Serial dilution results are applicable to target analytes that are present in the MS/MSD parent sample at or above 50 times the laboratory's default (undiluted) LOQ and PDS results are applicable to target analytes that are present in the MS/MSD parent sample at less than 50 times the laboratory's default LOQ. The evaluation of MS/MSD recoveries, PDS recoveries, and serial dilution percent differences and the qualification conventions will be specified by the project QAPP.

PDS results are subject to the same "4 times rule" that is used for MS/MSDs. There may be some situations where the MS/MSD and PDS results are out of control but are not applicable because of the 4 times rule, but the parent sample is below the 50 times LOQ rule for serial dilution results to be applicable. In such cases, the validator must evaluate the matrix data as a whole and decide whether qualification for matrix effects is required.

Other methods may require PDSs as method-specific QC elements. The evaluation requirements for non-metals PDSs will be included in the project QAPP, and generally these PDSs can be used alone to qualify data.

4.9 METHOD BLANKS

HGL's QAPPs list acceptance criteria for method blanks. These acceptance criteria are the levels above which blank contamination necessitates that the laboratory performs corrective action. However, *all* method blank concentrations that are greater than the associated DL or have a negative concentration with absolute value greater than the associated DL should be used to qualify the associated sample results. The data validator should note any concentrations of target analytes detected in method blanks that are greater than the associated acceptance limits, including metals method blanks showing negative concentrations with absolute value greater than the acceptance limits.

Target analyte concentrations detected in method blanks should be multiplied by 5; this calculated value is called the artifact threshold.[3] Concentrations of these analytes in associated samples that are less than the artifact threshold are considered artifacts and are qualified in accordance with the QAPP.

Concentrations of common laboratory contaminants are multiplied by 10 instead of 5 to determine the artifact threshold. Common laboratory contaminants for VOCs include methylene chloride, acetone, and 2-butanone (methyl ethyl ketone). Common laboratory contaminants for semivolatile organic compounds (SVOCs) are the phthalate esters.

When comparing method blank action levels to sample concentrations, the artifact threshold should be adjusted to account for sample-specific information, including percent moisture, subsample size, and dilution factor. Often, the easiest way to determine a sample-specific adjustment is to compare the LOQ of a target compound in the sample to the LOQ for that compound in the method blank.

> *Example*: Toluene is detected in a method blank at 4.3 micrograms per kilogram (µg/kg). The toluene LOQ is 5 µg/kg in the method blank and 7.4 µg/kg in sample ABC123. The sample-specific artifact threshold for toluene is 4.3 x (7.4/5) x 5 µg/kg = 32 µg/kg.

In most cases, it will be readily apparent that a result is above or below an artifact threshold and this sample-specific adjustment is necessary for only a minority of comparisons.

4.10 FIELD BLANKS

Field blanks are evaluated in a similar manner as method blanks (Section 4.8). Two main differences are (1) the artifact threshold calculated from concentrations in field blanks is *not* adjusted for sample-specific factors; and (2) most field blanks are aqueous and conversion to equivalent solid units is not straightforward for some analytical methods.

When evaluating the effect of aqueous field blank results on associated aqueous field samples, the artifact threshold associated with field blank contamination is 5 times the concentration detected in the blank (10 times the concentration in the case of common laboratory contaminants). When evaluating the effect of aqueous field blank results on associated solid matrix field samples, the field blank results must first be converted to the equivalent solid concentration.

[3] Note that the term "action level" was previously used to describe this value; the use of the term action level is discouraged because that term is also used in site characterization and has a different meaning when used in that context.

4.10.1 Water-to-Soil Conversion for Organic Extraction Methods

Aqueous field blank results for organic extraction methods can generally be converted to solid units by comparing the ratio of the aqueous LOQs to the LOQs reported in the solid matrix method blanks.

> *Example*: A rinse blank has a detected result of 7.8 micrograms per liter (µg/L) for diethyl phthalate. The aqueous LOQ is 10 µg/L and the solid LOQ is 330 µg/kg. The diethyl phthalate result in the rinse blank is the equivalent of a result of 257.4 µg/kg (7.8 x 330/10). Because diethyl phthalate is a common laboratory contaminant, the artifact threshold is 2,574 µg/kg.

4.10.2 Water-to-Soil Conversion for VOCs

For VOCs, the formula for converting a water result to a soil result is not straightforward; the laboratory should be consulted before the convention used for organic extraction methods can be used to evaluate VOCs field blank results. In some cases, the raw data will show an "on-column" result reporting the concentration in the extract not converted to the final units used for the matrix of the samples. In these cases, the on-column results for field blanks can be multiplied by 5 (or 10) and compared directly to the on-column results reported for the associated field samples. It is more likely; however, that the laboratory software will show the raw data results already converted to the matrix units and this method of comparison will be usable only in a limited number of cases.

4.10.3 Water-to-Soil Conversion for Metals

For metals, the conversion equation is as follows:

$$C_S = (C_W \times V_F)/M_E$$

Where:

C_S = the calculated equivalent solid concentration (in milligrams per kilogram [mg/kg])
C_W = the reported aqueous concentration in µg/L
V_F = The final volume of soil digestate extracts in liters (L)
M_E = The nominal mass extracted for solid samples in grams (g) (use the mass of a solid method blank)

> *Example*: A rinse blank has a detected zinc concentration of 5.3 µg/L. The laboratory's preparation forms show that the final volume of soil extracts is 50 milliliters (= 0.05 L) and the soil method blank was extracted using 1.00 g. The rinse blank result is the equivalent of 0.265 µg/g = 0.265 mg/kg, which leads to an artifact threshold of 1.325 mg/kg. Note that the laboratory may report an actual mass for the method blank that is not a "round" number. If it can be determined that that the nominal method blank mass is a round number

like 1.00 g or 0.50 g, use that value even if an individual method blank may be slightly off (for example, 1.02 g instead of 1.00 g or 0.49 g instead of 0.5 g).

4.11 FIELD DUPLICATE PRECISION

The evaluation of field duplicate precision depends on the concentration of each target analyte detected in the duplicate pair relative to the LOQ. Concentrations can be considered "low-level" or "high-level." The QAPP will specify the criteria for making this determination, and this determination should be made for every detected analyte before any further duplicate evaluation. One of the most common criteria for determining if a pair of results is high-level is if both results are greater than 5 times the associated LOQ.

General rules for evaluating field duplicate results include the following elements in the sequential order they are presented:

1. Two nondetected results are considered to be in control.
2. Two results detected below the LOQ, or one result below the LOQ and one nondetected result, are considered to be in control.
3. Two low level results or one low level-result and one high-level result are considered to be in control if the absolute difference of the two results is less than the value of the LOQ.
4. Two high-level results are considered to be in control if the RPD of the two results meets the RPD acceptance criterion listed in the QAPP.

The evaluation criteria presented in this section are also applicable to laboratory duplicate analyses that are performed for metals and other inorganic methods.

4.12 SURROGATE RECOVERIES

As discussed in Section 3.2 of the SOP, the validator should verify that the surrogate control limits reported by the laboratory match those required in the project QAPP. Although some data validation conventions assign individual surrogate compounds to lists of target compounds, HGL discourages this practice and the preferred approach is to assume that all surrogate discrepancies are associated with all target analytes. An exception to this is the evaluation of SVOCs surrogate results. When evaluating surrogate recoveries for this method, the acid extractible fraction surrogates should be associated with the acid extractible fraction target compounds (phenols and benzoic acid), and the base/neutral extractible surrogates should be associated with the base/neutral extractible fraction target compounds (all other analytes).

Surrogate recoveries that are above the acceptance limits are usually considered not to affect nondetected results. In cases of extremely high recoveries (approaching 200 percent or greater) the validator should consider whether an analytical system problem has occurred. If the cause for

abnormally high recoveries is not noted in the case narrative, the validator should contact the laboratory and request an explanation for such anomalies. In some cases, such discrepancies can be traced to accidental double-spiking, and the recoveries will meet acceptance criteria when calculated using the actual spiked concentration. However, the validator should consider the qualification of nondetected results associated with unusually high recoveries if the underlying cause indicates a problem in the analytical system.

Dilution of samples can affect surrogate recovery performance. For methods that have surrogate compounds added to a sample before any dilution steps, surrogate discrepancies can occur that are not caused by matrix or analytical effects but rather are caused by dilution effects. The validator should examine surrogate discrepancies in diluted analyses. In most cases, surrogate discrepancies reported in samples diluted greater than 5 times should be considered to be a dilution effect and qualification should not be applied to the affected sample results. Some methods, such as VOCs, can have surrogates added after dilution; in this case, dilution effects will not occur and the surrogate recoveries can be evaluated regardless of the dilution level.

4.13 METHOD-SPECIFIC QC CHECKS

Method-specific QC elements include such checks as pH buffer checks, cyanide distillation standards, synthetic precipitation leaching procedure extraction blanks, and replicate precision for total organic carbon. If these checks are reported in a Stage 2A data package, the validator should review these items as appropriate to the assigned level of validation. If the review guidelines are not included in the QAPP, the validator should consult with the project chemist to develop a review and qualification approach.

4.14 ANALYTE QUANTITATION

The validator should discuss any dilutions performed. In some cases, multiple analyses will be performed on a sample because of a required dilution or to verify results affected by a QC discrepancy. Some laboratories will report the entire analytical dataset for all analyses performed on a sample, while others will report only the "best" result for each analyte. If the laboratory reported multiple results for an analyte or set of analytes in a sample, the validator should select the best result for each analyte in each sample and indicate which result was chosen and why in the validation narrative. All results not selected for use are excluded from the dataset, and this is indicated by applying a # qualifier to the laboratory applied qualifiers (see Section 3.5).

Samples that are nominally solid samples may have very high percent moisture content. This is especially true of sediment samples that are very "soupy." Calculation of concentration on a dry weight basis for solid samples composed of less than 50 percent solids is complicated by the added nonhomogeneity of the samples. The validator should evaluate results from solid samples with high liquid content and apply qualification in accordance with professional judgment if qualification protocols are not specified in the QAPP.

5.0 STAGE 2B QC ELEMENTS

The Stage 2A validation guidelines presented in Section 4.0 are applicable to QC elements that are common to many analytical methods. Stage 2B validation guidelines build on the Stage 2A requirements and address QC elements that are more specific to individual extraction and analytical principles.

5.1 GC/MS ORGANICS

Gas chromatography (GC)/mass spectrometer (MS) organics include analyses for VOCs and for SVOCs, most commonly by SW-846 methods 8260B or C and 8270C or D, respectively, and the associated selected ion monitoring (SIM) modifications to these methods. Air sample analyses performed by Method TO-15 and TO-15-SIM are also performed by GC/MS; however, in most cases, method-specific requirements that apply to TO-15 analysis will differ from the general GC/MS requirements discussed in this section.

5.1.1 Instrument Tuning

SW-846 GC/MS methods require that the MS be tuned at the beginning of each 12-hour analytical sequence. MS tuning is a critical QC component, and no analyses may proceed without an acceptable MS tuning. Each GC/MS method document prescribes the ions of interest and the required relative abundances. If MS tuning data show discrepancies and sample analyses proceeded without corrective action, the project chemist should be contacted immediately to resolve this issue.

In some cases, laboratories report tuning criteria for CLP analysis methods for SW-846 analyses. Although this approach is permissible, it is not in accordance with the QAPP. When the validator observes incorrect MS tuning criteria applied to tuning results, they should immediately contact the project chemist to determine if the affected results are usable and to initiate corrective action at the laboratory.

In some cases, analytical samples and the closing calibration verification standard (CCV) of an analytical batch will be analyzed outside the 12-hour window that begins with an instrument tune. The validator should examine the magnitude of the exceedance to determine if the discrepancy is nominal. For larger discrepancies, the closing CCV results and other information should be reviewed to determine if any additional qualification is required.

5.1.2 Instrument Initial Calibration

Most GC/MS analytes will be calibrated to a mean relative response factor (RRF), which quantitatively relates the concentration of each target analyte to the associated internal standard.

There should be at least 5 calibration points for an initial calibration to a mean RRF to be valid. If the calibration relationship for a compound is linear or quadratic, a minimum of 6 and 7 points, respectively, is required.

5.1.2.1 Instrument Performance Criteria

For an initial calibration to be valid for GC/MS methods 8260B and 8270C, system performance check compounds (SPCCs) and calibration check compounds (CCCs) are critical QC elements and must meet acceptance criteria, even if these method-specified compounds are not target analytes for the associated samples. One exception to this statement is if SVOCs analyses are only requested for base/neutral-extractable compounds or acid extractable compounds, only the SPCCs and CCCs associated with the requested fraction need be reported and evaluated. Each SPCC must meet minimum mean RRF requirements, even if an individual SPCC is calibrated to a linear or quadratic relationship. Each CCC must meet maximum percent relative standard deviation (%RSD) requirements, even if an individual SPCC is calibrated to a linear or quadratic relationship. Failure of these compounds to meet acceptance criteria can indicate instrumental problems such as dirty injector ports, carrier gas flow problems, or reactive sites on the chromatography column. Consequently, analyses performed in association with failed SPCCs and CCCs are potentially compromised by instrument performance. Methods 8260C and D and 8270D and E do not have requirements for SPCCs and CCCs; SPCC and CCC performance is also not evaluated for the SIM modifications to Method 8260B and 8270C (see Section 5.1.2.2).

If SPCC or CCC discrepancies are noted, this information must be referred to the HGL senior chemist and project manager for immediate follow-up with the laboratory. SPCC and CCC discrepancies are serious QC deficiencies and can potentially result in the rejection of all data produced in association with that initial calibration. The HGL senior chemist, the HGL project manager, and the laboratory project manager and QC manager will determine (1) if the associated results can be used, (2) the appropriate instrument maintenance and recalibration procedures, and (3) the notification measures to ensure that SPCC and CCC deficiencies are appropriately addressed at the laboratory as soon as they are noted by the analyst.

Note that an SPCC or a CCC that is also a target compound will be evaluated against both the SPCC or CCC acceptance criteria and against the target analyte criteria presented in Section 5.1.2.2 below. These two evaluations are independent of each other.

> *Example*: VOCs CCC vinyl chloride is reported calibrated to a mean RRF with %RSD of 17.5 percent. The requirement for VOCs CCCs is that each have a %RSD of no greater than 30 percent. Vinyl chloride shows acceptable performance as a CCC; however, the target analyte criterion is for %RSD to be no greater than 15 percent. Vinyl chloride does not meet the acceptance criterion for target analytes. The effects, if any, of this discrepancy would be considered to affect vinyl chloride alone and not to be indicative of an instrument performance issue.

Laboratory initial calibration summary form formats will vary. If SPCCs are reported as calibrated to a linear or quadratic relationship, some laboratories' summary reporting forms may present the m1 term associated with the curve instead of the mean RRF. Other laboratories' summary forms may present both. If the summary forms do not include the mean RRF for one or more SPCCs, the validator should examine the associated continuing calibration verification forms; on occasion, the initial calibration mean RRF is reported there in addition to the continuing calibration RRF. The mean RRF also may be discussed in the case narrative if HGL has requested the laboratory to do so. If the mean RRF is not available in other locations in the data package, the data validator should contact the laboratory project manager and have this information transmitted.

As with SPCCs, laboratory summary forms may not present the CCC %RSDs for those CCCs calibrated to linear or quadratic relationships. This information is generally not presented elsewhere in the data package unless HGL has arranged with the project laboratory to present this information in the case narrative. Otherwise, the data validator should contact the laboratory project manager and have this information transmitted.

5.1.2.2 Target Analyte Performance Criteria

The linearity criterion for GC/MS initial calibration is %RSD no greater than 15 percent. The correlation (r^2) of linear or quadratic relationships should be no less than 0.99.

Although many laboratories are still using Method 8260B for VOCs analysis, some projects require the use of Method 8260C. Most laboratories have discontinued the use of Method 8270C and have updated the SVOCs method to 8270D. Methods 8260C and 8270D have replaced the mean RRF requirements for SPCCs with analyte-specific minimum mean RRFs and have discontinued the use of CCCs. The analyte-specific mean RRF requirements also apply to the SIM modifications to these methods. The mean RRF only needs to be checked for target analytes. The laboratory's summary forms may not present this information for target analytes calibrated to linear or quadratic relationships. If so, the validator should review the continuing calibration forms and case narrative to determine if this information is available from other sources, as described in Section 5.1.2.1 above. While some laboratories now have DoD accreditation for methods 8260D or 8270E, these methods not currently widely used although they are expected to become more common in the future.

Methods 8260B and 8270C do not have a requirement for minimum mean RRF for target analytes; however, some historical project QAPPs may include a requirement for all target analytes to show a mean RRF of no less than 0.050. This requirement comes from the requirements of the CLP scope of work and associated data validation protocols.

5.1.3 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP. SPCC and CCC performance evaluation or minimum mean RRF performance are not required for second source calibration verification standards.

5.1.4 Instrument Continuing Calibration

Continuing calibration standards must be analyzed immediately after an acceptable MS tuning has been performed. Continuing calibration standards are reviewed for SPCC, CCC, and target analyte performance in a manner similar to the evaluation performed for initial calibrations. SPCCs must meet method-specified continuing calibration RRF criteria and CCCs must meet method-specified percent difference (%D) criteria for methods 8260B and 8270C. Target analyte RRFs must meet criteria for methods 8260C and 8270D and for the SIM modifications to this method. Target analytes are evaluated against the target analyte criterion of no greater than 20 percent, and some QAPPs may also require that target compounds also meet minimum continuing calibration RRF criteria.

Some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias.

QSM version 5.0 introduced the requirement that GC/MS analyses to be bracketed by an end-of-sequence CCV, also known as a closing CCV. The first CCV standard analyzed after project sample analyses in a sequence is considered the ending CCV associated with those samples, even if there are additional CCVs analyzed later in the sequence. If samples are analyzed in a continuous sequence extending over more than 12 hours and involving multiple tunes and opening CCV standards, it is acceptable to consider each opening CCV to be the closing CCV for the preceding samples. Closing CCVs are required to have a %D requirement less than 50% for each target analyte. SPCC, CCC, and minimum target analyte RRFs do not need to be reviewed for closing CCVs.

5.1.5 Internal Standards

Internal standard compounds must be spiked into every sample, standard, and blank analyzed by GC/MS methods. Internal standards must meet the method area and retention time criteria for peak area and retention time. Older versions of the DoD QSM required that the peak area for each internal standard compound must be no less than 50 percent and no greater than 200 percent of the peak area for that internal standard compound in the midpoint standard in the associated initial calibration sequence. The retention time for each internal standard must be within 10 seconds of

the retention time of the midpoint standard in the associated initial calibration sequence. While this requirement was retained in DoD QSM version 5.1, this version of the QSM (and subsequent versions) expanded the internal standard acceptance criteria to allow for the daily initial CCV to be used for peak area and retention time comparison on days when initial calibration is not performed.

Discrepancies in internal standard performance are generally associated with the matrix characteristics of individual samples. Although internal standard discrepancies are not usually indicative of an instrument issue, the QSM presents a requirement for the laboratory to include an evaluation of the analytical system when assessing the potential causes and corrective action for internal standard discrepancies, as there are potential systematic issues that can also lead to poor internal standard performance. Internal standard discrepancies should always be associated with a corrective action by the laboratory, which will usually consist of re-extraction and reanalysis of the affected samples or perform instrument maintenance and recalibration if the internal standard discrepancies are attributable to an issue with the analytical system and not sample specific. The only exception is if the internal standards that exhibit discrepancies are not associated with any target analytes.

Each internal standard is associated with a specific set of analytes. When internal standards are out of control, only the associated target analytes are qualified in the affected sample. Many formats of initial calibration summary forms are organized to show the internal standard associations. If the internal standard associations are not shown on the initial calibration summary or other form, the validator should contact the laboratory to have the required information transmitted.

5.2 GC AND HPLC ORGANICS

GC and high-performance liquid chromatography (HPLC) organics include analyses for pesticides (organochlorine and organophosphorus), PCBs, explosives, herbicides, and petroleum products. GC and HPLC analyses use dual columns or dual detectors to identify target analytes. Some laboratories assign the same quantitative significance to both columns/detectors, while others specify a dedicated primary and secondary column/detector. If presented, the QC data for both the primary and secondary column/detector should be evaluated. In cases where instrument QC discrepancies affect one column/detector and not the other, some degree of interpretation by the validator is required to determine the effect on the associated samples. If the detector or column used to report the result for each analyte in a sample can be determined, discrepancies reported from other columns or detectors that were not used to report the results should not be used to qualify results.

5.2.1 Instrument Initial Calibration

As with GC/MS methods, initial calibrations must include at least five calibration points for calibration to response factor. Six calibration points are required for linear calibration and seven

calibration data points are required for quadratic calibration. Initial calibration to response factor is required to meet the method-specific requirement, which is usually a %RSD no greater than 15 percent or 20 percent.

The analysis of PCBs only requires multipoint calibration for PCB-1016 and PCB-1260, with single point calibration for all other reported PCB congeners. PCBs are quantified using five characteristic peaks. The *mean* %RSD of the PCB-1016 peaks and the mean %RSD of the PCB-1260 peaks are compared to the acceptance criteria. Individual characteristic peaks may exceed the %RSD criterion so long as the mean %RSD for each congener is acceptable. Discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260. If PCBs other than 1016 or 1260 are identified in any associated sample, the laboratory should perform a multipoint calibration for all identified congeners and reanalyze the samples to quantify the detected congeners. These reanalyses should be accompanied by all other QC elements spiked with the specific detected PCBs and not with the representative PCB-1016/1260 mixture.

5.2.2 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP.

Because of the existence of multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs second source calibration verifications are spiked with a mixture of PCB-1016 and PCB-1260. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

5.2.3 Instrument Continuing Calibration

GC and HPLC methods require a continuing calibration standard to be analyzed at the beginning of each analytical sequence, at regular intervals after a specified number of sample analyses (generally 10), and at the end of the end of the analytical sequence. Each continuing calibration standard is associated with all samples analyzed after the previous continuing calibration standard analysis and before the following continuing calibration standard analysis. Discrepancies in continuing calibration standard analyses will require evaluation of the affected analytes in the associated samples.

As a result of the existence of multiple overlapping peaks in the spectrum of each individual PCB congener, PCBs continuing calibration verification standards are spiked with a mixture of PCB-1016 and PCB-1260. Generally, discrepancies shown by PCB-1016 are considered to affect PCBs

1016, 1221, and 1232; and discrepancies shown by PCB-1260 are considered to affect PCBs 1242, 1248, 1254, and 1260.

Note that some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias.

5.2.4 Degradation Summary

Analysis for organochlorine pesticides requires that a 4,4′-dichlorodiphenyltrichloroethane (DDT) and endrin degradation standard be measured before samples are analyzed and at the beginning of each 12-hour shift. These compounds are easily degraded at the injection port. Generally, the acceptance criterion is that neither DDT nor endrin should have a breakdown of greater than 15 percent. Unacceptable DDT breakdown will cause the qualification of all associated DDT, 4,4′-dichlorodiphenyldichloroethene, and 4,4′-dichlorodiphenyldichloroethane results. Unacceptable endrin breakdown will cause the qualification of all associated endrin, endrin aldehyde, and endrin ketone results. However, this test should be performed as a test of the inertness of the analytical system even when DDT and endrin are not target analytes for a given project, unless otherwise specified in the QAPP.

5.2.5 Retention Times

There are no standardized summary forms for reporting chromatographic retention times, and each laboratory's forms will vary greatly in both format and content. In general, the validator should review all available retention time data. Retention time shifts, either in calibration standards or in sample results, must be accompanied by analyst documentation for the associated results to be accepted.

5.2.6 Confirmation

GC and HPLC methods require confirmation (except for petroleum hydrocarbon analysis) to differentiate target analytes from matrix interferences. Detected results are confirmed either by a second detector or by retention time on a second column that has different chemical properties than the primary column. Target analytes detected on one column/detector that are not confirmed are potentially interferences rather than a true detection. Such results should not be reported as detections by the laboratory unless the analyst and section leader provide documentation as to why the analytes should be considered detected in the absence of confirmation. Results that are detected and confirmed should have approximately the same quantitation on both columns/detectors; results that do not meet RPD criteria should be qualified as estimated.

5.3 METALS

Metals analyses are performed using SW-846 methods 6010C or D (inductively coupled plasma-atomic emission spectroscopy [ICP-AES]) and 6020A or B (inductively coupled plasma-mass spectrometry [ICP-MS]) for "full list" metals; cold vapor atomic absorption (CVAA) methods 7470A and 7471B for mercury in water and soil, respectively. Graphite furnace atomic absorption (GFAA) method 7010 can be used for select metals that can be affected by spectral interferences that prevent definitive analysis by ICP-AES; however, with improvements to ICP-AES and the emergence of ICP-MS as the metals method of choice, GFAA analysis is now rarely used.

5.3.1 Instrument Tuning

Methods 6020A and B use a mass spectrometer to identify target elements; the mass spectrometer must be tuned prior to use. Instrument tuning data is not always available on summary forms. If the required data is not available for review on summary forms, the data validator should contact the laboratory to request the required information. If the information is not available on summary forms, the raw data must be examined.

The QSM requires that tuning peaks show a resolution of no greater than 0.9 atomic mass units (amu) at 10 percent peak height. Some instrumental systems report the peak resolution at 5 percent of total peak height; this is more stringent than the QSM requirement and should not be considered a discrepancy provided that the resolution criterion of ≤0.9 amu is met.

5.3.2 Internal Standards

Methods 6020A and B use internal standards in the quantification of target elements. If an internal standard does not meet acceptance criteria and corrective action was not performed or was not successful, the target analytes associated with that internal standard should be qualified in the affected sample.

In some cases (especially with short analyte lists), there may be internal standards that do not meet acceptance limits but are not associated with target metals. Some laboratories also will choose a secondary internal standard to quantify a metal if the primary internal standard does not meet acceptance criteria.

5.3.3 Initial Multipoint Calibration

Initial multipoint calibration is required for CVAA and GFAA methods. It is not required for ICP-AES or ICP-MS analyses and there are QC elements described below that are intended to be performed instead of initial multipoint calibration; however, if a multipoint initial calibration is performed, it must meet the acceptance criteria in the QAPP. If the alternative QC checks are acceptable but the multipoint initial calibration was out of control, the associated results must be considered for qualification. The laboratory should not present such a situation as being in control.

5.3.4 Low-Level Calibration Verification

Low-level calibration verification standards at or below each target compound LOQ are required under projects with QC requirements from the QSM. This QC check should be performed for ICP-AES and ICP-MS methods regardless of whether an initial multipoint calibration is performed. Note that the DoD QSM requires that this check meet control limits of 80 to 120 percent even though the methods allow a window of 70 to 130 percent.

Some laboratories also perform what is called a CRDL check standard. This CRDL check standard is generally spiked at 2 times the LOQ. If the low-level calibration verification standard does not meet acceptance criteria, the usual response is to qualify detections with concentrations up to 10 times the LOQ and nondetections. However, if a low-level calibration verification does not meet acceptance criteria and an associated CRDL check standard is performed and is in control, stability at 2 times the LOQ has been demonstrated and only detected results up to 2 times the LOQ and nondetections require qualification.

5.3.5 High-Level Calibration Verification

High-level calibration verification standards are used to determine the upper end of the working range of the instrument. If the high-level calibration verification standard does not meet acceptance criteria, the validator should determine if a multipoint initial calibration has been performed. If so, and the high point on the calibrated curve has a concentration below that of the high-level calibration verification standard, only results above the high point on the curve (adjusted for matrix as necessary) require qualification.

Detected results above the high-level calibration verification should be qualified unless the laboratory performed appropriate dilutions so that the effective concentration measured by the instrument is less than the high-level calibration verification standard concentration.

5.3.6 Initial and Continuing Calibration Verification

Most laboratories use initial calibration verification (ICV) standard analyses as a second source verification check. HGL's preferred convention is to associate ICV results with all sample results in an analytical sequence and to the associated continuing CCV results only with sample results "bracketed" by a given CCV. A result is considered bracketed by a CCV if that CCV is the last CCV analyzed before that result was generated or is the first CCV analyzed after that result is generated.

More recent versions of Methods 6010 and 6020 include the analysis of low-level ICVs and CCVs. The QSM does not provide control limits for these low-level standards and HGL uses general acceptance criteria of 70-130 percent. If the project laboratory uses the low-level ICV as the DoD-

required low-level calibration verification standard (see Section 5.3.5), then the low-level ICV is required to meet the DoD acceptance criteria of 80-120 percent.

It is allowable to evaluate ICV/CCV results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable if the ICV or CCVs are from the same source as the initial calibration; however, if the ICV and/or CCVs are from a second source, the associated results should be considered for qualification.

5.3.7 Continuing Calibration Blanks

Continuing calibration blanks (CCBs), including initial calibration blanks (ICBs), are performed for inorganic methods. CCBs are evaluated like method blanks (Section 4.9). HGL's preferred convention is to associate ICB results with all sample results in an analytical sequence and to associated CCB results only with sample results bracketed by a given CCB. A result is considered bracketed by a CCB if that CCB is the last CCB analyzed before that result was generated or is the first CCB analyzed after that result is generated.

CCBs are aqueous but can be associated with both aqueous and solid matrix analyses. When determining the potential effect of CCB contamination on the associated solid matrix sample results, convert the CCB result to an equivalent soil concentration using the procedure presented for field blanks (Section 4.10.3).

The artifact threshold associated with field blank contamination is 5 times the concentration detected in the blank (10 times the concentration in the case of common laboratory contaminants). As with action levels associated with method blank contamination, both aqueous and solid-equivalent artifact levels should be adjusted on a sample-specific basis to account for sample-specific variables. In most cases, it will be clear that a result is above or below an action level and in practice this sample-specific adjustment is necessary for a minority of comparisons.

5.3.8 Interference Check Sample Results

Interference check samples (ICSs) are analyzed in pairs. ICS A (ICSA) is a blank spiked with high concentrations of aluminum, calcium, iron, and magnesium; in some cases, ICSAs will also be spiked with lower concentrations of other elements that are also potentially interfering. ICS AB (ICSAB) is spiked with the same levels of aluminum, calcium, iron, and magnesium as is the ICSA and contains lower spiked levels of the elements of concern. The purpose of analyzing ICSAs is to determine if interelement correction factors from naturally occurring elements that are often present at high concentrations cause false positive or false negative results due to over- or under-correction. The purpose of analyzing ICSABs is to determine if interelement correction factors for all elements, including those that occur at high concentrations naturally, are being applied correctly and provide correct quantitation. Generally, QAPPs will require a single ICSA and ICSAB be analyzed before sample analyses as a minimum requirement; however, if the laboratory reports

multiple ICSA/ICSAB results in an analytical sequence, the reviewer should evaluate the bracketing ICSA/ICSAB results both before and after the sample analyses and assign both sets equal significance.

According to QSM version 5.1, the ICSA acceptance criteria are a concentration with absolute value less than one-half the LOQ; however, note that QAPPs written in accordance with earlier versions of the QSM (through version 5.0) will present acceptance criteria of less than the LOD for target metals instead. ICSA discrepancies can be an indicator of problems with interelement correction. HGL has had experiences with false positive results ultimately traced to failure of the analytical system to take advantage of all mathematical tools available to correct for interferences. In cases where ICSA discrepancies are attributable to known contamination in the stock solution, this situation should be noted by the laboratory in the case narrative. In other cases, ICSA discrepancies can be attributed to instrument drift or system contamination. Indicators of this kind of issue will include positive or negative results in associated CCBs or method blanks. If ICSA discrepancies are potentially attributable to sources other than interelement interference, the reviewer should consider not qualifying the associated results or reducing the severity of qualification.

Most data validation conventions consider ICSA results with absolute value greater than the LOQ to constitute a severe discrepancy. If severe ICSA discrepancies are noted, the data reviewer should contact the HGL senior chemist before rejecting the associated results. ICSAs often contain higher levels of interfering element concentrations than are present in environmental samples, and alternatives to rejection may be available.

It is rare for ICSAB results to fail to meet control criteria, and often this is an indication of a spiking error rather than a problem with the analytical sequence.

5.3.9 Recovery Test Results

GFAA methods use recovery tests to determine if the sample matrix has affected reported results. The method requires a recovery test to be performed on a representative sample in each preparation batch, but in practice, laboratories perform recovery tests on a sample-specific basis.

5.3.10 Method of Standard Addition Results

The method of standard additions (MSA) is associated with GFAA analyses; this procedure is rarely performed as virtually all laboratories perform sample-specific recovery tests rather than batch-specific recovery tests. If MSA results are reported in a data package, the data validator should consult with the HGL senior chemist.

5.4 GENERAL CHEMISTRY

General chemistry parameters include a variety of analytical parameters and methodologies, including colorimetry, ion chromatography, GC, and infrared spectrometry. Usually, these parameters are secondary data that are used to determine the potential for a site to undergo monitored natural attenuation or the progress of monitored natural attenuation. Often, these tests will only require a Stage 2A data review; however, some parameters, such as cyanide, perchlorate, anions, or total organic carbon will, on occasion, require Stage 2B validation.

In many cases, the review of general chemistry QC parameters is similar to the review of the corresponding parameters for metals. Method-specific QC parameters should be discussed in the QAPP along with the acceptance criteria and qualification requirements. Some laboratories do not have summary forms for Stage 2B QC elements and the raw data will need to be examined by the validator to evaluate performance.

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ATTACHMENT D
Automated Data Review

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ATTACHMENT D
Automated Data Review

1.0 INTRODUCTION

The most common programs used to perform automated data review (ADR) are the web-based data validation functions provided by Environmental Synectics, Inc. (Synectics) of Sacramento, California, and the FUDSChem data validation and evaluation program developed by U.S. Department of Defense with Synectics. ADR programs identify quality control (QC) issues by comparing QC results in the laboratory-generated electronic data deliverable (EDD) against a data library generated in accordance with the requirements of the project Quality Assurance Project Plan (QAPP). This data library is often referred to as an electronic QAPP (eQAPP). ADR programs can streamline the data validation process by identifying QC issues and providing a listing of preliminary data qualification to be applied to the associated results; the extent of chemist review post-ADR will depend on project-specific requirements and objectives and on the EDD-generating capabilities of the laboratory.

2.0 ADR USES AND LIMITATIONS

ADR can reduce the amount of time spent reviewing laboratory data reports by generating a comprehensive list of QC discrepancies in a data package and identifying the associated affected results. ADR can be the primary data validation tool used for a project, integrated with only minimal "sanity check" review by a staff chemist, or it can be used as a tool to support manual data validation, relieving the validator from the task of reviewing each page of the laboratory data report and documenting all observed QC discrepancies.

ADR can support Stage 2A validation (as defined in Attachment A).

2.1 STAGE 2A REVIEW LIMITATIONS

ADR is not capable of evaluating the information in several critical areas of Stage 2A data review. In some cases, the QC element is not included in ADR. In other cases, ADR can perform an initial check of a QC element against the performance criteria but is not capable of incorporating additional sample- or method-specific information that is used to modify the initial evaluation. Following ADR, the ADR result should be reviewed by a staff chemist to ensure that all qualification applied by ADR is appropriate based on additional information not able to be evaluated by ADR.

2.1.1 Case Narrative

ADR cannot review any issues identified in the case narrative that may not be reflected in the associated QC data results. The case narrative should be examined by a chemist to ensure that

there are no additional issues that require corrective action, resolution, or qualification of the associated data.

2.1.2 Sample Delivery and Condition

ADR is capable of qualification based on sample temperature at receipt; however, it cannot evaluate other issues associated with sample delivery and condition, including broken bottles, misidentified samples, improper preservation, and bubbles greater than 6 millimeters noted in volatile organic compound sample vials. The staff chemist should review the chain of custody, the laboratory sample chronicle, and sample receipt documentation to verify that the samples were delivered to the laboratory in good condition, and properly identified.

2.1.3 Holding Times

Holding time can be evaluated by ADR. However, the holding time calculated from the time of collection on the chain of custody to the time of preparation or analysis at the laboratory can differ from the true holding time. This can be due to time zone differences between the sample location and the laboratory or a switch to or from daylight savings time occurring between the time of sampling and the time of preparation or analysis. The staff chemist should review the holding time calculations and ensure that these differences are accounted for.

Additionally, some projects require that the field teams assign "dummy" sample times to field duplicate samples to obscure the parent sample identity. The staff chemist should ensure that holding times for field duplicate samples have been calculated using the actual collection time and not an arbitrary collection time entered by the field sampling team.

In general, holding times longer than 72 hours are expressed in "days" and are evaluated to the nearest calendar day. The staff chemist should review any holding time discrepancies identified by ADR to determine if the affected analyses meet the holding time when evaluated against calendar days instead of the number of elapsed 24-hour periods. The Synectics ADR program is known to qualify samples based on 24-hour periods. This qualification may need to be corrected manually for those analyses with holding times expressed in days.

2.1.4 Surrogate Recoveries

Sample dilution can cause surrogate recovery discrepancies that are not associated with matrix interferences or analytical problems. When ADR identifies surrogate discrepancies in diluted samples, the staff chemist should review the affected data. Generally, data from sample analyses performed at dilution greater than fivefold should not be qualified for surrogate discrepancies unless a matrix effect is noted to have affected the sample even when analyzed under dilution. Most ADR programs can incorporate a dilution factor above which results will not be qualified for

surrogate discrepancies, and this maximum dilution factor should be identified on a method-specific basis in the eQAPP.

2.1.5 Matrix Spike/Matrix Spike Duplicate Recoveries

Matrix spike (MS)/matrix spike duplicate (MSD) recovery discrepancies are not considered to have significance if the native concentration of the affected analyte in the parent sample is more than four times the concentration resulting from the spike (see Section 4.7 of Attachment C). In some cases, the native concentration of one or more target analytes is so high that the MS/MSD will be analyzed under dilution. Discrepancies in diluted MS/MSDs are likely to be a result of dilution effects rather than matrix effects, as the majority of material in a diluted sample will consist of material not representative of the site (that is, it will be analyte-free laboratory water or solvent) and unlikely to contain interferences. In some cases, MS/MSDs are analyzed without dilution but with one or more spiked compounds quantitated above the calibrated range. Quantification of results above the calibrated range is inherently less reliable, and MS/MSD discrepancies can be caused by quantification errors.

