Lead Screening Study of Fish in Gills Creek Watershed

SC DEPARTMENT *of*
ENVIRONMENTAL SERVICES

Bureau of Water – Aquatic Science Division
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Publication and Contact Information

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On July 1, 2024, the South Carolina Department of Health and Environmental Control dissolved into two separate agencies, the South Carolina Department of Environmental Services and the South Carolina Department of Public Health.

Lead Screening Study of Fish in Gills Creek Watershed

by

Taylor Shearer

Bureau of Water – Aquatic Science Division
South Carolina Department of Environmental Services
Columbia, SC 29201

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Executive Summary

Lead is ubiquitous in our environment as this element is found in water, rocks, soils, plants, air and animals. It is a naturally occurring toxic metal, whose use by humans has led to widespread environmental contamination. These sources of contamination range from mining, smelting, metal processing, waste incineration, and ammunition manufacturing (Eisler, 1988; Lee & et al., 2019; South Carolina Department of Health and Environmental Control (SCDHEC), 2011; South Carolina Department of Health and Environmental Control (SCDHEC), 2024). In 2021 and 2022, the South Carolina Department of Health and Environmental Control (SCDHEC, now South Carolina Department of Environmental Services) conducted a Gills Creek Lead Study and found that a regularly monitored base station, C-078, which is located below a munitions range on Fort Jackson military base, consistently violated the chronic total lead standard for aquatic life (SCDHEC, 2024). These results prompted a special screening study to identify if there were elevated concentrations of lead in either fish fillets or whole fish from sites located downstream of C-078.

Five (5) sites were chosen within Gills Creek watershed and sampled by South Carolina Department of Environmental Services (SCDES) staff. At each site, 4-8 fish were collected for lead analysis in both fillet and whole tissue components. In total, 56 samples were analyzed for total lead by Access Analytical (now Eurofins) using EPA method 6010D. All sample results were below Reporting Limit (RL). One whole fish sample was reported as above the Method Detection Limit (MDL) but below the RL. Results below the RL are considered estimated values.

Introduction and Background

Surface water concentrations of lead exceeding South Carolina state water quality standards for aquatic life have been observed in the Gills Creek watershed in Richland County, South Carolina. Gills Creek headwaters begin north of Sesquicentennial State Park and eventually flows into the Congaree River upstream of Congaree National Park. This watershed is comprised of over 115 miles of streams and covers more than 47,000 acres (SCDHEC, 2011). In 2021 and 2022, the South Carolina Department of Health and Environmental Control (SCDHEC, now South Carolina Department of Environmental Services or SCDES) conducted a Gills Creek Lead Study, which resulted in the development of a lead TMDL for the watershed (SCDHEC, 2024). A regularly monitored ambient water quality base station (C-078), located below a munitions range on Fort Jackson military base prior to entering Rockyford Lake in the northeastern area of Gills Creek watershed, consistently violated the chronic total lead water quality standard for aquatic life. Specifically, 22 of the 24 water samples collected in the Gills Creek Lead Study were found to exceed these lead criteria for aquatic life use at C-078 (SCDHEC, 2024).

Elevated concentrations of lead in the aquatic environment can impair normal physiological functions and bioaccumulate in aquatic organisms. Therefore, the consumption of fish can directly impact and accumulate in humans (Ju-Wook Lee, 2019). In response, SCDES conducted a special screening study (Appendix 1) to evaluate concentrations of lead in fish fillets and whole fish at five (5) sites located downstream of C-078 (Table 1, Figure 1).

Samples were processed following SCDHEC Standard Operating Procedure (SOP) for Fish and Shellfish Tissue Collection (Appendix 2). This process includes measuring for total length and weight, scaling the right side of each fish, and removing the scaled fillet as a separate sample from the whole body. Each fish produced two samples: fillet sample and whole body sample. Samples were then prepared by Access Analytical (now Eurofins) following their SOP for Fish Tissue Preparation (Appendix 3). Samples were prepared for analysis using acid digestion method 3050B and analyzed for total lead following EPA method 6010D (Appendix 4).

Table 1: Fish site descriptions in Gills Creek watershed sampled for lead.

Waterbody Name	Site Name	Description	Latitude/Longitude	Sampling Method
Forest Lake	C-068	Dammed lake; receiving waters from Spring Lake & Rockyford Lake outfalls	34.02731, -80.95600	Electrofishing Boat
Lake Katherine	S-1013	Dammed lake; receiving waters from Eightmile Branch, Forest Lake & Pen Branch	34.00769, -80.96122	Electrofishing Boat
Gills Creek	C-017	Flowing creek; Gills Creek @ SC 48 (Bluff Road)	33.94814, -80.98909	Hook & line
Upper Rockyford Lake	S-1047	Dammed lake; receiving water from Boyden Arbor Pond outfall	34.03987, -80.94758	Hook & Line
Lower Rockyford Lake	S-1046	Dammed lake; receiving water from Upper Rockyford Lake outfall	34.03803, -80.95284	Hook & Line

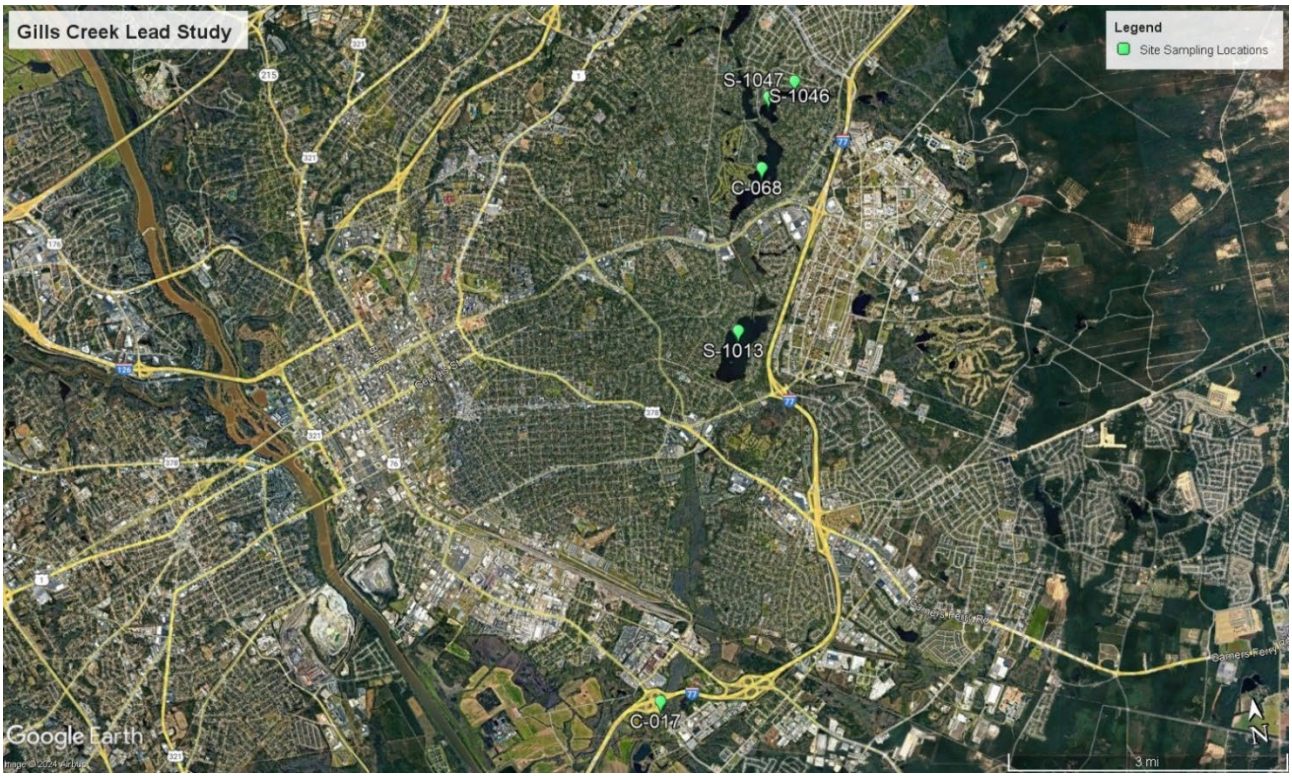

Figure 1: Sampling site locations.

Results

SCDES staff collected 4-8 fish from each waterbody using either the electrofishing boat or hook & line, totaling 56 individual samples (Table 2 & Table 3). The results for all samples were below laboratory reporting limits (Appendix 5).

Table 2: Whole fish (with right side fillet removed) results for lead by site. Total length and weight of each sample measured before fillet removal.

Gills Creek Watershed Lead in Fish Study: Whole Fish							
Date	**Site ID**	**Sample ID**	**Species**[a]	**Length (mm)**	**Weight (g)**	**Reporting Limit (mg/Kg)**	**Result (mg/Kg)**[b]
05/13/2024	C-068	FW-01	LMB	361	603	0.682	ND
05/13/2024	C-068	FW-02	LMB	342	593	0.824	ND
05/13/2024	C-068	FW-03	BKS	281	284	0.815	ND
05/13/2024	C-068	FW-04	BKS	173	68	0.706	ND
05/13/2024	C-068	FW-05	BGS	153	60	0.542	ND
05/13/2024	C-068	FW-06	BGS	152	58	0.795	ND
05/13/2024	C-068	FW-07	RES	177	99	0.742	ND
05/13/2024	S-1013	FW-08	LMB	383	768	0.598	ND
05/13/2024	S-1013	FW-09	LMB	314	404	0.634	ND
05/13/2024	S-1013	FW-10	W	187	149	0.667	ND
05/13/2024	S-1013	FW-11	W	173	116	0.804	ND
05/13/2024	S-1013	FW-12	W	171	109	0.695	ND
05/13/2024	S-1013	FW-13	W	154	82	0.690	ND
05/13/2024	S-1013	FW-14	BGS	149	61	0.644	ND
05/15/2024	C-017	FW-15	LMB	403	947	0.662	ND
05/15/2024	C-017	FW-16	LMB	244	167	0.664	ND
05/15/2024	C-017	FW-17	RES	177	106	0.822	ND
05/15/2024	C-017	FW-18	RES	158	68	0.780	ND
05/15/2024	C-017	FW-19	BGS	157	83	0.654	ND
05/15/2024	C-017	FW-20	W	153	80	0.634	ND
10/31/2024	S-1047	FW-21	LMB	260	207	1.200	ND*
11/05/2024	S-1047	FW-22	LMB	299	375	1.400	ND
11/05/2024	S-1047	FW-23	RES	243	258	1.500	ND
11/05/2024	S-1047	FW-24	RES	197	128	1.300	ND
11/05/2024	S-1046	FW-25	LMB	350	580	1.200	ND
11/05/2024	S-1046	FW-26	LMB	299	358	1.400	ND
11/05/2024	S-1046	FW-27	BGS	212	169	1.200	ND
11/05/2024	S-1046	FW-28	RES	230	264	1.300	ND

a. LMB= Largemouth Bass; BKS= Black Crappie; BGS= Bluegill Sunfish; RES= Redear Sunfish; W= Warmouth
b. ND = Result below laboratory Reporting Limit (RL)
*. Sample result below laboratory Reporting Limit but above Method Detection Limit (MDL). Results below RLs are estimated values.

Table 3: Fillet results for lead by site. Fillets removed from the right side of the fish after total length and weight was recorded.

Gills Creek Watershed Lead in Fish Study: Fillet					
Date	**Site ID**	**Sample ID**	**Species** [a]	**Reporting Limit (mg/Kg)**	**Result (mg/Kg)** [b]
05/13/2024	C-068	FF-01	LMB	0.605	ND
05/13/2024	C-068	FF-02	LMB	0.586	ND
05/13/2024	C-068	FF-03	BKS	0.601	ND
05/13/2024	C-068	FF-04	BKS	0.559	ND
05/13/2024	C-068	FF-05	BGS	0.653	ND
05/13/2024	C-068	FF-06	BGS	0.588	ND
05/13/2024	C-068	FF-07	RES	0.659	ND
05/13/2024	S-1013	FF-08	LMB	0.577	ND
05/13/2024	S-1013	FF-09	LMB	0.602	ND
05/13/2024	S-1013	FF-10	W	0.745	ND
05/13/2024	S-1013	FF-11	W	0.680	ND
05/13/2024	S-1013	FF-12	W	0.624	ND
05/13/2024	S-1013	FF-13	W	0.629	ND
05/13/2024	S-1013	FF-14	BGS	0.619	ND
05/15/2024	C-017	FF-15	LMB	0.576	ND
05/15/2024	C-017	FF-16	LMB	0.565	ND
05/15/2024	C-017	FF-17	RES	0.602	ND
05/15/2024	C-017	FF-18	RES	0.540	ND
05/15/2024	C-017	FF-19	BGS	0.612	ND
05/15/2024	C-017	FF-20	W	0.581	ND
10/31/2024	S-1047	FF-21	LMB	1.200	ND
11/05/2024	S-1047	FF-22	LMB	1.300	ND
11/05/2024	S-1047	FF-23	RES	1.100	ND
11/05/2024	S-1047	FF-24	RES	1.100	ND
11/05/2024	S-1046	FF-25	LMB	1.400	ND
11/05/2024	S-1046	FF-26	LMB	1.200	ND
11/05/2024	S-1046	FF-27	BGS	1.100	ND
11/05/2024	S-1046	FF-28	RES	1.200	ND

a. LMB= Largemouth Bass; BKS= Black Crappie; BGS= Bluegill Sunfish; RES= Redear Sunfish; W= Warmouth

b. ND = Result below laboratory Reporting Limit (RL)

References

Eisler, R. 1988. Lead Hazards to Fish, Wildlife, and Invertebrates: A Synoptic Review. *U.S. Fish and Wildlife Services Biological Report 85 (1.14)*.

Lee, J. W., & et al. 2019. Toxic Effects of Lead Exposure on Bioaccumulation, Oxidative Stress, Neurotoxicity, and Immune Responses in Fish: A Review. *Environmental Toxicology ad Pharmacology*, 68: 101-108.

South Carolina Department of Health and Environmental Control (SCDHEC). 2011. Watershed Water Quality Assessment Saluda River Basin.

South Carolina Department of Health and Environmental Control (SCDHEC). 2024. Total Maximum Daily Load Document: Gills Creek.

Appendix 1: Lead Screening Study of Fish in Gills Creek Watershed Quality Assurance Project Plan and Addendum

South Carolina Department of Environmental Control
Bureau of Water
Aquatic Science Programs

Section A. Project Management

Revision 1.0

A.1 Title and Approval

Project: Lead Screening Study of Fish in Gills Creek Watershed Quality Assurance Project Plan

Date of Initiation: May 1, 2024

APPROVED BY:

QAM: 04-30-2024
Paul Miller, EA Date

Project Manager: 5/1/2024
Taylor Shearer, ASP BOW Date

BOW Management: 2024-05-01
Bryan Rabon, ASP Manager BOW Date

BOW Management: 4-30-2024
Rob Devlin, Assistance Bureau Chief BOW Date

Contract Laboratory: 05/01/2024
Ashley Amick, Access Analytical Date

A.2 Table of Contents

A.3 Distribution List

Name	Region/Office	Phone	Email
Paul Miller	EA – Columbia	(803) 898-4272	millerpm@dhec.sc.gov
Bryan Rabon	ASP – Columbia	(803) 896-4402	raboneb@dhec.sc.gov
Rob Devlin	BOW – Columbia	(803) 898-3798	devlinrj@dhec.sc.gov
Taylor Shearer	ASP – Columbia	(803) 898-1538	shearetv@dhec.sc.gov
Chad Altman	ASP – Columbia	(803) 898-4017	altmankc@dhec.sc.gov
Caitlin Smith	ASP – Columbia	(803) 898-4961	smithc5@dhec.sc.gov
Ashley Amick	Access Analytical	(803) 781-4243	aamick@axs-inc.com

A.4 Project/Task Organization

Taylor Shearer – is the Project Manager and is responsible for developing and maintaining the QAPP. She will also serve as field personnel and will assist in the collection, processing and delivery of samples to the laboratory.