Some ADR programs cannot take into account the "four times" rule, the effects of dilution, or the effects of results quantitated above the calibrated range when assigning qualifiers for MS/MSD discrepancies. The staff chemist should evaluate the MS/MSD percent recovery discrepancies identified by ADR and determine if these results are truly indicative of a matrix effect or are caused by other factors that eliminate the need for qualification of the associated results.

In some cases, the laboratory will report MS/MSD results from a different sample delivery group (SDG) as batch control; such batch control MS/MSDs are often presented without the client sample identification (ID). When a batch control MS/MSD is reported, the staff chemist should use the laboratory sample ID to confirm whether the MS/MSD is actually from a site sample reported in a different SDG or from a nonsite sample. If the MS/MSD is from a site sample, it will be considered applicable to associated results and any data qualification selected by ADR will be considered applicable. If the MS/MSD cannot be associated with a site sample, the results should be noted but no qualification should be applied unless the underlying cause of the discrepancy is suspected to be a problem with the analytical system.

Serial dilution and post-digestion spike (PDS) results are considered part of Stage 2A evaluation. These QC checks can be used to modify the qualifiers applied due to MS/MSD percent recovery (%R) discrepancies; however, these elements are not usually provided in laboratory EDDs. Where ADR applies qualifiers to metals results based on MS/MSD %R discrepancies, the validator should examine the serial dilution or PDS results in accordance with the QAPP validation guidelines to determine if those qualifiers should be eliminated or reduced in severity.

2.1.6 Matrix Spike/Matrix Spike Duplicate Precision

As described in Section 4.7 of Attachment C, some laboratories compare the concentrations detected in the MS and the MSD to calculate precision rather than comparing the percent recoveries. This convention can lead to the resulting relative percent differences (RPD) being an incorrect representation of the analyte-specific precision. If the expected concentration in the MS is different than the expected concentration in the MSD, calculation of the RPD using a direct comparison of the detected concentrations is not relevant. The staff chemist should verify that the RPDs reported for MS/MSD results are calculated using the percent recoveries or that the expected concentration in the MS is the same or reasonably similar to the expected concentration in the MSD. If the RPDs are calculated using noncomparable results, the validator should contact the laboratory and request that the calculations be performed using percent recoveries. If this information cannot be produced by the laboratory, the validator will have to perform these calculations.

2.1.7 Field and Laboratory Duplicate Precision

ADR evaluates the performance of field and laboratory duplicates based on the calculation of the RPD of the results for the parent sample and duplicate. However, some ADR programs will not evaluate duplicate performance considering the commonly used convention for "low-level" results, usually defined as results that are less than 5 times the quantitation limit. Under most data validation protocols, low-level results are evaluated by comparing the absolute difference between the parent and duplicate result to the associated quantitation limits (see Section 4.11 of Attachment C). If ADR is used without supplemental manual review, there is a potential for data to be over-qualified for field or laboratory duplicate discrepancies.

2.1.8 PCB Discrepancy Associations

As described in Sections 4.6 and 4.7 of Attachment C, laboratory control samples (LCS) and MS/MSDs for polychlorinated biphenyls (PCBs) analysis are spiked with only two representative PCB congeners. Discrepancies affecting PCB-1016 are also considered to affect results for PCBs 1221 and 1232, and discrepancies affecting PCB-1260 are also considered to affect results for PCBs 1242, 1248, and 1254. If the ADR program is not able to extend the association of a QC issue reported for one compound to other compounds in accordance with the QAPP, this situation will have to be addressed by the staff chemist.

2.1.9 Selection of Final Result

In cases where multiple analysis results are reported for a sample because of dilution or reanalysis, all analyses are reviewed by ADR. Based on the body of QC data, the staff chemist should select one definitive result for each analyte in each sample in accordance with Section 3.5 of Attachment C. All other results for that analyte in that sample should be denoted as superseded by applying an # qualifier to the qualifiers applied by ADR.

2.2 STAGE 2B REVIEW LIMITATIONS

The QC elements included in a Stage 2B data validation are limited by the specific capabilities of the selected ADR program and the laboratory's ability to supply an EDD that addresses these QC elements. When an ADR program is used to perform Stage 2B validation, the data validator must be aware of the limitations of the laboratory EDD and the ability of ADR to address situations where the data is not reported in the standard format (e.g., the evaluation of system performance check compounds that have been calibrated to a curve and do not have the associated mean relative response factor reported.

3.0 ELECTRONIC QAPP AND DATA LIBRARY

All ADR functions require reference to the project-specific data library that is assembled into an eQAPP. It is critical that the eQAPP be prepared and the associated data library transmitted to the laboratory before project sampling activities. If the data library has not been constructed at the time of sample analysis, the required information may not be captured in the laboratory EDD, resulting in the need to regenerate EDDs that conform to the data library requirements or late EDD delivery, causing delays and potentially increased laboratory costs.

The eQAPP should encompass the sensitivity limits, control limits, validation protocols, qualification conventions, and qualifier priorities that have been established in the project QAPP. The data library requires the input from a HydroGeoLogic, Inc. (HGL) project chemist and the laboratory database manager at a minimum. After the draft eQAPP has been prepared, all information contained in it must undergo a QC review against the requirements of the QAPP by an HGL chemist. Any discrepancies between the eQAPP and the QAPP must be resolved before the eQAPP can be used to support ADR.

3.1 SENSITIVITY LIMITS

There are two principal conventions for establishing sensitivity limits. Both are in common use and are described in Attachment C, Table C.1. ADR file formats can support either sensitivity limit convention, as specified in the project QAPP.

3.2 CONTROL LIMITS

The method- and matrix-specific control limits listed in the QAPP should be incorporated into the eQAPP. Control limits can be differentiated by QC element (such as LCS/LCS duplicates and MS/MSDs).

3.3 VALIDATION PROTOCOLS

The project-specific validation protocols are entered into the eQAPP using the Qualification Scheme application of the ADR program. The Qualification Scheme for a project must match the

procedures presented in the project QAPP. The Qualification Scheme allows for qualifiers to be assigned based on whether each affected result is a detection or a nondetection. The Qualification Scheme also allows for discriminating between minor discrepancies and major discrepancies that require results to be rejected, i.e., several QC elements allow the entry of both an estimation limit and a rejection limit for that element.

3.4 QUALIFICATION CONVENTIONS

The Qualification Scheme includes the project-specific qualifiers that will be applied to analytical results either as a result of quantification (for example, results below the quantitation limit) or as a result of a QC discrepancy. The eQAPP can specify on a method-specific basis whether some QC elements, such as MS/MSD results, affect the parent sample only or all samples in the associated preparation batch.

3.5 QUALIFIER PRIORITY

ADR includes a Qualifier Hierarchy matrix that allows for the determination of the final qualifier applied to each data point. The Qualifier Hierarchy matrix for some ADR programs only allows for the simultaneous evaluation of two qualifiers; if more than two qualifiers are potentially applicable to a sample result, ADR will evaluate only the two highest priority qualifiers as defined in the QAPP.

4.0 ADR LABORATORY DELIVERABLES

The primary ADR programs can process a staged EDD-formatted EDD. The specifications for providing data for FUDSChem are provided on the FUDSChem website: http://fudschem.com/public/framework/bannerhtml.aspx?dsn=systm&idhtml=10642&themesuffix=default&banner=banner_fudschem.jpg&idMenu=78296&ddlDSN=SYSTM&Title=HOME.

5.0 ADR PROCEDURES

At a minimum, each ADR EDD delivered by the laboratory will undergo a QC review upon receipt and QC sample associations will be added to the file. If additional manual review is required after the QC and association step, the procedures described in Sections 5.1 and 5.2 must be followed.

5.1 ADR FILE QC

On receipt from the laboratory, each set of EDD files should be reviewed to ensure that all required fields have been populated correctly and that all information is complete and correct. Following this QC check, the field QC sample results in the laboratory data package must be associated with the field sample results. This step includes associating trip blanks and equipment blanks with the

corresponding field samples and associating designated field duplicate samples and MS/MSDs with the corresponding parent samples.

5.2 SUPPLEMENTAL MANUAL REVIEW – STAGE 2A

Manual chemist review of Stage 2A QC elements should include the following elements, in accordance with the referenced guidance presented in Section 2.1 of Attachment D and the referenced sections of Attachment C:

- Case narrative (Section 4.1), including any associated sample discrepancy reports;
- Chain of custody (Section 4.2);
- Sample receipt and log-in forms (Section 4.3);
- Sample ID cross reference (Section 4.4);
- Association of Aroclors 1016 and 1260 QC discrepancies with additional Aroclors (Sections 4.6 and 4.7);
- Evaluation of any MS/MSD results potentially not relevant to sample results (Section 4.7); and
- Evaluation of any low-level field duplicate and laboratory duplicate comparisons (Section 4.11).

Any changes made to the ADR results based on manual review must be documented and undergo a peer review.

5.3 SUPPLEMENTAL MANUAL REVIEW – STAGE 2B

A manual chemist review of Stage 2B QC elements should verify that all required QC elements were validated by the ADR program with manual review and validation to address any identified gaps or special circumstances outside the capabilities of the ADR program.

Any changes made to the ADR results based on manual review must be documented and undergo a peer review.

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ATTACHMENT E
Data Qualification Reason Codes

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ATTACHMENT E
Data Qualification Reason Codes

QC Element	Reason Code	Definition
Ambient Blank	ABH	Ambient blank result ≥ limit of quantitation (LOQ)
Ambient Blank	ABHB	Result is judged to be biased high based on associated ambient blank result
Ambient Blank	ABL	Ambient blank result <LOQ
Analyte Quantitation	ACR	Result above the upper end of the calibrated range
Analyte Quantitation	EXC	Result excluded; another data point for this analyte was selected for use (use with X-qualified results)
Analyte Quantitation	RTW	Target analyte outside retention time window
Analyte Quantitation	PSL	Solid matrix sample with percent solids less than 50%
Analyte Quantitation	PSLX	Solid matrix sample with percent solids less than 10%
Analyte Quantitation	TR	Result between the detection limit and LOQ
Calibration Blank	CBH	Initial or continuing calibration blank result ≥LOQ
Calibration Blank	CBHB	Result is judged to be biased high based on associated continuing calibration blank result
Calibration Blank	CBL	Initial or continuing calibration blank result <LOQ
Calibration Blank	CBN	Negative initial or continuing calibration blank result with absolute value <LOQ
Calibration Blank	CBNH	Negative initial or continuing calibration blank result with absolute value ≥LOQ
Continuing Calibration	CCCC	Calibration check compound did not meet percent difference (%D) criterion in continuing calibration standard
Continuing Calibration	CCVD	Continuing calibration standard did not meet %D criterion
Continuing Calibration	CRFL	Continuing calibration RRF below acceptance criterion
Continuing Calibration	CSPC	System performance check compound did not meet minimum RRF criterion in continuing calibration
Continuing Calibration	CVDX	Continuing calibration standard did not meet %D criterion, extreme discrepancy
Confirmation	CF	Confirmation precision exceeded acceptance criterion
Cyanide Method	DSH	High-level distillation standard did not meet %D criterion
Cyanide Method	DSL	Low-level distillation standard did not meet %D criterion
Equipment Blank	EBH	Equipment blank result ≥LOQ
Equipment Blank	EBHB	Result is judged to be biased high based on associated equipment blank result
Equipment Blank	EBL	Equipment blank result <LOQ
Field Duplicate	FDPA	Field duplicate results did not meet absolute difference criterion
Field Duplicate	FDPR	Field duplicate results did not meet RPD criterion
Holding Time	HTA	Analytical holding time exceeded
Holding Time	HTAX	Analytical holding time exceeded, extreme discrepancy
Holding Time	HTP	Preparation holding time exceeded
Holding Time	HTPX	Preparation holding time exceeded, extreme discrepancy
Initial Calibration	ICCC	Calibration check compound did not meet percent relative standard deviation (%RSD) criterion in initial calibration

ATTACHMENT E (continued)
Data Qualification Reason Codes

QC Element	Reason Code	Definition
Initial Calibration	ICLS	Initial calibration low-level standard >LOQ
Initial Calibration	ICR2	Initial calibration r^2 below acceptance criterion
Initial Calibration	ICRD	Initial calibration %RSD above acceptance criterion
Initial Calibration	ICRX	Initial calibration %RSD above acceptance criterion, extreme discrepancy
Initial Calibration	IRFL	Initial calibration RRF below acceptance criterion
Initial Calibration	ISPC	System performance check compound did not meet minimum mean RRF criterion in initial calibration
Initial Calibration	LQSH	LOQ check standard above acceptance criteria
Initial Calibration	LQSL	LOQ check standard below acceptance criteria
Initial Calibration	SSVD	Second-source standard did not meet %D criterion
Initial Calibration Verification	ICVD	Continuing calibration standard did not meet %D criterion
Initial Calibration Verification	ICVX	Continuing calibration standard did not meet %D criterion, extreme discrepancy
Interference Check Standard	ICAH	Non-spiked concentration above acceptance criterion in ICSA
Interference Check Standard	ICAN	Negative concentration with absolute value above acceptance criterion in ICSA
Interference Check Standard	ICHX	Non-spiked concentration above acceptance criterion in ICSA, extreme discrepancy
Interference Check Standard	ICNX	Negative concentration with absolute value above acceptance criterion in ICSA, extreme discrepancy
Interference Check Standard	ICSH	ICSA or ICSAB spiked analyte with high percent recovery (%R)
Interference Check Standard	ICSL	ICSA or ICSAB spiked analyte with low %R
Internal Standards	IRH	Internal standard peak area above upper limit
Internal Standards	IRL	Internal standard peak area below lower limit
Internal Standards	IRLX	Internal standard peak area below lower limit, extreme discrepancy
Internal Standards	ISRT	Internal standard retention time outside window
Labeled Standards	LSH	Labeled standard %R above acceptance criterion
Labeled Standards	LSL	Labeled standard %R below acceptance criterion
Labeled Standards	LSLX	Labeled standard %R below acceptance criterion, extreme discrepancy
Laboratory Control Sample	LCLX	LCS and/or LCSD %R below acceptance criterion, extreme discrepancy
Laboratory Control Sample	LCSH	LCS and/or LCSD %R above acceptance criterion
Laboratory Control Sample	LCSL	LCS and/or LCSD %R below acceptance criterion
Laboratory Control Sample	LCSP	LCS/LCSD RPD above acceptance criterion
Laboratory Duplicate	LDPA	Laboratory duplicate results did not meet absolute difference criterion
Laboratory Duplicate	LDPR	Laboratory duplicate results did not meet RPD criterion

QC Element	Reason Code	Definition
Low-Level Calibration Check	LLCH	Low-level calibration check above the upper limit
Low-Level Calibration Check	LLCL	Low-level calibration check below the lower limit
Low-Level Calibration Check	LLXL	Low-level calibration check below the lower limit, extreme discrepancy
Method Blank	MBH	Method blank result ≥LOQ
Method Blank	MBHB	Result is judged to be biased high based on associated method blank result
Method Blank	MBL	Method blank result <LOQ
Matrix Spike	MSH	MS and/or MSD %R above acceptance criterion
Matrix Spike	MSL	MS and/or MSD %R below acceptance criterion
Matrix Spike	MSLX	MS and/or MSD %R below acceptance criterion, extreme discrepancy
Matrix Spike	MSP	MS/MSD RPD above acceptance criterion
Post-Digestion Spike	PDH	Post-digestion spike recovery high
Post-Digestion Spike	PDL	Post-digestion spike recovery low
Post-Digestion Spike	PDLX	Post-digestion spike recovery low, extreme discrepancy
Post-Digestion Spike	PDN	Post-digestion spike not performed or not applicable and serial dilution result not performed or not applicable
Sample Delivery and Condition	BUB	Bubbles >5 millimeters in volatile organic compounds vial
Sample Delivery and Condition	DAM	Sample container damaged
Sample Delivery and Condition	PRE	Sample not properly preserved
Sample Delivery and Condition	TEMP	Sample received at elevated temperature
Sample Delivery and Condition	TMPX	Sample received at elevated temperature, extreme discrepancy
Serial Dilution	SDIL	Serial dilution did not meet %D criterion
Serial Dilution	SDN	Serial dilution not performed
Surrogate	SSH	Surrogate %R high
Surrogate	SSL	Surrogate %R low
Surrogate	SSLX	Surrogate %R low, extreme discrepancy
Surrogate	SSN	Surrogate compound not spiked into sample
Trip Blank	TBH	Trip blank result ≥LOQ
Trip Blank	TBL	Trip blank result <LOQ
Validator Judgment	VJ	Validator judgment (see validation narrative)

ICS = interference check sample
MS = matrix spike
MSD = matrix spike duplicate
QC = quality control
RPD = relative percent difference
RRF = relative response factor

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ATTACHMENT F
Review of Subcontracted Data Validation Reports

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ATTACHMENT F
Review of Subcontracted Data Validation Reports

1.0 INTRODUCTION

The goal of subcontracted data validation is to generate a validated project dataset that is qualified in accordance with Quality Assurance Project Plan (QAPP) requirements and ready for HydroGeoLogic, Inc. (HGL) to upload into the project database, and to do so at a cost savings to HGL's projects. Subcontracted data validation will be performed in accordance with the individual firm's internal procedures and policies; however, the overall procedure must include prereview, validation by qualified personnel, and peer or senior review of all data validation reports before delivery to HGL. All validation should be performed in accordance with the project QAPP and the scope of work provided by HGL.

Note that the guidance presented in this Attachment assumes that the project QAPP presents validation and qualification criteria based on the quality control (QC) requirements of the U.S. Department of Defense (DoD) Quality Systems Manual (QSM) version 5.3. Although a majority of project QAPPs will reference QSM version 5.3 or the similar requirements of QSM versions 5.1 or 5.2, there are still older QAPPs in use that have the data qualification protocols based on the QC requirements of DoD QSM version 4.2 or 5.0. If the guidance presented in this Attachment conflicts with the project QAPP qualification protocols, the requirements of the project QAPP should always take precedence.

2.0 DELIVERABLES

2.1 SUBCONTRACTED DATA VALIDATOR

Subcontracted data validators will deliver data validation reports to HGL. These reports may be in the validation firm's internally derived format; however, HGL prefers that an individual report be prepared for each sample delivery group (SDG) and analytical method within that SDG (although "bundling" methods for metals and wet chemistry parameters is acceptable, in the same fashion as HGL's internally produced data validation reports). Each report should include a summary of every QC element evaluated by the data validator, an identification of discrepancies, the qualification required by this discrepancy, and an identification of the associated samples. Subcontracted data validation reports are required to include a summary of all qualified data. This summary can be provided as a table of qualified results, as a listing of qualifiers assigned by QC element, or as copies of data reporting forms with validation qualifiers applied by hand.

In most cases, the subcontracted validator will also be responsible for providing qualified data electronically in a format that allows upload into HGL's project database (see Section 6.0 of the standard operating procedure [SOP]), usually in the form of an Excel file. The validation firm will

be responsible for data entry, data entry QC, and removal of any residual laboratory-applied flags before delivery to HGL.

2.2 HGL REVIEWER

The HGL reviewer should prepare a review report to document the findings of the review of each subcontracted data validation report. This review should include a discussion of any discrepancies noted in the data validation report, any follow-up communications with the data validator or the laboratory, and any changes to the final data qualifiers assigned by the validator (including qualifiers applied by the laboratory and accepted as the final qualifier by the laboratory). The HGL reviewer is also responsible for ensuring that any HGL modifications to the validator's data qualifiers and other fields applicable to the validation process (including the HGL Value, HGL Qual, Detected, Report Usability, and HGLReason Code fields) are correctly incorporated into the 100 percent QC Excel file generated by the project database and transmitted to the project's database administrator. The HGL reviewer should at a minimum indicate any changes made to the 100 percent QC Excel file by color coding any affected cells. An example of an HGL data validation review report is presented as Attachment F.1.

3.0 INITIAL HGL REVIEW

The initial data validation reports provided by the contractor should be reviewed in-depth by an HGL senior chemist as soon as possible to provide the data validator with timely feedback to guide ongoing validation efforts. Promptly alerting the data validators to any discrepancies allows for data validator to issue correct reports rather than reissuing revised reports. Performing and in-depth review will assist in identifying areas where the data validation contractor's interpretation of QC elements differs from the requirements of the QAPP.

This review should mimic HGL's peer review of an internally generated data validation report (see Section 3.4 of the SOP), including a re-examination of the laboratory data package to verify that no QC discrepancies have been overlooked by the validator. The most common cause for a QC element being overlooked or misinterpreted by the data validator is unfamiliarity with the specific requirements of the project QAPP, which should supersede any corporate validation conventions in place at the validation firm.

4.0 GENERAL HGL REVIEW GUIDELINES

The following are the general guidelines for reviewing data validation reports from subcontracted validators.

4.1 REPORT DETAIL

When conducting data validation, HGL's practice is to identify and discuss all QC discrepancies associated with an analytical fraction, whether those QC discrepancies cause data to be qualified or not. Data validation subcontractors and individual validators vary in the amount of detail that is provided in the report narrative, especially if no corresponding results require qualification. The HGL reviewer should be alert to cases where the validator has indicated no discrepancies for a QC element when, in fact, there were discrepancies, but no qualification is required or no project sample results are associated with that specific discrepancy. Many validation firms provide a checklist with the text of the validation report. If such a checklist is available for review, it should be compared to the report text to check if there are QC discrepancies noted that are not discussed in the report because no qualification was required. This comparison can also assist in verifying that the validation report does not contain any "template" errors.

4.2 APPLICATION OF FINAL QUALIFIERS

In all cases, the final qualifier applied by the data validator must be an allowable project qualifier. When more than one qualifier is applicable to a result, the final qualifier must have been assigned in accordance with the priority of qualifiers presented in the QAPP.

The HGL reviewer should examine the qualified electronic file to ensure that all the validator-applied qualifiers are allowable under the project QAPP and that there are no changes to laboratory qualifiers that do not make sense. For instance, if a laboratory qualifier is U and the final qualifier is B, the HGL reviewer should suspect that the B qualifier is in error and determine the correct final qualifier that should be applied.

5.0 REVIEW OF STAGE 2A DATA VALIDATION ELEMENTS

The HGL reviewer should examine the following elements of each data validation report. The common discrepancies associated with each QC element are also discussed in the following subsections.

5.1 SAMPLE RECEIPT AND DELIVERY

The HGL reviewer should review the validation report and verify that any qualification is performed in accordance with the QAPP.

5.2 HOLDING TIMES

The holding times for preparation and analysis for each analytical method should be presented in the project QAPP. The validator should have used the QAPP conventions for evaluating holding times or provide justification (such as nominal exceedance) for not qualifying results that are associated with holding time exceedances. The validator should have considered any time zone

differences, daylight savings time changes, or "dummy" sample collection times (such as on field duplicates) when evaluating short (≤72 hour) holding times.

5.3 LCS/LCSD RECOVERIES AND PRECISION

Laboratory control sample (LCS) (and laboratory control sample duplicate [LCSD]) recoveries greater than the control limits should not cause qualification of nondetected results unless there is a gross exceedance that is evidence of a problem with the analytical system.

LCS/LCSD relative percent difference (RPD) exceedances should not cause qualification of nondetected results.

Discrepancies shown by polychlorinated biphenyl (PCB)-1016 should be considered to affect PCBs 1016, 1221, and 1232; and discrepancies shown by PCB-1260 should be considered to affect PCBs 1242, 1248, 1254, and 1260. The validator should have taken this convention into account when applying qualifiers.

Some QAPP data validation protocols establish a two-tiered approach for evaluating LCSs. The HGL reviewer should verify that the validator distinguished between routine and extremely low percent recoveries (%Rs) when applying qualifiers to the associated results.

5.4 MS/MSD RECOVERIES AND PRECISION

The issues applying to LCS (and LCSD) performance also apply to matrix spike (MS)/matrix spike duplicates (MSDs). There are additional issues that affect the evaluation of MS/MSDs.

The association of MS/MSD results to project samples varies by method and by project. Ensure that any identified MS/MSD discrepancies are associated correctly.

Ensure that no qualification of project samples is performed based on discrepancies found in nonsite samples unless the validator has provided an appropriate rationale.

Ensure that no qualification has been performed based on MS/MSD %R discrepancies identified for analytes that are present in the parent sample at greater than 4 times the spiked concentration.

Ensure that project samples from other SDGs that were reported as batch control MS/MSDs were properly identified as project samples and used to qualify project data.

Verify that the RPDs reported for MS/MSD results are calculated using the percent recoveries or that the expected concentration in the MS is comparable to the expected concentration in the MSD. If the RPDs are calculated using non-comparable results (different spiked concentrations in the MS and MSD), the validator should have noted this in the evaluation of the RPDs. Note that it may

be justifiable to assign qualifiers based on MS/MSD RPD discrepancies even if MS/MSD recoveries are affected by the "4 times" rule.

Where there are MS/MSD %R discrepancies affecting metals results from methods 6010 or 6020, the laboratory should perform a serial dilution or post-digestion spike (PDS) using the same parent sample, whether the "4x rule" applies to the discrepancy (see Section 5.5).

On occasion, the laboratory will select a member of a field duplicate pair to perform MS/MSD analyses. For organics, the general convention is to qualify only the MS/MSD parent sample for when MS/MSD discrepancies are noted. If an MS/MSD is performed on one of the members of a duplicate pair, however, the MS/MSD results are applicable to both members of the pair, and the HGL reviewer should verify that both samples were qualified.

5.5 SERIAL DILUTIONS AND POST-DIGESTION SPIKES

The use of serial dilution and post-digestion spike results varies depending on when the QAPP was written. The current guidance used in HGL QAPPs follows, but the specific QAPP requirements should be used to evaluate these QC elements.

When a metals MS/MSD analysis shows %R discrepancies, the laboratory should perform a serial dilution and PDS on the MS/MSD parent sample. Serial dilution and PDS results should only be used to modify the qualifiers applied due to MS/MSD %R discrepancies in accordance with the qualification protocols presented in the project QAPP. If the MS/MSD %R is in control for a metal; qualification should not be applied for serial dilution or PDS discrepancies associated with acceptable MS/MSD %R results.

Serial dilution results are applicable to analytes that are present at ≥50 times the limit of quantitation (LOQ) in the MS/MSD parent sample, and PDS results are applicable to analytes that are presented at <50 times the LOQ in the MS/MSD parent sample. The "4x rule" that is used for MS/MSD results is also applicable to PDS results, so there may be situations where a parent sample concentration for a metal is high enough that MS/MSD and PDS results cannot be used to qualify the associated samples, but the concentration below the threshold for using serial dilution results. In these cases, the validators should use judgment to evaluate whether matrix effects are suspected. If the serial dilution results are in control and the parent sample concentration is greater than the LOQ, the serial dilution results can be used as corroborating evidence that there is no matrix effect, even if the concentration is below the ≥50 times the LOQ threshold.

The HGL reviewer should evaluate the validation narrative and verify that serial dilutions and PDSs were evaluated in accordance with QAPP criteria.

If the laboratory performed neither a serial dilution nor a PDS using a project sample, then matrix effects cannot be ruled out. The validator should have reviewed available MS/MSD data, site

results reported from other data packages, and the case narrative and determine whether qualification is necessary.

5.6 METHOD BLANKS

The evaluation of laboratory blank results is one of the few QC elements where the results can meet acceptance requirements for reporting data (instead of performing corrective action), but the associated results will still be qualified. HGL often sets acceptance criteria for laboratory blanks using the QSM criteria, which are "No analytes detected > ½ LOQ (>LOQ for common laboratory contaminants) or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater." These acceptance criteria are the thresholds above which the laboratory should take corrective action and evaluate the need to reanalyze any affected samples. However, HGL's convention is that any contamination detected in laboratory blanks at or above the associated detection limit (DL) must be used to establish an artifact threshold and qualify associated results below that threshold. This qualification must be applied whether the associated blank result is above the acceptance criterion or below it.

This division between acceptance criteria and qualification criteria is a common source of error in subcontracted evaluation of laboratory blanks. The HGL review must ensure that the validator has evaluated all blank results at or above the DL and applied qualification in accordance with the validation conventions. For metals, this will also include the evaluation of blanks with negative concentrations that have an absolute value greater than the DL.

5.7 FIELD BLANKS

Field blanks are evaluated in a similar manner as method blanks (Section 5.5). Two main differences are (1) the artifact threshold calculated from concentrations in field blanks is *not* adjusted for sample-specific factors; and (2) most field blanks are aqueous and conversion to equivalent solid units is not straightforward for some analytical methods.

Ensure that the data validator correctly calculated the artifact threshold and made any corrections for conversion from water to soil units.

5.8 FIELD DUPLICATE PRECISION

Ensure that the appropriate criterion, absolute difference for low-level results of RPD for high-level results, was used to evaluate each set of duplicate results, as specified in the QAPP.

The association of field duplicate results to project samples beyond the parent sample varies by method and by project. Ensure that any identified field duplicate discrepancies are associated correctly.

5.9 SURROGATE RECOVERIES

The HGL reviewer should examine any results qualified as a result of surrogate discrepancies noted in diluted samples. Generally, qualification should not be applied for surrogate discrepancies if the sample dilution factor was greater than 5 and the surrogates were added prior to dilution.

5.10 METHOD-SPECIFIC QC CHECKS

Method-specific QC elements include such checks as pH buffer checks, cyanide distillation standards, synthetic precipitation leaching procedure extraction blanks, and replicate precision for total organic carbon. If these checks are reported in a Stage 2A data package, the validator should review these items. If the review guidelines are not included in the QAPP, the validator should consult with the project chemist to develop a review and qualification approach.

6.0 REVIEW OF STAGE 2B DATA VALIDATION ELEMENTS

Stage 2B QC elements are specific to individual analytical methods.

6.1 GC/MS ORGANICS

Gas chromatography (GC)/mass spectrometry (MS) organics include analyses for volatile organic compounds (VOCs) and for semivolatile organic compounds (SVOCs), most commonly by SW-846 methods 8260B or 8260C and 8270D, respectively.

6.1.1 Instrument Tuning

It is rare for a laboratory data package to include mass spectrometer tuning discrepancies. Data validation reports for this QC element will rarely include more than a statement that tuning frequencies and results were acceptable.

6.1.2 Instrument Initial Calibration

A common source of error in subcontracted data validation reports is the confusion between instrument performance criteria for Method 8260B (and SVOCs method 8270C, which is now infrequently performed) and target compound performance criteria in the evaluation of initial calibration data. Subcontracted data validation reports should note that the following QC elements were reviewed, along with any noted discrepancies:

- System performance check compounds (SPCCs) evaluated against analyte-specific mean relative response factor (RRF)
- Calibration check compound (CCCs) evaluated against percent relative standard deviation (%RSD) of 30 percent

- Target analytes (including CCCs that are also target analytes) evaluated against %RSD of 15 percent (20% for analysis by 8270-SIM) or r^2 of 0.99

The failure of an SPCC or CCC to meet *the SPCC- or CCC-specific criteria* constitutes a failure of the entire calibration and can cause rejection of all associated results; whereas the failure of a target compound to meet the linearity criterion constitutes a failure for only that target compound and causes less severe qualification. In some cases, a CCC can pass the CCC criterion but fail the target analyte criterion. The reverse can also be true.

> *Example*: Method 8260B CCC vinyl chloride is reported calibrated to a mean RRF with %RSD of 17.5 percent. The requirement for VOCs CCCs is that each has a %RSD of no greater than 30 percent. Vinyl chloride shows acceptable performance as a CCC; however, the target analyte criterion is for %RSD to be no greater than 15 percent. Vinyl chloride does not meet the acceptance criterion for target analytes. The effects, if any, of this discrepancy would be considered to affect vinyl chloride alone and not to be indicative of an instrument performance issue.

> *Example*: Method 8270C CCC di-n-octyl phthalate is reported calibrated to a mean RRF with %RSD of 31.2 percent, but the laboratory elected to fit the calibration sequence to a curve with an r^2 of 0.996. The requirement for SVOCs CCCs is that each has a %RSD of no greater than 30 percent. Even though a r^2 of 0.996 meets the acceptance criterion for a target analyte, this CCC does not meet the acceptance criterion of %RSD $\leq$30 percent. Although mean RRF is not used as the calibration relationship for this compound, the laboratory should have performed corrective action in this case.

Some QAPPs include a requirement that target analytes also be evaluated against analyte-specific mean RRF requirements. This should only be done if included as a QAPP requirement, such as for Methods 8260C and 8270D and the selected ion monitoring (SIM) modifications to these methods; if the data validator has qualified data based on target compound mean RRF when not required by the QAPP, the data validation reports should be revised to remove this extraneous qualification.

6.1.3 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP. SPCC and CCC performance evaluation is not required for second source calibration verification standards.

6.1.4 Instrument Continuing Calibration

The data validator should have evaluated continuing calibration verification (CCV) standards for SPCC, CCC, and target analyte performance in a manner similar to the evaluation performed for initial calibrations. The data validation report should note that the SPCCs met method-specified continuing calibration RRF criteria and CCCs met method-specified percent difference (%D)

criteria. For GC/MS methods, CCV standards performed at the end of the analytical sequence are only required to meet the %D requirement for target analytes; SPCC, CCC, and minimum target analyte RRF performance evaluation is not required for ending CCVs.

Target analytes are evaluated against the target analyte criterion of no greater than 20 percent. Some QAPPs may also require that target compounds also meet minimum continuing calibration RRF criteria in the opening CCV standards, such as for Methods 8260C and 8270D and the SIM modifications to these methods. If the QAPP does not require the evaluation of target compound RRFs, the data validation report should not use this QC element to assign qualifiers to target analyte data.

Note that some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias. The data validation report should not use the direction of bias when evaluating continuing calibration results.

6.1.5 GC/MS Internal Standards

Internal standard compounds must be spiked into every sample, standard, and blank analyzed by GC/MS methods. Internal standards must meet the method area and retention time criteria for peak area and retention time. Older versions of the DoD QSM required that the peak area for each internal standard compound must be no less than 50 percent and no greater than 200 percent of the peak area for that internal standard compound in the midpoint standard in the associated initial calibration sequence. The retention time for each internal standard must be within 10 seconds of the retention time of the midpoint standard in the associated initial calibration sequence. While this requirement was retained in DoD QSM version 5.1 and subsequent versions, internal standard acceptance criteria were expanded to allow for the daily initial CCV to be used for this comparison on days when initial calibration is not performed.

6.2 GC AND HPLC ORGANICS

GC and high-performance liquid chromatography (HPLC) organics include analyses for pesticides (organochlorine and organophosphorus), PCBs, explosives, herbicides, and petroleum products. GC and HPLC analyses use dual columns or dual detectors to identify target analytes. Some laboratories assign the same quantitative significance to both columns/detectors, while others specify a dedicated primary and secondary column/detector. If presented, the QC data for both the primary and secondary column/detector should have been evaluated. In cases where instrument QC discrepancies affect one column/detector and not the other, some degree of interpretation by the validator is required to determine the effect on the associated samples.

6.2.1 Instrument Initial Calibration

The interpretation of GC initial calibration is generally straightforward. If any discrepancies are identified in the initial calibrations associated with PCBs analyses, the HGL reviewer should ensure that the validator considered discrepancies shown by PCB-1016 to affect PCBs 1016, 1221, and 1232; and considered discrepancies shown by PCB-1260 to affect PCBs 1242, 1248, 1254, and 1260.

6.2.2 Second Source Calibration Verification

A second source calibration verification standard should be analyzed immediately after the initial calibration is performed. The performance of each target analyte should be evaluated against the acceptance criteria presented in the QAPP. If any discrepancies are identified in the second source calibration verifications associated with PCBs analyses, the HGL reviewer should ensure that the validator considered discrepancies shown by PCB-1016 to affect PCBs 1016, 1221, and 1232; and considered discrepancies shown by PCB-1260 to affect PCBs 1242, 1248, 1254, and 1260.

6.2.3 Instrument Continuing Calibration

If any discrepancies are identified in the continuing calibration verifications associated with PCBs analyses, the HGL reviewer should ensure that the validator considered discrepancies shown by PCB-1016 to affect PCBs 1016, 1221, and 1232; and considered discrepancies shown by PCB-1260 to affect PCBs 1242, 1248, 1254, and 1260.

Note that some laboratories evaluate continuing calibration results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. HGL's preferred convention is to consider all continuing calibration discrepancies to affect detections and nondetections regardless of direction of bias. The data validation report should not use the direction of bias when evaluating continuing calibration results.

6.2.4 Degradation Summary

The evaluation of this QC element is straightforward and should not be a source of error in the validation report.

6.2.5 Retention Times

Verify that retention time shifts were evaluated in the data validation report.

6.2.6 Confirmation

Verify that confirmation for detected results was evaluated and that confirmed results were qualified if confirmation agreement criterion (RPD ≤40%) was not met.

Most GC and HPLC methods use a second column or second detector to confirm detected results, and the QSM requires that QC results for the confirmation column/detector meet the same QC criteria as the primary column/detector. HGL's preferred convention for qualifying results is by the detector used to report the results for each analyte. This reporting can vary on a sample-specific basis to address sample matrix characteristics that affect one column/detector more than the other.

> *Example*: The laboratory has designated column X as the primary column for reporting herbicide results by Method 8151A. The initial calibration associated with all sample analyses has an acceptable %RSD for dinoseb in column X but a high %RSD for dinoseb in column Y. All reported dinoseb results are nondetections; however, of the nine samples associated with this initial calibration, six have dinoseb reported from column X and three have dinoseb reported from column Y. The three dinoseb results reported from column Y should be qualified UJ; the six dinoseb results reported from column X would not require qualification for an initial calibration discrepancy.

6.3 METALS

Metals analyses often contain discrepancies between the validation criteria applied by the validator and the QAPP criteria. The HGL reviewer should be especially alert to errors in evaluating continuing calibration blanks (CCBs) (Section 6.3.7), and interference check samples (ICSs) (Section 6.3.8).

6.3.1 Instrument Tuning

Instrument tuning data is not always available on summary forms. Verify that the validators were able to evaluate instrument tuning data, including mass windows, peak widths, and %RSD of scans.

6.3.2 Internal Standards

Verify that the validators reviewed internal standard results. In some cases (especially with short analyte lists), there may be internal standards that do not meet acceptance limits but are not associated with target metals. Some laboratories will also choose a secondary internal standard to quantify a metal if the primary internal standard does not meet acceptance criteria.