Chad Altman- is the Field Manager and will assist with the collection, processing and delivery of samples to the laboratory.

Caitlin Smith – is the Data Validator for this project.

Paul Miller – will approve and review the QAPP.

Ashley Amick- is the owner of Access Analytical and liaison for the study.

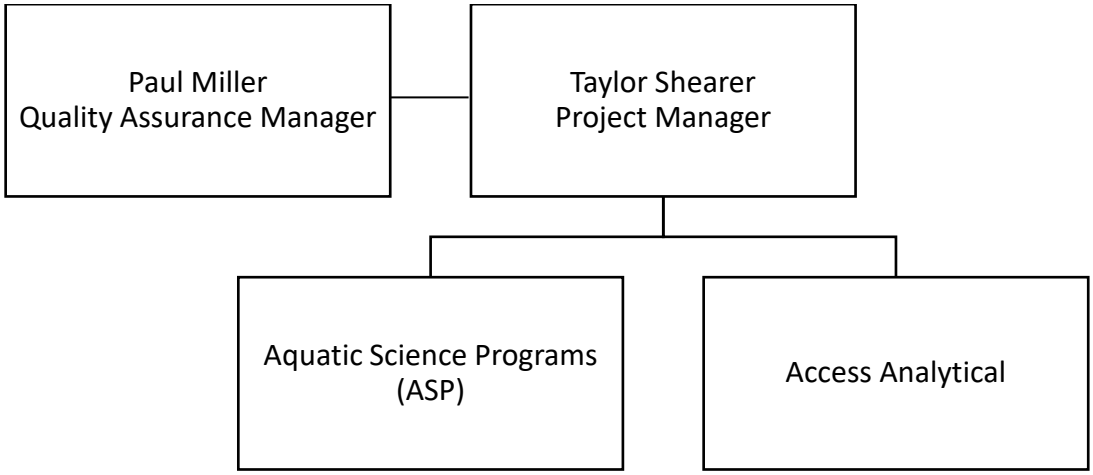

Figure 1: Organization chart.

A.5 Problem Definition/Background

Lead is ubiquitous in our environment as this element is a component found in water, rocks, soils, plants, air and animals. It is a naturally occurring toxic metal, whose use by humans has led to widespread environmental contamination. These sources of contamination range from mining, smelting, metal processing, and waste incineration to ammunition manufacturing and waste (Eisler, 1988).

Elevated concentrations of lead in the aquatic environment can impair normal functions and bioaccumulate in aquatic organisms. Therefore, the consumption of fish can directly impact and accumulate in humans (Ju-Wook Lee, 2019).

Concentrations of lead exceeding SC state water quality standards for aquatic life have been observed in the Gills Creek watershed, which is in Richland County. Gills Creek watershed headwaters begin north of Sesquicentennial State Park and eventually flows into the Congaree River upstream of Congaree National Park. This watershed is comprised of over 115 miles of streams and covers more than 47,000 acres of land (SC DHEC, 2011). In 2021 and 2022, the South Carolina Department of Health and Environmental Control (SC DHEC) conducted a Gills Creek Lead Study, which resulted in the development of a lead TMDL within the watershed (SC DHEC, 2024). A regularly monitored base station, C-078, which is located below a munitions range on Fort Jackson Military reserve prior to entering Rockyford Lake in the northeastern area of Gills Creek watershed, consistently violated the chronic total lead standard for aquatic life. Twenty-two (22) of the 24 water samples collected in the Gills Creek Lead Study were found to exceed these criteria for lead for aquatic life use at that location (SC DHEC, 2024). Consequently, SC DHEC will conduct a special screening study to identify if there are elevated concentrations of lead in either fish filets or whole fish from sites located downstream of C-078.

A.6 Project/Task Description

The purpose of this screening project is to determine if fish from Gills Creek watershed contain elevated concentrations of lead. Three (3) sites will be sampled within the watershed (Table 1, Figure 2). There will be a minimum of one (1) round of sampling at each site; however, repeated sampling events will be conducted if necessary. Approximately 5-7 fish will be collected by SC DHEC staff from each site for a total of 20 fish. Collection will be performed via boat electroshocking and dip netting, backpack electroshocking, and/or hook and line following the SC DHEC Standard Operating Procedure (SOP) for Fish and Shellfish Tissue Collection (Appendix 1). SC DHEC Bureau of Water Aquatic Science Programs staff will be targeting five (5) sunfish, ideally Readear sunfish or Bluegill, and one to two (1-2) Largemouth bass from each site. The target for size of fish are those large enough to be kept by fishermen; however, the fish must at a minimum be large enough to perform tissue analysis. Fish will then be transported on ice back to the SC DHEC Aquatic Biology Lab, where fish samples will be weighed, measured for total length, scaled on the right side, and the scaled filet removed. The separated filet will be placed in a Ziplock bag and placed in the freezer at or below -20 C. The remaining fish will also be placed in a Ziplock bag and placed in the freezer at or below -20 C. Each fish produces two separate samples. These samples will then be transported on ice to Access Analytical within the holding time. Samples will be prepared by Access Analytical by following their SOP for Fish Tissue Preparation (Appendix 2). Samples will then be prepared by acid digestion via method 3050B and analyzed for lead following EPA method 6010D (Appendix 3). Fish samples will only be collected for lead analysis.

Table 1: Fish sites in Gills Creek watershed sampled for lead.

Waterbody Name	Site Name	Description	Latitude/Longitude	Sampling Method
Forest Lake	C-068	Dammed lake; receiving waters from Spring Lake & Rockyford Lake outfalls	34.02731/ -80.95600	Electrofishing Boat
Lake Katherine	S-1013	Dammed lake; receiving waters from Eightmile Branch, Forest Lake & Pen Branch	34.00294/ -80.96475	Electrofishing Boat
Gills Creek	C-017	Flowing creek; Gills Creek @ SC 48 (Bluff Road)	33.94814/ -80.98909	Hook & Line/ Electrofishing Backpacks