6.3.3 Initial Multipoint Calibration

Initial multipoint calibration is required for cold vapor atomic absorption and graphite furnace atomic absorption (GFAA) methods. It is not required for inductively coupled plasma (ICP) atomic emission spectroscopy or ICP-MS analyses; however, if a multipoint initial calibration is performed, it must meet the acceptance criteria in the QAPP. If the supplemental calibration checks

described in Section 6.3.4 or 6.3.5 are acceptable but the multipoint initial calibration was out of control, the associated results should have been qualified by the validator.

6.3.4 Low-Level Calibration Verification

The integration of the results for initial calibration, low-level calibration standards, and contract required detection limit standards is a common source of validator error. The HGL validation reviewer should ensure that the validator understands how to evaluate these three QC elements in totality and apply the correct final qualifier to any results affected by discrepancies associated with the initial calibration QC checks.

6.3.5 High-Level Calibration Verification

Verify that the validator evaluated high-level calibration standards and qualified any results reported from above the calibrated range.

6.3.6 Initial and Continuing Calibration Verification

Most laboratories use initial calibration verification standard (ICV) analyses as a second source verification check. HGL's preferred convention is to associate ICV results with all sample results in an analytical sequence and to associate CCV standard results only with sample results "bracketed" by a given CCV. A result is considered bracketed by a CCV if that CCV is the last CCV analyzed before that result was generated or is the first CCV analyzed after that result is generated.

Note that some laboratories evaluate ICV/CCV results with respect to the direction of the bias and consider nondetected sample results associated with a discrepancy biased high to be acceptable. For metals methods, HGL considers it to be acceptable to evaluate the direction of the bias when qualifying associated results. The HGL validation reviewer should ensure that the data validator correctly identified ICV/CCV results that did not meet acceptance criteria and that any discrepancies were associated in accordance with the QAPP conventions.

6.3.7 Continuing Calibration Blanks

CCBs present the same common source of error as do method blanks: the confusion caused by the qualification criteria differing from acceptance criteria (see Section 5.5). The HGL reviewer should ensure that all CCB contamination at or above the DL was evaluated for the potential effect on associated sample results, not just the CCB contamination that was present above the acceptance criteria.

CCBs are always aqueous; the concentrations should be converted to the equivalent soil concentration when comparing the blank results to the concentrations found in any associated soil

samples. The HGL reviewer should verify that the appropriate conversion was made by the validator.

HGL's preferred convention is to associate initial calibration blank (ICB) results with all sample results in an analytical sequence and to associate CCB results only with sample results bracketed by a given CCB. A result is considered bracketed by a CCB if that CCB is the last CCB analyzed before that result was generated or is the first CCB analyzed after that result is generated. The HGL reviewer should verify that the association conventions used by the data validator are those in the QAPP.

The HGL validation reviewer should ensure that the data validator correctly identified ICB/CCB results that did not meet acceptance criteria and that any discrepancies were associated in accordance with the QAPP conventions. The HGL reviewer should also verify that any blank contamination with concentrations or absolute values of concentrations greater than the acceptance levels were noted by the validator with a discussion of any laboratory corrective action.

6.3.8 Interference Check Sample Results

The evaluation of ICS data is another common source of error in data validation reports. One of the primary reasons for this is that laboratory data summary reporting forms generally provide inadequate information for the data validator to be able to evaluate the results that are presented. The HGL reviewer should evaluate whether the data validator evaluated ICS A (ICSA) results in accordance with the QAPP and applied the correct qualifiers. Common errors are:

- Failure to evaluate ICSA results at all (some firms consider this a Stage 4 item);
- Failure to identify severe discrepancies (results greater than the LOQ or converted water-to-soil LOQ); and
- Failure to interpret discrepancies and apply qualification in accordance with the QAPP.

Note that QAPPs written to include QSM version 5.1 (or later) requirements will require the absolute value of each unspiked analyte in the ICSA to be less than one-half the LOQ; QAPPs written in accordance with older versions of the QSM will include a requirement that the absolute value of each unspiked analyte to be less than the limit of detection.

The evaluation of ICS AB results is generally straightforward, and this QC element rarely shows discrepancies.

6.3.9 Recovery Test Recoveries

GFAA methods use recovery tests to determine if the sample matrix has affected reported results. The method requires a recovery test to be performed on a representative sample in each preparation

batch, but in practice, laboratories perform recovery tests on a sample-specific basis. The HGL reviewer should verify that this QC element was evaluated in accordance with QAPP requirements.

6.3.10 Method of Standard Addition Results

The method of standard additions (MSA) is associated with GFAA analyses; this procedure is rarely performed as virtually all laboratories perform sample-specific recovery tests rather than batch-specific recovery tests. If MSA results are reported in a data package, the HGL reviewer should consult with the HGL Senior Chemist.

6.4 GENERAL CHEMISTRY

General chemistry parameters include a wide variety of analytical parameters and methodologies, including colorimetry, ion chromatography, GC, and infrared spectrometry. Usually, these parameters are secondary data that are used to determine the potential for a site to undergo monitored natural attenuation or the progress of monitored natural attenuation. Often, these tests will only require a Stage 2A data review; however, some parameters, such as cyanide, perchlorate, anions, or total organic carbon, will on occasion require Stage 2B validation.

In many cases, the review of general chemistry QC parameters is similar to the review of the corresponding parameters for metals. Method-specific QC parameters should be discussed in the QAPP along with the acceptance criteria and qualification requirements. Some laboratories do not have summary forms for Stage 2B QC elements and the raw data will need to be examined by the validator to evaluate performance.

The HGL reviewer should ensure that each general chemistry parameter was validated to the appropriate stage, and that all appropriate QC elements were validated. If it is found that the subcontracted data validator is not applying the correct stage of validation to one or more general chemistry parameters, this should be brought to the attention of the HGL project manager and the project chemist.

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APPENDIX B

ENERGY LABORATORIES QUALITY ASSURANCE MANUAL

ENERGY LABORATORIES-BILLINGS, MT
QUALITY ASSURANCE MANUAL

Revision February 09, 2022

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Date: 2022.02.17 14:40:54 -07:00

Quality Assurance Officer:

TABLE OF CONTENTS

ELI COMMITMENT

Energy Laboratories, Inc. Strives Toward:

1. Being highly skilled in the field of analytical chemistry.
2. Delivering quality and service with integrity.
3. Encouraging the professional development of our staff.
4. Offering our employees a safe and positive work environment.
5. Being profitable and using resources wisely for a sustainable future.

INTRODUCTION

Energy Laboratories, Inc. provides chemical, industrial hygiene, and environmental analytical services to private industry, agricultural industry, engineering consultants, government agencies, and private individuals. Analytical services include: analysis of waters and soils for inorganic and organic constituents, aquatic toxicity testing, hazardous waste analysis, radiochemistry, industrial hygiene, microbiology, soils and water physical parameters, and petroleum analysis.

Founded in 1952, Energy Laboratories currently incorporates four separate testing laboratories. The corporate headquarters are located in Billings, MT, with laboratories located in Casper, WY; Gillette, WY; and Helena, MT.

ELI, as a coordinated company of four participating laboratories, has developed a QA program that takes into account the various method types and EPA programs, while also considering sample matrices, to develop a single comprehensive set of QA guidance. We have used scientific approaches, Good Laboratory Practices, EPA Methods and Guidance documents, and accreditation audit guidance to develop our overall QA Program.

The Quality Assurance Program establishes acceptable performance criteria for all routine analytical procedures being performed by laboratory personnel. The Quality Assurance Assessment Program provides a formal system for evaluating the quality of data being generated and reported. The ELI Laboratory Safety Manual & Chemical Hygiene Plan defines the safety and monitoring procedures used by laboratory personnel in laboratory operations. These, in addition to the experience and expertise of our analysts, provide a comprehensive Quality Assurance Program. Energy Laboratories, Inc., in Billings, Montana, is certified under the Safe Drinking Water Act by Region VIII EPA for Wyoming, and the States of Montana, Idaho, Colorado, Nevada, Texas, Florida, Nebraska, North Dakota, South Dakota, Washington, and Georgia. ELI-Billings also holds accreditation for Clean Water Act, Safe Drinking Water Act and Resource Conservation Recovery Act (RCRA) parameters through the National Environmental Laboratory Accreditation Program (NELAP) managed by TNI (The NELAC Institute), which is supported by the USEPA. The primary NELAP certification is maintained through the state of Florida. Individual State approval for SDWA, RCRA and CWA (NPDES) is managed through the Federal/State DMRQA program or through reciprocal certifications when required by a specific state. ELI obtains these certifications either through reciprocal recognition

of ELI's primary Montana State, NELAP, or ISO/IEC 17025/DoD certifications. Department of Defense (DoD) and international lab certification under ISO/IEC 17025 and DoD requirements is provided through ANSI ASQ National Accreditation Board (ANAB). To perform radon testing, ELI is certified under the National Radon Proficiency Program (NRPP) administered by the National Environmental Health Association. Copies of current ELI certificates, including the ISO/IEC 17025/DoD certificate, are maintained on ELI's website: www.energylab.com.

The ELI Quality Assurance Manual and the ELI Professional Services Guide (Fee Schedule) together are used to outline the ELI Quality Assurance/Quality Control Program. This Quality Assurance Manual is appropriate to all departments of Energy Laboratories-Billings. The procedures discussed or referenced in this manual describe our day-to-day laboratory practices and adhere to USEPA Safe Drinking Water Act, and TNI (The NELAC Institute) requirements as well as Good Laboratory Practices (GLPs). A list of certifications that the ELI Billings laboratory holds can be found in Appendix A of this plan. Where possible, ELI uses EPA, AOAC, ASTM, APHA, NIOSH, OSHA, or published analytical methods and follows the procedures with strict adherence to described protocol and recommended QA/QC parameters. The analytical methods approved and in use are described in Standard Operating Procedures, and are available for review at the laboratory. Vital parts of our Quality Assurance Program, Quality Control and Quality Assessment programs are outlined in Chapters One and Two of this manual.

To generate data that will meet project-specific requirements, it is necessary to define the type of decisions that will be made and identify the intended use of the data. Data Quality Objectives (DQOs) are an integrated set of specifications that define data quality requirements and the intended use of the data. Project-specific DQOs will be established as needed for both field and lab operations. Through the DQO process, appropriate reporting limits, extraction/digestion methods, clean-up methods, analytical methods, target analytes, method quality control samples, sample security requirements, method validation criteria, quality control acceptance ranges, corrective action procedures, validation procedures, reporting formats and reporting limits can be specified. Professional laboratory project managers are available to assist clients in specifying appropriate laboratory analyses and reporting procedures necessary to meet project requirements.

Client-specific DQOs can be coordinated with the laboratory through our Project Managers via quotations or contracts, or with relevant documentation provided to the laboratory prior to (or at time of) sample receipt. Client-specific requirements are communicated to analysts and final report validators through the laboratory LIMS system. By default, our methods, analytes, and QC parameters are set up to meet the DQOs specified in the referenced method and/or federal/state regulations. ELI encourages clients to provide ELI documentation of any client-specific, regulatory or project monitoring requirements. Project samples requiring analysis under DoD accreditation are managed as having project specific requirements to meet client DQO requirements in addition to Quality System and method requirements as specified within the DoD Quality System Manual (QSM) Version 5.4.

Certain types of requests may not be suitable to standardized analytical methods. These custom requests are handled individually with laboratory management and staff scientists.

Project-specific methods and reporting packages are available. Attention to documentation of the analytical procedure and use of suitable QC parameters is maintained according to good scientific discipline and Good Laboratory Practice guidelines.

The ELI-Billings laboratory Director, or the designee, will evaluate all new contracts to determine that the laboratory is capable of performing the requested work. This process includes ensuring that the laboratory maintains the required accreditation, equipment and resources. In the event that sample analysis is not performed at our Billings location, clients are notified on the laboratory analytical report if the work is subcontracted to a qualified ELI laboratory or an outside laboratory (See Subcontracting Policy – Chapter 6 in this QA Manual).

This Quality Manual and related quality documentation meet requirements of the National Environmental Laboratory Accreditation Program (NELAP), which is an EPA approved accreditation program, and on a project specific basis include additional Department of Defense DoD accreditation requirements as specified in their Quality System Manual Version 5.4 (DoD QSM 5.3, May 2019) or current approved version.

CHAPTER 1 – QUALITY CONTROL PROGRAM

Quality Policy Statement

Energy Laboratories, Inc. is committed to producing laboratory data of known and documented quality that is scientifically valid, meets method specifications, satisfies regulatory requirements, and accomplishes the data quality objectives of the client and project. ELI's Management and Quality Systems ensure that the laboratory maintains current certifications and is in compliance with accreditation and regulatory requirements through USEPA, Federal and State, NELAP/TNI, and DoD/ ISO/IEC-17025 accreditations. Those method, regulatory, and client requirements (as well as the policies, procedures, and all referenced documents) are incorporated into our Quality Assurance Program; which is outlined within this Quality Assurance Manual. The Quality Systems are designed to comply with the standards as defined by the most current approved version of the NELAC accreditation standards (TNI 2016) and includes procedures to manage risk and requirements as discussed in ISO/IEC 17025-2017. To ensure compliance with these standards, all laboratory personnel are required to be familiar with quality documentation and implement those policies and procedures in their work. ELI is dedicated to the continual improvement of the management system's effectiveness by providing appropriate corporate resources to set objectives, offering training opportunities, and monitoring the quality performance of our testing. ELI also provides facilities, resources, and equipment adequate and appropriate to these objectives.

Quality Assurance Program

The purpose of the Quality Assurance Program is to ensure that the analytical services provided by Energy Laboratories are of high quality, data is within established accuracy and precision limits (required by the referenced method or Standard Operating Procedure), and each analytical result produced meets or exceeds our accreditation requirements. Management ensures that the integrity of the management system is maintained. The Technical Director, or their designee, ensures that changes to the management system are planned, implemented and documented.

Management establishes and maintains data integrity by providing the following to ELI's data integrity system:

1) Data Integrity Training (Including the highest standards of ethical behavior)
2) Periodic review of data integrity procedural documentation
3) Annual review of data integrity procedures with updates as needed
4) Periodic, in-depth monitoring of data integrity
5) Maintenance of signed data integrity documentation for all laboratory employees

All employees are expected to implement and follow the policies contained within the Quality Assurance Program.

The quality systems in the program consist of the policies and procedures, and all referenced documents, described in this Quality Assurance Manual. The Quality Control Program also functions to maintain the laboratory's compliance with accreditations through USEPA, State

Agencies, NELAP, and ANSI-ASQ National Accreditation Board (ANAB) for DoD and ISO/IEC-17025 accreditation.

The Quality Control Program requires that the following points be met for each applicable analytical method:

- Performance of any analytical method requires that the proper equipment and instrumentation are available. A list of major equipment is listed in Appendix E. The procedure for operation of an analytical instrument is described in the equipment manufacturer's operating manual, and may also be supplemented with a specific Standard Operating Procedure (SOP) for the instrument and/or the method.

- Specific SOPs cover operation of the instrument including the sequence of operations involved in instrument start-up, calibration, analysis, and shut down. Chapter 13 of this manual includes recommended preventative maintenance, and/or a list of parameters used to identify other types of maintenance. SOPs outline any special safety precautions for operation of the instrumentation.

- SOPs of detailed EPA, AWWA Standard Methods, ASTM, NIOSH, APHA, OSHA, or other published procedures include, as appropriate, a list of any method-specific items or variances, a list of QC parameters and their recommended method performance ranges, recommended or example analytical sequences, specific or unique safety information, method references, and a signed signature page. SOPs details, and format of method SOPs, follow NELAP requirements. Detailed SOPs may be prepared for those procedures that do not have published methods. Further details of SOP format and information required in method SOPs can be found in the ELI SOP, *Preparation, Numbering, Use, and Revision of Standard Operating Procedures.* Written Standard Operating Procedures referenced within this manual are available at the laboratory for review. ELI SOPs are considered confidential proprietary information.

- For radiochemical analysis performed at the ELI-Casper Laboratory, each method undergoes Method Validation as outlined in EPA's specific method and/or the Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP), Chapter 6.

- The required detection level (RDL) for radiochemical analysis of drinking water samples is calculated based on the requirements in 40 CFR 141.25(c), which is a sample specific determination. The equation is specific for each method and noted in the method-specific SOP where appropriate.

- The initial test method evaluation for referenced EPA procedures, or new instrument setups applied to a procedure for chemical analysis involves Method Detection Limit (MDL) studies, including confirmation of the Limit of Detection (LOD) and Practical Quantitation Limit (PQL), also known as the Limit of Quantitation (LOQ) (refer to ELI SOP, *Determination of Method Detection Limits (MDL), Quantitation Limits and Initial Method and New Instrument/Equipment Validation)* and evaluation of method performance by successful completion of an Initial Demonstration of Capability (refer to

ELI SOP, *Personnel Training and Training Records*, the successful completion of appropriate Performance Evaluation (PT) studies (when available), evaluation of the method selectivity and sensitivity, and any additional method or client-specific requirements.

- ELI demonstrates that laboratory staff is qualified and capable of performing the method. Analysts are assigned duties based on their skills and experience. Training records are maintained for all analysts. Curricula vitae of key management and personnel are described in Appendix D.

- It is the responsibility of the analyst to become thoroughly familiar with the methodology and instrument operation before performing the analysis. It is the responsibility of the person providing training to monitor all laboratory results generated for a reasonable time. The amount of time necessary may vary depending on the method and the experience of the analyst. At a minimum, the analyst's performance is to be monitored until the analyst demonstrates the ability to generate results of acceptable accuracy and precision according to the method.

- All analysts are required to demonstrate and maintain a record of proof of competency by routinely analyzing quality control samples appropriate to the analytical procedures they perform. Proof of competency is documented in analysts' training files per NELAP requirements (for more information, see ELI SOP, *Personnel Training and Training Records*. For those analyses where external proficiency testing (PT) samples are not routinely analyzed, competency is documented by including the results of routine analysis of method-specific quality control samples (prepared by laboratory staff) and/or a verifying statement of procedural review by a supervisor or trained analyst.

- Each analytical method is subjected to quality control monitoring. The purpose is to demonstrate that results generated meet acceptable accuracy and precision criteria for the method. Precision and bias are determined for standard and non-standard methods. Precision and bias are determined for standard methods through control charting of data from quality control samples. Precision and bias using non-standard, modified standard or laboratory-developed methods are compared to the criteria established by the client (when requested), the method, or the laboratory.

- Quality control requirements are outlined in the methods and ELI, at a minimum, follows the guidelines specified in the methods used. Additional QC requirements are also added as appropriate. Statistical method performance is periodically evaluated against method requirements using control charts.

- Quality control monitoring to measure accuracy for each method generally requires that five to ten percent of all samples analyzed be fortified (spiked) with a known concentration of target analytes tested by the method. The percent recovery is then calculated. This provides a means for monitoring method accuracy and evaluating sample matrix effects. Where appropriate, surrogates are included in the method to monitor method performance on each individual sample. Blank spike samples replace

matrix spike samples for certain methods, or when there is insufficient sample for a matrix spike analysis. Historical, routine batch QC sample performance can be used to estimate the precision and accuracy of the method.

- Quality control monitoring to measure precision for each method requires replicate samples be prepared and analyzed when appropriate. Actual requirements are outlined in the specific SOP. When replicate samples or matrix spike duplicates are analyzed, relative percent difference is calculated and used to monitor precision of the method. In instances where there are no specific method requirements, it is the policy of this laboratory to analyze five to ten percent of all samples in duplicate. Duplicate test results must be within the control limits established for each analysis type or data is qualified. Acceptance limits generally follow specifications listed in the method. Matrix spike duplicates replace sample duplicates for most methods.

- When not defined in the method, and as appropriate, method blanks and/or instrument blanks are analyzed one in every 20 samples at a minimum. Method blanks are used to verify that contamination from laboratory reagents and glassware is not present in the analytical sample process. Generally, the method blank should be less than the reporting limit, or 10 times less than the concentration amount in the sample, for the analytical parameter being tested, whichever is greater.

- When method spike frequency is not defined in the method and as appropriate, method spikes (blank spikes) are analyzed, at a minimum one in every 20 samples.

- Calibration standards are analyzed and calibration curves are developed for all applicable methods. For additional information on instrument calibration, see Chapter 7 of this QA manual.

- The initial calibration is continuously monitored by analyzing a continuing calibration standard every 10 to 20 samples, or within a specified time frequency, and at the end of each analytical sequence; depending on the method and instrumentation. Results must be within an established range as described by the method SOP. Initial calibrations are verified against a standard from a second source.

- Proficiency testing samples and further quality control check samples may be required for various methods. Refer to Chapter 2 of this QA manual for further details.

Estimation of Uncertainty

The estimation of uncertainty consists of the sum of the uncertainties of the individual steps or processes of an analytical procedure and the field sampling variabilities. The variability of the sampling plan, sample heterogeneity, extraction procedure, instrument calibration, instrument drift, systematic bias, and many other factors all contribute to the uncertainty of a measurement or sample result.

ELI estimates uncertainty utilizing Confidence Intervals defined as ±2σ (95%) and ±3σ (99%) where σ is the standard deviation of the recovery of quality control samples. The confidence intervals calculated from these QC samples are based on the spike level concentrations for each method. For most procedures, uncertainty at the reporting limit or Limit of Quantitation (LOQ) is determined by Limit of Quantitation spike recovery studies or by MDL study spike recovery evaluations. LOQ/MDL verifications are also performed quarterly to verify ongoing method accuracy, precision and sensitivity. LCS limits are used to set method accuracy and precision overall. PT Acceptance criteria are also a guide for evaluating interlaboratory method accuracy, and the reasonableness of ELI assigned method QC limits. Real world samples, depending on matrix interferences, may have a greater amount of uncertainty associated. Due to limitations in assessing the uncertainty for each matrix type, the confidence intervals calculated from method QC samples provides an estimate of laboratory method uncertainty.

Energy Laboratories, Inc. uses the procedures outlined in ELI SOP, *Control Chart Generation and Maintenance*, for the purpose of evaluating estimation of uncertainty for chemical analyses and uses the determination of uncertainty on a sample-specific basis for all radiochemistry measurements. These estimates of uncertainty have formulas documented in the individual SOP.

Maintenance of Performance Records

All quality control monitoring is recorded and documented. Quality control data is recorded in laboratory notebooks, electronic summary files, and/or analysis sheets. Generally, review of QC data and trends is managed within the Laboratory LIMS system. QC data management and control chart generation, maintenance, and usage are described in ELI SOP, *Control Chart Generation and Maintenance*. It is the responsibility of the analyst to see that all results are recorded in a timely manner.

All quality control data is filed and available for inspection and assessment by analysts, supervisors, management, and quality control personnel.

Method Quality Control Specifications

Summaries of Quality Assurance/Quality Control specifications for a selected subset of procedures offered by ELI are outlined in Appendix B. These types of method QC Element tables are available upon request for our clients to use in the preparation of Quality Assurance Project Plans (QAPPs). Exact details of method QC can be found in the applicable method SOPs.

CHAPTER 2 – QUALITY ASSESSMENT PROGRAM

The function of the Quality Assessment Program is to provide formal evaluation of the quality of data being generated and reported by the laboratory. External and internal quality control measures are used in this assessment. These measures include proficiency testing samples, laboratory quality control check samples, and routine internal and external audits on methodology and documentation procedures.

Proficiency Testing (PT) Samples

PT samples are supplied by an outside entity and contain known amounts of constituents. The laboratory does not have access to known values of the samples. Only the PT provider has knowledge of constituent levels prior to the formal publishing of the test results.

PT samples are received on a routine basis, with results sent to the providing entity for evaluation. Proficiency Testing (PT) samples for USEPA, NELAP and various State certifications are Water Pollution Study samples (WP or DMRQA), Water Supply Study samples (WS), and LPTP Soil PT samples provided by NELAP approved PT providers - either Millipore Sigma and/or Environmental Resource Associates (ERA). Routine participation in LPTP, WS and WP PT sample studies is used to maintain certifications for Safe Drinking Water Act (SDWA), Clean Water Act (CWA), National Pollutant Discharge Elimination System (NPDES), Discharge Monitoring Report Quality Assurance (DMRQA), permit monitoring analyses, Resource Conservation and Recovery Act (RCRA) analyses, as well as for other states and projects requiring method accredited parameter analyses. The samples are analyzed in the same manner as any routine sample in the laboratory. Acceptable results are those that fall within a defined range as determined by the vendor; based on multi-laboratory study results. The provider sends results to the appropriate certifying agencies as requested by the laboratory. PT study results are posted on the ELI website www.energylab.com.

A list of current certifications maintained by ELI Billings is included in Appendix A. For a list of accredited matrix/method/analytes refer the current certifications available on the ELI website at www.energylab.com. The Montana primary certification includes a list of parameters/methods for which drinking water certification has been granted. The NELAP certificate also includes RCRA methods used for hazardous waste characterizations and CWA parameters/methods which are used for NPDES monitoring permits. Reciprocal accreditation in other states is based on either of these, or both, depending on specific state certification requirements/parameters. ISO/IEC 17025/DoD certification is maintained for Department of Defense and international projects requiring that certification type. ELI also participates in the Federal/State DMRQA programs for clients which require/request this with their NPDES permits. Reciprocal accreditation in other states is based on either of these, or both, depending on the specific state certification requirements for accreditations.

Proficiency testing samples for Radon Proficiency testing are from approved NRPP PT providers. Our own radon sampling canisters are submitted for known levels of radon exposure. Acceptable results are those that fall within a defined range based on multi-laboratory study results.

Blind Quality Control Check Samples are samples submitted as regular lab samples and are processed through the system in the same manner as any other routine environmental sample. The analysts do not know the true values of these samples when performing the analyses. Method performance reports are returned to the analysts. Clients occasionally submit these types of samples for their QAPP.

Inter-Laboratory comparison samples are samples containing known or unknown concentrations of analytes that are split and analyzed by more than one laboratory.

Quality Control Check Samples

Quality Control Check Samples are performance evaluation samples used for routine method performance monitoring. As appropriate, analytical procedures include the analysis of a quality control sample with every sample batch analyzed. The materials are obtained from a commercial source when available, or they may be prepared in-house. Acceptable results are within a defined range based on certified ranges, or against statistically-determined control limits, method-defined criteria, or client-defined Data Quality Objectives. Routinely used methods not subjected to PT sample monitoring are evaluated with Quality Control Check Samples, as appropriate.

QC samples are processed through the system in the same manner as any other sample, except the analyst is aware of the source, concentration, and acceptance ranges of target analytes and calculates analyte recoveries to evaluate method performance in real time.

Quality Assurance Audits

Quality Assurance Audits consist of internal and external laboratory inspections designed to monitor adherence to Quality Systems and quality control requirements. These audits check general laboratory operations, overall Quality Systems, adherence to QA program requirements, sample tracking procedures, sample holding times, storage requirements, adherence to procedures during analysis, calculations, completion of required quality control samples within the group surrounding the sample, and proper record-keeping.

Internal quality control audits are conducted or coordinated by the Quality Assurance Officer of the laboratory. See ELI SOP, *Internal Audits*, for further information. ELI conducts internal inspections on a regular basis to monitor adherence to quality control requirements. Results of formal audits are given to management with recommendations for corrective action in the event any discrepancies are found. As necessary, a follow-up review is conducted to determine that identified problems have been addressed. Annually, the overall quality systems of the laboratory are reviewed and a summary report is prepared.

Per current NELAP/ISO/IEC 17025- requirements, the management of the laboratory will conduct an annual review of the Quality System, including policies, procedures and environmental testing activities in a meeting with key laboratory management and supervisory staff. This is done to ensure the continuing suitability and effectiveness of the QA systems, as

well as provide the opportunity to introduce necessary changes or improvements. The review shall take into account, at a minimum, the following:

- Changes in internal and external issues that are relevant to the laboratory
- Fulfilment of objectives
- The suitability of policies and procedures
- Status of Actions from previous management review reports from managerial and supervisory personnel
- Outcome of recent internal audits
- Corrective and preventative actions
- Assessments by external bodies
- The results of inter-laboratory comparisons or proficiency tests
- Changes in the volume and type of work
- Client and personnel feedback
- Complaints
- Recommendations for improvement and effectiveness of any implemented improvements
- Results of risk identification
- Other relevant factors, such as quality control monitoring activities, data integrity, data accuracy and precision, risks to impartiality, resources, and staff training

The findings from management reviews and the corrective actions that arise from these findings shall be recorded. The management shall ensure that any corrective actions are carried out within an appropriate, pre-determined time frame and with provision of required resources.

ELI also conducts Peer Audits as part of an internal auditing program established within the company. This process utilizes analysts and supervisors from other ELI laboratories to evaluate a designated ELI branch. The Peer Audits serve to not only address conformance issues, but also provide ELI with a tool to continuously improve process and consistency throughout the company. The goals of the Peer Audits are to:

- Encourage relationships between analysts
- Transfer technical knowledge between peers
- Establish consistency of analytical process/method between ELI laboratories
- Identify the depth of analysts' knowledge at each position by observing what analysts are doing at the bench
- Determine training needs of personnel
- Document process/method and verify that issues are being corrected when found
- Work with, and in support of, QA department efforts

Depending on the size of the laboratory, a large number of methods and processes can be examined during a Peer Audit. Results from these audits are provided to the branch management, as well as Corporate Management. Corrective Action Plans of a Peer Audit are initiated with the assistance of the Quality Assurance Officer for resolution of any findings.

ELI welcomes external Quality Assurance Audits, by qualified outside auditors, for review and comment on the overall QA program. To maintain certifications, accrediting authorities from the State of Montana, ANAB, and NELAP conduct periodic comprehensive external audits. External audits to meet Quality Assurance Project Plans (QAPPs), as applicable to environmental remediation projects, or for major industries, are conducted as requested. For more information, see ELI SOP, *External Quality Assurance Audits*.

CHAPTER 3 – LABORATORY FACILITIES

The facility for Energy Laboratories, Inc. – Billings, MT consists of multiple buildings with over 35,000 square feet of total space; these buildings are located in Billings at 1120 South 27th Street, Billings MT 59101.

The phone number for Billings Energy Laboratories, Inc. is (406) 252-6325, the fax number is 406-252-6069, the toll free number is 800-735-4489, and the email address is eli@energylab.com.

Laboratory space includes adequate bench top and floor space to accommodate periods of peak work load. Working space includes sufficient bench top area for processing samples; storage space for reagents, chemicals, glassware, bench and portable equipment items; floor space for stationary equipment; and adequate associated area for cleaning glassware. Laboratory departments are organized and the facilities are designed for specific laboratory operations in order to protect the safety of analysts and to minimize potential sources of contamination between and within department areas (for more information, see ELI SOP, *Facility Description, Access, and Security*.

The laboratory is appropriately ventilated and illuminated, and is not subject to excessive temperature changes. Specific laboratory areas are temperature and humidity controlled as required. Ample cabinets, drawers and shelves are available for storage and protection of glassware. Exhaust fume hoods are available as needed for use during preparation, extraction, and analysis of samples. Employee exposure monitoring is conducted to provide a safe working environment.

To maintain security, all visitors must enter their name on the ELI sign-in log at the front desk and wear a visitor's badge, undergo safety awareness training, and are escorted.

The laboratory has provisions for the disposal of chemical and microbiological wastes. These provisions are described in Standard Operating Procedures as well as outlined in the Laboratory Safety Manual & Chemical Hygiene Plan along with other safety and health guidelines. For more information, see ELI SOP, *General Laboratory Waste Disposal*.

CHAPTER 4 – PERSONNEL REQUIREMENTS AND LABORATORY ORGANIZATION

Relationship between Management, Technical Operations, Support Services and the Quality System

Laboratory Organization

The corporate organization of the four ELI laboratories located in Montana (2), and Wyoming (2), is provided in Appendix C. The Billings laboratory is the center for all corporate functions. Each laboratory is managed and operated individually under the supervision of a Laboratory Manager/Director. All ELI laboratories have fiscal and QA/QC responsibilities to the corporate office, as well as general operating policies and goals. Quality Assurance Manuals are prepared individually for each laboratory and follow the QA/QC program outlined in the ELI-Billings QA manual.

The ELI-Billings Organizational Chart is also included in Appendix C with curricula vitae of key ELI-Billings laboratory personnel maintained in Appendix D of this manual. Job descriptions are maintained by the Human Resources Department.

Quality Assurance receives direct support from senior management. Laboratory Quality Assurance Officers report directly to the Corporate Quality Assurance Officer as well as their Laboratory Director. Quality Assurance Officers provide independent oversight of Quality Systems within the overall Energy Laboratories structure. When Quality Assurance Officers fill more than one role within the organization, they operate independently of direct environmental data generation while fulfilling quality assurance responsibilities. Quality Assurance Officers facilitate development of and maintain the Quality Assurance Manual, provide assistance to personnel on quality assurance / quality control issues, maintain a quality assurance training program, and review quality documentation including SOPs.

Management ensures the development and implementation of programs and policies to continuously improve the effectiveness of ELI's QA Program and Management Systems. Management performs an annual review of the laboratory's Quality System (policies, procedures, work instructions) to assure their continuing suitability and effectiveness (See ELI SOP, *Management Reviews*, for detailed procedures. As appropriate, management identifies and implements any necessary changes or improvements. In addition, management performs meetings with supervisory and key staff members throughout the year. Supervisors and QA personnel provide input on their specific areas of responsibility and evaluate the following:

1) Client-Related Items
2) Internal and External Audit Reports
3) Proficiency Testing Results
4) Review of Performance by Department
5) Corrective and Preventive Actions
6) Personnel Training Needs
7) Quality System Policies and Procedures

8) Resources including Personnel, Equipment and Facilities

Laboratory Management Review findings are compiled into a summary report. The report includes deficiencies identified and areas for improvement. The QA department ensures items from the Management Review are tracked, including actions that must be addressed, assignment of parties responsible for the actions to be taken, and recommendations on improvements to the Quality System. The Technical Director, Laboratory Director, Quality Assurance Officer or designee, shall assign specific persons to address management review findings and establish deadlines for their completion. The Technical Director, Laboratory Director, Quality Assurance Officer or designee, reviews and approves all QA documents issued to personnel in the laboratory as part of the management system. The Technical Director, or designee, has overall responsibility for the technical operations of the laboratory. Any procedural deviations to SOPs that are client- or project-specific must receive approval either from the Technical Director, Laboratory Director, or Quality Assurance Officer. Work is stopped when identification of any of the following is made: unapproved departures from the management system, unauthorized deviations from the procedures for performing tests and/or calibrations, and data quality or data integrity issues. The Technical Director, Laboratory Director, QA Officer, or designee, is responsible for providing authorization for the work to resume once the identified issue has been addressed.

Personnel Requirements

ELI maintains experienced staff and management. Below is a summary of the primary roles, responsibilities and qualifications for the designated positions. Laboratory experience can be substituted for academic requirements. At ELI's smaller laboratory operations, the technical director may serve multiple roles. Detailed job descriptions are maintained by the Human Resources department. Specific titles of employees are at the discretion of the Laboratory Director.

Laboratory Director

The Laboratory Manager/Director is required to have education and/or experience equivalent to a Bachelor of Science degree in Chemistry or a related science. Five years of relevant laboratory experience is required.

The Laboratory Director is responsible for all operations, client management, analysis scheduling, and equipment acquisition, as well as compliance with all employment, safety, environmental and NELAP /ISO/IEC17025 regulations. The Laboratory Director may delegate daily activities of these work aspects to appropriate personnel. The Laboratory Director reports directly to the Corporate Director of Operations. All Laboratory Directors have both technical and management responsibilities.

Quality Assurance Officer

The Quality Assurance Officer is required to have an education and/or experience equivalent to a Bachelor's of Science degree in Chemistry or a related science. Five years of relevant laboratory experience is preferred.

The Quality Assurance Officer is responsible for quality systems development, implementation, and management. The Quality Assurance Officer is also responsible for maintaining and improving compliance with all applicable state and federal regulations as well as maintaining compliance with NELAP/ISO/IEC17025 regulations regarding Quality Systems. The Quality Assurance Officer or his/her designee with the help of the Laboratory Director manages the laboratory's certification programs to meet government regulatory and specific client requirements. The QA program is implemented in cooperation with all levels of management and staff. Quality Assurance Officers report directly to the Corporate Quality Assurance Officer. The Laboratory Director will direct daily laboratory-specific QA/QC requirements. The Corporate Quality Assurance Officer reports directly to the ELI President.

Technical Director

The Technical Director is required to have a Bachelor of Science degree in Chemistry or a related science and meet all applicable education requirement listed in the current NELAP standard. Five years of relevant laboratory experience is preferred.

The Technical Director is responsible for ensuring compliance with all laboratory policies and that the analyses conducted under their supervision are compliant with all state, EPA, and NELAC/ISO17025 required standards and regulations. Technical Directors report directly to the Laboratory Director.

The Technical Director may serve multiple roles. Laboratory Directors serve as one of the laboratory Technical Directors.

Laboratory Supervisor

A Laboratory Supervisor is required to have education and experience equivalent to a Bachelor of Science degree in Chemistry or related science. Two years of relevant laboratory experience is required.

ELI's Laboratory Supervisors are responsible for the day-to-day operation of the laboratories: scheduling testing, assigning work, and completing the technical review of laboratory data. Supervisors are responsible for ensuring compliance with all laboratory policies and ensure that the analyses conducted under their supervision are compliant with all state, EPA, and NELAC/ISO17025 standards and also client- or project-specific requirements. They report directly to the Laboratory Director.

Analysts

Laboratory Analysts are required to have an education equivalent to a Bachelor of Science degree in Chemistry (or related science), or a High School diploma with experience as an analyst in training. New analysts require on-the-job training, under direct supervision of a qualified analyst until authorized by management to perform assigned tasks. The training shall be relevant to the present and anticipated tasks required and the effectiveness of the training must be evaluated (for more information, see ELI SOP, *Personnel Training and Training Records)*. After the initial training period, and on a continuing basis thereafter, the analyst must demonstrate acceptable skills through the successful participation in the analysis of applicable performance evaluation and quality control samples.

Analysts perform the following duties: Preparation of samples and reagents, analysis and preliminary data input, as well as various other tasks assigned by the supervisor. Analysts are responsible for complying with all laboratory policies and procedures.