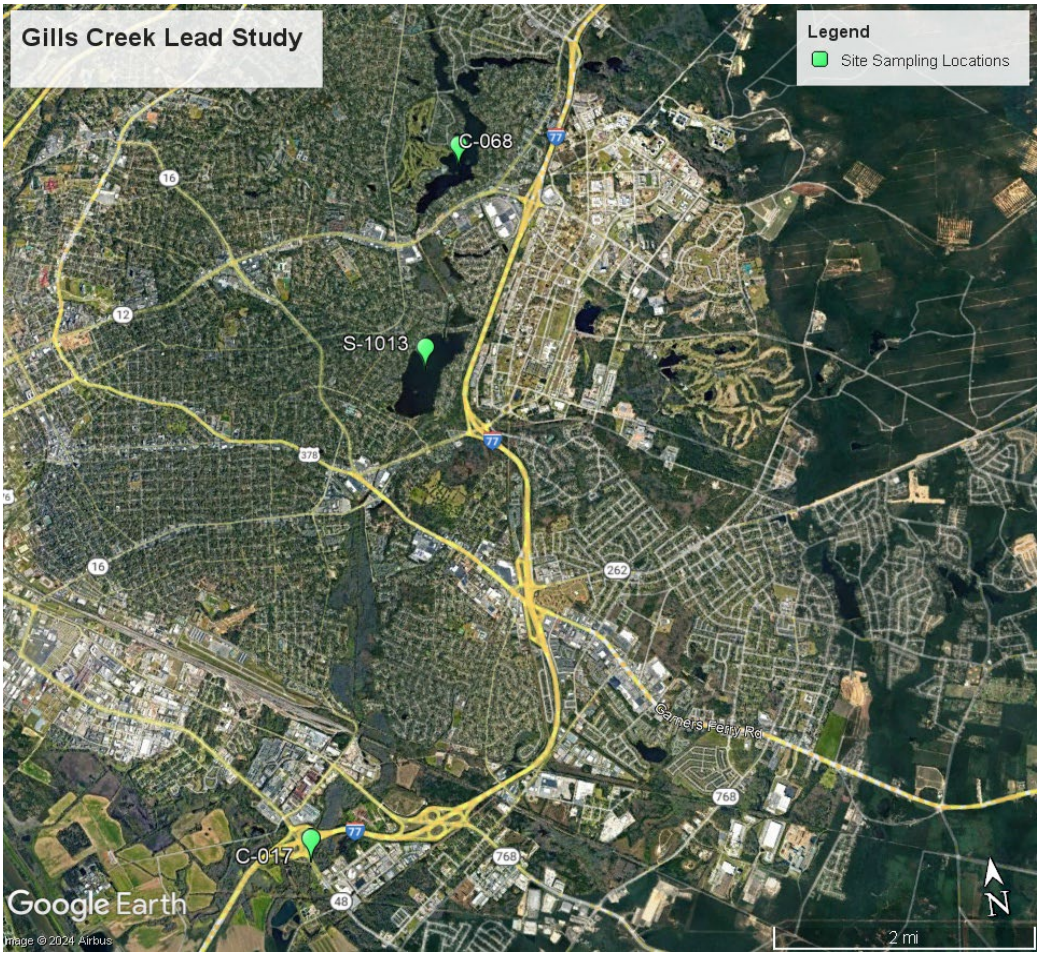

Figure 2: Sampling site locations.

The following table gives project activities and their anticipated date of initiation and completion.

Table 2: Estimated dates of initiation and completion.

Activity	Name/Organization	Anticipated date of Initiation	Anticipated date of Completion
QAPP Approval	Taylor Shearer	03-25-2024	05-01-2024
Sampling	BOW- ASP	05/06/2024	05-31-2024
Data Verification	Access Analytical	As Samples are submitted	Within one month of final sample
Data Validation	BOW- ASP	As soon as verification is complete	08/30/2024

The dates shown in Table 2 above are estimates only. No sampling will be conducted outside of standard workdays. Only ice is needed for the samples, and the holding time is 180 days. Fish samples will be analyzed for lead by Access Analytical (refer to Access Analytical SOP in Appendix 3). Transportation of fish samples will be coordinated between BOW ASP staff and Access Analytical.

A.7 Data Quality Objectives (DQOs) and Data Quality Indicators (DQIs)

The data collected during this screening study will be used to determine if there are elevated concentrations of lead in either fish filets or whole fish from Gills Creek Watershed. All sites were selected by BOW ASP due to access by boat or access by walking with backpack electroshocking and/or hook and line fishing. Data quality indicators provide a measure of bias, accuracy, comparability, representativeness and completeness. Data bias and accuracy are managed through the implementation of SOPs for field sampling and laboratory analysis. Bias and accuracy are managed through verification and validation processes to ensure all project QA and QC requirements are met. Representativeness is ensured by collecting samples consistent with SC DHEC SOPs for Fish and Shellfish Tissue Collection. A high level of completeness is achieved by preventing sample loss through verification and validation of all data at the project's conclusion. Any samples that are missed or invalid will be omitted from the data set. The Department will determine if a sampling event should be repeated due to missing lab analytical data. Refer to Access Analytical's SOP (Appendix 3) for analytical protocol. The data meeting the quality criterion in Access Analytical's SOP is deemed acceptable. Laboratory analytical methods for lead are performed by inductively coupled plasma mass spectrometry (EPA method 6010D), and the laboratory reporting limit is 1 mg/kg. SC DHEC BOW ASP staff estimates up to 40 total samples for the study if there are no sample issues.

Sampling Protocols and Standard Operating Procedures

Standard operating procedures (SOPs) for fish sample collection, holding times, and chain of custody are detailed in sections within the Fish and Shellfish Tissue Collection SOP and Access Analytical SOPs.

A.8 Training

Current sampling and laboratory methods are already established and in practice per SOPs.

A.9 Documentation and Records

The fully executed QAPP and any subsequent revisions will be sent to the Distribution List in A.3 electronically by the project manager, Taylor Shearer.

The Field Sample Logbook is completed and maintained by BOW ASP personnel and is reviewed by the Project Manager for completeness. These logbooks are maintained indefinitely.

The Analysis Data and Analytical Support from Access Analytical will be delivered electronically in excel-compatible, executable file.

Section B. Measurement/Data Acquisition

B.1-B.7 Sampling Process Design and Requirements

The sampling locations were chosen by SC DHEC to adequately determine if there are elevated concentrations of lead in fish collected from the Gills Creek Watershed. Since it was determined that site C-078 has been impaired for lead, sites were chosen at various intervals downstream of C-078 due to access with either a boat or walk-in backpack electrofishing/hook & line fishing. Sample collection, sample handling, and filling out chain of custodies will be conducted by BOW ASP staff following all protocols given in the Fish and Shellfish Tissue Collection SOP.

Sample analysis and process are covered in the SCDHEC SOPs and the Access Analytical SOPs. Refer to A.6 for sample design.

The Department will determine if a sampling event should be repeated due to missing lab analytical data. The other results from a sampling event that is repeated will be kept for informational purposes.

B.8 Inspection/Acceptance for Supplies and Consumables

The Project Manager is responsible for the consumables needed in the field and the BOW ASP laboratory, as well as ensuring the collection, handling, and delivery of grab samples in accordance with the SC DHEC Fish and Shellfish Tissue Collection SOP is already addressed. Review of the field logbooks is also the responsibility of the Project Manager.

Consumables required by the analytical laboratory are the responsibility of that laboratory based on the analytic procedure used and must be appropriate state certified reference standards and American Chemical Society (ACS) certified reagent grade chemicals. Acceptance for supplies and consumables will follow the current Access Analytical SOP.

B.9 Non-direct Measurements

None.

B.10 Data Management

Analytical results (raw and QA/QC) produced by Access Analytical will be forwarded to the Project Manager via email. Data produced by the BOW ASP laboratory will be documented in a fish collection

logbook. The Project Manager is responsible for collating results from all sources into one spreadsheet that is maintained indefinitely on SC DHEC internal server which is backed up daily.

Section C. Assessments and Oversight

C.1 Assessment and Response Actions

Access Analytical Laboratory is evaluated and certified by SC DHEC laboratory certification program. The laboratory is evaluated every three years. The laboratory also participates in WP Proficiency Testing. These results are sent to the Laboratory Director and SC DHEC's laboratory certification program. Contract laboratories are responsible for any corrective actions for internal assessments.

C.2 Reports to Management

Analytical results (raw and QA/QC) produced by Access Analytical will be forwarded to the Project Manager via email. Data produced by the BOW ASP laboratory will be documented in a fish collection logbook. The Project Manager is responsible for collating results from all sources into one spreadsheet that is maintained indefinitely on SC DHEC internal server which is backed up daily.

Section D. Data Validation and Usability

D.1 Data Review, Verification, and Validation

All sample COC forms and field logbooks will be reviewed by the Project Manager after each sampling event. Any errors/discrepancies noted will be brought to the attention of the Field Manager for resolution. The Project Manager will be responsible for implementing any corrective actions needed. Items that will be reviewed are:

Sample Collection

- Records reviewed: Collection logs and COC forms
- Responsible party: Field Manager and Project Manager
- Discrepancies reported to: Project Manager

Sample Receipt

- Records reviewed: COC forms from Field Manager
- Responsible party: Laboratory Sample Custodian
- Discrepancies reported to: Project Manager

Sample Analysis

- Records reviewed: Email communication
- Responsible party: Laboratory Director
- Discrepancies reported to: Project Manager

Error/Corrective Actions

- Records reviewed: Report from laboratory

- Responsible party: Project Manager/Data validator
- Discrepancies reported to: Project Manager

Laboratory Data Verification

Data verification performed by Access Analytical is detailed in their laboratory SOPs (Appendix 3). Verification by Caitlin Smith will consist of a completeness check. This check will ensure that all sample data was received. Verification will also include a review of field data to make sure documentation was complete. Any problems will be noted and forwarded to the Project Manager for review and validation.