Laboratory Technicians

Laboratory Technicians are required to have a High School Diploma or equivalent. Laboratory Technicians work under the supervision of the primary analyst performing general laboratory tests.

Under the supervision of a primary analyst, Laboratory Technicians perform the following duties: preparation of samples and reagents, analysis, and preliminary data input, as well as various other tasks assigned by the supervisor.

Laboratory Technicians are responsible for complying with all laboratory policies and procedures.

Approved Signatories

Signatures for policies are based on individual roles and responsibilities as determined by the policy being reviewed and approved. A list of significant signatories is included below. Additional signatures may be required for specific procedures.

- Laboratory Director
- Technical Director
- Quality Assurance Officer
- Corporate Officer - ELI Board of Directors

A master list including signatures and initials for all employees is maintained for reference and signature verification.

CHAPTER 5 – SAMPLING PROCEDURES

Private individuals or companies, who are responsible for using proper collection procedures, collect most of the samples processed in this laboratory. Members of the staff are acquainted with proper sample collection and handling procedures and advise those who need help in this area. Instructions and forms for initiating Chain-of-Custody are available from ELI. Laboratory procedures for logging in samples for analysis and maintaining Chain-of-Custody are described in ELI SOP, *Sample Receipt, Login, and Labeling*.

When the laboratory has been assigned the responsibility of sample collection, there is strict adherence to correct sampling protocols, initiation of chain-of-custody, sampling documentation, complete sample identification, and prompt transfer of sample(s) to the laboratory. Procedures are described in ELI SOP, *Field Sampling*.

This laboratory provides proper sample containers and preservatives as specified for the procedure. Certified sample bottles may be ordered upon request. Sample containers, preservatives, coolers for shipping, re-sealable plastic bags for ice containment, trip blanks for monitoring contamination during shipping, temperature blanks for accurately monitoring sample receiving temperatures, Chain-of-Custody forms, Chain-of-Custody seals, sample bottle labels, instructions for sampling, sample labeling, sample preservation, and sample packaging/shipping are provided upon request. Container traceability is available upon pre-arranged request. Sample container type, sample volume, preservation requirements, and maximum holding times, are detailed for each analyte/method in the ELI Professional Services Guide.

Energy Laboratories maintains a strict Sample Acceptance Policy. The client is immediately notified (as appropriate) upon sample receipt, or as soon as possible, if there is any doubt concerning the sample's suitability for testing, including but not limited to, when:

- Samples are out of temperature compliance;
- Samples are received in unacceptable containers;
- Samples have not been properly preserved;
- Samples have labels or chain-of-custody procedures that are incomplete;
- Samples cannot be analyzed within method recommended holding time; or
- The custody seal has been broken.

Notification of sample receipt condition is available through the final report, Energy Source, Email, telephone, and/or voice.

Samples not collected or documented properly can be rejected for any regulatory-based analysis with re-sampling recommended. If re-sampling is not possible, or the client cannot be contacted, the sample may be analyzed, and if analyzed, the sample will be clearly qualified in the data package.

The laboratory will preserve samples at the time of sample login if samples are unpreserved and preservation is required by the methodology. Aqueous samples for volatile analysis are checked

for preservation at the time of analysis. Samples for microbiological analysis are collected in pre-sterilized 120 mL plastic bottles containing sodium thiosulfate.

Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at collection. Refer to ELI Professional Services Guide for detailed information on sample preservation requirements per applicable method and regulatory requirements.

The laboratory initiates a sample condition report titled Work Order Receipt Checklist at the time of sample receipt. The sample condition report contains Chain-of-Custody procedures, sample preservation status, carrier used for sample shipment, sample receipt temperature, and general comments concerning sample condition. The sample condition report is provided with the analytical data report package. For more information, see ELI SOP, *Sample Receipt, Login, and Labeling*.

When any sample is shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements as described in the ELI Professional Services Guide, the Office of Hazardous Materials, Material Transportation Bureau, and Department of Transportation have determined the Federal Hazardous Materials Regulations do not apply to the following:

A) Hydrochloric Acid - (HCl) in water solutions of 0.04 % by weight or less (pH of 1.96 or greater).
B) Nitric Acid - (HNO_3) in water solutions of 0.15 % by weight or less (pH of 1.62 or greater).
C) Sulfuric Acid - (H_2SO_4) in water solutions of 0.35% by weight or less (pH of 1.15 or greater).
D) Sodium Hydroxide - (NaOH) in water solutions of 0.080% by weight or less (pH of 12.30 or less).

For regulatory compliance monitoring, it is required that all samples be analyzed within the prescribed holding times. Holding times are the maximum times allowed between sampling and analysis for results to still be considered valid. Samples should be delivered to the laboratory as soon as possible following collection to assure that holding times can be met. Samples are analyzed as soon as possible after sample receipt. When maximum holding times cannot be met, re-sampling is requested. If samples are analyzed out of hold, data is appropriately qualified.

To ensure that drinking water analysis requirements for radiochemistry analyses are met, the requirements for sample handling, preservation, and instrumentation for radiochemical analysis are included in ELI SOP, *Sample Receipt, Log-In and Labeling*. (For additional information, refer to “Manual for the Certification of Laboratories Analyzing Drinking Water”, Table VI-2: Sample Handling, Preservation, and Instrumentation, EPA 5th Edition, January 2005).

CHAPTER 6 – SAMPLE HANDLING

All ELI laboratories utilize a sample tracking policy that includes client-initiated chain of custody. Upon receipt, the security of the samples is maintained by the implementation of the laboratory access and security policies. See ELI SOP, *Facility Description, Access and Security*.

Sample Receipt

All samples arriving at the laboratory are logged in the Laboratory Information Management System (LIMS). Each sample container is given a unique laboratory sample number. The sample receipt checklist evaluates Chain-of-Custody procedures, sample preservation status, carrier used for sample shipment, sample temperature, and provides general comments concerning sample condition. The completed checklist is provided with the analytical report package. Chain-of-Custody forms are checked for pertinent information. If necessary information has been omitted, the collector is notified, if possible, and the missing information is requested.

Samples requiring preservation are checked to determine if the client performed preservation. If requested, ELI staff will preserve or filter samples as appropriate. Samples that degrade quickly or cannot be opened (such as aqueous samples for volatiles) are not preserved at the time of sample login. If samples are improperly preserved, or the maximum holding times are exceeded upon arrival at the laboratory, the client is notified and re-sampling may be recommended.

Samples are stored per method specifications, or as method/parameter storage requirements are updated per later EPA guidance in Federal Regulations posted in 40CFR Part 136 and Part 140.

During sample login, all sample information such as sample description, client name and address, analyses requested, special requirements, etc. are entered into the computer database of the Laboratory Information Management System (LIMS). Requested analysis parameters and special requirements are communicated to the analysts via their LIMS work lists. Project-specific requirements are maintained in the LIMS for any samples received from a special project. This process ensures that individual requirements are maintained.

Chain-of-Custody

Evidence level internal chain-of-custody (COC) procedures are available on a project-specific basis. For these procedures, internal COC sample custody is maintained down to the individual analyst level. When transferring the possession of the samples, the transferee must sign and record the date and time on the chain-of-custody record. Every person who takes custody must fill in the appropriate section of the chain-of-custody record. For all sample sets received by ELI, sample identification information on the sample containers is compared to the custody report form. The sample is inspected and information regarding the condition of the sample and seal (if used) is recorded on a report form; the method of shipping is also documented on the report form. A copy of the report form is kept with the sample data file and a copy is sent to the

client with the analysis report. Internal chain-of-custody forms are used, when appropriate to document the progress of the sample through the laboratory. ELI's routine COC policy is maintained at the laboratory level through our laboratory access and security policies. See ELI SOP, *Facility Description, Access, and Security*.

Sample Tracking

Samples are tracked through the analytical process by the LIMS. Completed analyses, which have been approved by the appropriate reviewer as valid data, are reported in the LIMS. When all analyses are complete, the data is reviewed as a whole to ensure results pass data quality checks. The completed report is signed by an approved signatory. The signed report is sent to the client via requested delivery format. Generation of the invoice automatically completes the work order in the LIMS and removes the samples from the status report. For more information, see ELI SOP, *Laboratory Records, Notebooks, and Document Management, Control and Archiving*.

Sample Disposal

It is preferred that remaining hazardous sample material be returned to the originator (client) for disposal. When this is not possible or reasonable, ELI will dispose of remaining hazardous sample materials with a waste disposal surcharge added to the cost of the analysis.

The disposal of laboratory wastes will be performed in accordance with local, state, and federal regulations which apply to such activities. Each method SOP addresses waste minimization and management specific to the method procedure. See ELI SOP, *General Laboratory Waste Disposal,* for more information.

Subcontracting Policy

The ELI Billings laboratory utilizes the expanded ELI branch laboratory capability and expertise to provide comprehensive analytical services. This occurs when the laboratory is requested to perform an analysis outside of the laboratory's capabilities: if sample overload is experienced, if equipment is out of service, or when the laboratory is not accredited for the particular analysis. Upon completion of the analyses, the subcontracted ELI laboratories report the sample results, and their quality control package, to the primary laboratory. The results are reviewed before being reported.

All ELI laboratories are certified to perform drinking water analysis in their state and in selected neighboring states. Samples are forwarded to our branch laboratories only if the laboratory is certified in the state from which the sample originated per the individual State certification requirements. Individual ELI laboratory Quality Assurance Programs are consistent with the Corporate Quality Assurance Program and are monitored through internal laboratory audits.

To support Energy Laboratories, Inc. Billings' analytical services, ELI branch laboratories (which maintain specific instrumentation for specialized analysis) are utilized to provide complete

analytical services. Current accreditation certificates for All ELI laboratories are available on the Energy Laboratories website at www.energylab.com.

ELI Billings routinely subcontracts the following parameters/methods to other ELI laboratories:

Total Organic Halogens (TOX) by SW-846 9020
Total Arsenic and Arsenic speciation by CVAA per ASTM 3114
Carbamates by EPA 531.1
Glyphosate by EPA 547
Diquat by EPA 549.2
Total Organic Carbon (TOC/DOC) by A5310C or A5310B, and SW-846 9060A
Oil & Grease by SW-846 1664A
All Radiochemistry except Radon in air

In the event that ELI is dependent on the service of an outside laboratory for analyses not available through our facility or our other branch laboratories, the client is notified that their samples are subcontracted to a pre-approved outside laboratory. The outside laboratory reports the results to ELI and these results become part of the final report. Any external or internal subcontracted analyses that require accredited analyses will be performed by a laboratory accredited for those parameters as required in the State from which the sample originated and/or to meet client-specified required accreditation programs. All final reports indicate where the analyses were performed. Certification files of pre-approved subcontract laboratories are maintained by the ELI QA departments.

CHAPTER 7 – INSTRUMENT OPERATION AND CALIBRATION

Laboratory instruments and equipment are operated and calibrated according to the manufacturer's instructions and according to the requirements of the method being used. Exact calibration procedures are outlined in the appropriate SOP. For most instruments, a calibration curve composed of three to five standards covering the concentration range of the samples is prepared. The acceptance criteria for the calibration curves are listed in the individual methods. Unless otherwise specified in the method, at least one of the standards is at or below the practical quantitation limit (PQL) of the method. Routine PQLs for each method are given in the ELI Professional Services Guide. Calibration standards are routinely compared to second source calibration standards to verify accuracy. These second source standard results must fall within an established range, as described by the SOP, to be considered acceptable. Whenever possible, the laboratory uses calibration standards prepared from certified stock standards. Initial instrument calibration curves are verified and routinely monitored by analyzing a continuing calibration standard every 10 to 20 samples (or within a specified time frequency) and at the end of every analytical sequence, depending on the analysis method and instrumentation. When applicable to the method, high-level samples, which produce an analytical response outside the calibrated range of the instrument, are diluted (or reduced in mass) and re-analyzed until a response within the calibrated range is obtained and/or the result is appropriately qualified.

System cleanliness is verified through the analysis of reagent/instrument blanks prior to analysis, between highly contaminated samples, and at regular intervals during the analysis.

Use of measuring equipment and reagents (glassware, water, chemical reagents, and industrial gases) conform to Good Laboratory Practice guidelines. Good Laboratory Practices (GLPs) are laboratory guidelines which were established by the Food and Drug Administration and published in the Federal Register (21 CFR, part 58). The GLP guidelines were adopted by the Environmental Protection Agency. SOPs are developed in accordance with GLP and NELAP guidelines. Laboratory volumetric glassware conforms to National Institute of Standards and Technology (NIST/SI), American Society for Testing and Materials (ASTM) Class A or B standards. All mechanical pipettes are calibrated at least quarterly. Laboratory balances are serviced and calibrated by certified technicians annually. Calibration checks of balances are performed each day of use, using ASTM Class 1 or 2 weights. Laboratory thermometers are calibrated annually against a reference thermometer traceable to the International System of Units (SI) through a national metrological institute, such as NIST. Laboratory drying ovens, incubators, freezers, refrigerators, and water bath temperatures are monitored and recorded each working day, or at frequencies as described in the specific SOP. Laboratory pure water is generated by commercial water purification systems and is monitored and documented each working day in accordance with specifications needed for applicable methods. The routine analysis of laboratory blanks is used to verify laboratory water quality and the suitability of sampling containers. Chemical reagents and gases meet or exceed purity requirements for their intended uses. Laboratory stock and working standards are derived from ISO/IEC17025 and/or 9001 (or equivalent-certified) commercially available primary standards whenever possible. Standard preparation notebooks document the reagent/standard type, source, purity, content, concentrations, preparation date, and analyst. All calibration standards are

documented in each the analytical records such that they are uniquely identified and traceable to stock standards and their source.

Standard Operating Procedures (SOPs) detail the sequence of operations involved in instrument start-up, calibration, analysis, shut-down, and routine maintenance. Suggestions for corrective action are included with the SOPs and parameters are identified which dictate certain types of maintenance. Instrument and method detection limit studies are performed at the method required frequency or whenever there is a significant change in instrumentation. Method Detection Limits are determined according to EPA guidelines found in 40 CFR, part 136, Appendix B (except for the few methods that are not amenable to MDLs). Refer to ELI's Professional Services Guide for routine practical quantitation limits (method reporting limits). Acceptable instrument response/performance criteria are based upon the manufacturer or the analytical method specifications.

Instrument logbooks and/or electronic logbooks are used to document instrument maintenance and repairs. Instruments that are no longer being utilized are documented in the applicable instrument logbook as "out-of-service" with the date the instrument was taken out of use noted. All out-of-service instruments are labeled with an out-of-service tag that identifies the effective date the instrument was taken out of use.

Laboratory analysts record and document all instrumental sequences in Laboratory Instrument Logbooks, LIMS system, or computer files. Instrument Logbooks and/or dated computer files record instrument performance data, analytical sequences, instrument maintenance, calibration standards data, and any other additional information pertinent to operation of the instrument.

CHAPTER 8 – RECORDS AND REPORTING

Document Management

Energy Laboratories Inc. manages three types of documents: 1) controlled, 2) approved, and 3) obsolete.

A CONTROLLED document is one that is uniquely identified, issued, tracked, and kept current as part of the Quality or Management System. Controlled documents may be internal documents or external documents. Controlled documents are considered to be all documents issued to personnel in the laboratory as part of the management system such as accreditation standards, forms, test and/or calibration methods, and company policies and procedures. All internal ELI controlled documents are written and reviewed by personnel technically competent to perform the procedure and are approved for use by the Laboratory Director, or Director's designee(s).

APPROVED document is one that has been reviewed and approved for use by the Laboratory Director or Director's designee(s).

OBSOLETE document is a document that has been superseded by more recent versions or is no longer being used. Obsolete documents are retained for legal use or historical knowledge preservation. Old or archived SOPs are available for review using the laboratory's electronic document system. ELI's OBSOLETE document records are maintained for at least ten years.

Documents are reviewed on an annual basis to ensure their contents are suitable and in compliance with the current quality systems requirements, and accurately describe current operations. SOPs include a Record of Revision page, which details revisions or reviews. The Quality Assurance Officer maintains a master list of controlled documents.

Procedures for identification, collection, access, filing, storage, and disposal of records are found in ELI SOP, *Laboratory Records, Notebooks, and Document Management, Control and Archiving*.

Laboratory Notebooks

Several different types of Laboratory Notebooks are maintained at the ELI Laboratory. These include, but are not limited to, the following:

Method/Parameter Notebooks
Project Notebooks
Instrument/Equipment Use and Maintenance Notebooks
Standard Preparation Logbooks
Balance Calibration Logbooks
Pipet Calibration Logbooks
General Logbooks

The general purpose of maintaining each of these Laboratory Notebooks is to record the details that may be important in repeating a procedure, interpreting data, or documenting certain operations. Entries in the notebook may include data such as standard and sample weights, pH measurements, instrument operating parameters, preparation of calibration curves, analytical sequences, calculations, recording of instrument operating parameters, sample condition, etc. The analyst's notebook is particularly important in documenting analyses that deviate in any way from routine or standard practices. It can also be an important training record. All pertinent data is to be recorded directly in the notebook. Most notebooks or data records are maintained in electronic format (LIMS, spreadsheets, or databases). Electronic data records are duplicated using hardcopy and/or alternate electronic backup techniques.

It is the responsibility of each analyst to maintain a laboratory notebook according to Good Laboratory Practices (GLP) Guidelines. All physical laboratory notebooks are assigned a unique logbook control number and are assigned to an analyst and/or supervisor. These notebooks remain the responsibility of the ELI staff member to whom they are assigned until they are formally transferred to another staff member, until they are completely filled and returned to the ELI QA Department for archiving, or until the staff member resigns and returns them as a part of the check-out process. ELI staff members, other than the individual to whom the laboratory notebook is issued to, may make entries in the notebook as long as those entries are consistent with the intended use of the notebook and such entries are initialed and dated. Procedures for use and maintenance of laboratory notebooks are detailed in ELI SOP, *Laboratory Records, Notebooks, and Document Management, Control and Archiving*.

Records

The laboratory maintains records of all chemical analyses, including all quality control records, for a minimum of ten years. In the event that Energy Laboratories, Inc., or any individual laboratory transfers ownership or goes out of business, the records will be transferred to the new owners. If an ELI laboratory is closed, records will be maintained by Energy Laboratories Corporate office in Billings, Montana. Energy Laboratories, Inc. reserves the right to offer the records to the clients in the event of complete closure. Details are described in ELI SOP, *Laboratory Records, Notebooks, and Document Management, Control and Archiving*.

Data Reduction

Data reduction refers to the process of converting raw data to reportable units. The reporting units used and analytical methods performed are described in the ELI Professional Services Guide.

Wherever possible, the instrument is calibrated to read out directly in the units reported. In this case, the value is recorded directly into a laboratory notebook, logbook, bench sheet, or electronic file and presented for review.

In cases such as titration, gravimetric measurements, or other techniques that require calculation prior to reporting, raw data is recorded in the appropriate laboratory notebook or electronic file, or on the appropriate laboratory form. The calculations specified in the methods are used to determine the reported value. That value is also entered into the laboratory

notebook or bench sheet. Most calculations are automated to reduce the chance of arithmetic or transcription errors.

Wherever possible, electronic data results are transmitted throughout the laboratory via the LIMS computer network. This process is intended to minimize manual data transcriptions within the laboratory. Additional advantages include the opportunity for rapid comprehensive data validation by supervisors, and more rapid data reporting.

Validation

Data validation includes the procedures used to ensure that the reported values are consistent with the raw data, calculated values, sample type, sample history, and other analysis parameters requested. Data validation also includes review that client-specific DQO's are met.

The data recorded is validated with several review steps. The analyst who submits the analytical results checks all the values reported for omissions and accuracy. Elements of this review also evaluate all instrument and method QC results. Automated data management programs are designed with an interactive step allowing data review by the analyst. Results to be reported are approved by the analyst or supervisor.

The report is reviewed for the suitability of the data according to project and method performance specifications. Analytical results for each requested parameter may be evaluated against other requested parameters, project specifications, other samples within the set, historical files associated with the project/client, and/or any other information provided with the sample.

The reports are generated, proofread, and reviewed by designated reporting staff.

The Laboratory Director, project managers, supervisors, Quality Assurance Officer or their designees, may also examine the data included in the final report.

Internal and external laboratory audits review selected sets of data to ensure that the analytical results are correct and accurate, analytical methods are appropriate, documentation and record keeping procedures are complete, and that there is compliance to the overall objectives of the Quality Assurance Program. Data integrity is monitored on an on-going basis. See ELI SOP, *Assessment of Data Integrity*, for details.

All controlled automated programs used to process and report data are initially verified using manually calculated results. Whenever a modification is performed to a program, re-verification of overall software function is performed.

One step of the Quality Control process involves data outlier detection; data that falls outside of established limits. If an outlier is observed, corrective action is taken as appropriate, to investigate and/or correct the cause. Actions to correct these causes may include, but are not limited to, inspection of the instrumentation, checking calibrations, checking sample numbers or dilutions, re-analyzing samples or calibrations.

Reporting

One copy of the report is distributed to the client, via requested delivery format, after the report is validated and signed. A standardized report format is used unless otherwise specified. Client-specified report formats are available upon request. Results can be sent via physical media, email, EDD, website FTP and/or FAX when requested by the client. Energy Laboratories, Inc. offers its clients access to electronic records through our Energy Source Portal.

Various levels of data reporting are available. All analytical results, regardless of the level of reporting used, have record keeping procedures which allow an appropriate "data validation package" to be produced. Note that a comprehensive "data validation package" is most easily generated at the time of sample analysis. Example data packages are available upon request. Maximum contaminate limits and/or decision rules per applicable regulation may be included on analytical reports per type of regulatory analysis being requested.

Safe Drinking Water Act (SDWA) compliance monitoring samples for microbiological and chemistry samples that exceed the SDWA maximum contaminant level (MCL) may require notification to the appropriate state agencies. Generally, notification to the client, and to the state, of any SDWA MCL exceedance must be within 24 hours of completion of analysis/review, or by noon the next business day. If requested by the client, additional copies of the report will be sent to a specified address or person.

The final copy of a completed report is maintained in an electronic format. An electronic copy of this file is available upon request. Energy Source is a client resource of ELI that provides secure online access for clients to view their data and documents. Clients are able to access their electronic files through ELI's secure website at *https://energysource.energylab.com/*. For more information, see ELI SOP, *Laboratory Records, Notebooks, and Document Management, Control and Archiving*.

In addition to traditional ink signatures, Energy Laboratories has approved the use of electronic signatures within our company-produced PDF documents. These signatures comply with Title 15 of the US Code Section 101 regarding legal requirements of a digital signature.

Electronic signatures verify that the document has not changed after it was produced. Upon opening the document, notifications automatically display to inform the recipient of the validity of the sender's electronic signature and all included certificates. Should any changes be detected, an alert message is automatically displayed, noting that the signatures cannot be validated due to changes made to the document. Detailed instruction on how to view/validate ELI's electronic signatures is available.

CHAPTER 9 – GENERAL LABORATORY PRACTICES

Chemicals and Reagents

When available and appropriate, chemicals used in the laboratory are ACS (American Chemical Society) analytical reagent grade chemicals purchased from reliable suppliers, preferably ISO accredited suppliers, and which meet referenced method specifications. Reagents are prepared, standardized, and made fresh as mandated by the method, their stability, and according to Good Laboratory Practices. Procedures for purchasing of materials may be found in ELI SOP, *Property Procurement, Inventory, and Control*.

Normalized standards are checked regularly against independently prepared reference materials.

All standards and reagents are dated when received, opened, or prepared, and each is labeled with an expiration date when applicable. Standards and reagents are checked for discoloration or signs of degradation and are discarded if these are observed.

Certified primary standards are obtained from ISO accredited commercial sources when available. Standards used for calibration are verified against second source standards. Secondary and working standards are accurately prepared with volumetric flasks, or other calibrated lab ware, from primary standards and stored in appropriate containers.

ELI has determined twenty years to be a reasonable expiration date for stable salts where the manufacturer does not supply such information. Titrants, standards, and other solutions used for analytical purposes are frequently standardized upon preparation with certified or traceable standards. Method SOPs specify if standardization is necessary. The date and analyst's initials must be recorded on the container whenever re-standardized and these records are maintained in a laboratory notebook or in the LIMS.

Individual SOPs may also provide additional details for reagent requirements.

Reagent Interference

To determine the extent of reagent interference, method blanks are analyzed prior to sample analysis whenever appropriate.

If any interference cannot be eliminated, the magnitude of the interference is considered when calculating the concentration of the specific constituent in the sample, but only when permitted within the applicable method.

If reagents, materials, or solvents contain substances that interfere with a particular determination, they are replaced.

Individual method SOPs may also provide additional requirements for handling reagent interferences.

Glassware Preparation

All glassware used for inorganic and radiochemical analysis is washed in warm detergent solution and thoroughly rinsed in tap water. Glassware is then rinsed well three times with laboratory-purified water. This cleaning procedure is sufficient for many analytical needs, but individual SOPs detail additional procedures when necessary. Glassware washing procedures for inorganic analyses are described in ELI SOP, *Inorganic Glassware Washing.*

All glassware used for organic analysis is washed in warm synthetic detergent solution and thoroughly rinsed in tap water. The glassware is then rinsed well with laboratory-purified water, followed by rinses with acetone to remove any residual organics. Prior to use, the glassware is rinsed three times with the organic solvent to be used with the glassware. Glassware washing procedures for cleaning glassware for organic analysis are described in ELI SOP, *Cleaning of Glassware Used in Volatile and Semivolatile Analyte Sample Preparation and Analysis*.

All glassware used for microbiological analysis is washed in warm detergent solution. The detergent must be proven to contain no bacteriostatic or inhibiting substances. The glassware is rinsed thoroughly with laboratory-purified water. Specific details are described in method specific SOPs.

Disposable, glassware/plastic ware is preferred for many procedures in the laboratory. The cleanliness and suitability of disposable glassware/plastic ware is continuously evaluated for each test with the routine analysis of method blanks.

All volumetric glassware used in precise measurements of volume is Class A or laboratory calibrated.

Laboratory Purified Water

Laboratory-purified water is used in the laboratory for dilution, preparation of reagent solutions and final rinsing of glassware. For organic analysis, organic-free water is prepared and used. Energy Laboratories, Inc. uses water purification systems that are designed to produce deionized water that meets the requirements of the methods. Use and maintenance of laboratory reagent water systems are described in ELI SOP, *Use and Maintenance of the Milli-Q Water System*.

Water quality is monitored for acceptability in the procedure in which it is used. Specific details are listed in the appropriate SOPs.

Employee Training

All new ELI employees and contract personnel are given an initial general orientation and tour of the laboratory facilities. Personnel are shown the locations of safety equipment such as safety showers, eye wash fountains, fire extinguishers, and first aid supplies. Personal protective equipment such as lab coats, disposable gloves, and safety glasses (if applicable) are issued at this time.

Safety considerations are a vital part of the training process. All hazards associated with the performance of a procedure or with the operation of an instrument are to be understood by the trainee before training can be considered complete. General laboratory safety procedures are a part of the new and current employee training. Specific safety procedures are outlined in SOPs and in instrument Operator's Manuals. Training in use of protective clothing, eye protection, ventilation, and general safety are provided to each employee. Each employee is required to read and sign the *Laboratory Safety Manual & Chemical Hygiene Plan*.

All new and existing employees must demonstrate capability prior to performing an analytical procedure independently (see Chapter One). Method performance on Quality Control Samples is used to document employee training and work quality. Employees are required to read the Quality Assurance Manual and all appropriate SOPs. Employees are required to sign, for all applicable Manuals and SOPs, a Record of Acknowledgement Form that states they have read, understood, and agree to abide by the Manual/SOP.

Employees also receive training on general laboratory policies including ethics and conflict of interest. All employees are required to read, understand and comply with the Corporate Compliance & Ethics Manual. Data integrity training is provided for all employees initially upon hire and annually thereafter. In addition to the *Corporate Compliance & Ethics Manual*, the ELI Quality Assurance department maintains a *Laboratory Ethics & Data Integrity Manual*, which supplements the corporate manual and provides specific training on data integrity. All employees are required to read, understand and comply with the ELI *Laboratory Ethics & Data Integrity Manual*. An annual Ethics training course is given to all laboratory employees. Attendance is required and is recorded with a signature attendance sheet or other form of documentation that demonstrates all staff members have participated and understand their obligations related to data integrity and ethics policies. For details pertaining to ethics training and additional ethical procedures and policies refer to ELI SOP, *Personnel Training and Training Records*.

ELI encourages attendance at courses, workshops and other forms of continuing education available from on-site seminars, webinars, private institutions, local schools, and State and Federal regulatory agencies. Staff and department meetings are held routinely to communicate company policies and procedures. All training on procedures and policies is documented, per NELAP guidelines, in employee training files. For more information see ELI SOP, *Personnel Training and Training Records*.

Data Integrity

To provide data of known quality Energy Laboratories Inc. activities, policies, and procedures are structured and managed to safeguard impartiality. In order to provide for the security and integrity of ELI and client data, the laboratory has multiple controls on the network, LIMS and applications used. These controls limit access to and the ability to change data as well as provide for redundancy in case of loss.

These include but are not limited to:

- Users connecting to ELI computer systems are authenticated through a user name and password combination.
- Passwords are required to be changed on a regular basis.
- Permissions within ELI applications are role based with different roles having various levels of access and control. Users (analysts, supervisors, and Directors) are assigned to these roles.
- In the LIMS, analytical data locks after a period of time and cannot be modified without special handling.
- Certain information has been identified for additional tracking and logging. Changes to this information is not only tracked in an audit log but also reported to select personnel.
- Information on ELI servers including the ELI LIMS system is backed up and recoverable.

Standard Operating Procedures

Laboratory operations and procedures are documented in Standard Operating Procedures (SOPs). SOPs provide information regarding the consistent and safe operation of the laboratory. For analytical methods, SOPs provide information on the details of the analysis that may not be specified in the published reference analytical method(s). For routine procedures other than analytical methods, SOPs define the steps required in accomplishing a given task. All SOPs are reviewed and updated periodically to reflect any changes in laboratory operations. Method SOPs follow NELAP requirements. For more information on generation and distribution of SOPs, see ELI SOP, *Preparation, Numbering, Use, and Revision of Standard Operating Procedures*.

Client Confidentiality

Each employee has the responsibility to maintain confidentiality in all matters pertaining to our clients, samples submitted, and Energy Laboratories, Inc. Information obtained during employment with this laboratory, regarding the specific business of this laboratory, or its clients shall at no time be revealed to any outside sources without permission from the owner of the data.

Sample submittal, analysis and the report contents are considered confidential information of the client. When requested to provide results (either in person, via telephone or email), the employees shall verify that the requestor is either the person associated with the project, on the COC, or on a list provided by the client who are authorized to receive data. If a person who is not associated with the project personnel (or is not on the approved list), the base client will be contacted to inquire about authorization to release data. These contacts are documented and associated with the work order in the LIMS system to provide archival proof of authorization to release data. If the client does not authorize a release of data, the requestor will be contacted and informed of this decision.
Client confidentially is maintained electronically through the use of password-protected logins on all laboratory computer systems. Additionally, the laboratory maintains network security such as anti-virus programs and firewalls that prevent any unauthorized outside access. All copies of the original report are stored on the laboratory's document archival system, which is also

protected from unauthorized use by the network security systems. Raw data, reports, and LIMS records are kept in a secure location of the laboratory or off-site. All client confidential paper waste, including printouts, is shredded.

When the laboratory is required by law or authorized by contractual arrangements to release confidential information, the customer or individual concerns shall, unless prohibited by law, be notified of the information provided. As example, samples provided for Safe Drinking Water Act compliance monitoring, as per individual state regulatory requirements, may also need to be reported to the applicable state agency.

An individual acting on the laboratory's behalf shall keep confidential all information. Information about the customer obtained from sources other than the customer (e.g. complainant, regulators) shall be confidential between the customer and the laboratory. The provider (source) of this information shall be confidential to the laboratory and shall not be shared with the customer unless agreed by the source.

CHAPTER 10 – QUALITY CONTROL MONITORING

Routine Monitoring

Temperatures of incubators, water baths, refrigerators, and ovens are checked and recorded according to a prescribed schedule and using an automated continuous monitoring system. In the event that the automated monitoring system is inoperable, the temperatures will be recorded manually on instrument specific forms.

Conductivity of the laboratory-purified water is continuously monitored using an automated monitoring system and as method blanks in routine analytical sequences.

Reagents are dated and initialed at the time of receipt. Expiration dates are assigned as a fundamental component of their receipt and/or preparation. Reagents are not used after manufacturer's expiration date is exceeded.

Balances are checked daily, or as required, against ASTM Class 1 or 2 weights traceable to the International System of Units (SI) and are calibrated and serviced by certified technicians annually.

Method and Quality System SOPs are reviewed annually for accuracy.

Laboratory Notebooks are reviewed periodically for correctness and accuracy by supervisors and by internal and external auditing.

Proficiency Testing (PT) Samples are analyzed as required (See Chapter 2 of this QA Manual).

Quality Control Check Samples are analyzed with each analytical batch.

Internal and external audits are performed as specified or requested (See Chapter 2 of this QA Manual for additional discussion).

Additional monitoring requirements may also be specified in individual SOPs.

The Laboratory maintains an active fraud protection program that is implemented through the laboratory ethics policy. Additionally, the potential of fraud is monitored through analyst supervision, management supervision, regular internal audits, PT study participation, and an active quality assurance program.

Instruments/Methods

Calibration is performed as outlined in Chapter 7 of this QA Manual.

Generally, and depending on method requirements, the standard curve is verified with a known second source reference sample. The reference sample results must fall within the appropriate target range for the calibration to be considered acceptable.

In most cases, the calibration stability is checked by analyzing a continuing calibration standard every 10 to 20 samples, depending on the analysis and instrumentation. The verification standard results must fall within an established range as described by the SOP. Corrective actions steps are defined by SOP or by project specific requirements.

All laboratory instruments are subjected to preventive maintenance schedules. Preventive maintenance schedules are specified in instrument maintenance logbooks.

As appropriate, instrument and/or method detection limits are determined annually, or more frequently if changes in instrument performance are noted or per method requirements. Procedures for the determination of instrument detection and method detection limits are described in ELI SOP, *Determination of Method Detection Limits (MDL) and Quantitation Limits and Initial Method and New Instrument/Equipment Validation.* The MDL procedure includes a verification of the statistically-determined MDL with a Limit of Detection (LOD) verification sample analysis spiked at a level near the MDL to verify the reasonableness of the calculated MDL and to determine/verify a minimum LOD level. ELI-Billings follows for all applicable procedures, DoD QSM Version 5.4 guidance/requirements and definitions for performing MDL, LOQ, and LOD analysis. If within assigned accuracy acceptance criteria, LOQ analyses may be done at levels lower than the PQL and closer to the LOD.

Precision and accuracy requirements for each method are specified in the SOPs. General guidelines are given below.

- Each analytical batch will contain QC samples to measure the accuracy of the method. Each QC sample result is monitored to be within QC specifications of the method. Results of blank spiked sample analysis must be within the established control limits. Quality Control Limits are specified in the SOPs and meet recommended QC limits as described in the referenced method.

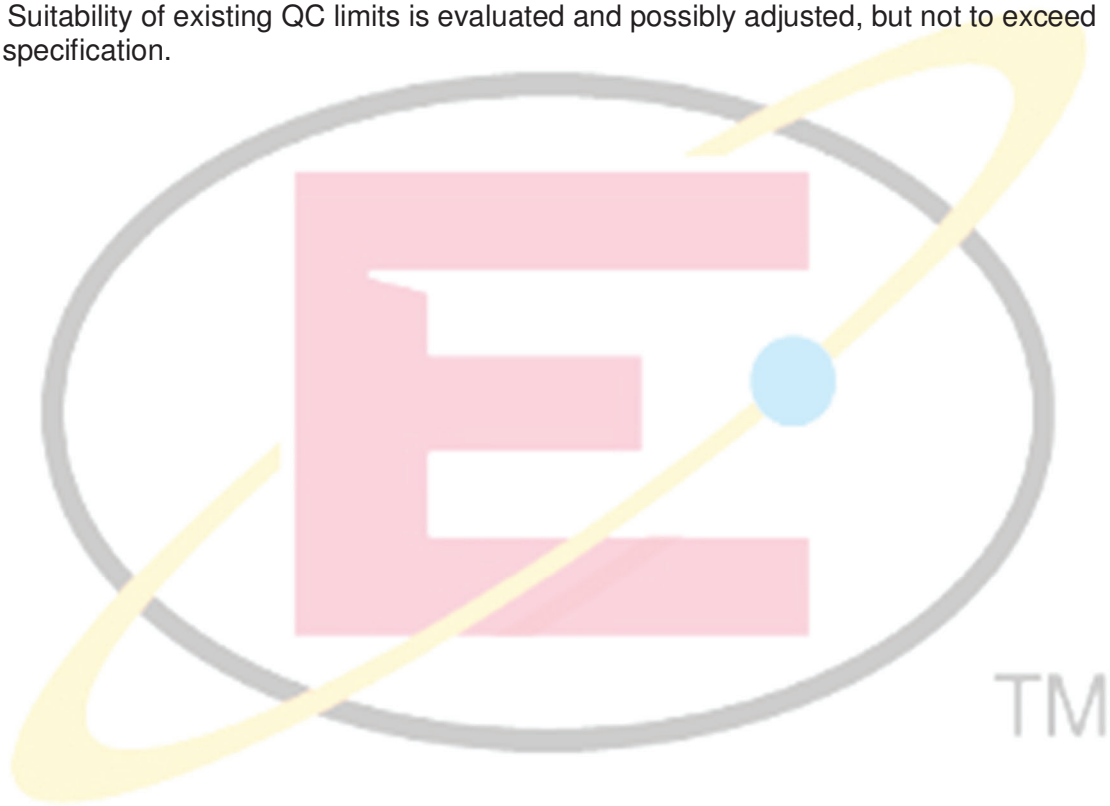
- Each analytical batch will contain QC samples to measure the precision of the method. (See Chapter One for discussion on duplicate sample analysis.) Criteria for duplicate sample acceptance are found in the SOP and are generally taken from the referenced method.

- Each analytical batch will contain QC samples to measure the performance of the method on the sample matrix. These are typically identified as a matrix spike analysis and may be performed in duplicate to assess method precision. Typically the sample is fortified with a known amount of target analyte and spike recoveries are calculated. Results outside of method QC guidance are flagged. Quality control limits and

appropriate corrective actions steps are specified in the method SOP or by client-specific project requirements.

- Several methods are considered to be concurrent methods in that they are either nearly identical or are identical to a method with a different citation. Even if two methodologies are identical in procedure, slight differences in the QC requirements might be the only difference between the two methodologies. These types of methods may also be considered "concurrent" if the procedures are identical and the more stringent of the two method criteria are used. During data reduction and reporting, the referenced method specifications and criteria will always take priority.

As appropriate, the performance trends of QC sample results are evaluated with Quality Control Charts. Suitability of existing QC limits is evaluated and possibly adjusted, but not to exceed method specification.



CHAPTER 11 – CORRECTIVE ACTION

When the quality control checks indicate that an analysis is not within the established control limits, corrective action is needed. This section gives general guidelines for corrective action. Corrective actions for each method or instrument are detailed in individual SOPs. Records are maintained of non-conformances requiring corrective action to show that the root cause(s) was investigated, and includes the results of the investigation. The Quality Assurance Officer will monitor implementation and documentation of the corrective action to assure that the corrective actions were effective.

Method QC samples that fail to fall within QC control limits may be analyzed again to verify if a problem exists. However, matrix spike or matrix spike duplicate QC samples are not required to be re-analyzed if the performance can be attributed to matrix effects; data results are then reported and properly qualified.

If the repeat analysis is not within control limits, the particular instrument or procedure is checked according to the specific protocols outlined in the method or according to the instrument manufacturer's guidelines. Results within acceptable control limits must be reestablished before the instrument can continue analysis. Analysis of all samples that were analyzed while the procedure was out of control must be repeated. In the case of radiochemical analysis, the term “analyze again” means to recount the final sample on the same (or different) detector.

If the analyst is unable to achieve acceptable results after following the corrective action guidelines detailed in the SOP, or by project specifications, a supervisor and/or technical director is consulted. If necessary, the appropriate service personnel are contacted if the problem is determined to be due to instrument error, and cannot be resolved. It is also possible that the result is due to statistical variation of the results based on the tolerable error rate that has been determined for the analysis (usually 0.05). In certain cases, where control limits are exceeded, it is possible that problems cannot be corrected to satisfy QC criteria. This could be due to problems such as matrix interference, instrument problems, lack of sufficient sample, missed holding times, high blank contamination, etc. If all possible solutions available to correct the problem are examined and the sample results are still considered valid, qualifying comments are attached to the sample report describing the non-compliance and probable cause.

In the case of a single radiochemistry detector being returned to service, this refers only to the samples counted on that detector. For example, an individual gas proportional counter instrument may have up to 16 detectors; if only one does not pass the QC check the others are still valid and sample analyses performed on the others do not need to be repeated.

In the event that a QC audit or other informational review shows an analysis report to be incorrect, incomplete, or adversely compromised, a revised report and explanation is submitted to the client within ten business days unless otherwise communicated to the client with another time period. The report will clearly be identified as a revised report. As appropriate, an explanation submitted to the client should give a detailed review of the problem and document

any unapproved deviations from the regulations, standard operating procedures, or project-specific scope of work that may have caused it. The explanation to the client may include, but not be limited to, the following components:

1) What actions have been taken regarding the affected data set(s),
2) Identification of the cause, and
3) Corrective action(s) taken to prevent future occurrence.

In the event that a QC check fails, the analyst will follow the procedures outlined in the QA/QC summary of the SOP.

Quality Control Checks for each method or instrument may vary. Energy Laboratories Inc. follows the QC checks set by each governing method. Due to the wide variations between methods, specifics are listed within each SOP for the given method. Please reference the SOP for specific QC checks for the given method. The QC checks may include: ICV, MB, CCV, CCB, LCS, LCSD, LOD, MS, MSD or others specific to that method.

A summary of Quality Assurance/Quality control specifications and QC corrective actions for representative methods is outlined in Appendix B. Any deviation from the SOP/method shall be documented in laboratory records.

Procedure for Dealing with Complaints

DEFINITIONS

Complaint: For the purposes of this procedure, a complaint is an expression of dissatisfaction from a client, a user of our data, or employee. The complaint might cover issues about the quality of our data, sample turnaround time, method used, pricing, or other expectations and for which a response is expected.

Client: The client is a person or company that ordered and paid for the services.

Procedure: The staff person receiving the complaint exercises judgment in deciding the severity and disposition of every complaint. The judgment must be used to decide whom, if anyone is alerted to the complaint and what actions are appropriate. The complaint issued should be handled with a high degree of discretion and tact by the supervisor or Director involved. The individual handling the complaint is instructed to follow ELI's guidelines provided in this section on how to handle the complaint. This involves listening to the client and getting adequate information so the complaint can be investigated and resolved. The appropriate laboratory staff are notified and a response plan is made with a timeline for action, which is communicated to the client. Records are maintained regarding the complaint and of the investigations and corrective actions being taken.

After the complaint is investigated or resolved, as necessary, the client is made aware of the results and determination is made as to what further actions are needed. Complaints and investigations may result in the need to submit a revised report or invoice. Complaints that are straightforward and can be resolved using the resources available to the person handling the complaint should be resolved there. These include such things as minor revisions of reports or invoices. If other decisions need to be made, the appropriate person should be contacted.

It may be appropriate to initiate or prepare a corrective action report. This report should be completed with the intention of informing the affected staff about the problem so that all relevant staff can use it as a learning opportunity, change our procedures and improve our service. A procedure to document corrective action reports is in ELI SOP, *Nonconformance, Root Cause Analysis and Corrective Action Procedures.*

If an employee sees an issue, they are encouraged to report concerns regarding Quality Systems, unethical behavior, and/or financial mismanagement. This issue should initially be brought to the attention of their supervisor. The supervisor will take appropriate action to resolve the concern. If the employee is uncomfortable with approaching their supervisor or feels that the issue was not properly dealt with, they may approach higher levels of management with their issue.

Energy Laboratories, Inc. has also implemented a program to facilitate confidential reporting to upper management. This tool allows employees to report situations or behaviors that they consider to be unethical, immoral, or improper. It also allows the reporting of suggestions or comments. The program has been implemented at ELI so that anyone reporting a situation can be assured that there will not be retaliation for reporting. It is meant to encourage parties to

communicate with upper management when there appears to be no alternative for resolving the types of issues already described. Access to the program is available on the ELI internal website. Complaints, suggestions or comments from clients, vendors, auditors, and other interested parties can be submitted directly to project or laboratory management who will initiate resolution.

Penalty for Improper, Unethical or Illegal Actions

Energy Laboratories, Inc. employees are expected to work in an ethical, proper, and legal manner. They are expected to perform laboratory analyses according to the cited method(s) and in conjunction with the SOP and the Quality Assurance Plan. Employees are expected and required to report any violations of this policy. All employees are mandated to participate in an ethics-training program as part of their orientation upon hire.

Improper, unethical, or illegal actions by an employee will be addressed on a case-by-case basis as determined by the seriousness of the offense. Corrective actions may include disciplinary action up to and including discharge.

CHAPTER 12 – MANAGEMENT OF CHANGE

Management of change is the process used to review and manage proposed changes to materials, technology, equipment, procedures, personnel and facility operations. These changes may be permanent or temporary depending on circumstances. Change is managed, communicated, and documented as appropriate to the level of change, by the Laboratory Director, QA Officer, and Supervisors of each department. Significant revisions to controlled documents may require employees to sign a record of acknowledgement.

- New Equipment Validation – Documented in the Instrument Maintenance Module. Supporting studies are documented in the LIMS.
- Implementation of new test methods and method updates – Documented in the method SOP and the Instrument Maintenance Module. Supporting studies are documented in the LIMS.
- The QA Manual and SOPs – Documented in the Record of Revision and stored in the Document Control Software.
- Work order changes - Documented in the work order report and stored in the LIMS or Document Control Software.
- LIMS changes - Documented in a version control repository.
- Personnel changes - Documented in employee training records or personnel records.

CHAPTER 13 – MAJOR EQUIPMENT AND METHODS

A summarized listing of major instrumentation utilized in the laboratory is included in Appendix E. Refer to ELI's Professional Services Guide, located on the ELI website at www.energylab.com, for a complete list of available analytes and methods supported by ELI.

CHAPTER 14 – PREVENTIVE MAINTENANCE

Preventive maintenance is performed on laboratory equipment according to the manufacturer's guidelines and our operational experience. Repairs and maintenance are accomplished in-house by experienced laboratory personnel whenever possible. Other than consumable equipment items, an inventory of spare parts is not maintained. Spare parts are available from outside vendors on an as needed basis. (To ensure method capability, some methods have more than one instrument available). An example of maintenance performed follows:

Instrument	**Maintenance**	**Frequency – Note that Daily is based on use.**
Balances	Check with appropriate Class weights	Daily
	Perform Internal Calibration	As needed – when daily check does not meet acceptance criteria
	Independent Calibration and Service	Annually
Thermometers	Calibration Verification	Annually-Liquid Quarterly-Electronic
Pipettes	Check volume	Quarterly, DoD daily prior to use
Ion Chromatograph	Replace Guard Column	As Needed
	Replace Analytical Column	As Needed
	Calibrate	Monthly, after maintenance, or as needed
	Clean Stator Plate	Annually
	Replace tubing	As needed
	Calibrate Conductivity Cell	Every 6 months
ICP-Atomic Emission	Check Pump Tubing	Daily
	Check Coolant Levels	Monthly
	Lubricate Autosampler	As needed
	Air Filter	Quarterly
	Optics Servicing	As needed
ICP-Mass Spectrometry	Check Pump Tubing	Daily
	Check Coolant Levels	Monthly
	Check Electron Multiplier	Daily
	Lubricate Autosampler	As needed
	Air Filter	Quarterly
Gas Chromatograph	Replace Septum	As needed/per # of injections
	Check Injection Liner	Daily
	Clean Detector	As needed
	Change Gas Cylinders	At 200 psi
	Change Column	As needed
Auto Analyzers		
	Check For Leaks	Daily
	Change Tubing	When wear is visible
	Lubricate Pumps	Annually
	Lubricate Sampler	Annually
Metrohm Auto-titrator	Visually inspect all probes/ stirrer/ thermometer and fill probes	Daily/As needed
	Flush pH probe/ Fluoride probe	Every 15 days
	Calibrate sample dosing pump	Quarterly
	Replace Tubing	Annually/ As needed
	Clean out titration vessel and rinse station	Quarterly/ As needed
	Clean buret	Quarterly

Instrument	**Maintenance**	**Frequency – Note that Daily is based on use.**
	Calibrate buret	Monthly
	Replace pH/ Fluoride probe	As needed
	Replace Tubing	As needed
	Replace Lip seals gland washers on dosing pump	As needed
Metrohm-automated pH, conductivity, ion electrode analyzer	Visually inspect all probes/ stirrer/ thermometer and fill probes	Daily/As needed
	Flush pH probe/ change storage solution	Monthly/ As needed
	Replace Tubing	As needed
	Calibrate buret	Monthly
	Replace pH probe	As needed
Mass Spectrometers	Monitor Vacuum Pressures	Daily
	Monitor Background Levels	Daily
	Monitor Electron Multiplier	Daily
	Change Pump Oil	As Needed
Microbiology	Monitor Room Temperature	Twice daily
	Monitor Incubator Temperature	Twice daily
	Autoclave Maintenance	Annually
	Monitor Water Bath Temperature	Twice daily
Reagent Water Systems	Change/Check Cartridges	Quarterly, or as needed
Compressed Gases	Change Gas Cylinders	At 200 psi, monitor daily
Liquid Chromatograph	Flush System	Daily
	Replace Filters	As needed
	Replace Seals	As needed
Continuous Temperature Monitoring Systems	Check Temperatures	Daily, calibrate annually

CHAPTER 15 - REFERENCES

ANSI N42.23-1996, American National Standard Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories.

ASTM Annual Book of Standards, Part 31 (water), American Society for Testing and Materials.

ASTM D 7282-06 Standard Practices for Set-up, Calibration, and Quality Control of Instruments Used for Radioactive Measurements.

Handbook for Analytical Quality Control in Water and Wastewater Laboratories, Environmental Protection Agency. EPA 600/4-79-019

ELI Professional Services Guide (Fee Schedule), Current Revision, Energy Laboratories, Inc.

Manual for the Certification of Laboratories Analyzing Drinking Water, 5th Ed., EPA 815-R-05-004, 2005.

Manual for the Certification of Laboratories Analyzing Drinking Water, Supplement to 5th Ed., EPA 815-F-08-006, June 2008.

Methods for Chemical Analysis of Water and Wastes Environmental Protection Agency, 600/4-79-020.

Methods for the Determination of Metals in Environmental Samples – Supplement I, EPA/600/R-94-111, May 1994.

Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93-100, August 1993.

Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, December 1998.

Methods for the Determination of Organic Compounds in Drinking Water – Supplement I, EPA/600/4-90/020, July 1990.

Methods for the Determination of Organic Compounds in Drinking Water – Supplement II, EPA/600/R-92/129, August 1992.

NELAC Chapter 5: Quality System Standard, 2003, 2009, or 2016, most current version approved by Florida and Texas NELAC Accreditation program.

NELAP, National Environmental Laboratory Accreditation Program, The NELAC Institute (TNI) https://nelac-institute.org/index.php

Standard Methods for the Examination of Water and Wastewater; 20th, 21st 22nd and -23rd Editions, APHA.

Technical Notes on Drinking Water Methods, EPA/600/R-94/173, October 1994.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW846), Environmental Protection Agency. https://www.epa.gov/hw-sw846

Management and Technical Requirements for Laboratories Performing Environmental Analysis, TNI Standard, Volume 1 (EL-V1-2009), The NELAC Institute.

Management and Technical Requirements for Laboratories Performing Environmental Analysis, TNI Standard, Volume 1 EL-V1-2016 Rev2.1, ELV1M4-2017-Rev2.2, The NELAC Institute.

DoD Quality System Manual (QSM) for Environmental Laboratories, Version 5.4, U.S. Department of Defense, October 2021.

General requirements for the competence of testing and calibration laboratories, ISO/IEC 17025, Second edition, 2005; Third edition 2017-11

Risk Management – Guidelines, ISO 31000, 2nd Edition 2018-02

CHAPTER 16 – GLOSSARY OF TERMS

Acceptance Criteria - Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accreditation - The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy - The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.

Analyte - A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed.

Analyst - The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Sample - Any solution or media introduced into an instrument on which an analysis is performed, excluding QC samples such as: instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification, and continuing calibration blank.

Assessment - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation).

Audit - A systematic and independent examination of facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Batch - Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours unless otherwise specified by method SOP. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples.

Blank (BLK) - A sample of clean matrix, which accompanies the samples through different aspects of sampling and/or sample preparation. It is used to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and

measurement process to establish a zero baseline or background value. There are various types of blanks: equipment blank, field blank, instrument blank, method blank, and reagent blank.

> **Method Blank** - A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Blank Spike - See Laboratory Fortified Blank.

Blind QC Check Samples - Samples whose analyte concentrations are not known to the analyst. That the sample is a QC check sample may or may not be known to the analyst.

Calibration - A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).
2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Check Standard - See Check Standard.

Calibration Curve - The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Standard - A substance or reference material used for calibration.

Chain of Custody Form - Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. See also Legal Chain of Custody Protocols.

Check Standard - A material of known composition that is analyzed concurrently with test samples to evaluate a measurement process.

Clean Water Act - Public Law PL 92-500. Found at 40 CFR 100-140 and 400-470. The act regulates the discharge of pollutants into surface waters.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) - The enabling legislation (42 USC 9601 - 9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 USC 9601 et seq.), to eliminate the health and environmental threats posed by hazardous waste sites.

Confirmation - Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: Second column confirmation, Alternate wavelength, Derivatization, Mass spectral interpretation, Alternative detectors, or Additional cleanup procedures.

Constant Weight - The repeated process of drying, cooling, desiccating, and weighing a sample until readings are ≤4% of the previous weight or does not vary more than ≤0.5mg.

Continuing Calibration Blank (CCB) – A sample of laboratory purified water or matrix similar to calibration standards, in which no analytes of interest are present at concentrations that impact results, measured periodically throughout an analytical run. Evaluates baseline drift, contamination in the analytical system, and analyte carryover.

Continuing Calibration Verification (CCV) - A mid-range calibration standard measured periodically throughout an analytical run that evaluates instrument drift throughout analytical run.

Control Limits - A range within which specified measurement results must fall to be compliant.

Control Standard - See Check Standard.

Corrective Action (CA) - An action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

Data Integrity - The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.

Data Reduction - The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more useful form.

Data Quality Objectives (DQO) - An integrated set of specifications that define data quality requirements and the intended use of the data.

Decision Rule – Rule that describes how measurement uncertainty is accounted for when stating conformity with a specific requirement.

Demonstration of Capability - A procedure to establish the ability of the analyst to perform analyses with acceptable accuracy and precision.

Detectability – For radiochemical analysis, detectability as a Lower Limit Detection (LLD) or Minimum Detection Concentration (MDC), is assessed based on the requirements of 40 CFR

141.25(c) and is a sample-specific determination. The equation is specific for each method and noted in the method SOP.

Detection Limit - See Practical Quantitation Limit and Method Detection Limit. Reporting of detection in radiochemistry is based on specific formulas identified in individual procedures. Single activity point standards are used for efficiency calibration. When required, multiple energy emitters are used for energy calibration.

Document Control - The act of ensuring that documents and revisions are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Duplicate (DUP) - A second aliquot of a sample that is treated the same as the original sample to determine the precision of the method.

Duplicate Sample - See Duplicate.

Field of Accreditation - Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Finding - An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.

Fortified Sample - See Matrix Spike.

Holding Times (Maximum Allowable Holding Times) - The maximum time that can elapse between two (2) specified activities. Sample holding time is based on Date/Time of Collection and Date/Time of the beginning of sample analysis. Time is based on hour/minute by default or by the accreditation requirements for a project. The maximum time is the longest time period that samples may be held prior to analysis and still be considered valid or not compromised.

In-depth Data Monitoring - When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.

Internal Standard - A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Impartiality - The presence of objectivity which is managed by procedures and processes to avoid conflict of interest, freedom from bias, lack of prejudice, neutrality, fairness, open-mindedness, even handedness, detachment and balance so as not to adversely influence subsequent activities of the laboratory.

Initial Calibration Verification (ICV) - A sample of known concentration, from a source other than that of the calibration standards, analyzed following calibration to demonstrate validity of the calibration and standards used.

Instrument Blank - See Calibration Blank.

Internal Standard – A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, Initial calibration verification (ICV) or QC check sample) - A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Laboratory Control Sample Duplicate (LCSD) - A second laboratory control sample of known concentration and similar matrix as samples. Evaluates overall method accuracy/bias and precision for the batch.

Laboratory Fortified Blank (LFB) – A sample of laboratory purified water or matrix similar to the calibration standards to which a known amount of target analyte(s) is added. Evaluates spiking technique and when prepared from a source independent of the calibration standards can also be used to measure method performance.

Laboratory Inter-comparison Sample - A sample, typically a performance evaluation sample of same or similar composition, analyzed by two or more laboratories in accordance with predetermined conditions. Acceptance criteria are often based statistically on the analysis results.

Laboratory Intra-comparison Sample - A sample, of same or similar composition, analyzed within the same laboratory with predetermined conditions. Sample may be used for evaluation of new instruments or methodology.

Legal Chain of Custody Protocols - Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Limit of Detection (LOD) - For chemical analysis, the LOD is an estimate of the minimum amount of a substance that an analytical process can reliably detect with 99% confidence. At the LOD the false negative rate (type II error) is 1%. An LOD is analyte- and matrix-specific and

may be laboratory-dependent. Generally, the LOD is assigned as 1-3X of the MDL. See Limit of Detection (LOD) Verification.

Limit of Detection (LOD) Verification - This is an analysis of a sample spiked with a concentration near the calculated MDL. The spike concentration should be at a level of 1-4 times the calculated MDL for multiple analyte tests and 2-3 times the calculated MDL for single analyte tests. Lower spike concentration may be used if LOD verification criteria are met.

Limit of Quantitation (LOQ) – For chemical analysis, the LOQ is the smallest concentration that produces a quantitative result with known and recorded precision and bias. The LOQ must be equal to or greater than the LOD, and the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range. The LOQ is comparable to the PQL (Practical Quantitation Limit) or RL (Reporting Limit) as defined by the laboratory. The lowest LOQ available is the lowest limit of quantitation (LLOQ).

LIMS - Laboratory Information Management System.

Matrix – The substrate of a test sample.

Matrix Duplicate - A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision. (Also see MSD)

Matrix Spike (spiked sample or fortified sample) - A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. Generally, for valid recovery calculations the parameter spike level should be greater than 1-4X of the sample parameter level.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate) - A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Maximum Contaminant Level (MCL) – Regulatory action level for a contaminant of concern.

Measurement System - A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).

Method - A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.

Method Detection Limit (MDL) - A measure of the limit of detection for an analytical method determined according to the procedure given in 40 CFR Part 136 Appendix B. The MDL is the minimum concentration of a substance that can be reported with 99% confidence that the

measured concentration is distinguishable from a zero or blank concentration. At the MDL the false positive rate (Type I error) is 1%. This MDL is referred to as the DL (Detection Limit) by DoD.

Method Validation - The confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled (NELAC 2003) (MARLAP 2004 for radiochemical methods).

Metrological Traceability – Property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty.

NELAC - National Environmental Laboratory Accreditation Conference.

NELAP - National Environmental Laboratory Accreditation Program (Now TNI).

National Institute of Standards and Technology (NIST) - A federal agency of the US Department of Commerce's Technology Administration that is designated as the United States national metrology institute (NMI). SI is the international metrological traceability term which NIST includes.

NPDES - National Pollutant Discharge Elimination System- A discharge permit system authorized under the Clean Water Act.

Performance Evaluation (PE) Sample - A sample with a composition unknown to the analyst that is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance limits.

Physical Parameter - A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical or biological components.

Practical Quantitation Limit (PQL) – See LOQ definition.

Precision - The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation - Refrigeration and/or reagents added at the time of sample collection to maintain the chemical and/or biological integrity of the sample.

Preventative Action – A pro-active process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

Proficiency Testing - A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency Testing Program - The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.

Proficiency Testing (PT) Sample - A sample with a composition unknown to the analyst/laboratory which is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

Protocol - A detailed, written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

Quality Assurance (QA) - An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
.
Quality Assurance Project Plan (QAPP) - A formal document describing the detailed quality control procedures pertaining to a specific project. For environmental clean-up projects, this is typically produced by an engineering firm with references to include a laboratory's Quality Assurance Manual.

Quality Control (QC) - The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.

Quality Control Sample - A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.

Quality Manual - A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System - A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities.

Quality System Matrix - These matrix definitions are to be used for purposes of batch and QC requirements:

Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.
Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.
Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.
Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.
Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Raw Data - The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, tabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.

Reference Material - Material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Method - To be used to determine the extent of method validation in Modules 3-7. A reference method is a published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a “standard method”, that term is equivalent to “reference method”). When a laboratory is required to analyze an analyte by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is not a regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.

Reference Standard - Standard used for the calibration of working measurement standards in a given organization or at a given location.

Replicate - See Duplicate.

Reporting Limit (RL) – The lowest level of concentration reported for an analyte.

Resource Conservation and Recovery Act (RCRA) - The enabling legislation under 42 USC 321 et seq. (1976) that gives EPA the authority to control hazardous waste.

Safe Drinking Water Act (SDWA) - The enabling legislation, 42 USC 300f et seq. (1974), which requires the USEPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Sampling - Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Sample (SAMP) - A portion of material to be analyzed.

Selectivity - The ability to analyze, distinguish, and determine a specific analyte from another component that may be a potential interferent or that may behave similarly to the target analyte within the measurement system.

Sensitivity – The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g. concentrations) of a variable of interest.

Spiked Sample – See Matrix Spike.

Standardization - See Calibration.

Standard Operating Procedures (SOPs) - A written document that details the method for an operation, analysis, or action, with a thorough description of techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.

Technology - A specific arrangement of analytical instruments, detection systems, and/or preparation techniques

TNI – The NELAC Institute

Traceability - The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.

Trip Blank - One type of Field Blank. An aliquot of analyte-free water or solvent transported to the field in a sealed container and returned to the laboratory with the sample containers.

Validation – The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification - Confirmation by examination and objective evidence that specified requirements have been met. Regarding instrumentation and measuring equipment, verification is a confirmation the difference between measured values and known values are within maximum allowable error as defined by a method, regulation or specification for the instrument.

Acronyms and Abbreviations

AA	-	Accrediting Authority
AB	-	Accrediting Body
ANSI	-	American National Standards Institute
AOAC	-	The Scientific Association Dedicated to Analytical Excellence
APHA	-	American Public Health Association
ASQC	-	American Society for Quality Control
ASTM	-	American Society for Testing and Materials
Bq	-	Becquerel
BLK	-	Blank
Bg	-	Background
°C	-	Degrees Celsius
Cal	-	Calibration
CAS	-	Chemical Abstract Service
CCB	-	Continuing Calibration Blank
CCV	-	Continuing Calibration Verification
COC	-	Chain of Custody
DOC	-	Demonstration of Capability
DO	-	Dissolved Oxygen
DoD	-	Department of Defense
DQO	-	Data Quality Objectives
DMRQA	-	NPDES Discharge Monitoring Report Quality Assurance
DUP	-	Duplicate
ELI	-	Energy Laboratories, Inc.
EPA	-	Environmental Protection Agency
FDA	-	Food and Drug Administration
g/L	-	Grams per Liter
GC	-	Gas Chromatography
GC-MS	-	Gas Chromatography-Mass Spectrometry
ICP-AES	-	Inductively Coupled Plasma Atomic Emission Spectrophotometry/Spectroscopy
ICP-MS	-	Inductively Coupled Plasma-Mass Spectrometry
ICV	-	Initial Calibration Verification
ISO	-	International Organization for Standardization
LCS	-	Laboratory Control Sample
LFB	-	Laboratory Fortified Blank
LIMS	-	Laboratory Information Management System
LLD	-	Low Limit Detection
LOD	-	Limit of Detection
LOQ	-	Limit of Quantitation
MDC	-	Minimum Detection Concentration
MDL	-	Method Detection Limit
MBLK	-	Method Blank
MS/MSD	-	Matrix Spike/Matrix Spike Duplicate
NEHA	-	National Environmental Health Association
NELAC	-	National Environmental Laboratory Accreditation Conference
NELAP	-	National Environmental Laboratory Accreditation Program
NIOSH	-	National Institute for Occupational Safety and Health
NIST	-	National Institute of Standards and Technology
NPDES	-	National Pollutant Discharge Elimination System
OSHA	-	Occupational Safety and Health Administration

pCi/L	-	Picocuries per Liter
PT	-	Proficiency Testing
QA/QC	-	Quality Assurance / Quality Control
QS	-	Quality Systems
QAM	-	Quality Assurance Manual
QAPP	-	Quality Assurance Project Plan
RCRA	-	Resource Conservation and Recovery Act
RL	-	Reporting Limit
RPD	-	Relative Percent Difference
RSD	-	Relative Standard Deviation
SOP	-	Standard Operating Procedure
SPK	-	Spike
SI	-	International System of Units
SVOC	-	Semi-Volatile Organic Compound
TNI	-	The NELAC Institute
ug/L	-	Micrograms Per Liter
UV/VIS	-	Ultraviolet/Visible Spectroscopy
VOC	-	Volatile Organic Compound
WET	-	Whole Effluent Toxicity

APPENDIX A

Laboratory Certifications

Current certifications and performance evaluation studies are available at www.energylab.com website and include:

- Primary Montana DPHHS Certification
- Primary Florida DOH NELAP Certification
- Alaska State Certification
- ANSI-ASQ National Accreditation Board, ISO/IEC-17025 and Department of Defense Certification
- Colorado State Certification
- Idaho State Certification
- Louisiana State Certification
- Nebraska State Certification
- Nevada State Certification
- North Dakota State Certification
- South Dakota State Certification
- Texas Dakota State Certification
- Washington State Certification
- Wyoming State Certification (EPA Region VIII)
- Recent EPA WS and WP/DMRQA Study Results
- Recent NELAC Water/Soil Study Results

APPENDIX B

Quality Assurance / Quality Control Specifications

APPENDIX B
Quality Assurance / Quality Control Specifications
Example Methods: 245.1/7470A, 200.7/6010B, 200.8, VPH, EPH, 8260B/D, 8270C/D/E

MERCURY ANALYSIS FOR AQUEOUS ANALYSIS BY COLD VAPOR ATOMIC ABSORPTION (AA) EPA METHODS 245.1/7470A

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Sample Preparation	All samples digested	Meet method QC criteria for the matrix.	1) Re-analyze sample. 2) Re-prepare sample/batch.	
Instrument Calibration (IC)	Daily, after maintenance, or when needed. At least 5-point calibration including blank. Calibration Standards are not digested per 245.1 except at trace levels.	Correlation coefficient ≥0.995 also includes visual interpretation for quadratic or higher order calibration fit types.	1) Perform instrument maintenance. 2) Re-calibrate. 3) Prepare new standard.	Establishes calibration curve over a range of analyte concentrations to quantify analytes of interest. Calibration validity Tested by ICV and ICB.
Initial Calibration Verification (ICV) =QCS per 245.1	Immediately follows calibration or when new standards are prepared. Analyzed each analytical sequence.	%R= 90-110	1) Recalibrate and reanalyze. 2) Prepare fresh standards and/or ICV. 3) Instrument maintenance.	Evaluates calibration accuracy and method performance. Must be prepared from Second source standard.
Method Blank (MBLK) =LRB per 245.1	Minimum 1/20 samples or for each batch-whichever is more frequent.	Must be less than the larger of: 1) ± 1*lowest reporting limit or 2) 2.2 X MDL. (245.1) < Reporting limit (7470)	1) Re-analyze MBLK. 2) Re-digest samples from batch which fail acceptance criteria or flag and report data. 3) Test/re-prep all reagents for contamination.	Evaluates calibration accuracy, reagent/glassware contamination, and instrument carryover.
Laboratory Control Sample (LCS) = LFB per 245.1	Minimum 1/20 samples or for each batch-whichever is more frequent.	%R = 80-120 (7470) %R = 85-115 (245.1)	1) Repeat analyses 2) Prepare new standards 3) Re-calibrate 4) Re-extract and re-analyze samples associated with failed LCS.	Evaluates method accuracy. Must be Second Source Standard per NELAC. Also used to evaluate spiking technique for MS/MSD analysis.
Continuing Calibration Verification (CCV) = Instrument Performance Check (IPC) per 245.1	Analyzed at beginning of run, every 10 samples and at end of run. Same source standard.	%R = 95-105 Immediately after IC (245.1 only) %R = 90-110 as continuing calibration check.	1) Recalibrate and reanalyze all samples since last valid CCV. 2) Check for sample matrix problem.	Evaluates Instrument calibration drift.
Continuing Calibration Blank (CCB)	Analyzed after every CCV. Run every 10 samples and at end of run.	Must be less than the larger of: 1) ± 1*lowest reporting limit or 2) 2.2 X MDL.	1) Check for high concentration sample. 2) Re-analyze CCB. 3) Re-analyze all samples associated with failing CCB.	Evaluates baseline drift, contamination in the analytical system, and analyte carryover.
Reporting Limit Check Solution (CCV2)= RLCS for SM3112	Immediately follows calibration or when new standards are prepared. Analyzed each analytical sequence.	%R= 50-150 (3112)	1) Recalibrate and reanalyze. 2) Prepare fresh standards and/or CCV2. 3) Instrument maintenance.	Evaluates calibration accuracy at reporting limit. Must be made identically to lowest level standard used in calibration.

MERCURY ANALYSIS FOR AQUEOUS ANALYSIS BY COLD VAPOR ATOMIC ABSORPTION (AA) EPA METHODS 245.1/7470A

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Matrix Spike Sample and Matrix Spike Duplicate (MS/MSD) = LFM per 245.1	Minimum 1 set/10 samples for 245.1 Minimum 1 set/20 samples for 7470	%R = 70-130 for 245.1 %R = 75-125 for 7470 RPD < 30% for 245.1 RPD < 20% for 7470	1) If matrix interference suspected report as found, or 2) Re-analyze and re-spike if no matrix interference suspected, or 3) Use "A" qualifier for sample amount > 4X spike level.	Evaluates effect of matrix on method performance. Results not evaluated when sample analyte concentration > 3X spike level. Spike with same source as LCS. Control limits valid for spike level 1/3 of sample amount or higher.
Serial Dilution Sample (SD)	Minimum 1/20 samples for method 7470A	RPD 10%	1) Repeat dilution analysis. 2) Investigate cause. 3) Redigest batch or flag data results.	Measures method precision/sample homogeneity.
MDL Studies	Two MDL_{spike} solutions are prepared and analyzed quarterly. The MDL study is evaluated annually by calculating the MDL_{spike} and MDL_{blank}. A minimum of six months of method blank results or 50 data points (whichever is greater) analyzed from the previous year are used to calculate the MDL_{blank}.	< PQL	1) Repeat if obvious problem occurs or new analyst begins operation of the instrument. 2) Adjust reporting limit to > MDL.	Evaluates overall method detection limits in clean sample matrix. Actual samples may have higher MDL.
LOD verification	Quarterly	Positive result above signal to noise	1) Examine method or preparatory steps. 2) Verify MDL study 3) Repeat analysis	Spike at 2-4X the calculated MDL for multiple analyte tests.
LOQ Verification	Quarterly	%R= 70-130%	LOQ≤ reporting limit; if it is not then re-run at a higher concentration, within the calibration range, until acceptance criteria are met.	Generally 3-10X the MDL
Linear Dynamic Range (LDR)	Annually, or whenever method changes might affect sensitivity.	Calculated standard values within 10% of expected.	1) Repeat. 2) Correct problem. 3) Adjust upper calibration limit.	Used to determine upper linear range for instrument.
External PE Samples	Semi-annually, WS (245.1) and WP 7470) study samples.	PT sample defined acceptance limits (Must pass 2 out of last 3 PT studies).	1) Complete corrective action report. 2) Repeat with another make-up study (for failure of 2 out of 3).	External review of analytical method accuracy.
Control Charting	Annual statistical review of method performance.	Data statistically within control limits.	1) Trend Analysis/Method Review. 2) Correct method/instrument problem. 3) Replace Analyst.	For statistical process control.
Batch Definition	Each batch of 20 samples	Must pass all method QC criteria as specified above	Re-analyze batch or qualify results.	A group of samples and associated QC.

ELEMENTAL ANALYSIS OF WATER AND WASTES BY ICP-AES EPA METHODS 200.7/6010B

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Sample Preparation	Dissolved Waters: Analyze direct. Drinking Waters: Turbidity <1 Analyze direct. Turbidity >1 Digest using 200.2. CWA samples: Digest using 200.2 6010B Total Waters: 3010 Digestion. Soils: 3050 Digestion. Extracts: 3010 Digestion.	Meet method QC criteria for the matrix.	1) Reanalyze sample. 2) Re-prepare sample/batch.	
Instrument Calibration (IC)	Daily, or when needed. Minimum 1-point calibration and blank.	If used, multipoint calibration must have correlation coefficient ≥0.996	See QC Samples.	Calibration of Instrument. Calibration validity tested by ICV, ICB.
Quality Control Sample (QCS) /Initial Calibration Verification (ICV)	Immediately follows calibration. Second source standard used.	6010B %R =90-110 200.7 %R=95-105 Immediately after IC when new standards are prepared.	1) Recalibrate and reanalyze. 2) Prepare fresh standards and/or ICV.	Evaluates accuracy of calibration standards.
Initial Calibration Blank verification sample (ICB)	Analyzed at beginning of run.	Must be less than the larger of: 1) ± 1*lowest reporting limit or 2) 2.2 X MDL.	1) Re-pour blanks, recalibrate, and reanalyze. 2) Prepare fresh blank.	Evaluates instrument calibration, reagent contamination, and instrument carryover.
Low Level Calibration Verification (LLRV/CRI)	Analyzed at beginning of run. Count as sample for CCVs.	%R = 50-150, except for Be, Cd where %R = 70-130	None – Limits are advisory only.	Verifies Instrument ability to detect/quantitate analytes near the reporting limit.
Interference Check Sample "A" (ICSA)	Analyzed at beginning of run. Count as sample for CCVs.	%R = 80-120 for interferents. Advisory limit ± 2* reporting limit for other analytes	1) Evaluate sample data. Results near reporting limit suspect if failing. 2) Reanalyze samples as needed.	Evaluates spectral interference correction factors.
Interference Check Sample "AB" (ICSAB)	Analyzed at beginning of run. Count as sample for CCVs.	%R = 80-120 for interferents and analytes	1) Re-determine IECs if failures persist. 2) Reanalyze samples as needed.	Evaluates spectral interference correction factors.
Continuing Calibration Verification (CCV) /Instrument Performance Check (IPC)	Analyzed at beginning of run, every 10 samples and at end of run. Same source standard.	200.7: %R=95-105 Immediately after Initial Calibration. %R = 90-110 as continuing calibration check.	1) Remake and reanalyze 2) Correct problem and reanalyze all samples since last valid CCV	Evaluates instrument drift throughout analytical run. Typically uses midpoint calibration standard or ICV
Continuing Calibration Blank (CCB)	Analyzed after every CCV.	Must be less than the larger of: 1) ± 1*lowest reporting limit or 2) 2.2 X MDL.	1) Check for high concentration sample carryover. 2) Reanalyze CCB. 3) Reanalyze samples as needed.	Measures instrument drift and/or analyte carryover.