Criteria for Accepting, Rejecting, or Qualifying Project Data

Item	Criteria[a]	If the criteria are not met are samples flagged or rejected
Holding Times	Samples must be analyzed within holding time	Rejected
Temperature	Samples must be frozen and received on ice.	Rejected

a: Access Analytical SOP (Appendix 3)

D.2 Verification and Validation Methods

Verification:

Verification is done by the laboratories per their respective SOPs. Verification by Caitlin Smith will consist only of a completeness check. This check will ensure that all sample data was received. Any problems will be noted in an email to Taylor Shearer who will validate the data.

Validation:

The Project Manager will note the problems seen by the verifiers. They will then examine the data and ensure that sample results match what was expected at the site and compare the data against historical data, where available, and determine if the data agrees with the project data. After these assessments, the Validator researches the data and/or documentation that are inconsistent. This is done by contacting Lab and Field Personnel to correct and/or explain inconsistencies. After the Validation steps have been completed, the Validator will submit the data to BOW ASP.

D.3 Reconciliation with User Requirements

Any issues with the data found during the verification or validation will be transmitted to data users. This includes the process for reconciling project results with DQOs and reporting limits of data use.

References

Eisler, R. 1988. Lead Hazards to Fish, Wildlife, and Invertebrates: A Synoptic Review. U.S. Fish and Wildlife Services Biological Report 85 (1.14)

Lee, J. W., et al. 2019. Toxic Effects of Lead Exposure on Bioaccumulation, Oxidative Stress, Neurotoxicity, and Immune Responses in Fish: A Review. Environmental Toxicology and Pharmacology, 68, 101-108.

South Carolina Department of Health and Environmental Control (SC DHEC), 2011. Watershed Water Quality Assessment Saluda River Basin.

South Carolina Department of Health and Environmental Control (SC DHEC), 2024. Total Maximum Daily Load Document: Gills Creek.

South Carolina Department of Environmental Services
Bureau of Water
Aquatic Resource Monitoring

Section A. Project Management

Revision 1.1

A.1 Title and Approval

Project: Lead Screening Study of Fish in Gills Creek Watershed Quality Assurance Project Plan Addendum

Date of Revision: October 2, 2024

APPROVED BY:

QAM:

10-15-2024
Paul Miller, EA — Date

BOW Management:

2024-10-15
Bryan Rabon, ARM Manager BOW — Date

Project Manager:

10/15/24
Taylor Shearer, ARM BOW — Date

Contract Laboratory:

10.21.24
Ashley Amick, Access Analytical — Date

Revision Number	Date	Revisions
1.1	October 2, 2024	Determined two additional sites needed to be sampled upstream of original sites.

Table 1: Distribution list.

Name	Region/Office	Phone	Email
Paul Miller	EA – Columbia	(803) 898-4272	Paul.miller@des.sc.gov
Bryan Rabon	ARM – Columbia	(803) 896-4402	Bryan.rabon@des.sc.gov
Rob Devlin	BOW – Columbia	(803) 898-3798	Rob.devlin@des.sc.gov
Taylor Shearer	ARM – Columbia	(803) 898-1538	Taylor.shearer@des.sc.gov
Chad Altman	ARM – Columbia	(803) 898-4017	Chad.altman@des.sc.gov
Caitlin Smith	ARM – Columbia	(803) 898-4961	Caitlin.smith@des.sc.gov
Ashley Amick	Access Analytical	(803) 781-4243	aamick@axs-inc.com

Background

After initiation of the QAPP, it was requested to have the South Carolina Department of Environmental Services (SCDES) sample fish from two (2) additional sites within Gills Creek Watershed for lead analysis. These sites are located in Rockyford Lake, directly upstream from the project's initial testing (Table 2 and Figure 2).

Table 2: Fish sites in Gills Creek watershed sampled for lead.

Waterbody Name	Site Name	Description	Latitude/Longitude	Sampling Method
Upper Rockyford Lake	S-1047	Dammed lake; receiving water from Boyden Arbor Pond outfall	34.039872, -80.947584	Electrofishing boat / Hook & Line
Lower Rockyford Lake	S-1046	Dammed lake; receiving water from Upper Rockyford Lake outfall	34.038034 -80.952839	Electrofishing boat / Hook & Line

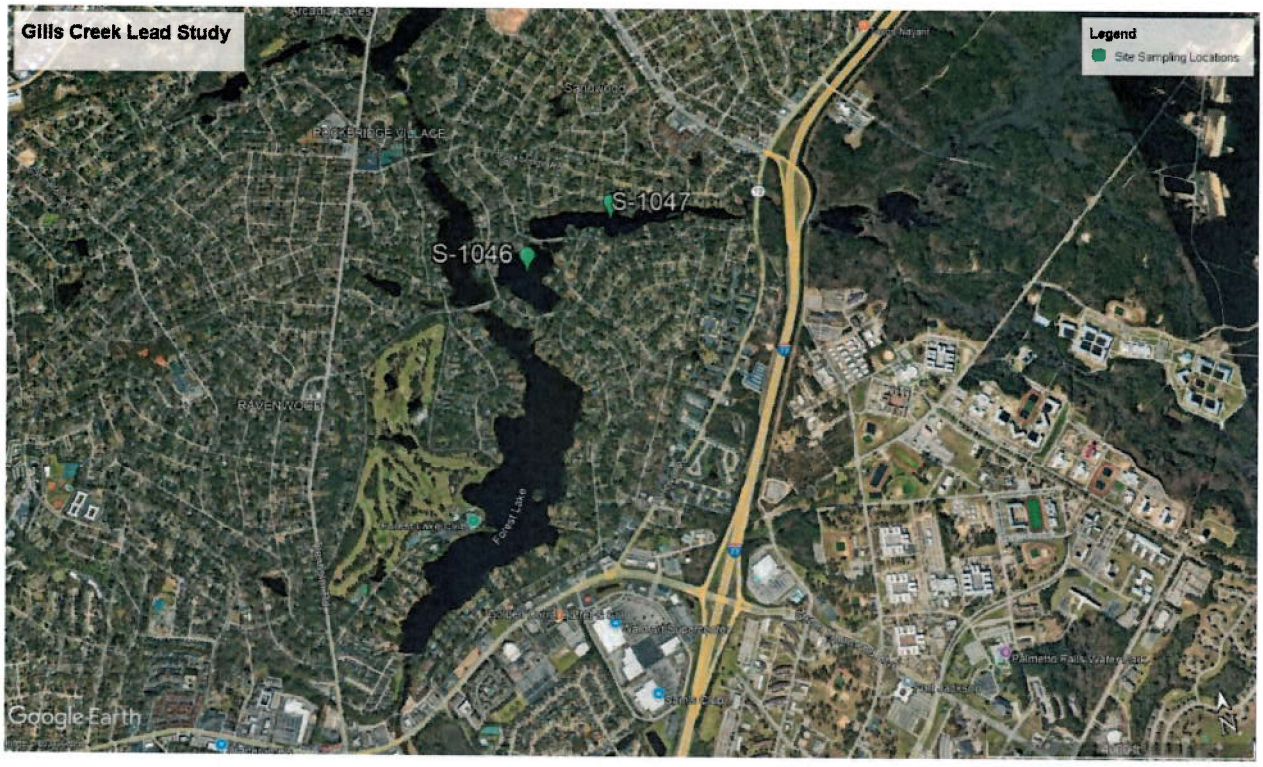

Figure 2: Sampling site locations.