ELEMENTAL ANALYSIS OF WATER AND WASTES BY ICP-AES EPA METHODS 200.7/6010B

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Analytical Matrix Spike Sample (Direct analysis) (MS2)	200.7: Minimum 1/10 samples. 6010B: Minimum 1/20 samples.	6010B: %R = 75-125 200.7: %R = 70-130	1) Evaluate LCS/LFB performance. 2) Report spike as analyzed if LCS/LFB is acceptable.	Evaluates effect of matrix on analytical part of method performance. Results not evaluated when sample analyte concentration > 4X spike level.
Analytical Spike Duplicate (MSD2), or Analytical Duplicate Sample	200.7: Minimum 1/10 samples. 6010B: Minimum 1/20 samples.	Larger of 3 * PQL or 20% RPD %R see MS2	1) See LCS/LFB performance. 2) Report spike as analyzed if LCS/LFB is acceptable.	Measures method precision/sample homogeneity.
Serial Dilution Sample	When new matrix is encountered or 1 per batch or 1 per 20 samples	%R = 90-110 for analytes greater than 50 * PQL	1) Reanalyze samples. 2) Analyze samples on dilution.	Used for screening analyses evaluating new matrices.
Method Blank (MBLK) /Laboratory Reagent Blank (LRB)	1 per analytical run for direct samples, or 1 per digestion batch.	Must be less than the larger of: 1) ± 1*lowest reporting limit or 2) 2.2 X MDL.	1) Reanalyze LRB/MBLK. 2) Re-digest samples from batch which fail acceptance criteria or flag and report data.	Evaluates possible contamination in reagents and glassware.
Laboratory Fortified Blank (LFB) /Laboratory Control Sample (LCS)	1 per analytical run for direct samples, or 1 per digestion batch.	200.7: %R = 85-115 6010B: %R = 80-120	1) Reanalyze. 2) Re-digest sample batch or flag data.	Evaluates preparation method accuracy.
Soil/Solid Standard Reference Material (SRM)	Prepared and analyzed quarterly or as needed.	Within SRM-established acceptance ranges.	1) Reanalyze SRM. 2) Re-digest SRM. 3) Evaluate prep method.	Evaluates preparation method accuracy.
Pre-digestion Spike / Laboratory Fortified Sample Matrix (MS3)	200.7: Minimum 1/10 samples or 1/digestion batch. 6010B: Minimum 1/20 samples or 1/digestion batch.	200.7: %R =70-130 6010B: %R =75–125	1) See LCS performance. 2) Report spike as analyzed if LCS/LFB is acceptable. 6010B TCLP: When %R < 50% analyze PDS for MSA, adjust sample results for MSA recovery.	Evaluates effect of matrix on overall method performance. Results not evaluated when sample analyte concentration > 4X spike level.
Internal Standards (IS), when used.	All sample & QC in sequence.	50-150% Recovery Advisory Limits	1) Evaluate data for sample matrix affects	Quantitation using Internal Standards improves method accuracy. IS recoveries can be affected by sample matrix.
MDL Studies	A minimum of 2 MDLspike solutions are prepared and analyzed quarterly. The MDL study is evaluated annually by calculating the MDLspike and MDLblank. A minimum of six months of method blank results or 50 data points (whichever is greater) analyzed from the previous year are used to calculate the MDLblank	< PQL	1) Repeat if obvious problem occurs. 2) Adjust reporting limit to >MDL.	Evaluates overall method detection limits in clean sample matrix. Actual samples may have higher MDL.

ELEMENTAL ANALYSIS OF WATER AND WASTES BY ICP-AES EPA METHODS 200.7/6010B				
QA SAMPLE/ INDICATOR	**FREQUENCY**	**ACCEPTANCE CRITERIA**	**CORRECTIVE ACTION**	**COMMENTS**
LOD Verification Required for each analyte/method to verify calculated MDL.	Quarterly	Positive Result With signal to noise ratio of at least 3.	LOQ≤ reporting limit; if it is not then re-run at a higher concentration, within the calibration range, until acceptance criteria are met	Spike at 1-4X the calculated MDL for multiple analyte tests.
LOQ Verification	Quarterly	200.7: %R = 65-135 6010B: %R = 60-150	LOQ≤ reporting limit; if it is not then re-run at a higher concentration, within the calibration range, until acceptance criteria are met	Generally 3-10X the MDL
Inter-Element Correction Factor Studies	Annually, or whenever instrument changes might affect inter-element effects. Verified every 6 months.	Comparison to historical data.	1) Repeat. 2) Correct problem.	Correction factors to account for spectral overlap between differing elements.
Upper Linear Range Studies	Annually, or whenever method changes might affect sensitivity.	Comparison to historical data.	1) Repeat. 2) Correct problem. 3) Adjust upper calibration limit.	Used to determine upper linear range for instrument.
External PE Samples	WS and WP, LPTP (soil) and internal blind samples	EPA/PE Provider-defined control limits.	1) Repeat. 2) Correct problem.	External review of analytical method accuracy.
Batch Definition	Each daily analytical sequence. Prepped samples: Each batch of 20 samples/matrix or when there is a change of reagents, whichever is more frequent.	Must pass all method QC criteria.	Reanalyze batch, re-prepare samples, or qualify results.	A group of samples and associated QC.

ANALYSIS OF TRACE ELEMENTS IN AQUEOUS SAMPLES BY ICP/MS EPA METHOD 200.8

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Sample Preparation	Dissolved Waters: analyze direct Drinking Waters: Turbidity <1 analyze direct Turbidity >1 digest using 200.2 CWA samples: digest using 200.2	Meet method QC criteria for the matrix.	1) Reanalyze sample. 2) Re-prepare sample/batch.	
Instrument Calibration (IC)	Daily, after maintenance, or when needed. Multipoint calibration, usually 9 points and blank.	$R^2 \geq 0.995$ Highest 3 standards within ±10% Lowest standard (LOQ) ±30%	1) Perform instrument maintenance 2) Re-calibrate 3) Prepare new standard	Establishes calibration curve over a range of analyte concentrations to quantify analytes of interest. Calibration validity tested by ICV.
Initial Calibration Verification/ Quality Control Sample (ICV/QCS)	Immediately follows calibration. Must be prepared from second source standard.	%R = 90-110	1) Recalibrate and rerun. 2) Prepare fresh standards and/or ICV. 3) Instrument maintenance.	Evaluates calibration accuracy and method performance.
Initial Calibration Blank (ICB)	Analyzed at beginning of run.	Larger of ±1* lowest reporting limit, 10% sample concentration, or 2.2 X MDL	1) Prepare fresh blank. 2) Re-pour blanks, recalibrate, and rerun.	Evaluates instrument calibration, reagent contamination, and instrument carryover.
Interference Check Sample "A" (ICSA)	Analyzed at beginning of run. Count as sample for CCVs.	%R = 70-130 For interferents ± 2* reporting limit	1) Evaluate sample data. Results near reporting limit suspect if failing. 2) Rerun samples as needed.	Evaluates elemental equations and collision cell performance (when in use).
Interference Check Sample "AB" (ICSAB)	Analyzed at beginning of run. Count as sample for CCVs.	%R% = 70-130 For analytes present in the standard	1) Confirm elemental equations per method. 2) Recalibrate/rerun samples as needed.	Evaluates elemental equations and collision cell performance (when in use).
Laboratory Reagent Blank (LRB)/Method Blank (MBLK)	1 LRB per analytical run for direct samples 1 MBLK per digestion batch	≤ 2.2 * MDL < Reporting limit	1) Reanalyze LRB/MBLK. 2) Re-digest samples from batch which fail acceptance criteria or flag and report data.	Evaluates calibration accuracy, reagent/glassware contamination, and instrument carryover.
Laboratory Fortified Blank (LFB)/Laboratory Control Sample (LCS) Water Sample	1 LFB per analytical run for direct samples 1 LCS per digestion batch	%R = 85-115	1) Reanalyze LFB/LCS 2) Re-calibrate and reanalyze 3) Redigest samples associated with failed LCS.	Evaluates method accuracy. LCS must be second source standard. Also used to evaluate spiking technique for MS/MSD analysis.
Continuing Calibration Verification (CCV	Run every 10 samples and at end of analysis	R% = 90-110	1) Remake and reanalyze twice consecutively- both CCVs must pass in order for data sequence to be valid 2) Correct problem and reanalyze all samples since last valid CCV	Evaluates instrument drift throughout analytical run. Typically uses midpoint calibration standard or ICV
Continuing Calibration Blank (CCB)	Analyzed after every CCV	Larger of ±1* lowest reporting limit, 10% sample concentration, or 2.2 X MDL	1) Check for high concentration sample carryover. 2) Reanalyze CCB. 3) Reanalyze samples as needed.	Evaluates baseline drift, contamination in the analytical system, and analyte carryover
Matrix Spike (MS) Direct Analysis	Minimum 1/10 samples	%R = 70-130	1) Evaluate LFB performance (must be passing) 2) If matrix interference suspected report as found, 3) Re-spike and reanalyze if no matrix interference suspected 4) Use "A" qualifier for sample amount > 4X spike level.	Evaluates effect of matrix on method performance. Results not evaluated when sample analyte concentration > 4X spike level. Use the same solution and concentration as LFB.
Direct Analysis Matrix Spike Duplicate (MSD) Or Analytical Duplicate Sample	Minimum 1/10 samples	%R = 70-130 Larger of 3* PQL or 20% RPD	1) Evaluate LFB performance (must be passing) 2) If matrix interference suspected report as found, 3) Re-spike and reanalyze if no matrix interference suspected 4) Use "A" qualifier for sample amount > 4X spike level.	Duplicate analysis measures method precision/ sample homogeneity.

ANALYSIS OF TRACE ELEMENTS IN AQUEOUS SAMPLES BY ICP/MS EPA METHOD 200.8

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Digestion Matrix Spike (MS4)	Minimum 1/10 samples	%R = 70-130	1)Evaluate LCS performance (must be passing) 2) If matrix interference suspected report as found 3) Re-spike and reanalyze if no matrix interference suspected 4) Use "A" qualifier for sample amount > 4X spike level.	Evaluates effect of matrix on method performance. Results not evaluated when sample analyte concentration > 4X spike level. Use the same solution and concentration as LCS.
Matrix Spike Duplicate (MSD4) Or Digestion Duplicate Sample	Minimum 1/10 samples	%R = 70-130 Larger of 3* PQL or 20% RPD	1) Evaluate LCS performance (must be passing) 2) If matrix interference suspected report as found 3) Re-spike and reanalyze if no matrix interference suspected, or 4) Use "A" qualifier for sample amount > 4X spike level.	Duplicate analysis measures method precision/ sample homogeneity.
Internal Standards (IS)	All samples & QC in sequence	60-125% Recovery	Reanalyze samples on dilution, as needed.	Corrects data for sample matrix effects. Quantitation using Internal Standards is required for ICP-MS.
MDL Studies	A minimum of 2 MDLspike solutions are prepared and analyzed quarterly. The MDL study is evaluated annually by calculating the MDLspike and MDLblank. A minimum of six months of method blank results or 50 data points (whichever is greater) analyzed from the previous year are used to calculate the MDLblank.	< PQL	1) Repeat if obvious problem occurs or new analyst begins operation of the instrument. 2) Adjust reporting limit to > MDL.	Evaluates overall method detection limits in clean sample matrix. Actual samples may have higher MDL.
LOD Verification Required for each analyte/method to verify calculated MDL.	Quarterly	Positive Result With signal to noise ratio of at least 3	1) Examine method or preparatory steps, 2) Verify MDL study, 3) Repeat analysis.	Spike at 2-4X the calculated MDL for multiple analyte tests.
Linear Dynamic Range	Daily	±10%	1) Repeat. 2) Correct problem. 3) Adjust upper calibration limit.	Used to determine upper linear range for instrument.
External PE Samples	WS and WP and internal blind samples.	PT sample defined acceptance limits (Must pass 2 out of last 3 PT studies)	1) Complete corrective action report 2) Repeat with another make-up study (for failure of 2 out of 3)	External review of analytical method accuracy.
LOQ verification	Quarterly per DoD	%R=70-130	LOQ≤ reporting limit; if it is not then re-run at a higher concentration, within the calibration range, until acceptance criteria are met	Generally 3-10X the MDL
Control Charting	Quarterly	Data statistically within control limits.	1) Trend Analysis/Method Review 2) Correct method/instrument problem 3) Replace Analyst	For statistical process control
Batch Definition	Each daily analytical sequence. Prepped samples: Each batch of 20 samples/matrix or when there is a change of reagents, whichever is more frequent.	Must pass all method QC criteria as specified above	Reanalyze batch or qualify results	A group of samples and associated QC

ANALYSIS BY FLAME IONIZATION/PHOTOIONIZATION DETECTOR (FID/PID) VOLATILE PETROLEUM HYDROCARBONS (VPH) PER MASSACHUSETTS METHOD				
QA SAMPLE/ INDICATOR	**FREQUENCY**	**ACCEPTANCE CRITERIA**	**CORRECTIVE ACTION**	**COMMENTS**
Sample Preparation	Soils: Extracted by 5035, then analyzed by Purge & Trap. 10 grams Soil/10mL of methanol VPH Surrogates added to all samples before extraction. Waters: VOA Vials, preserve to a PH<2.	Meet all method QC criteria for the matrix.	1) Re-analyze sample	VPH surrogates added to all sample before extraction. Waters are introduced into the GC using Purge & Trap. Soils are extracted into methanol and the methanol extract is added to water and analyzed by Purge and Trap/GC.
Instrument Calibration (IC)	5 Point calibration to precede analyses. Use average response factors. Certain compounds are selected for FID calibration and other compounds are used for PID calibration.	25% RSD of Mean Response Factors. Includes individual compound response factors and range response factors. Relative error (RE) when calculated as a percent recovery of the standard against the curve is recommended to be evaluated against statistically set criteria with default limits being the CCV criteria excepting the lowest point (s) which should have a 50% - 150% recovery.	1. Correct problem. 2. Prepare new standards. 3. Recalibrate.	Establishes calibration curve over a range of analyte concentrations to quantify analytes of interest. Calibration of instrument and check of response linearity. Consists of a 13 component standard containing both aliphatic and aromatic hydrocarbons
Initial Calibration Verification (ICV)	Follows valid initial calibration (See Blank Spike)	75-125%	1. Correct problem. 2. Re-calibrate and rerun ICV.	Evaluates accuracy/bias in calibration standards.
Continuing Calibration Verification (CCV)	Every 24 Hours and at the end of every analytical sequence	75-125% of Initial Calibration for the CCV preceding sample analyses.	1. Correct problem. 2. Re-analyze CCV. 3. Recalibrate and re-analyze all samples since last valid calibration check.	Evaluates instrument drift throughout analytical sequence. Typically uses midpoint calibration standard or ICV.
Method Blank	Before samples, and at least one MB every 24 hours.	½ of PQL for target analytes	1. Repeat analyses once. 2. Correct problem. 3. Re-extract and re-analyze all samples associated with failing method blank.	Evaluates overall method including possible contamination in reagents and glassware utilized in preparatory batch. Soil method blanks use clean sand.
Matrix Spike and Matrix Spike duplicate (MS/MSD)	Each batch of 20 samples/matrix or when there is a change of reagents, whichever is more frequent.	%R = 70-130 %RPD < 20	1. Repeat analyses. 2. Re-extract and re-analyze MS, (if sufficient sample).	Evaluates effect of matrix on method performance.
Lab Control Sample (LCS) (Blank Spike)	Minimum 1/20 samples Soils are prepared using a blank sand matrix.	%R = 70 - 130	1. Repeat analyses. 2. Prepare new standards. 3. Recalibrate. 4. Re-extract and re-analyze all samples associated with failing LCS (laboratory fortified blank).	Evaluates overall method precision and accuracy. Method specifies 70-130.

ANALYSIS BY FLAME IONIZATION/PHOTOIONIZATION DETECTOR (FID/PID) VOLATILE PETROLEUM HYDROCARBONS (VPH) PER MASSACHUSETTS METHOD				
QA SAMPLE/ INDICATOR	**FREQUENCY**	**ACCEPTANCE CRITERIA**	**CORRECTIVE ACTION**	**COMMENTS**
Surrogates	Present in all extracted samples (including QC)	Trifluorotoluene %R = 70-130	1. Repeat analyses. 2. Recalibrate with fresh fortification standard. 3. Re-extract samples.	Evaluates method performance on each individual sample analyzed.
Analyte Confirmation in Samples	Confirm target VPH analytes by GC/MS analyses.	Upon client request.	None	Analyte identifications in samples are not routinely confirmed. GC/MS confirmation done only per client request.
MDL Studies Per CFR Part 136	MDL - Quarterly for water and soils and initially for each new instrument setup or whenever method changes might affect sensitivity.	MDL< ½ PQL	1) If the result for any individual analyte from the MDL spiked samples does not meet the method qualitative criteria or does not provide a numerical result greater than zero, repeat the spiked samples at a higher concentration. 2) Repeat initial MDL spike or adjust reporting limit to > 2X of calculated MDL.	The minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results.
LOD Verification Required for each analyte/method to verify calculated MDL.	Quarterly based on MDL Study frequency.	Positive Result, (Above background)	1) Examine method or preparatory steps. 2) Verify MDL study. 3) Repeat analysis. 4) Consult QA.	Spike at 1 - 4X calculated MDL.
External PE Samples	Semi-annually, WP study samples.	PT sample defined acceptance limits (Must pass 2 out of last 3 PT studies)	1. Complete corrective action report. 2. Repeat with another make-up study (for failure of 2 out of 3).	External review of analytical method accuracy.
Control Charting and Proof of Competency	Quarterly, statistical review of method.	Data statistically within control limits.	1. Trend Analysis/ Method Review. 2. Correct method/instrument problem. 3. Replace analyst.	For statistical process control.
Batch	Each batch consists of a maximum of 20 samples	Must pass all method QC criteria	Re-analyze batch or qualify results	
LLOQ Study	Performed initially to verify LLOQ for each instrument and preparation method. Prepare and analyze 7 replicate samples. MDL study may be used if criteria met.	Within established in-house limits or advisory limits of +/- 20% of the LCS limits (i.e. low limit -20% upper limit +20%).	1) Repeat if obvious problem occurs. 2) LLOQ Recovery should be reasonable relative to default advisory limits. Results should be within statistically based limits when available.	Evaluates overall method precision and accuracy at the lowest reporting limit. Actual samples may have higher RL.
LLOQ Verification	Quarterly, after initial study.	Within established in-house limits or advisory limits of +/- 20% of the LCS limits (i.e. low limit -20% upper limit +20%).	1) Repeat if obvious problem occurs. 2) LLOQ Recovery should be reasonable relative to default advisory limits. Results should be within statistically based limits when available.	Used to verify ongoing instrument quantitative accuracy at the LLOQ. Can be control charted to verify and determine statistical LOQ limits.

ANALYSIS BY FLAME IONIZATION DETECTOR (FID) EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) PER MASSACHUSETTS METHOD

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Sample Preparation	Methods: Soils: 3550 (30 grams to 2mL) Waters: 3510 or 3520 (1 Liter to 2 mL) EPH extraction surrogates added to all samples prior to extraction. EPH fractionation surrogates added to extract just prior to fractionation.	Meet all method QC criteria for the matrix.	1) Re-analyze sample	Samples are extracted using Methylene chloride solvent and then the extract is concentrated. Following separation of extract into an aliphatic and aromatic fraction each fraction is independently analyzed by GC/FID. Sample amount and final extract volume may be adjusted based on analyte levels and/or sample matrix.
Fractionation Check	Per each Lot # of Separation Cartridges Used	Effective separation of target analytes into appropriate fraction. R%=40-140 except the more volatile target analytes with R%=40-140	1. Repeat once 2. Correct problem (adjust elution volumes) 3. Prepare new standards 4. Recalibrate	Uses aliphatic and aromatic hydrocarbon standards in hexane. The more volatile aromatic and aliphatic compounds may have lower recoveries than method specified limits.
Initial Calibration (IC)	5 point initial calibration each for aliphatics and aromatics, external standardization option of method chosen. Aliphatic Standard Solutions Aromatic Standard Solutions 1, 20, 50, 200, and 500 ug/mL in each component. (EPH Screen: aliphatic standard solutions 1, 20, 200, 500, and 1000 ug/mL). To precede sample analyses.	25% RSD MnRF 25%RSD each component. Relative error (RE) percent recovery for calibration level 1-5 should be 75% - 125% .	1. Repeat once 2. Correct problem 3. Prepare new standards 4. Recalibrate	Used to Calibrate instrument, evaluates chromatographic separation effectiveness, and instrument response linearity. .
Chromatography	1) Each IC or CCV- Resolution is verified 2) Retention Time Windows –Use RRT and analyst discretion for instrument stability.	Chromatographic resolution: Monitored against historical performance levels. 50% separation of phenanthrene and anthracene.	1. Repeat once 2. Adjust column conditions 3. Perform instrument maintenance 4. Replace GC column	Verifies that gas chromatographic system is operating properly. Resolution criteria for two selected PAH pairs are not met as per method specifications.
Initial Calibration Verification (ICV)	Follows the IC, using second source calibration standards. DRO standard used to verify aliphatic IC standard and a separate PAH standard is used for aromatics.	+/- 25% of MnRF +/- 25% RF each component	1. Repeat once 2. Prepare fresh standards and reanalyze. 3. Recalibrate and re-analyze all affected samples.	Evaluates accuracy of calibration standards.
Continuing Calibration Verification (CCV)	Mid-level standard analyzed every 12 hours and at the end of every analytical sequence	+/- 25% of MnRF +/- 25% RF each component	1. Repeat once 2. Correct problem 3. Re-calibrate and re-analyze all samples since last valid calibration check.	Verifies instrument calibration and stability throughout analyses. No QC criteria for the CC following sample analyses.

ANALYSIS BY FLAME IONIZATION DETECTOR (FID) EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) PER MASSACHUSETTS METHOD

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Method Blank	Each batch of 20 samples/matrix or when there is a change of reagents, whichever is more frequent.	<½ PQL.	1. Repeat analyses once 2. Correct problem 3. Re-extract and re-analyze all samples associated with method blank.	Measures and evaluates possible contamination in reagents and glassware used in method.
Instrument Blank	Each 12 hour sequence or as indicated, such as after a heavily contaminated extract. A method blank analysis can be substituted for an instrument blank.	<½ PQL	1. Repeat analyses once 2. Perform Instrument maintenance 3. Re-analyze all associated samples in sequence where contamination level may affect result.	Measures and evaluates possible contamination in gas chromatographic analysis system.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Each batch of 20 samples/matrix or when there is a change of reagents, whichever is more frequent. Fortified with all aliphatic and aromatic compounds present in ICAL standards. Uses a second source standard.	%R = 40-140 except for the more volatile aromatic and aliphatic compounds which may have lower recovery. %RPD = 50% (advisory)	1. Repeat GC analyses 2. Re-extract and reanalyze MS/MSD, (if sufficient sample) or select another sample to MS. 3. Evaluate LCS performance.	Evaluates effect of individual matrix on method performance and method precision. Poor MS/MSD QC performance does not necessarily reject extraction batch group. Control limits are advisory due to sample matrix effects.
Laboratory Control Sample (LCS)	Minimum 1/20 samples/matrix and each batch of samples, whichever is more frequent. Same spiking solution as for MS/MSD	%R = 40-140 Except for nonane, %R = 30-140. Concentration of naphthalene or 2-methylnaphthalene in the aliphatic fraction must not exceed 5% of the total concentration of naphthalene or 2-methylnaphthalene.	1. Repeat analyses 2. Prepare new standards 3. Recalibrate 4. Re-extract and re-analyze all samples associated with LCS.	Evaluates method accuracy. Used for ongoing proof of competency.
Extraction Surrogate	Added to all samples prior to extraction (including QC). Ortho-Terphenyl (Aromatic f and 1-Chloro-octadecane (Aliphatic fraction).	%R = 40-140 Control limits are advisory due to possible sample matrix effects.	1. Repeat analyses 2. Evaluate for matrix effects 3. Re-extract samples if method batch performance is suspected.	Evaluates extraction and separation method performance on each individual sample analyzed. Water samples containing sediment may have reduced analyte and surrogate extraction efficiency. Extraction performance alone can be evaluated from an EPH screening result.
Fractionation Surrogates	2-Bromonapthalene and 2-Fluorobiphenyl surrogates are added to sample extract prior to fractionation, These and OTP from extractions are Aromatic Surrogates. 1-Chloro-octadecane (from extractions) is Aliphatic Surrogate.	%R = 40-140 in Aromatic fraction. Control limits are advisory due to possible sample matrix effects.	1. Repeat analyses 2. Evaluate for matrix effects 3. Re-extract samples if method batch performance is suspected.	Evaluates the effectiveness of the aliphatic/aromatic separation step. Proportional Level of presence of either surrogate in the aliphatic fraction suggests incomplete separation of the more volatile PAHs from the aliphatic fraction.
EPH Screening	Analyses of extract prior to the separation step of the EPH method.	%R = 40-140 for OTP extraction surrogate. Full EPH recommended if TEH result >0.1 mg/L for waters or 200 mg/kg for soils.	1. Repeat analyses 2. Evaluate for matrix effects 3. Re-extract samples if method batch performance is suspected.	Evaluates method extraction performance on each individual sample analyzed. Target analyte levels in result are used to determine if full EPH analyses is necessary.

ANALYSIS BY FLAME IONIZATION DETECTOR (FID) EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) PER MASSACHUSETTS METHOD

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
PAH Target Analyte Confirmations	Analyses performed by 8270 on Aromatic fraction if PAH target analytes are present above MTDEQ limits.	Meets 8270 analyses criteria	1. Repeat analyses to meet all 8270 method QC criteria	Confirms and accurately quantitates PAH levels in aromatic extract. 8270 method is considered less sensitive to false positives than the EPH method.
MDL Studies	Annually using quarterly MDL data or whenever method changes might affect sensitivity.	½ PQL	1) If the result for any individual analyte from the MDL spiked samples does not meet the method qualitative criteria or does no provide a numerical result greater than zero, repeat the spiked samples at a higher concentration. 2) Repeat initial MDL spike or adjust reporting limit to > 2X of calculated MDL.	The minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results
LOD Verification	Following MDL study to confirm calculated MDL value and then quarterly.	Positive Result	1) Examine method or preparatory steps, 2) Verify MDL study, 3) Repeat analysis.	Spike at 1-4X MDL for multiple analyte tests.
External PE Samples	Twice a year.	PT sample defined acceptance limits (Must pass 2 out of last 3 PT studies).	1) Complete corrective action report 2) Repeat with another make-up study (for failure of 2 out of 3).	External review of analytical method accuracy.
Control Charting and Proof of Competency	Quarterly, statistical review of method QC data. The control charts are a year's worth of data or more if needed.	Data statistically within control limits.	1. Correct method problem 2. Adjust control limits 3. Replace analyst	For statistical process control and demonstration of capability for analysts.
Batch Definition	Prepped Samples = Each batch of 20 samples/matrix or when there is a change of reagents, whichever is more frequent.	Must pass all method QC criteria.	Re-analyze batch or qualify results	A group of samples and associated QC
LLOQ Study	Performed initially to verify LLOQ for each instrument and preparation method. Prepare and analyze 7 replicate samples. MDL study may be used if criteria met. Annually.	Within established in-house limits or advisory limits of +/- 20% of the LCS limits (i.e. low limit -20% upper limit +20%).	1) Repeat if obvious problem occurs. 2) LLOQ recovery should be reasonable relative to default advisory limits. Results should be within statistically based limits when available.	Evaluates method precision and accuracy at or below the lowest reporting limit. Actual samples may have higher RL.
LLOQ/LOQ Verification	Quarterly, after initial study.	Within established in-house limits or advisory limits of +/- 20% of the LCS limits (i.e. low limit -20% upper limit +20%).	1) Repeat if obvious problem occurs. 2) LLOQ/LOQ recovery should be reasonable relative to default advisory limits. Results should be within statistically based limits when available.	Used to verify ongoing instrument quantitative accuracy at the LLOQ/LOQ. Can be control charted to verify and determine statistical LLOQ/LOQ limits.

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS (VOCS) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) EPA METHODS 8260B, 8260D, AND 624.1

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Initial Calibration	7-point initial calibration range: 12.5, 25, 50, 125, 250, 375, 500 ng to the GC. 8th point at 2.5 ng to the GC for low level. For analytes with a normal purging efficiency. Analyte concentrations vary based on purging efficiency; please see attachment 17.3 Spike and Calibration Protocols.	If %RSD < 15 may use average RF, if %RSD > 15 use 1st or 2nd order calibration curve with R2 > 0.99 weighted 1/C. Relative error (RE) for the lowest calibration point is set to have a 50% - 150% recovery and recovery for calibration points above the lowest point is 70% - 130%. 8260B: CCC = Continuing Calibration Check Compounds %RSD must be < 30. Average RF for SPCCs must be > 0.3000 for Chlorobenzene and 1,1,2,2-Tetrachloroethane; and must be > 0.1000 for Chloromethane, 1,1-dichloroethane, and Bromoform.	1. Perform instrument maintenance. 2. Recalibrate. 3. Prepare new Standards.	Establishes calibration curve over a range of analyte concentrations to quantify analytes of interest.
Tuning	BFB Initially and every 12 hours thereafter. Ongoing tuning is optional for 8260D unless changes to the instrument conditions have been made.	Meet criteria in Table 3 of Method 8260D.	1. Re-analyze BFB 2. Perform instrument maintenance. 3. Run software tuning programs.	Evaluate mass sensitivity, mass resolution, isotope ratio, and baseline threshold.
Continuing Calibration Verification (CCV)	Mid-level standard analyzed every 12 hours	RF Drift ± 20% of Initial Calibration for CCCs, RF Drift ± 30% for all other compounds. RF for SPCCs must be > 0.3000 for Chlorobenzene and 1,1,2,2-Tetrachloroethane; and must be > 0.1000 for Chloromethane, 1,1-dichloroethane, and Bromoform. EICP Area of the Internal Standards must be 50-200% of the Initial Calibration and the retention time must not shift more than 30 seconds.	1. Remake and rerun CCV. 2. Perform instrument maintenance 3. Recalibrate or demonstrate 2 consecutive passing CCV's.	Evaluates instrument drift throughout analytical sequence. Typically uses midpoint calibration standard.
Method Blank (MBLK)	Each batch of 20 samples or when there is a change of reagents, whichever is more frequent.	<½ PQL	1. Repeat analyses. 2. Correct problem. 3. Re-extract and re-analyze all samples associated with failing method blank.	Evaluates overall method including possible contamination in reagents and glassware utilized in preparatory batch.
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Each batch of 20 samples or when there is a change of reagents, whichever is more frequent.	Statistical Control Limits	1. Repeat analyses. 2. Re-extract and re-analyze MS (if sufficient sample). 3. Evaluate LCS performance.	Evaluates effect of matrix on method performance.
Lab Control Sample (LCS)	Minimum 1/20 samples/matrix and each batch of samples, whichever is more frequent. Use second source standards to check calibration.	Statistical Control Limits	1. Repeat analyses. 2. Prepare new standards. 3. Recalibrate. 4. Re-extract and re-analyze all samples associated with failing LCS.	Evaluates overall method precision and accuracy.

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS (VOCS) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) EPA METHODS 8260B, 8260D, AND 624.1

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Internal Standards (All Samples & QC Standards)	Monitor total areas in each analyses: Fluorobenzene Chlorobenzene-d5 1,2-Dichlorobenzene-d5	CCV area 50-200% of Initial Calibration and Sample / QC area 50-200% of preceding CCV. RT = ± 30 seconds of Initial Calibration / CCV.	1. Repeat analyses. 2. Re-extract samples. 3. Re-analyze at higher dilution.	Measures instrument stability and sensitivity.
Surrogates	Present in all samples (including QC): Dibromofluoromethane (8260B and 8260D only) 1,2-Dichloroethane-d4 Toluene-d8 p-Bromofluorobenzene	Statistical Control Limits	1. Repeat analyses. 2. Re-extract samples. 3. Re-analyze at higher dilution. 4. Re-calibrate.	Evaluates method performance on each individual sample analyzed.
MDL Studies Per CFR Part 136	Quarterly or per SOP Determination of Method Detection Limits (MDL) and Quantitation Limits, and Initial Method and New Instrument/Equipment Validation requirements or whenever method changes might affect sensitivity.	MDL< PQL	1) If the result for any individual analyte from the MDL spiked samples does not meet the method qualitative criteria or does not provide a numerical result greater than zero, repeat the spiked samples at a higher concentration. 2) Repeat initial MDL spike or adjust reporting limit to > 2X of calculated MDL.	The minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results.
LLOQ Study	Performed initially to verify LLOQ for each instrument and preparation method. Prepare and analyze 7 replicate samples. MDL study may be used if criteria met.	Within established in-house limits or advisory limits of +/-20% of the LCS limits (i.e. low limit -20% upper limit +20%).	1) Repeat if obvious problem occurs. 2) LLOQ Recovery should be reasonable relative to default advisory limits. Results should be within statistically based limits when available.	Evaluates overall method precision and accuracy at the lowest reporting limit. Actual samples may have higher RL
LLOQ/LOQ Verification	Annually, after initial study and quarterly LOQ verification for DoD.	Within established in-house limits or advisory limits of +/-20% of the LCS limits (i.e. low limit -20% upper limit +20%).	1) Repeat if obvious problem occurs. 2) LLOQ/LOQ recovery should be reasonable relative to default advisory limits. Results should be within statistically based limits when available.	Used to verify ongoing instrument quantitative accuracy at the LLOQ/LOQ. Can be control charted to verify and determine statistical LOQ limits
LOD Verification	Required for each analyte/method to verify calculated MDL. Quarterly for DoD. Annually based on MDL Study frequency.	Positive Result, (Above background)	1) Examine method or preparatory steps. 2) Verify MDL study. 3) Repeat analysis. 4) Consult QA.	Spike at 2-4 times the calculated MDL.
External PT Samples	Performed semi-annually.	PT sample defined acceptance limits (Must pass 2 out of last 3 PT studies)	1. Complete corrective action report. 2. Repeat with another make-up study (for failure of 2 out of 3).	External review of analytical method accuracy.
Control Charting and Demonstration of Capability	Quarterly control charting annual demonstration of capability, or as needed.	Data statistically within control limits.	1. Trend Analysis/ Method Review. 2. Correct method/instrument problem.	For statistical process control.

ANALYSIS OF VOLATILE ORGANIC COMPOUNDS (VOCS) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) EPA METHODS 8260B, 8260D, AND 624.1				
QA SAMPLE/ INDICATOR	**FREQUENCY**	**ACCEPTANCE CRITERIA**	**CORRECTIVE ACTION**	**COMMENTS**
Individual Analyte QC Failures	When re-analysis and corrective action does not solve the issue; or when re-analysis is not possible or deemed necessary to meet quality objectives.	QC failures must be reported in the case narrative and/or flagged on QC Reports	Perform instrument maintenance and re-calibrate if QC failures continue.	

ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS (SVOCS) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) EPA METHODS 8270C, 8270D, 8270E AND EPA 625.1

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Sample Preparation Extraction	SW-846 Methods: Soils: 3550B or 3545 Waters: 3510C or3520C Wastes: 3550B, 3545, 3580 Surrogates added to all samples.	Meet Method QC criteria for the matrix	1) Re-analyze sample or re-extract sample. If re-extraction outside of holding time, report both sets of data.	Minimum sample volume required per sample. Soils: 30 grams Water: 1 Liter
Instrument Calibration (IC)	7-point calibration Range: 10, 20,50,75,100,120, 150ug/mL Bottom point or two may be dropped for reactive compounds as long as five consecutive points are used at a minimum	See Note #1 at bottom Relative error (RE) when calculated as a percent recovery of the standard against the curve is recommended to be evaluated against statistically set criteria with default limits being the CCV criteria excepting the lowest point (s) which should have a 50% - 150% recovery.	1) Perform instrument maintenance. 2) Recalibrate. 3) Prepare new Standards.	Establishes calibration curve over a range of analyte concentrations to quantify analytes of interest.
Instrument Blank	Following instrument calibration or beginning of each analytical sequence. May be substituted with batch method blank.	Clean baseline. No target analytes.	1) Rerun. 2) Perform instrument maintenance.	Evaluates instrument performance chromatographic baseline.
Tuning	DFTPP Initially and every 12 hours thereafter	Meet method-tuning criteria (Attachment 17.4)	1) Adjust instrument. 2) Recheck tune. 3) Until successful.	Evaluates mass sensitivity, mass resolution, isotope ration, and baseline threshold.
Initial Calibration Verification (ICV)	Immediately following calibration.	±30% difference from IC for 8270C, suggested for 8270E. RF for SPCC>0.050 %R of CCCs must be ±20% difference from IC. 625.1 and 8270D Method: %R for all compounds is ±20%.	1) Repour and rerun. 2) Prepare fresh calibration standards and/or ICV. 3) Recalibrate and rerun.	Evaluates calibration accuracy and method performance. Must be prepared from second source standard.
Method Blank (MBLK)	Immediately follows ICV. Each batch of 20 samples/matrix or when there is a change of reagents, whichever is more frequent.	< ½ PQL excepting phthalates <PQL for SIM	1) Prepare fresh blank 2) Re-extract and re-analyze all samples associated with failing method blank.	Evaluates calibration accuracy, reagent/ glassware contamination, and instrument carryover.
Continuing Calibration Verification (CCV)	Mid-level standard analyzed every 12 hours to update internal standard response factors (RF). Closing CCV required for DoD work.	±30% difference from IC for 8270C. RF for SPCC>0.050%R of CCCs must be ±50% difference from IC for closing CCV for DoD. 625.1 and 8270E: %R for all compounds is ±20%.	1) Remake and rerun. 2) Rerun instrument tune. 3) Recalibrate and rerun samples since last valid CCV	Evaluates instrument drift throughout analytical sequence. Typically uses midpoint calibration standard or ICV.
GC Performance Analyte Degradation	Each tuning; Evaluate TIC areas of DDT breakdown products and chromatographic profile.	< 20% breakdown	1) Instrument maintenance. 2) Re-check tune.	Evaluates chromatographic system for reactivity.
Minimum Response Factor	Check bottom ICAL point RF against values in Attachment 17.9	See Attachment 17.9	No action necessary. This is considered advisory criteria only.	The RFs are provided as guidance only and are not intended to be a requirement per 8270E.

ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS (SVOCS) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) EPA METHODS 8270C, 8270D, 8270E AND EPA 625.1

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
Matrix Spike (MS/MSD)	Each batch of 20 samples/matrix or when there is a change of reagents, whichever is more frequent. For 8270C-a representative list. For 625.1, 8270D/E- all target analytes	See LCS limits. Statistical control limits. RPD: 40%	LCS must be passing 1) If matrix interference suspected report as found, or 2) Re-extract and re-analyze MS if no matrix interference suspected (if sufficient sample) 3) Evaluate LCS performance (See Note #3 at bottom)	Evaluates effect of matrix on method performance. MSD also evaluates method precision.
Duplicate Sample (DUP)	If used in place of a MSD, 1/20 samples	5, 10, 20% RPD or 2X PQL depending on method	1) Rerun sample pair, evaluate for sample homogeneity or 2) Report with qualifiers	Evaluates method precision. MSD duplicate analyses preferred on some methods.
Laboratory Control Sample (LCS)	Minimum 1/20 samples/matrix and each batch of samples, whichever is more frequent.	Reference Material specified limits or laboratory statistical limits. 625.1 method: Limits don't exceed method criteria. DoD samples have LCS limits in Attachment 17.14	1) Prepare new Standards. 2) Re-calibrate. 3) Re-extract and re-analyze all samples associated with failing LCS.	Evaluates spiking technique and when prepared from a source independent of the calibration standards can also measure method performance.
Internal Standards	Monitor total areas in each analyses Acenapthene-d10 Phenanthrene-d10 Chrysene-d12 1,4-Dichlorobenzene-d4 Napthalene-d8 And Perylene-d12	Samples: Area %50-150% of IC. RT = ±30 sec of IC.	1) Repeat analyses 2) Re-prepare samples. 3) Analyze different sample. 4) Re-extract and re-analyze set of samples.	Measures instrument stability and sensitivity.
Mass Spectra	Review all target analytes in standards and reported analytes in samples.	Spectra must be consistent with library database.	1) Verify calibration spectra and retention times. 2). Repeat analyses.	Used to qualitatively identify target compound hits in samples.
Surrogates	Present in all extracted samples (Including QC).	Reference Material specified limits or laboratory statistical limits. 625.1 Method: Limits don't exceed method criteria.	1) Repeat analyses. 2) Recalibrate with fresh fortification standard. 3) Re-extract samples.	Evaluates method performance on each individual sample analyzed.
MDL Studies Per CFR Part 136	Bi-annually or annually per method requirement or whenever method changes might affect sensitivity	Spike at ~PQL, PQL = 10 ug/L or 0.33 ug/g with exceptions (See Note #4 at bottom).	1) If the result for any individual analyte from the MDL spiked samples does not meet the method qualitative criteria or does not provide a numerical result greater than zero, repeat the spiked samples at a higher concentration. 2) Repeat initial MDL spike or adjust reporting limit to > 2X of calculated MDL.	Evaluates overall method detection limits in clean sample matrix. The minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results Actual samples may have higher MDL.
LLOQ Study	Performed initially to verify LLOQ for each instrument and preparation method. Prepare and analyze 7 replicate samples. MDL study may be used if criteria met.	Within established in-house limits or advisory limits of +/-20% of the LCS limits (i.e. low limit -20% upper limit +20%).	1) Repeat if obvious problem occurs. 2) LLOQ Recovery should be reasonable relative to default advisory limits. Results should be within statistically based limits when available.	Evaluates overall method precision and accuracy at the lowest reporting limit. Actual samples may have higher RL.

ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS (SVOCS) BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) EPA METHODS 8270C, 8270D, 8270E AND EPA 625.1

QA SAMPLE/ INDICATOR	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	COMMENTS
LLOQ Verification	Annually, after initial study.	Within established in-house limits or advisory limits of +/-20% of the LCS limits (i.e. low limit -20% upper limit +20%).	1) Repeat if obvious problem occurs. 2) LLOQ Recovery should be reasonable relative to default advisory limits. Results should be within statistically based limits when available.	Used to verify ongoing instrument quantitative accuracy at the LLOQ. Can be control charted to verify and determine statistical LOQ limits.
LOD Verification	Bi-annually or annually per method MDL requirement following each MDL Study	Positive Result, S/N greater than 3 (above typical Method Blank performance)	1) Examine method or preparatory steps, 2) Verify MDL study, 3) Repeat analysis. 4) Consult QA	Spike at 1-4X MDL for multiple analyte tests.
External PE Samples	WP and LPTP PT studies. Biannual WS and/or WP and internal blind and double blind samples.	PT sample defined acceptance limits (Must pass 2 out of last 3 PT studies).	1) Complete corrective action report 2) Repeat with another make-up study (for failure of 2 out of 3).	External review of analytical method accuracy.
Control Charting and Proof of Competency	Annual statistical review of method.	Data statistically within control limits. Evaluate statistical limits reasonableness.	1) Trend Analysis/ Method Review. 2) Correct method/instrument problem. 3) Replace analyst.	For statistical process control.
Batch Definition	Prepped Samples = Each batch of 20 samples/matrix or when there is a change of reagents, whichever is more frequent.. 24 Hours	Must pass all method QC criteria.	Re-analyze batch or qualify results	A group of samples and associated QC

Note #1 %RSD for CCC (Table 4 SOP ELI 50-009) <30. RF for SPCC's (N-nitroso-di-n-propyl amine, hexachlorocyclopentadiene, 2,4 Dinitrophenol, and 4-Nitrophenol) > 0.050. If % RSD for a compound is < 15, linearity is assumed and average RF is used (<20% for 8270D). If % RSD > 15 (and less than 30 for CCC), use a calibration curve with correlation coefficient >= 0.990. Lower calibration levels are not used for certain compounds. PQLs are adjusted as appropriate.
Note #2 RF for SPCC>0.050, RF of CCC's must be <20% difference from IC. RF of all other compounds must be <30% difference from IC.
Note #3 If any analyte in the MS/MSD fails, QC limits for failed compounds must be within acceptable recovery limits for the blank spike laboratory control sample.
Note #4 PQL for Benzidine, 3,3' Dichlorobenzidine, and pyridine = 20ug/L. 4-Nitrophenol, Pentachlorophenol, 2,4-Dinitrophenol, 4,6-Dinitro-2-methylphenol = 50 ug/L.

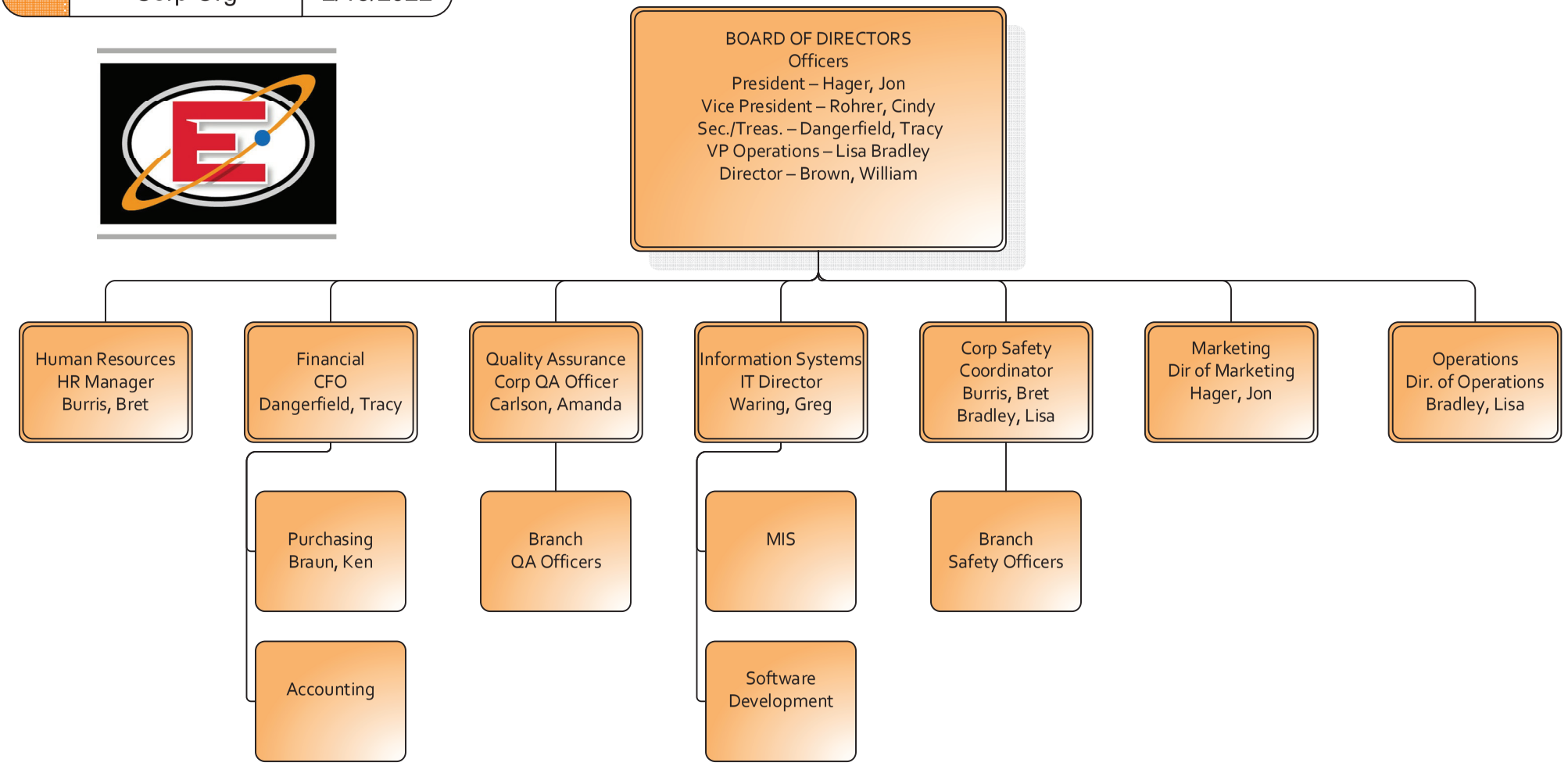
APPENDIX C

Organizational Charts

Corporate Organizational Chart
Billings Branch Lab Organizational Chart

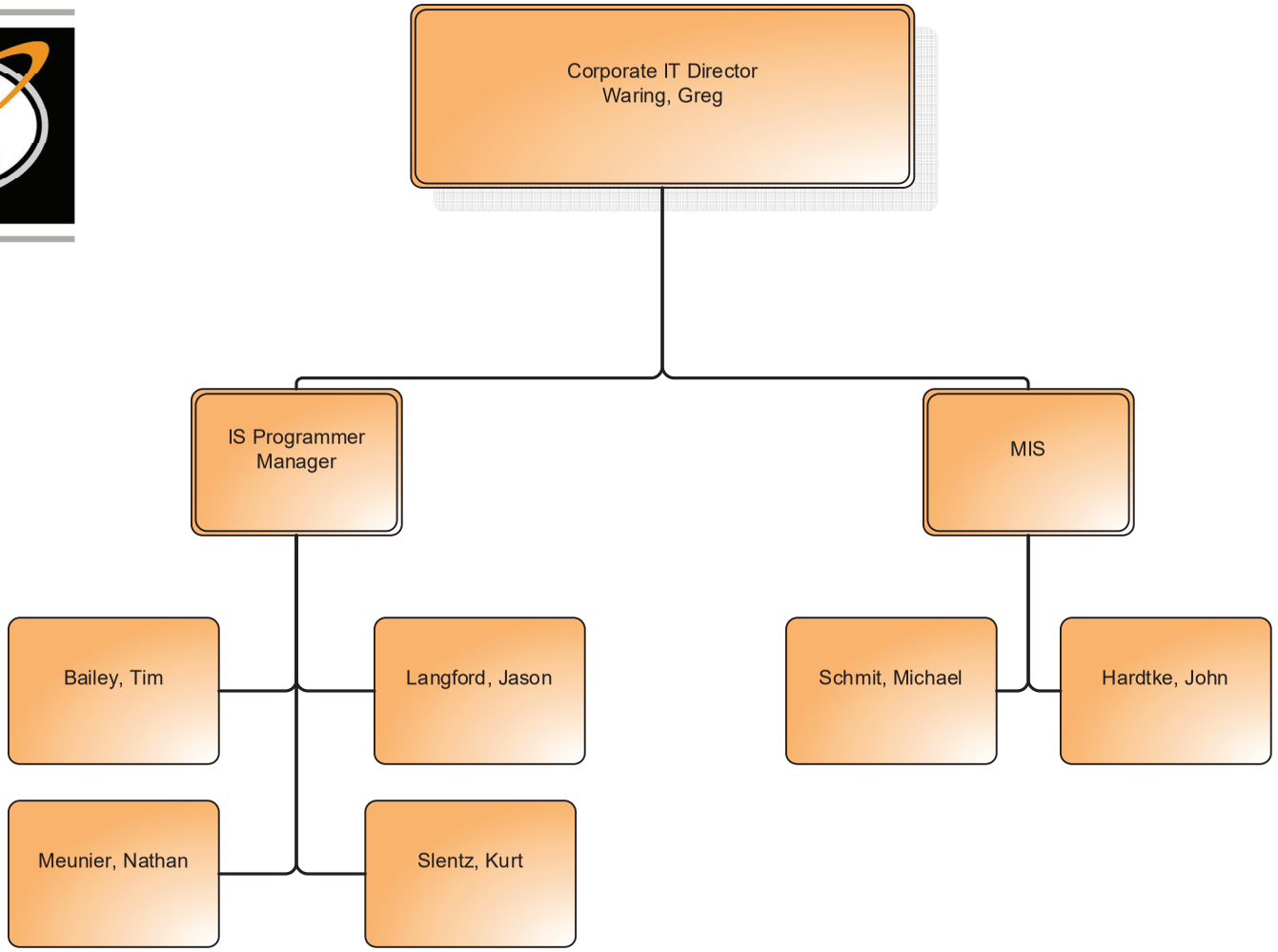
Energy Laboratories	
Corp Org	2/15/2022

Energy Laboratories / Corporate Structure



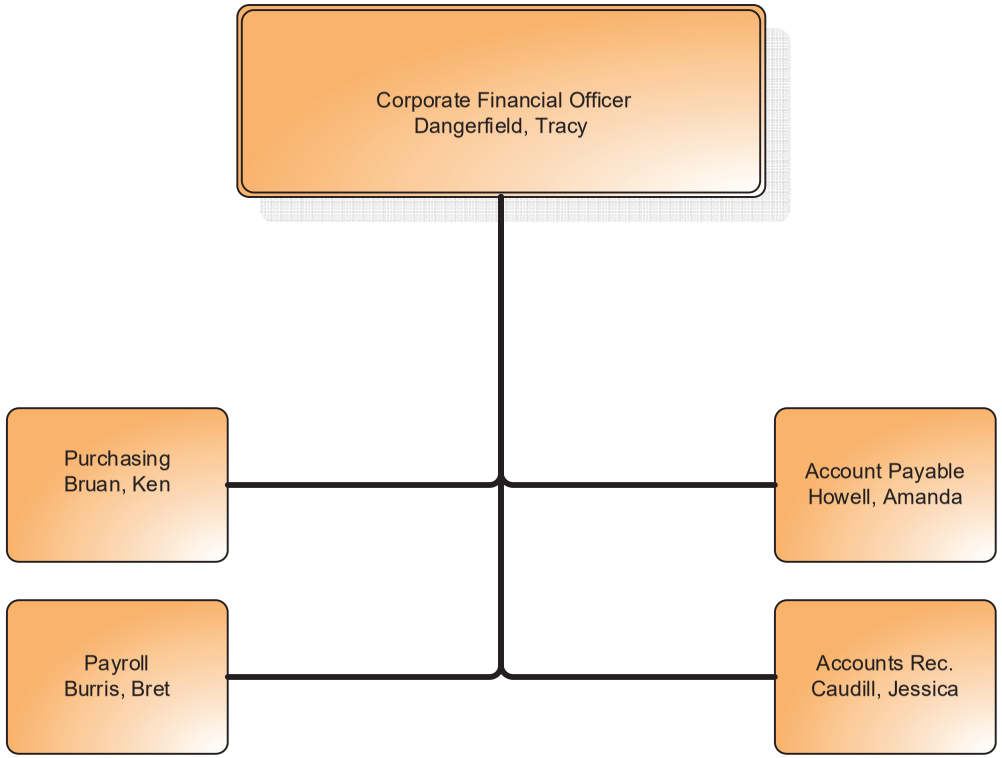
Energy Laboratories

IT | 2/15/2022



Energy Laboratories

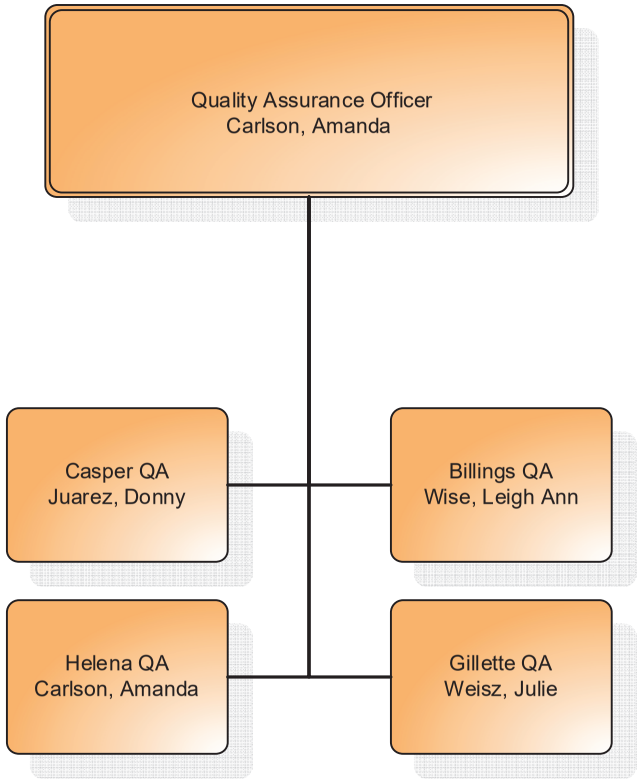
Accounting | 2/15/2022



Energy Laboratories

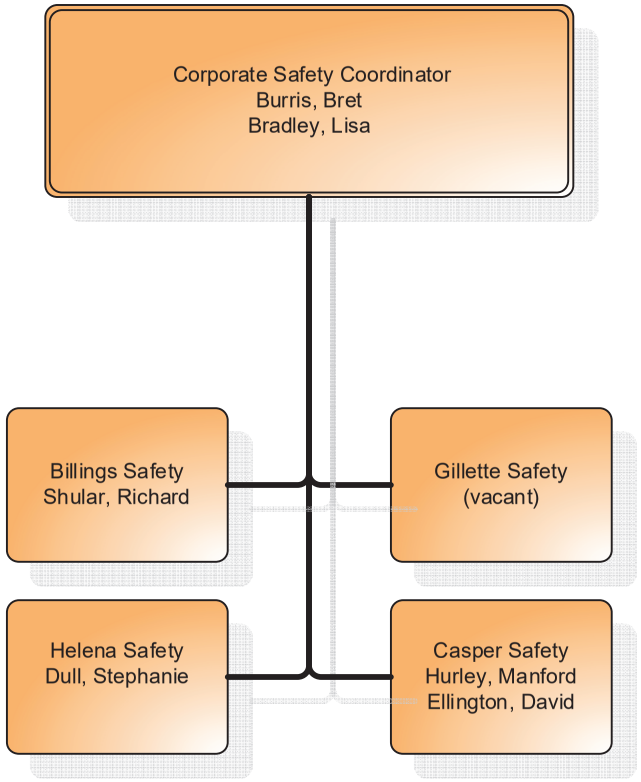
Operations | 2/15/2022

VP Operations
Bradley, Lisa


Quality Assurance Officer
Carlson, Amanda
Casper QA
Juarez, Donny
Billings QA
Wise, Leigh Ann
Helena QA
Carlson, Amanda
Gillette QA
Weisz, Julie

Energy Laboratories

Safety	2/15/2022



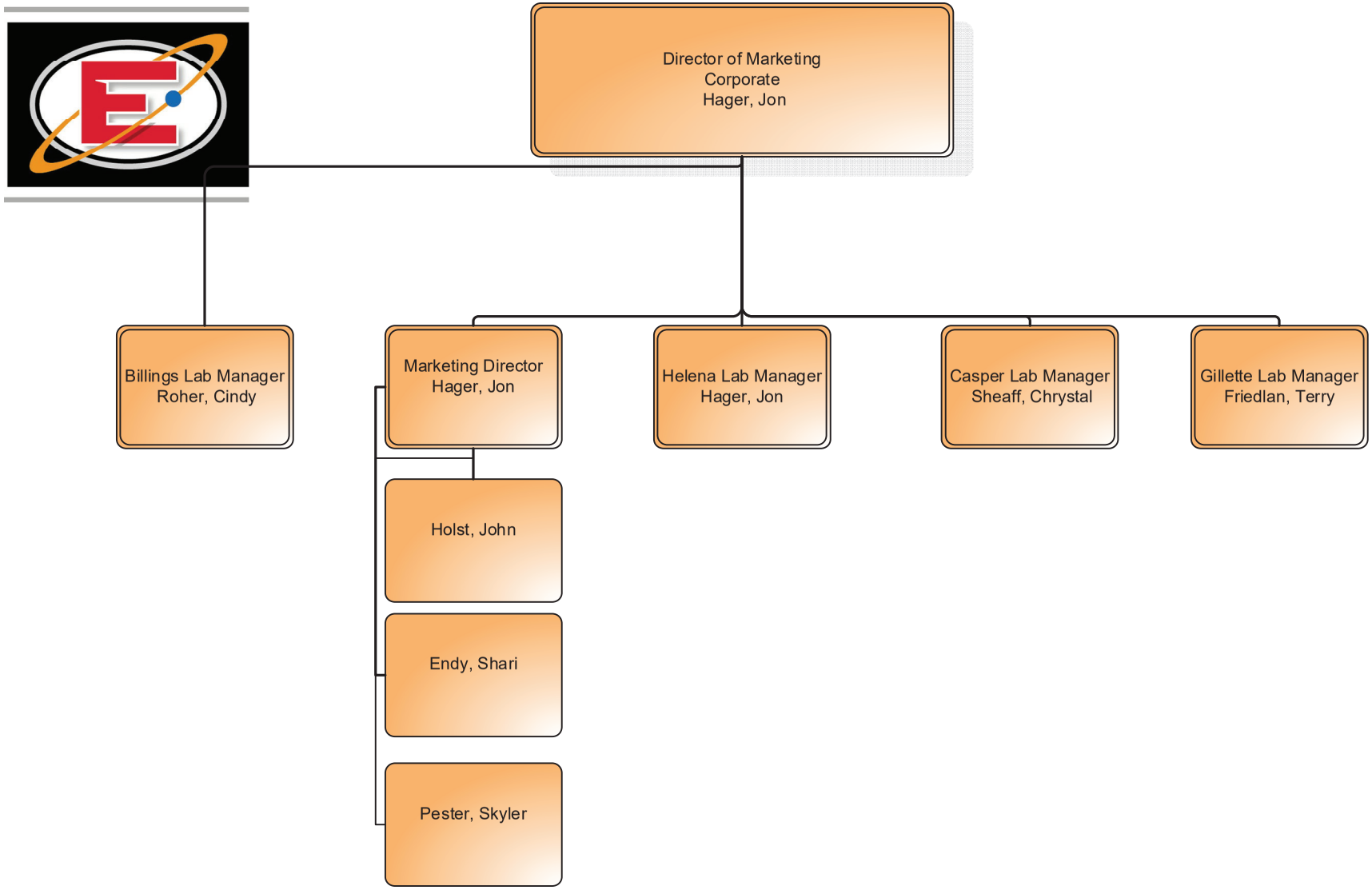
Energy Laboratories

HR	2/15/2022

HR Manager
Burris, Bret

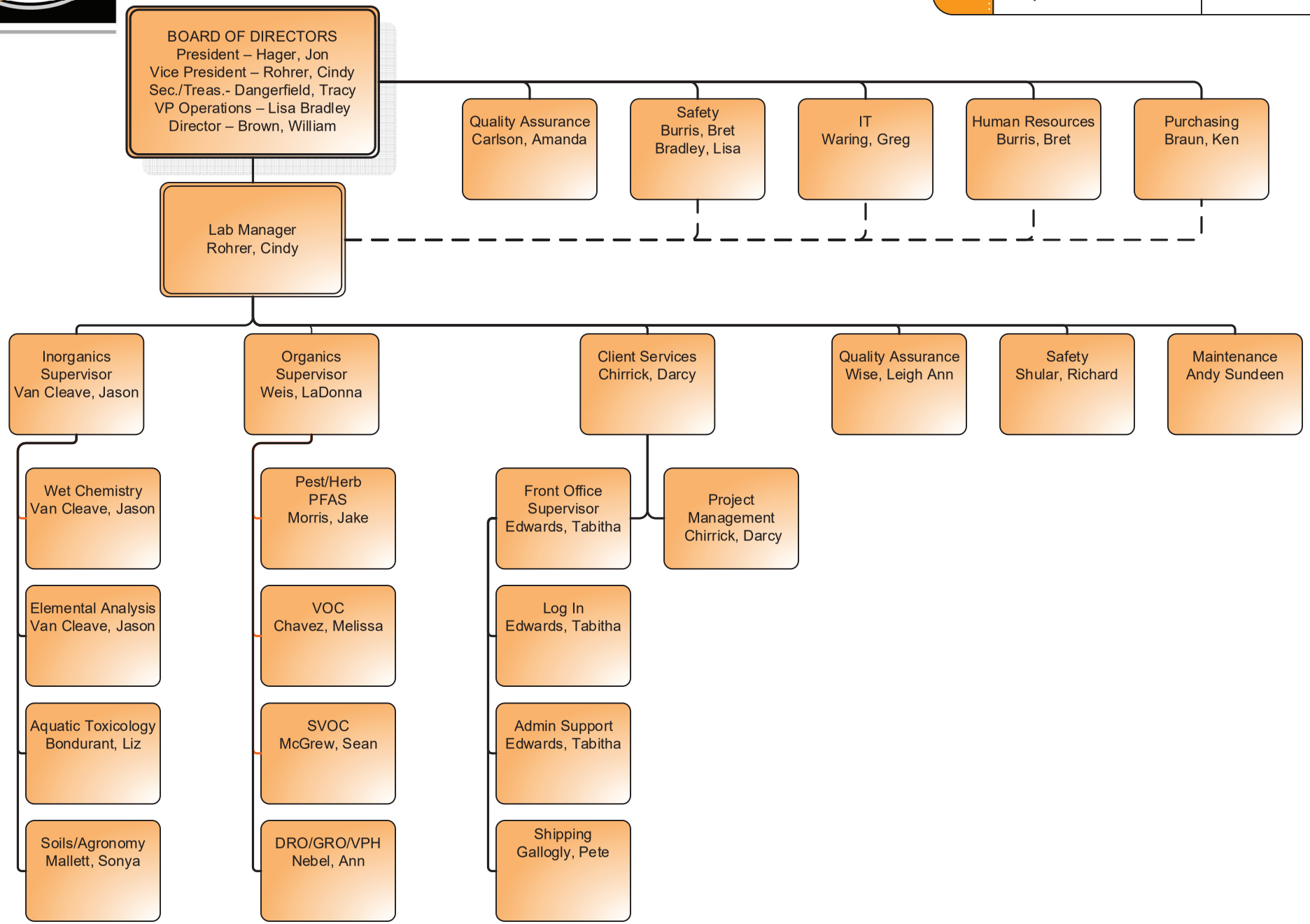
Energy Laboratories

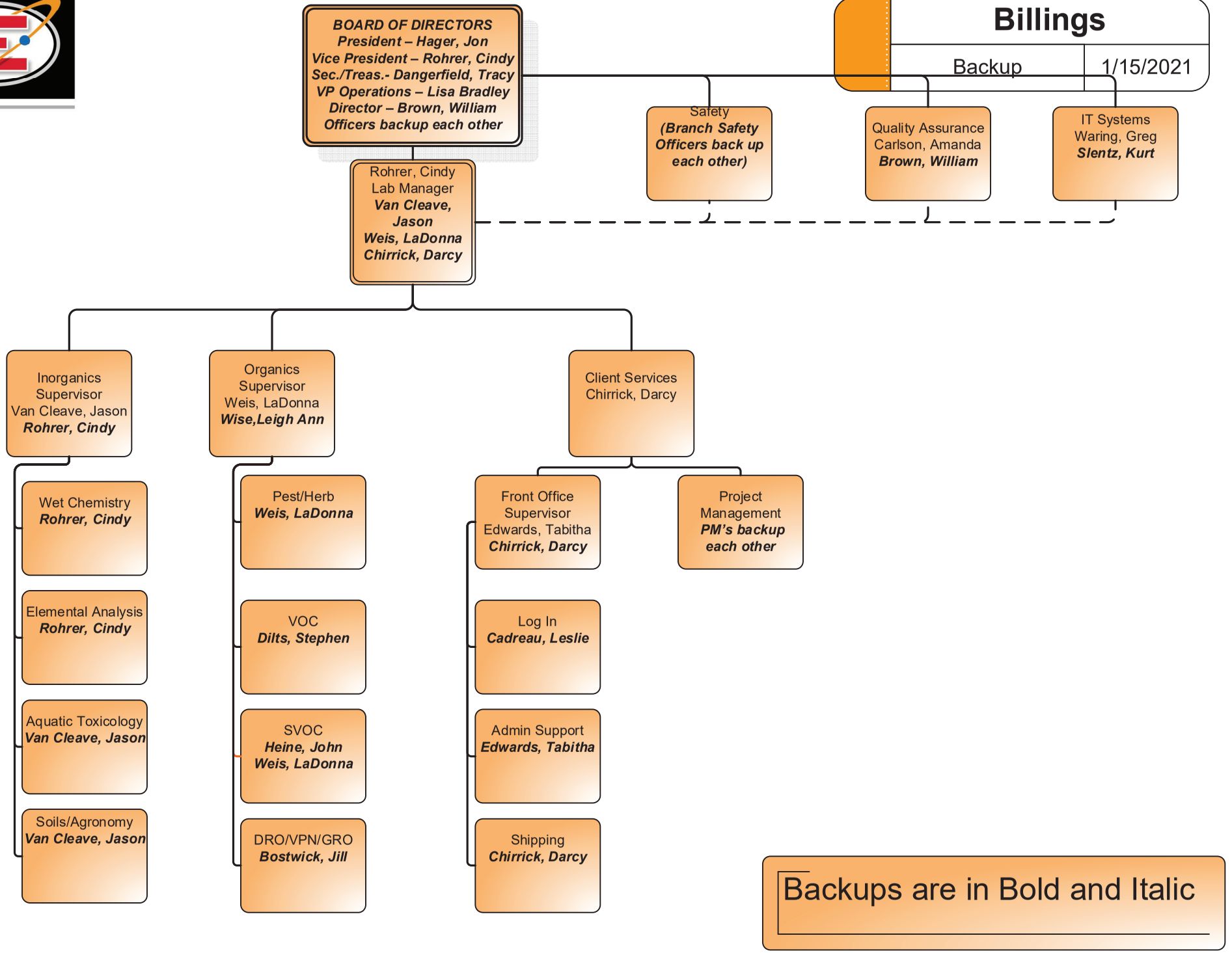
Marketing | 2/15/2022



Billings

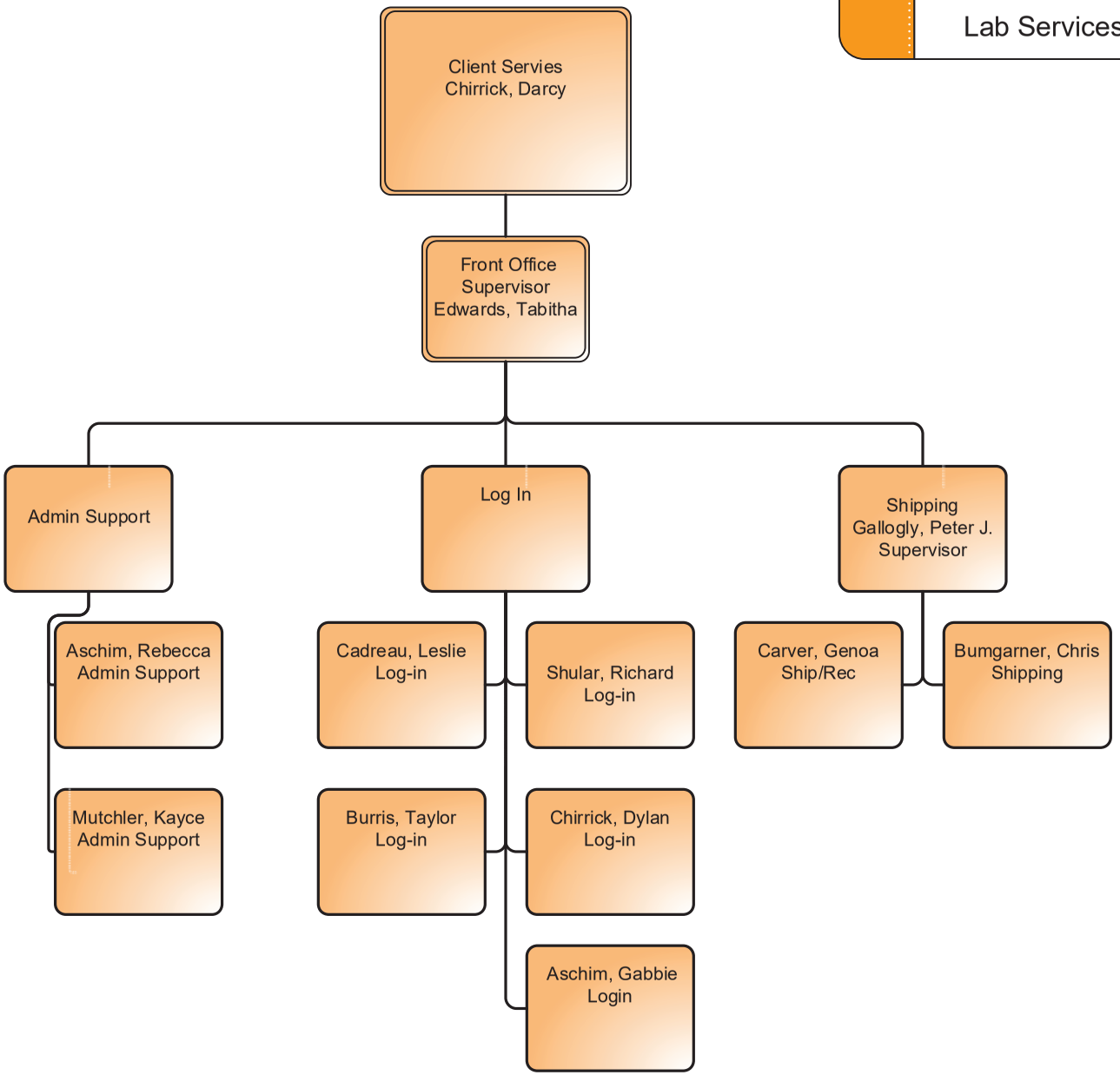
Department Heads | 1/15/2021




Billings
Backup
1/15/2021
BOARD OF DIRECTORS
President – Hager, Jon
Vice President – Rohrer, Cindy
Sec./Treas.- Dangerfield, Tracy
VP Operations – Lisa Bradley
Director – Brown, William
Officers backup each other
Safety
(Branch Safety Officers back up each other)
Quality Assurance
Carlson, Amanda
Brown, William
IT Systems
Waring, Greg
Slentz, Kurt
Rohrer, Cindy
Lab Manager
Van Cleave, Jason
Weis, LaDonna
Chirrick, Darcy
Inorganics Supervisor
Van Cleave, Jason
Rohrer, Cindy
Organics Supervisor
Weis, LaDonna
Wise,Leigh Ann
Client Services
Chirrick, Darcy
Wet Chemistry
Rohrer, Cindy
Elemental Analysis
Rohrer, Cindy
Aquatic Toxicology
Van Cleave, Jason
Soils/Agronomy
Van Cleave, Jason
Pest/Herb
Weis, LaDonna
VOC
Dilts, Stephen
SVOC
Heine, John
Weis, LaDonna
DRO/VPN/GRO
Bostwick, Jill
Front Office Supervisor
Edwards, Tabitha
Chirrick, Darcy
Project Management
PM's backup each other
Log In
Cadreau, Leslie
Admin Support
Edwards, Tabitha
Shipping
Chirrick, Darcy
Backups are in Bold and Italic