Analysis Performed

Aquatic Resource Monitoring (ARM) staff will collect up to five (5) fish from each site for a total not to exceed ten (10) fish. Collection will be performed via boat electroshocking and dip netting, backpack electroshocking, and/or hook and line following the SC DHEC Standard Operating Procedure (SOP) for Fish and Shellfish Tissue Collection. ARM staff will be targeting five (5) sunfish, ideally Readear sunfish or Bluegill, and one to two (1-2) Largemouth bass from each site. The target fish size are those large enough to be kept by fishermen; however, the fish must at a minimum be large enough to perform tissue analysis. After collection, samples will be transported on ice back to the SCDES Aquatic Resource Monitoring Lab, where fish samples will be weighed, measured for total length, and scaled on the right side. Each fish produces two separate samples; a scaled fillet and the remaining whole body. These samples will be sent to Access Analytical for analysis. Samples will be prepared utilizing acid digestion via method 3050B and analyzed for lead following EPA method 6010D. These protocols are outlined in Access Analytical's SOP for Fish Tissue Analysis and Determination of Metals in Water, Soils and Wastes by ICP-OES by EPA SW-846 Method 6010D and Prep Methods 3010A/3050B. Collection at the two (2) sites is scheduled to begin in October.

Data Reporting

Data will be retrieved electronically from Access Analytical and stored with the other project's data. Data completeness will follow the overall project's goals.

Appendix 2: SCDHEC Fish and Shellfish Tissue Collection Standard Operating Procedure

Table of Contents

1.1 Introduction

The collection of fish and shellfish for the purpose of tissue analysis is necessary to detect the presence and levels of heavy metals, pesticides and toxic organic compounds in edible tissue which may concentrate through aquatic food chains and threaten the health of human consumers. Aquatic organisms may accumulate contaminants through gills and epithelial tissue directly from water and sediment (bioconcentration), a combination of bioconcentration and dietary sources (bioaccumulation), or a process by which the tissue concentrations increase as the contamination is passed up the food chain (biomagnification). Data collected is used to issue consumption advisories for the protection of public health when necessary and to access adverse biological effects caused by environmental contaminants. A collecting permit is required from South Carolina Department of Natural Resources to collect fish for scientific research.

1.1.1 Species Selection

In most cases a piscivorous species will be targeted for collection. In most fresh waters of the state the targeted species for collection is the largemouth bass, *Micropterus salmoides*. Five largemouth bass with a minimum weight of one pound each should be collected from each waterbody that is capable of being sampled with an electrofishing boat. A minimum weight of one pound is not always possible, especially in some of the smaller rivers and ponds. Bowfin, *Amia calva*, are available in most low country waterbodies (lakes, rivers, and swamps) and a few piedmont water bodies. When available five bowfin at least one pound each should be collected and analyzed for mercury only. In waterbodies where the targeted species are not available in sufficient numbers or sizes, substitutions are made based on the field crews judgement. All fish collected must be of legal size according to South Carolina Department of Natural Resources Rules and Regulations. Substitutions for targeted species may include:

Chain pickerel (*Esox niger*)
Blue catfish (*Ictalurus furcatus*)
Channel catfish (*Ictalurus punctatus*)
Flathead catfish (*Plyodictis olivaris*)
Smallmouth bass (*Micropterus dolomieui*)
Spotted bass (*Micropterus punctutus*)
Rainbow trout (Oncorhychus mykiss)
Brown trout (*Salmo truta*)

Incidentals are any nontarget species readily taken for human consumption of edible size and may be collected while sampling for target species. No more then five fish of each species should be collected from each site. Incidentals may include, but are not limited to the following species:

White catfish (*Ictalurus catus*)
White bass (*Morone crysops*)
Redbreast sunfish (*Lepomis auritis*)
Warmouth (*Lepomis gulosus*)
Bluegill sunfish (*Lepomis macrochirus*)
Redear sunfish (*Lepomis microlophus*)

White crappie (*Pomoxis annularis*)
Black crappie (*Pomoxis nigromaculatus*)
Yellow perch (*Perca flavecens*)

In estuaries, oysters, *Crassostrea virginica,* and blue crabs, *Callinictes sapidus,* are collected for tissue analysis. The South Carolina Department of Natural Resources provides fish from estuarine environments for tissue analysis. Targeted species collected from estuarine environments are red drum, *Sciaenops ocellatus,* spotted seatrout, *Cynoscion nebulosus,* and southern flounder, *Paralichthys lethostigma*. Incidental species may be collected from marine environments and include striped mullet, *Mugil cephalus,* and spot, *Leiostomus xanthurus*. No more than five of each species will be collected from each site per year. All fish collected must fall within size limits set by the South Carolina Department of Natural Resources.

Occasionally the South Carolina Department of Natural Resources will provide edible portions of alligator meat collected during their nuisance alligator trappings.

1.1.2 Fish Collection Equipment

Smith Root 16S Electrofishing Boat
Duracraft 16' Electrofishing Boat
Duracraft 14' Electrofishing Boat
Backpack Electrofisher
Gillnets
Jugs, Trotlines, Limblines

1.1.3 Electrofishing Introduction

A current is passed between submerged electrodes. The conductivity of the water and the conductivity of the fish's flesh affect electrofishing the most. The quantity of dissolved salts and minerals determine the conductivity of the water. Low conductivity water (0.5 to 5.0 microSiemens/cc) requires high voltage , up to 12,000 volts, to pass a current thru fish. High conductivity water (greater then 2,000 microSiemens/cc) requires low voltages and high currents, up to 60 amps. Brackish water and industrial waste water may have conductivities as high as 10,000 microSeimens/cc.

The current flowing through the water is directly proportional to the amount of voltage applied. The higher the voltage, the greater the current will be. There are two types of current available, alternating current (AC) and direct current (DC). Alternating current is an electrical current in which the direction of the electrical current reverses a number of times each second between the anode and the cathode. During electrofishing with alternating current the fish attempt to face the anode and the cathode successively. Alternating current results in strong contractions of the body muscles. At high voltages these muscle contractions may be so severe that vertebrae are fractured and brain damage occurs. Alternating current should not be used for the collection of fish for tissue analysis due to its ability to kill unwanted fish during collections.

Direct current is electrical current that flows in only one direction. The current passes from the negative electrode (cathode) to the positive electrode (anode). The reaction of the fish

is to turn and swim towards the anode until it reaches a field strong enough to stun it (galvanonarcosis). There are no severe muscle contractions and therefore less injury to the fish. Only direct current should be used for the collection of fish for tissue analysis.

1.1.4 Smith Root Boat Electrofishing Procedures

1. Allow the outboard motor to warm idle for 1- 2 minutes before leaving the landing.

2. Remove caps from end of booms. Have a crew member move the booms from the trailering position to the upper underway position and attach boom extensions, and folded umbrella arrays. Attach the folded arrays connecting the quick connector first and then the attached safety line. Connect the quick connector by sliding the female quick connect fitting from the array over the male quick connect fitting on the end of the boom extension. Release the sleeve and pull on the female end to determine the connection is secure.

3. Throttle up slowly and head for the work site.

4. Trim bow to suit boat load and water conditions.

5. Throttle down slowly when reaching the work site.

6. Unfold the umbrella arrays and adjust the boom extensions to the desired position.

7. With the electrofisher off, start the generator after the booms are extended and allow the generator to warm up for a few minutes. The generator must be on water to run. The generator has a "water cooled exhaust", and damage will occur if the generator is ran out of water.

8. Adjust the foot switch system to desired sequence. On the lower right-hand corner of the console control panel is a foot switch workdeck control switch. In the " both position" both workdeck foot switches and the boat operators foot switch must be engaged simultaneously to activate the electrofisher. In the "separate position" only one workdeck foot switch and the boat operators foot switch need to be engaged to activate the electrofisher. The boat operators foot-switch has a separate active/inactive switch in the patch panel on the front of the console. The boat operators foot switch can be disengaged by placing this switch in the active position. The electrofisher can perform with use of only one workdeck foot switch (either the port or starboard foot switch).

9. Smith Root Boat Electrofisher Controls:

* Mode selects the type of output pulses. Direct current pulse rates are selectable in pulses per second. Alternating current frequency is fixed at 60 pulses per second.
* Range selects the output voltage range, or switches the output off.
* Percent Of Range limits the peak voltage of the pulses to a percentage of whatever range is selected.
* High Voltage Indicator Lamp indicates when voltage is present.
* Enunciator Volume controls the audio alarm that indicates an output voltage.
* Output Current shows the current flowing between the anode and cathode in amps.
* Time In Seconds records the actual time high voltage is applied and can be reset to

zero by pushing the small red button on front panel.