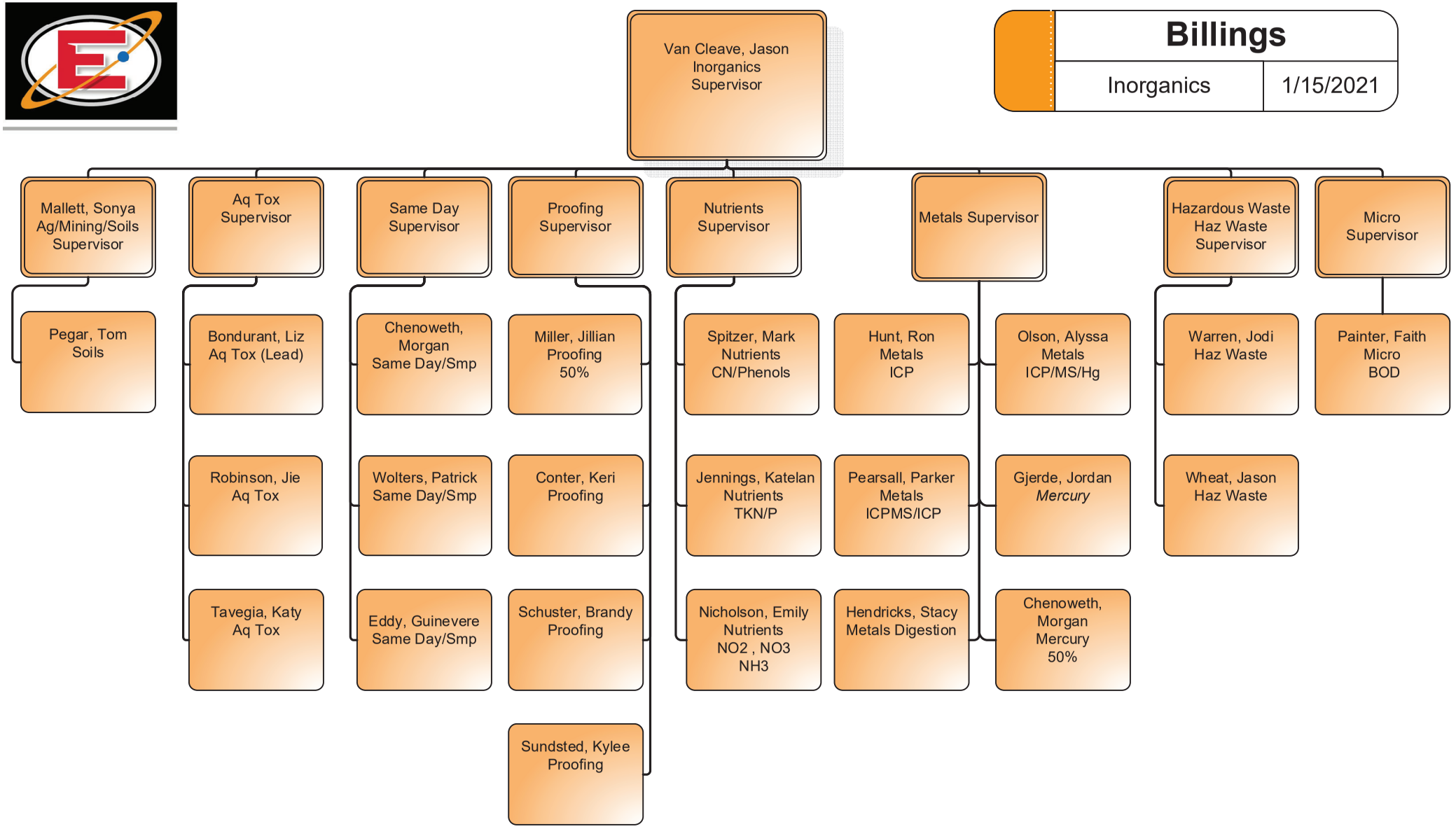
Billings

Lab Services | 1/15/2021



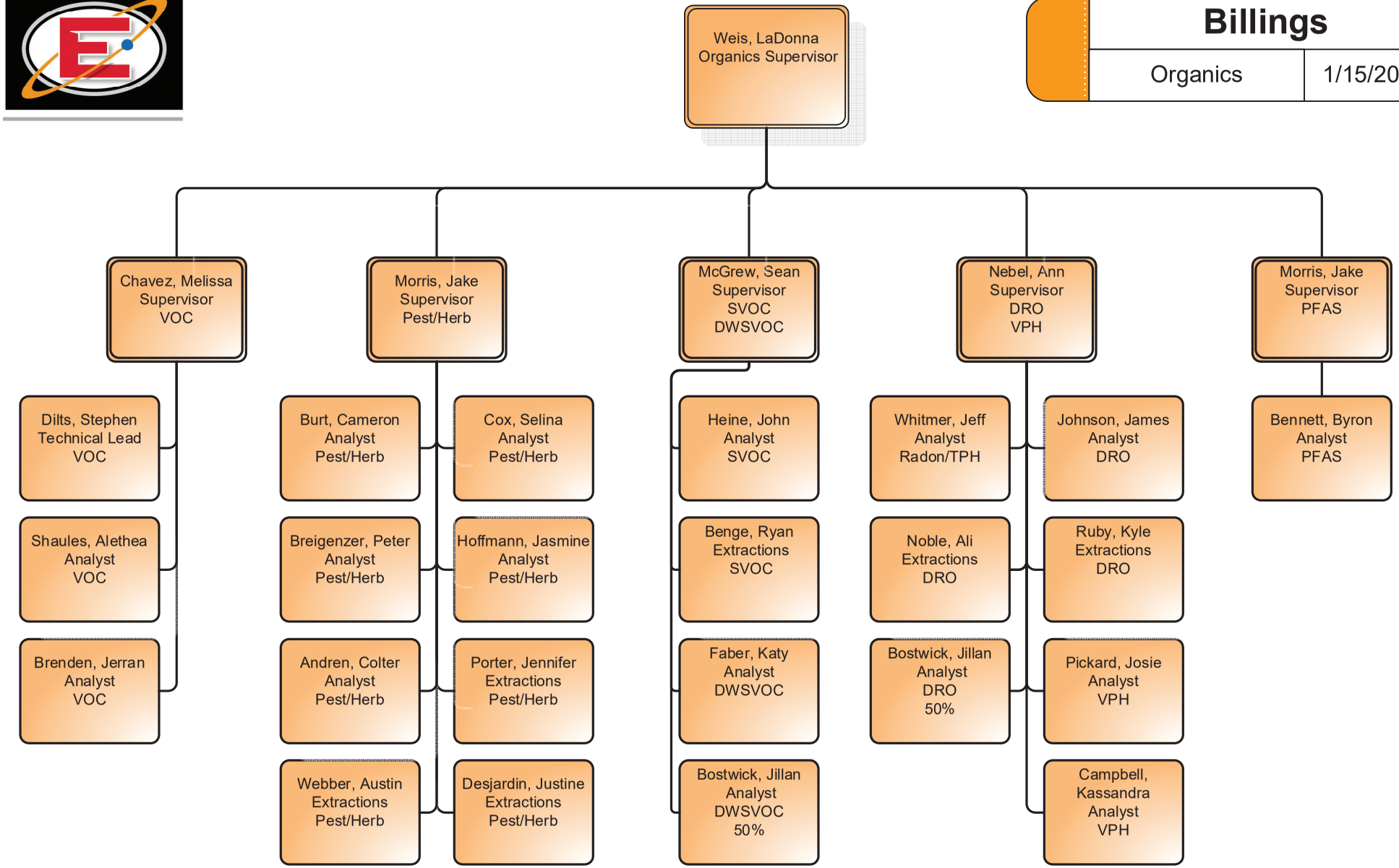
Billings

Inorganics | 1/15/2021



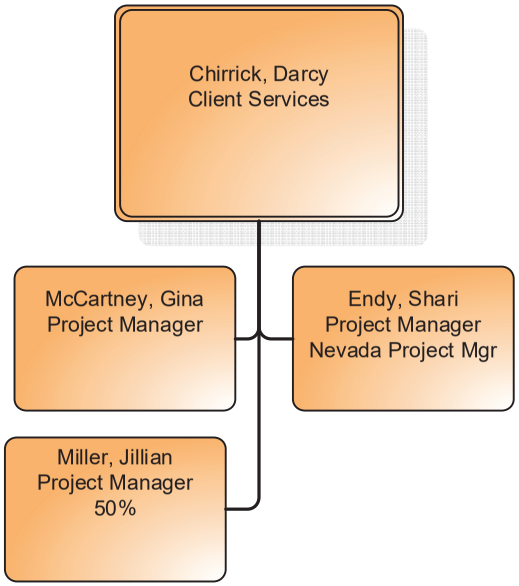
Billings

Organics | 1/15/2021



Billings

Project Management | 1/15/2021



APPENDIX D

Curricula Vitae of Key Laboratory Personnel

Curricula Vitae of Key Laboratory Personnel

Contents

JONATHAN D. HAGER

President / Helena Laboratory Manager

Academic Experience

Bachelor of Arts in Biology, Chemistry Minor, Carroll College, Helena, MT, May 2003
GC/MS Training Seminar, Restek 8 hour seminar, Sept 2005
Interaction Management, 40 hr. class, Billings, MT, 2008

Professional Experience

May, 2001-Present: Laboratory Manager -Energy Laboratories, Inc., Helena, Montana.

Responsible for ensuring work is performed with ethics, quality and safety as a primary concern. Encourages a quality-oriented and cooperative atmosphere that promotes collaboration and company-wide success.

Coordinates laboratory analysis with client contracts. Responsible for direction, training, and supervision of the analytical laboratory staff. Involved in new procedural and equipment development, quality assurance program, client relations, and report preparation.

Experienced in the analysis of soils and water in a variety of applications.

Technical Training:

GC/MS Training Seminar, Restek 8 hour seminar, Sept 2005
Interaction Management, 40 hour class, Billings, MT, 2008
Leadership Helena, Helena Chamber of Commerce, 2018

Professional Organizations

American Chemical Society
Treasure State Resource Industry Association
Alaska Miners Association
Soil Society of America

CINDY ROHRER

Vice President/Billings Laboratory Manager

Academic Experience
Bachelor of Science, Rocky Mountain College, Billings, Montana, 2000

Professional Experience
Experienced in supervision and management of staff, training analysts, technical review of data reports, and performing the following analyses: anion, alkalinity, acidity, metals analysis (ICP-MS), mercury analysis, metals digestions, Flame FAA, UV, solids and pH.

2020 – Present: Vice President, Energy Laboratories, Inc. - Responsible for development and oversight of operations for Energy Laboratories, Inc.

2014 – Present: Laboratory Manager, Energy Laboratories, Inc., Billings, MT
Supervises department operation, staff training, and maintains QA/QC criteria. Oversees audits, coordinates tasks with other departments, and performs data validation.

2011 – 2014: Inorganics and Aquatic Toxicology Supervisor, Energy Laboratories, Inc., Billings, MT
Responsible for daily operations and management of Inorganics and aquatic toxicology department. Responsibilities include supervision of Inorganics and Aquatic Toxicology staff, maintain QA/QC criteria, oversee audits, review and improve Inorganics and Aquatic Toxicology department operations, coordinate tasks with other departments, and proofing data.

2008 – 2014: Inorganics Supervisor, Energy Laboratories, Inc., Billings, MT
Responsible for daily operations and management of Inorganics department. Responsibilities include supervision of Inorganics staff, maintain QA/QC criteria, oversee audits, review and improve Inorganics department operations, coordinate tasks with other departments, and proofing data.

2006 – 2007: Inorganics Assistant Supervisor, Energy Laboratories, Inc., Billings, MT
Responsibilities included training of new analysts, QC method development, oversee audits, and management of samples.

1999: Montana State University, Billings, MT
Researched SOD mimetics, studied SOD mimetic activity of Copper Kinetin. Ran UV Spectrometry, pH meter, Mass Spec, and Flame AA.

Technical Training
Radon Measurement Provider Certification 2019
Interaction Management, 40 hour class, Billings, MT, 2008
Dale Carnegie Course 2004

LISA A. BRADLEY PH.D.

Vice President/Director of Corporate Laboratory Operations

Academic Experience
Ph.D., Analytical Chemistry, Indiana University - Bloomington, Indiana, 1996
Bachelor of Science, Chemistry, Montana State University, Bozeman, Montana, 1990

Professional Experience
2013 – Present: Vice President, Energy Laboratories, Inc., Billings, MT
Responsible for development and oversight of technical operations for Energy Laboratories, Inc.

2007 – Present: Director of Corporate Technical Operations, Energy Laboratories, Inc., Billings, MT

2008: Interim Laboratory Manager, Energy Laboratories, Inc., Casper, WY
Responsible for the supervision of the Casper laboratory.

2005 – 2008: Supervisor, Inorganics Dept., Energy Laboratories, Inc., Billings, MT
Responsible for supervision and management of inorganics laboratory. Experienced in atomic absorption spectroscopy (AA), inductively coupled plasma optical emission (ICPOES), and mass spectrometry (ICP-MS).

2000 – 2005: Supervisor, Metals Department, Energy Laboratories, Inc., Billings, MT
Supervised metals department; performed chemical analyses using laboratory instrumentation.

1996 – 2000: Analytical Chemist, Energy Laboratories, Inc., Billings, MT
Performed atomic absorption spectroscopy (AA), inductively coupled plasma optical emission (ICP-OES), and mass spectrometry (ICP-MS) analyses.

1990 – 1995: Research Assistant/Department of Chemistry, Indiana University, Bloomington, IN

1990 – 1992: Associate Instructor of Chemistry, Indiana University, Bloomington, IN

1989: Laboratory Technician, Intermountain Laboratory, Bozeman, MT

1986 – 1990: Undergraduate Research Assistant, Montana State University, Bozeman, MT

TRACY A. DANGERFIELD, CPA, MBA

Treasurer and Chief Financial Officer

Academic Experience

Master of Business Administration, University of Montana, Missoula, Montana, 2013
Certified Public Accountant, 1992
Bachelor of Science, Business Administration, Minor in Accounting, Eastern Montana College, Billings, Montana, 1989

Professional Experience

Experienced in business leadership, management, and strategic development. Extensive background in accounting, finance, and organizational development.

1989 – Present: Chief Financial Officer, Energy Laboratories, Inc., Billings, MT
Responsible for initiating, developing, and directing administrative operations including finance, human resources, taxation, and marketing. Steered the implementation of an Employee Stock Ownership Plan, transacted the ensuing 30% purchase of ELI, and continues to serve as Plan Trustee. Board Member of Tribute Insurance PIC for Self-Insured Health Plan Captive Re-insurer.

1985 – 1989: Office Management, Energy Laboratories, Inc., Billings, MT
Responsible for daily office operations and management of staff.

WILLIAM T. BROWN

Director

Academic Experience
Bachelor of Science in Fish and Wildlife, Montana State University, Bozeman, Montana, 1977

Professional Experience
Forty plus years of experience in environmental laboratory operations including Laboratory Manager, Supervisor of Organic Analysis, and Senior Organic Chemist. Experienced in Gas Chromatography, Gas Chromatography/Mass Spectrometry (GC/MS), sample preparation and extraction, ion chromatography, and chromatography data systems.

1986 – Present: President, Energy Laboratories, Inc.
Responsible for corporate direction and operations of Energy Laboratories, Inc.

1981 – 1987: Manager, Energy Laboratories, Inc., Branch Laboratory, Gillette, WY
Responsible for routine analysis and quality control of water, natural gas, and petroleum products. Involved in field on-site sampling and testing, meter calibrations, and supervision of branch laboratory staff.

1979 – 1981: Laboratory Technician, Energy Laboratories, Inc., Billings, MT
Responsible for the natural gas and petroleum products department of the lab including field natural gas testing. Involved with various work in water and soil analysis including formal training in ion chromatography.

1977 – 1979: Fisheries Biologist, Water and Forests Department of the Government of Niger, Africa while in the Peace Corps. Responsible for developing fisheries management programs in a specific region including monitoring water quality by on-site.

AMANDA B. CARLSON

Corporate Quality Assurance Officer/Helena Assistant Laboratory Manager

Academic Experience
Bachelor of Arts in Chemistry, Carroll College, Helena, Montana, 2004

Professional Experience
2019 – Present: Corporate Quality Assurance Officer, Energy Laboratories, Inc.
Responsible for Quality Assurance procedures and monitoring. Assists with method development, prepares and updates standard operating procedures, performs technical training, and involved with special projects.

2013 – Present: Assistant Laboratory Manager, Energy Laboratories, Inc., Helena, MT
Assists in the supervision of the daily operations of the laboratory while promoting collaboration and communication between analysts. Supervise Inorganics Department.

2008 – Present: Quality Assurance Manager, Energy Laboratories, Inc., Helena, MT
Ensures the laboratory maintains client satisfaction by meeting quality requirements. Maintains training records for employees and provide ongoing training of QAQC topics. Maintains a general knowledge of methods performed in the laboratory and the appropriate method corrective actions. Coordinate client relations from bottle preparation and sample receipt through reporting, invoicing, and data review of technical reports issued to clients.

2004 – 2008: Inorganics and Organics Analyst, Energy Laboratories, Inc. Helena MT
Certified analyst for total coliform and E.coli in both public and private water samples.

Technical Training
Basic Assessor Training TNI Standard 2016, 3 day course, 2019
Small Laboratory TNI Standard Implementation, 21 hour course, 2017
Contaminant Vapor Migration and Intrusion, 13 hour class, Helena, MT, Feb 2013
Interaction Management, 40 hour class, Billings, MT, 2008
GC/MS Training Seminar, Restek 8 hour seminar, Sept 2005

Professional Organizations
American Water Works Association
American Chemical Society
TNI

LEIGH ANN WISE

Billings Laboratory Quality Assurance Officer

Academic Experience

Bachelor of Science, Chemistry, Montana State University, Billings, Montana, 2003
Bachelor of Science, Biology, Montana State University, Billings, Montana, 2000

Professional Experience

2019 – Present: Quality Assurance Officer, Energy Laboratories, Inc., Billings, MT
Coordinates and monitors the laboratory quality assurance (QA) program. Works closely with supervisors to schedule and implement QA related activities and ensures the laboratory meets all accreditation requirements. Coordinates or performs QA performance audits through proficiency testing programs and method internal audits. Reviews and approves laboratory reports and provides ongoing training of QA topics.

2013 – 2019: Co-Supervisor Organics Department, Supervisor of Semi Volatile Drinking Water and Volatile Organic Analysis Energy Laboratories, Inc., Billings, MT. Supervises the various areas of the Billings Organics Department, encourages the professional development of staff and continually maintains and refines quality assurance and control criteria. Oversees audits, sample load, technically reviews data and reports, and assists with the requirements and maintenance of laboratory certifications.

2009 – 2013: Supervisor of Semi Volatile Drinking Water Analysis, Energy Laboratories, Inc., Billings, MT
Coached staff and managed sample load and analysis. Developed modules and guidelines for training, employee performances, and compensation reviews. Provided goals and expectations to staff and monitored the progress. Managed department and laboratory issues as they arose and addressed employee performance as needed. Maintained method standard operating procedures and technically reviewed data and reports.

2000 – 2009: Chemist, Energy Laboratories, Inc., Billings, MT
Certified in the analysis of volatile organic, semi volatile organic, pesticide, herbicide, and polychlorinated biphenyl compounds in various sample matrices. Maintained and operated various types of instrumentation including Gas Chromatography, Gas Chromatography/Mass Spectrometry, Electron Capture Detector, Chemical Ionization, and Purge and Trap. Managed sample loads, maintained quality assurance and control criteria, and performed method development and improvements.

Technical Training

Interaction Management Essentials of Leadership, Billings, MT 2012
Excelling as a Manager or Supervisor, SkillPath Seminar, Billings, MT 2010
GC/MS Training Seminar, Restek 8 hour seminar, Butte, MT 2005

JASON VAN CLEAVE

Inorganics Supervisor
Supervisor of Inorganics, Hazardous Waste, Soils, and Aquatic Toxicology Departments

Academic Experience
Bachelor of Science, Biology, Montana State University, Billings Montana, 2008

Professional Experience
Experienced in Supervision and Management of staff, training analysts, and performing the following analyses: Inductively Coupled Plasma-Mass Spectrometry, and Inductively Couples Plasma Atomic Emission Spectrometry (metals); Ion chromatography for anion analysis, colorimetric analysis of Nutrients-Segmented Flow, Colorimetric analysis of Nutrients- Discrete analyzer, Biochemical Oxygen demand, Chemical Oxygen demand, and UV254 (inorganics); Certified Microbiologist by the State of Montana for Drinking Water analysis, Colilert, Colilert 18, Colisure, MF-Total coliform, MF-fecal coliform, MF-*E. c*oli, multiple tube fermentation MPN, Sulfate reducing bacteria, and Iron-related bacteria BART (microbiology).

2018 – Present: Inorganics Supervisor, Energy Laboratories, Inc., Billings, MT
Responsible for the operations of the inorganic laboratory. Manage personnel and ensure that SOPs are followed. Coordinate testing with other departments and project managers. Assisting in maintenance and troubleshooting of instrumentation.

2011 – 2018: Inorganic analyst, Energy Laboratories, Inc., Billings, MT
Performed microbiological testing on drinking water and waste water samples. Performed inorganic analysis including the following: ion chromatography, spectrophotometric analysis (for many analytes including cyanide, phosphorus, ammonia, and nitrogen), and metals analysis by inductively coupled plasma-mass spectroscopy. Reviewed methods and aided in the development of analytical methods. Oversaw and trained analysts in the nutrients department.

2008: Adjunct faculty, Montana State University-Billings, Billings, MT
Instructed introductory biology and physics laboratories. Planned and set-up for laboratory experiments and created quizzes and tests. Managed teaching assistants who aided in set-up and take down of labs as well as assisted students during the lab.

LADONNA WEIS

Supervisor Billings Organics Department

Academic Experience
Bachelor of Science in Biology, Chemistry minor, Montana State University, Billings, MT 2003

Professional Experience
2013 – Present: Supervisor Organics Department, Energy Laboratories Inc., Billings, MT
Responsibilities include training of new analysts, EPA method development, maintaining instrumentation, overseeing audits, and management of samples. Handle and resolve critical quality problems using research abilities and hands-on experience. Provides team leadership, data review, and project management.

2009 – 2013: Supervisor of Pest/Herb Department, Energy Laboratories Inc., Billings, MT
Supervised and trained extraction analysts with an emphasis on proper laboratory technique and accurate, reproducible data. Combined effective communication, organizational skills and planning for successful time management. Assigned duties/shifts to employees, monitored performance of the employees and maintained/documented work completed. Participated in the development and implementation of Peer Audits throughout the company branch labs. Managed sample loads, maintained quality assurance and control criteria, and recommended new/modified method developments.

2005 – 2009: Chemist, Energy Laboratories Inc., Billings, MT
Performed analyses of pesticide, herbicide, and polychlorinated biphenyl compounds in various sample matrices. Maintained and operated Electron Capture Detectors (ECD). Increased knowledge of quality control measures. Documented and prepared timely reports on the tests conducted and the results obtained.

2003 – 2005: Lead Pest/Herb Extractions, Energy Laboratories Inc. Billings, MT
Began as analyst of pesticide, herbicide and polychlorinated biphenyl compounds; became lead analyst in 2004. Became proficient and knowledgeable with regulatory guidelines, managed incoming samples and prioritized sample load based on sample collection date, hold time, and client's needs. Mastered all software associated with the analysis process.

2002: Aquatic Toxicologist, Energy Laboratories Inc. Billings, MT
Performed toxicity reduction evaluations for chronic and acute testing of water samples and determined causative toxicity in effluent waters. Determined electrical conductivity, concentrations of dissolved oxygen, alkalinity, ammonia, total residual and free chlorine in aqueous solutions. Calculated inhibition concentration point and determined lethal and effective concentration end points using analytical graphical methods.

Technical Training
Supervisory Leadership Skills Training, Development Dimensions International, 2011
Interaction Management, 40 hour class, Billings, MT, 2008

TIMOTHY D. BAILEY PH.D.

Senior Analytical Chemist/Software Architect

Academic Experience

Ph.D., Analytical Chemistry, University of Wisconsin-Madison, Madison, Wisconsin, 1989
Bachelor of Arts, Chemistry, Montana State University, Bozeman, Montana, 1980

Professional Experience

Experienced in working for a commercial laboratory and for a major international chemical producer. Knowledgeable with inductively coupled plasma optical emission (ICP-OES) and mass spectrometer (ICP-MS), and atomic absorption (AA) techniques. Experienced with implementation of EPA Good Laboratory Practices programs, statistical quality management for laboratory analysis, and analytical methodologies such as EPA SW-846, 500, and 600 series. Aids in architecting solutions that improve the quality and efficiency of the laboratory analytical operations ranging across the Laboratory Information Systems (LIMs), metals and radiochemistry applications.

1994 – Present: Senior Analytical Chemist/Software Architect, Energy Laboratories, Inc., Billings, MT

1989 – 1994: Project Leader/Senior Research Chemist, the Dow Chemical Company, Midland, MI

1988 – 1989: Graduate Technical Assistant/Chemistry Department Instrument Center, University of Wisconsin-Madison, Madison, WI

1984 – 1988: Graduate Teaching Assistant/Analytical and General Chemistry, University of Wisconsin-Madison, Madison, WI

1980 – 1984: Analytical Chemist, Energy Laboratories, Inc., Billings, MT

STEPHEN B. DILTS, PH.D.

Senior Analytical Chemist

Academic Experience
Ph.D., Analytical Chemistry, Washington State University, Pullman, WA, 1993
M.S., Analytical Chemistry, Washington State University, Pullman, WA, 1985
B.S., Chemistry, Montana State University, Bozeman, MT, 1981

Professional Experience
1994 – Present: Senior Analytical Chemist, Energy Laboratories, Inc., Billings, MT
Volatile Organics GC/MS analyst.

1989 – 1993: Research Assistant, Department of Civil and Environmental Engineering, WSU, Pullman, WA. Performed field research in the analysis of atmospheric organic compounds.

1986 – 1989: Chemist, Montana Department of Agriculture-Laboratory Bureau, Bozeman, MT
Performed pesticide, hazardous waste, and toxicological analysis for regulatory purposes.

1982 – 1985: Research Assistant, Department of Civil and Environmental Engineering, WSU, Pullman, WA. Performed field research in the analysis of atmospheric sulfur compounds.

1982: Laboratory Technician, Halliburton Services, Inc., Evansville, WY
Performed oil field water, cement, and soils analysis.

Professional Organizations
American Chemical Society

DARCY E. CHIRRICK

Client Services Lead/Project Management

Academic Experience

HS Graduate, General Studies-Business Courses, Skyview High School, Billings, MT 1988
Attended Eastern Montana College majoring in Biology and Analytical Chemistry 1990-1994

Professional Experience

2018 – Present: Project Manager, Energy Laboratories, Inc., Billings, MT
Responsible for client services, including sample receipt and login, sample container shipping, data reporting, and project management.

2014 – 2018: General Manager, Homewood Suites by Hilton, Billings, MT
Responsibilities included all aspects of hotel operations, revenue management, personnel management, and guest relations.

2007 – 2014: Corporate Accounts Receivable Manager, Energy Laboratories, Inc., Billings, MT
Responsible for corporate accounts receivable for six branch offices including monthly invoicing, collections, and ensuring delivery and posting of all payments and invoices to the corporate accounting system.

1998 – 2014: Owner/Operator, Reifschneider Investments, Billings, MT
Owned and operated five Taco Johns Restaurants in Billings and Laurel, MT. Responsibilities included revenue management, personnel management, and franchise business operations.

1990 – 2007: Office Manager, Energy Laboratories, Inc., Billings, MT
Responsibilities included client relations, sample login, sample container shipments, data reporting, EDD generation, and personnel management.

SHARI ENDY

Senior Project Manager

Academic Experience
B.S. Petroleum Engineering, Montana College of Mineral Science and Technology, Butte, MT, 1988
Masters credits in Petroleum Engineering, Montana College of Mineral Science and Technology, Butte, MT, 1988

Professional Experience
2002 – Present: Project Manager, Energy Laboratories, Inc., Billings, MT
Twenty-five plus years of experience with management of environmental analytical projects for a wide variety of clients in the public and private sector. Perform marketing duties for new and existing business areas.

2000 – 2002: NELAP Coordinator, Energy Laboratories, Inc., Billings, MT
Responsible for coordination of achieving national certification status for Billings, MT laboratory and assisted in achieving equivalent certification status for Casper, WY laboratory.

1994 – 2000: Project Manager, Maxim Technologies, Inc., Billings, MT
Responsible for managing client projects and developing business for new market areas.

1988 – 1993: Environmental Engineer, Exxon Billings, Refinery, Billings, MT
Responsible for maintaining environmental compliance of hazardous waste operations permit. Included field sampling, monitoring of environmental data, management of land treatment units and responding to refinery upsets.

Technical Training
40 hours Hazardous Waste Operations Training
Licensed Wastewater Treatment Operation – State of Montana
Refinery Safety Training

GREG WARING

IT Director

Academic Experience
Bachelor of Science in Computer Science, Minor in Business Management, Montana Technological University, Butte, MT, 1996

Professional Experience
Experienced in information technology operations and management including the following: infrastructure support, hardware provisioning, software development, and vendor management.

2011 – Present: IT Director, Energy Laboratories Inc., Billings MT
Responsible for all aspects of IT operations including the following: personnel management, process improvement, software maintenance and development, desktop support operations, server and network management, and vendor management.

2007 – 2010: Client Care Manager, Zoot Enterprises, Bozeman, MT
Responsible for delivery, client satisfaction and growth of major client accounts including some of the largest financial institutions in the nation.

2005 – 2007: PM and Consulting Group Manager, Zoot Enterprises, Bozeman, MT
Managed the operation of the Project Management and Consulting teams. Responsible for process development and delivery standardization, resolution of client escalations, and personnel management.

1997 – 2005: Project Manager, Electronic Data Systems (EDS) a component of HP
Managed projects and delivered IT initiatives for multiple clients and industries. Projects ranged from upgrade and testing initiatives to large multi-system application development for Fortune 100 companies and government agencies.

RICHARD SHULAR

Safety Officer, Waste Manager, and Industrial Hygiene Coordinator

Academic Experience
Bachelor of Chemistry, Montana State University, Billings, MT 2015

Professional Experience
2019 – Present: Safety Officer, Waste Manager, and Industrial Hygiene Coordinator, Energy Laboratories Inc., Billings, MT. Responsible for all safety training including new hire safety orientation and continuing safety training for all staff. Oversees all required OSHA programs including the following: hearing conservation program, respiratory protection program, hazard communication, personal protective equipment, emergency action plans, emergency equipment, and record keeping. Manages all waste streams from the faculty per Montana Department of Environmental Quality and Environmental Protection Agency requirements. Responsible for the coordination of client sampling for Industrial Hygiene sampling and PM10 analyst.

2016 – 2019: Log-In Technician, Energy Laboratories Inc., Billings, MT.
Received client samples and processed them for the laboratory. Identified analysis needed from the clients' Chain of Custody and from state and federal requirements. Coordinated client needs and entered information into the LIMS system

2011 – 2015: Research assistant for Dr. Stuart Snyder, Professor of Physics at Montana State University Billings. AREIS grant recipient for Two-Photon Laser Induced Fluorescence of Atomic Sodium.

2013 – 2014: Research assistant for Dr. Matthew Marlow, Professor of Chemistry at Montana State University Billings. Researched into Polycyclic Aromatic Hydrocarbons identification in sugar glasses using Raman spectrometry.

Technical Training
40 hours OSHA 1910.120 Hazardous Waste Operations and Emergency Response Training 2019
8 hours OSHA Compliance Training 2019

APPENDIX E

Equipment and Methods List

APPENDIX E
Major Equipment and Methods

Equipment	Quantity	Methods
Gas Chromatograph - FID with auto sampler	5	MA-EPH, DRO, SW8015C
Gas Chromatograph - PID/FID with purge and trap and auto sampler	4	MA-VPH, GRO, SW8015C, SW8021B
Gas Chromatograph - Dual ECD with auto sampler	5	SW8011, SW8081B, SW8082A, SW8151A, E504.1, E508A, 515.4, E552.2, E608.3
Gas Chromatograph - Mass Spectrometer with auto sampler	6	SW8270C/D/E, E525.2, E507Mod, E548.1, E625.1
Gas Chromatograph - Mass Spectrometer with purge and trap and auto sampler	5	SW8260B/D, E524.2, E624.1
Liquid Chromatography/Tandem Mass Spectrometry	1	E537.1
Closed Cup Flashpoint Analyzer	1	SW1010M
Ion Chromatography System (IC)	2	E300.0
Inductively Coupled Atomic Emission Spectrophotometer (ICP-AES)	2	E200.7, SW6010B/D
Inductively Coupled Mass Spectrometer (ICPMS)	3	E200.8, SW6020/B
Block Digestors	7	E200.2, SW3010A, SW3050B, SW7471B
Cold Vapor Atomic Absorption (CVAA) Analyzer	2	E245.1, SW7470A, SW7471B, SM3112 B
Cold Vapor Atomic Fluorescence (CVAFS) Analyzer	1	E245.7
Flow Injection Analyzer (FIA)	3	E335.4, E350.1, E351.2, E353.2, E365.1, A4500-CN L
Total Kjeldahl Nitrogen (TKN) Block Digestor	2	E351.2
Total Phosphorus Block Digestor	1	E365.1
AutoAnalyzer	1	E353.2, E365.1
Segmented Flow Analyzer (SFA)	1	A4500-CN G, SW9012, Kelada-01, E335.4, A4500-CN-F, D2036C, E420.1, E420.4
Automatic Titrator	2	A2310 B, A2320 B, A4500-F C
Turbidimeter	2	A2130 B
Automated pH/SC	1	A2510 B, A4500-H B
pH /Conductivity/DO/ISE meters and probes	multiple	A2510 B, A4500-H B, A4500-O G, A4500-F C, A4500-CN-F
Automated Biochemical Oxygen Demand (BOD) Analyzer	1	A5210 B, A5210 C
Fixed Wavelength IR Spectrophotometer	1	E413.1, E413.2, E418.1
UV-Vis Spectrophotometer	2	410.4, A3500-CR B, A4500-S D, N3500M, A4500-CN M, A5550 B
Leco Carbon Sulfur Analyzer	2	D1552, Leco
Balances	multiple	A2540 C, A2540 D, A2540 G, A2540 B
Autoclave, Ovens, Incubators	multiple	

APPENDIX C

FIELD FORMS

- Change Request Form
- Equipment Maintenance and Calibration Record
- Safety Meeting/Training Log
- Field Sampling Report
- XRF Usage Log
- XRF Calibration Form
- XRF Daily Sampling Log

HGL
CHANGE REQUEST FORM

Contract/Project: _____ Date: _____

Requested by: _____

Description of requested change: _____

_____

_____

_____

_____

Reason for change: _____

_____

_____

_____

_____

Expected results or impact: _____

_____

_____

_____

Submit this form to the project manager immediately.

Required before implementation of major changes:

Approved by: _____(Project Manager) Date: _____

Approved by: _____ (Title: _____) Date: _____

cc: QA Staff Member

HGL HydroGeoLogic, Inc
Exceeding Expectations

EQUIPMENT MAINTENANCE AND CALIBRATION RECORD

Contract/Project: _____ Equipment Description: _____

Activity: _____ Equipment ID: _____

Equipment Serial No.: _____

Calibration Date/Time	Parameter	Standard Used (Concentration)	Lot Control No./ Expiration Date	Post Calibration Reading	Comments Pass/Fail	Signature

Maintenance Performed: _____

HydroGeoLogic, Inc
Exceeding Expectations

SAFETY MEETING/TRAINING LOG

- ☐ Tailgate (daily)
- ☐ Activity Hazard Analysis
- ☐ Pre-Task Hazard Analysis (prior to new task or operation)
- ☐ Site Safety Orientation (new personnel)
- ☐ Supervisor's (monthly)
- ☐ Supervisor's (weekly)
- ☐ UXO Awareness
- ☐ Asbestos Awareness
- ☐ Health and Safety Plan Addendum: _____
- ☐ Other: ___

Date/Time: _____ Client: _____

Location: _____ Job No.: _____

Meeting/training conducted by: _____

Work Activities: _____

Safety / Training Topics Presented

Chemical Hazards: _____

Physical Hazards: _____

Specific Safety Topic(s): _____

_____

Specific Training Covered: _____

_____

Attendees

Name Printed and Employee Number:	Signature:

FIELD SAMPLING REPORT

LOCATION: PROJECT NAME:

SITE: PROJECT NO:

SAMPLE INFORMATION

SAMPLE ID: DATE:_____ TIME:_____

MATRIX TYPE:

SAMPLE COLLECTION METHOD:

LOW-FLOW BAILER PASSIVE OTHER_____

LOT CONTROL #: ___ ___ ___ ___

(Ambient Blank # - Equipment Blank # - Trip Blank # - Cooler #)

CHAIN-OF-CUSTODY #: _____

SAMPLE BEG. DEPTH (FT):

SAMPLE END DEPTH (FT):

GRAB ( ) COMPOSITE ( )

ENTER SAMPLE NUMBERS FOR QC SAMPLES/ BLANKS ASSOCIATED WITH THIS SAMPLE:

MATRIX SPIKE (MS): _____

MATRIX SPIKE DUP (SD): _____

FIELD DUP (FD): _____

AMBIENT BLANK (AB): _____

EQUIPMENT BLANK (EB): _____

TRIP BLANK (TB): _____

CONTAINER SIZE/TYPE	#	PRESERVATIVE/ PREPARATION	ANALYTICAL METHOD	ANALYSIS

NOTABLE OBSERVATIONS

PID READINGS	SAMPLE CHARACTERISTICS	MISCELLANEOUS
1st (TOC):	COLOR:	
2nd (BZ):	ODOR:	
	OTHER:	

pH_____ Temperature_____(C) Dissolved Oxygen_____(mg/L) Specific Conductivity_____(mS/cm)

Ferrous Iron_____(mg/L) Oxidation/Reduction Potential_____(mv) Turbidity_____(NTU

GENERAL INFORMATION

WEATHER: SUN/CLEAR_____OVERCAST/RAIN_____WIND DIRECTION_____AMBIENT TEMPERATURE _____

SHIPMENT VIA: FEDEX_____HAND DELIVER_____COURIER_____OTHER _____

SHIPPED TO:_____

COMMENTS: _____

SAMPLER:_____OBSERVER: _____

MATRIX TYPE CODES

DC=DRILL CUTTINGS
SL=SLUDGE
WG=GROUND WATER
SO=SOIL
LH=HAZARDOUS LIQUID WASTE
GS=SOIL GAS
SH=HAZRDOUS SOLID WASTE
WS=SURFACE WATER
SE=SEDIMENT
SW=SWAB/WIPE
W=WATER

SAMPLE COLLECTION METHOD CODES

B=BAILER
HA=HAND AUGER
BP=GAS OPERATED BLADDER PUMP
HY=HYDRASLEEVE
CS=COMPOSITE SAMPLE
NS=NON-SUBMERSIBLE PUMP
EC/TC=ENCORE/TERRA CORE SAMPLER
PP=PERISTALTIC PUMP
GB=GEOPROBE
SP=SUBMERSIBLE PUMP
H=HOLLOW STEM AUGER
SS=SPLIT SPOON
OTHER
G = GRAB
TR=TROWEL

XRF Usage Log

Site Name:	Operator Name	Project No.:	Page:	
Date		Number of Analyses		Finger Ring Dosimeter Worn (Y/N)?
		Handheld Operation	Testing Stand Operation	

Notes:
Keep original copy of this log with project files.
Please forward s copy of this log to the Radiation Safety Officer at the end of the calendar or at completion of fieldwork.

XRF Calibration Form

Site:_________________ Dates: From_________________ Through_________________

Date	Time	Operator Name	Instrument Model/ Serial No.	Calibration Energy Reading	Comments

XRF Daily Sampling Log

Analysis Date: _________　　　　**Site Name: ___________________________**
Page ____ of ____　　　　**Analysist: __________________________**

Location ID		**Sampling Data**		**Prep**			**XRF Data**		**Conc (ppm)**					**Split to Lab**	**Other Comments**
Location	**Depth**	**Date**	**Time**	**Bag**	**Lab**	**Frag?**	**Read No.**	**Count (sec)**	**Pb**	**Std**	**As**	**Std**	**Other Metals**		