* Emergency Shutdown provides an override of remote switches. The electrofisher can be shut down by pushing this large red switch down. Switch is located on top of front panel.

10. Set the electrofisher to the desired mode. Turn the direct current/alternating current switch from the off position to the direct current position. Turn the Mode selector to 120 pulses per second DC.

11. Set the Percent Of Range to the minimum.

12. Set the Range selector switch to low.

13. Set the Emergency Shutdown switch to on.

14. Set the Enunciator Volume to a range crew members can hear.

15. With anode and cathode in water activate the electrofisher by stepping on the foot switch. The enunciator and high voltage indicator lamp should both come on. Look at the ammeter to determine amount of amps generated.

16. Deactivate the electrofisher by stepping off the foot switch and adjust the Percent Of Range and the Range selector switch to achieve optimum amperage. Generally 4 amps is an optimum range for our fish collections. Most often the Range is set at 1000 volts DC and the Percent Of Range is adjusted until 4 amps is reached. On some of the upstate reservoirs 4 amps is not possible and collections are made using as little as 1.5 amps due to the low conductivity of the water. Do not adjust the Range selector switch while the electrofisher is activated. Damage may occur.

17. If erratic operation occurs in the high range, switch to low range. Do not operate the generator above power ranges indicated on the meter.

18. Electrofish the site at likely fish habitat. Place fish in live well.

19. After completing fish collections allow the generator to run for a few minutes to allow it to cool down. Switch the Mode and Range controls to off. Switch the Percent Of Range and Enunciator Volume controls to the lowest possible settings.

20. Fold and disassemble the umbrella arrays, disconnect the boom extensions, and place caps on end of booms. Boat is ready to be loaded on trailer.

21. Place fish in a labeled cooler with ice. Include station location and date on label.

1.1.5 Duracraft Boat Electrofishing Procedures

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1. Generator and electrofisher should be placed in boat and connections made before launching boat. Connections are the same for the 14' and 16' Duracraft Boat.

2. Check generator engine oil level and replace with SAE 10-30 detergent oil classified for service SF, SE, SD, or SC. Do not overfill.

3. Refuel generator outdoors. Use gas with a minimum rating of 85 octane.

4. Place Smith Root Type VI-A Electrofisher in front of the generator.

5. Join the generator, anode booms, and electrofisher together by connecting the three pin male end black cable to the female three pin receiver on the left front of the electrofisher. Plug the male end of the adjoining cable into the 240V AC outlet on the front of the generator.

6. Join the netters foot switch, operators switch, and electrofisher together by connecting the four pin male end black cable to female four pin receiver on the front of the electrofisher.

7. Join the boat ground to the electrofisher by connecting the two pin male end black cable to the female two pin receiver on the right front of the electrofisher. Connect the opposite male end into the outlet located in front of the steering console. This outlet is connected to the boat hull to provide a ground for electrofishing.

8. Allow the outboard motor to warm idle for 1-2 minutes before leaving the landing.

9. After arriving at the electrofishing site deploy the anode booms in front of the boat by sliding them forward. Plug the boom ends into the outlet box connected to the electrofisher.

10. Turn the electrofisher Input Power Switch (located on the electrofisher console) to off.

11. Turn the generator Power Switch from “off” to “on”.

12. Start the generator by pulling the pull cord.

13. Adjust the Mode Selector Switch to 120 PPS DC.

14. Turn on the power switch (labeled Input Power). The red light located to the left of the power switch should come on.

15. Adjust the Pulse Switch Control to approximately 3.5 ms.

16. Place the Voltage Selector Switch to the lowest setting.

17. Insert the key into the key switch labeled Ready on the front panel and, turn it to the right (on position).

18. Lift the cover (bright red) on the Emergency Shutdown switch and move the switch to the right (on position)

19. Boat operator should activate the control switch by flipping the operators switch to the on position.

20. The netter can now stand on the foot control switch and activate the electrofisher. The High Voltage indicator lamp located to the left of the Voltage Selector should come on. The ammeter should deflect and the timer (labeled Seconds on the front panel) should start recording seconds.

21. Adjust the Pulse Width Control and Voltage Selector Switch as necessary to obtain the desired amperage to stun fish (usually approximately 4 amps). Never adjust the Voltage Selector or the Mode Selector under load. Turn the Key Switch off or depress the Emergency Shutdown Switch before making adjustments. Damage to switches may occur while switching under a load**.**

22. Adjust the Pulse Width to achieve approximately 4 amperes. Often 4 amperes is not possible and electrofishing is done with less amperes. The Output Mode and Voltage Selector may have to be adjusted downward if to many amperes are generated.). Generally the Voltage Selector Switch is set at 1061 VDC and the Output Mode at 120 PPS, and the Pulse Width is adjusted to obtain needed amperes.

23. Electrofish the site at likely fish habitat and place collected fish in a labeled cooler with ice. Label should include the station location and date.

24. After collections are completed turn the Pulse Width to the minimum setting, Voltage Selector to off, Output Mode to off, and Input Mode to off .

25. Allow the generator to run for a few minutes to allow it to cool off.

26. Retract anode booms. Boat is ready to be loaded on trailer.

1.1.6 Backpack Electrofishing Procedures

Backpack electrofishing is performed in wadable streams in pools and around snags, boulders, and other likely fish habitat. Waders must be worn at all times, and rubber gloves should be worn. Backpack electrofishing is performed with a Smith Root Model 12-B POW Electrofisher.

1. Make sure power switch is in the off position, and secure battery in battery box. Connect input power plug to the battery.

2. Connect the cathode (rat tail) to the electrofisher by connecting the four pin male end of the cathode to the four pin female connection on the electrofisher labeled “Cathode”.

3. Connect the anode (aluminum ring and fiberglass pole) to the electrofisher by connecting the four pin male end of the anode to the four pin female connection on the electrofisher labeled “Anode”.

4. Select voltage and frequency ranges. Set voltage ranges to 100V, and select mode settings of D and 4 when water conductivity is unknown.

5. Place power switch in the “on” position.

6. Place anode and cathode in water, and press pole switch to generate electricity. Audio tone and self test indicator should come on.

7. Observe reaction of fish. Voltage can be increased after releasing the pole switch. If electrofisher is not holding fish, increase pulse width or frequency. If fish are being stunned before reaching anode, decrease the voltage, pulse width or frequency. While the person wearing the electrofisher activates the electrofisher, other field crew can adjust the voltage, frequency, and pulse width until the needed voltage and amperes is obtained. Do not make adjustments with the pole switch pressed.

8. Electrofishing is performed by holding the anode pole button down and holding the anode ring in likely fish habitat.

9. The person dipping should stay in close proximity to the person wearing the backpack to assist with any problems that may arise.

10. After completing collections the fish are placed in a labeled cooler. Station name and date are on the label.

11. Turn the Power Switch to off, Frequency Switch, Pulse Width Switch, and Voltage Switch to minimum settings before removing the battery.

12. Recharge battery before next sampling event. Batteries should be recharged as soon as possible. Connect charger to battery, and connect the charger to the AC power supply, and switch on. Charging time will depend on size and depth of discharge of battery. A minimum of one hour is needed and possibly twelve hours may be needed to recharge a battery. Allow charger to complete its full cycle, indicated by green "Ready" LED. The charger will not overcharge the battery.

13. Surface of anode must be conductive to operate properly. It may become anodized and nonconductive during normal operation. To restore conductivity to anode clean with a Scotch-Brite pad until it shines. Wire brushes and cleaning solutions may also be used.

14. Model 12-B POW Electrofisher Controls and Features:

* Voltage Range Switch is located on bottom left side of electrofisher and has ten ranges. The range can be adjusted according to the conductivity of the water. Use 100 to 300 volt ranges for high conductivity waters (400 to 1600 microSiemens/cc), 400 to 700 volt ranges for medium conductivity waters (200 to 400 microSiemens/cc), and 800 to 1000 volt ranges for low conductivity waters (10 to 200 microSiemens/cc).
* Mode Switches are located on the middle of the left side of electrofisher and are able to produce 256 different waveforms. One switch is labeled A-P and the other 1-16.
* Output Voltage Indicator is an audio indicator that produces a tone warning field crew that voltage greater than 30 volts is being generated between the anode and cathode. The indicator beeps slowly when an input of 4 Amps is generated. The indicator beeps faster as the input increases.
* Timer is a six digit timer located on top of the left side of the electrofisher. The timer records actual shocking time in seconds and can be reset by placing a magnet over the word "reset" next to the timer.

* Input Power Connector is a quick-twist positive locking connector with index tabs for proper polarization of the connector halves.
* Input Power Switch is a 25A toggle circuit breaker switch that protects electrofisher from excessive input currents.
* Self Test Indicator is a SelfTest LED indicating that the control circuit wiring and pole switch are operating correctly under normal conditions. Problems exist with the battery or control circuit if the indicator doesn't come on when the pole switch is pressed.
* Batt/Gen is a LED that comes on only when the battery is discharged. It can be cleared by turning the electrofisher off and placing a charged battery in the unit.
* Average Current Overload is an LED indicator that turns on if the electrofisher draws to much current from the battery. Turn down the voltage range, select a narrower pulse width, select a lower frequency, or a combination of all three to correct the problem.
* Peak Current Overload is indicated by the Overload LED flashing, and SelfTest Led will also be on. Release pole switch and reduce voltage setting to correct the overload. Anode and cathode touching will also cause a peak current overload.
* A Tilt Switch will trip at approximately 15 degrees backward tilt, and 30 degrees sideways or forward tilt. Correct the problem by standing straight and releasing the pole switch.
* Operator Error is caused by changing the mode switches with output on or by having the pole switch pressed while the on/off circuit breaker is turned on. Release the pole switch to correct this problem.
* Over Temperature begins once the internal temperature of the unit reaches 182 degrees Fahrenheit (83 degrees Celsius). The unit will shutdown automatically. Allow the unit to cool for at least 15 minutes with the on/off circuit breaker turned off to correct the overheating problem.
* Startup Failure indicates an internal problem, and Smith-Root should be contacted.

1.1.7 Electrofishing Safety

1. Members of the electrofishing crew should be trained in cardiopulmonary resuscitation and artificial respiration.

2. Rubber gloves and boots should be worn.

3. Never touch an electrode while the circuit is energized.

4. Do not work on the system while the generator is running.

5. Do not enter the water while the system is running.
6. Never electrofish alone.

7. Inspect all equipment before each sampling event.

8. Use only nonconductive dip nets.

9. Wear personnel flotation devices.

10. Do not operate an electrofisher if you have had prior heart aliments.

11. Ground the generator to the boat hull.

12. Do not electrofish during rain or hazardous weather.

1.1.8 Gill-netting procedure

Gill-netting is used only when the target species is not readily available by electrofishing (e.g. striped bass). Gill-netting is usually performed in reservoirs. If gill-netting is to be performed in rivers, the net should be set parallel to the current.

1. The net is rigged with weights and floats before setting.

2. Place a weight (anchor) on the bottom of the net and a float with a section of rope on the top of the net.

3. Before setting the net, drop the anchor over the bow and back the boat as the net is played out. Remove tangles while keeping the net relatively taut.

4. When the end of the net is reached placed an anchor on the bottom of the net and a float on the top of the net and release the net while making sure it is relatively taut.

5. The nets are set near nightfall and collected at daylight the next morning.

6. Start retrieving the net at the downwind end of the net.

7. Remove anchor and float from downwind end.

8. Remove fish as they come out of the water and place on ice in a labeled cooler. The label will include the station location and date.

9. The net is stacked in a basket as it is retrieved.

10. Remove remaining anchor and float.

1.1.9 Jugs, Trotlines, and Limbline Procedure

Jugs, trotlines, and limblines are used for the collection of catfish when necessary. They are fished overnight and collected as soon as possible the next morning. Trotlines should be marked with clearly labeled floats. Cut bait (shad) is the preferred bait. The number of hooks, jugs, and limblines fished depends on the study requirements.

1.1.10 Sample Collection And Preservation

When the collection of fish or shellfish samples are complete, care should be taken to insure the freshness and integrity of each sample. Fish or shellfish samples collected from the

same site should be immediately placed in a cooler on wet ice for transport to the lab. Each cooler should be labeled with the station information including the site description, station number and date of collection (Ex. Congaree River @ Hwy 601, C-007, 8/11/98). When samples are left unattended, coolers should be placed inside the vehicle and locked to avoid theft and tampering.

.1.1.11 Fish Work-up Procedure

Fish should be worked-up as soon as possible after collection.

1. Record station number in log book.

2. Record date station was sampled in log book.

3. Record sample collectors in log book.

4. Record gear used for collection in log book.

5. Cover table used for working up fish with clean aluminum foil.

6. Place fish on table to be worked up. Only fish from one station can be on fish work-up table at a time.

7. Identify each fish to species and record in fish log book.

8. Measure total length of each fish to the nearest millimeter and record in log book.

9. Weigh each fish 800 grams or smaller to the nearest gram and record in log book. Weigh each fish over 800 grams to the nearest 10 grams and record in log book. Use platform scale (800g x 1g) or electronic scale for fish 800 grams or smaller. Use hanging scale (15kg x 20g) for fish greater then 800 grams.

10. Assign a collection number to each fish, and record collection number in log book.. The first two numbers of the collection number will be the year the fish was collected. The next three numbers of the collection number will be the order in which the fish are worked-up. The 200th fish worked-up in 1998 would be assigned a collection number of 98-200. After fish number 98-999, the 9 is dropped from the year and 1000 will be the last four digits. The 1200th fish worked-up in 1998 would be assigned a collection number of 8-1200.

11. The right side of each fish is scaled. Catfish and other scaleless fish are skinned on the right side.

12. Standard fillets are taken from the right side of each fish for contaminant analysis. Standard fillets are skin on and scales off with the belly flap included. When filleting, care must be taken to ensure fish entrails are not punctured and visible bones are removed. Fish are filleted on clean aluminum foil or on a plastic fillet board that has been cleaned and rinsed first with deionized water and then isopropyl alcohol. Using an electric fillet knife with stainless steel blades, fillet the right side of the fish. The electric knife blades are cleaned and rinsed first with

deionized water and then isopropyl alcohol when the species being filleted changes or the station changes. The fillet board is also cleaned and rinsed with deionized water and isopropyl alcohol whenever the species or station changes.

13. The sex of each fish is determined during filleting and recorded in the log book.

14. Fat deposits, visible bones, and viscera are removed from the fillet with a stainless steel knife and deionized water. This stainless steel knife is cleaned and rinsed first with deionized water and then isopropyl alcohol when the species or the station changes.

15. The fillets from each fish are weighed and the weights recorded in the log book. The stainless steel platform scale pan is cleaned and rinsed first with deionized water and then with isopropyl alcohol when the species or station changes. Fillets are weighed to the nearest gram with the platform scales.

16. After weighing, the fillets are wrapped in clean aluminum foil(dull side to fillet), labeled with the assigned lab number, and frozen until processed for the SCDHEC Columbia Lab.

1.1.12 Fish Processing Procedure

After freezing, the fillets are ground and homogenized for analysis at the Aquatic Biology Section Lab.

1. Assign a lab sample number to each fish. The lab number is a 10 digit number. The first six numbers of the lab sample number will be the date processing of those fish began. The last four numbers will be the order the sample was processed beginning with the number 1000. The 20th sample processed on a start date of March 3, 1998 would be assigned lab sample number 0303981019.

2. Remove tissue samples (fillets) from the freezer as needed to prevent thawing.

3. Place the frozen fillet on a clean chopping board and cut into approximately 10 mm cubes using a stainless steel knife and hammer. The chopping board, knife, and hammer are cleaned and rinsed first with deionized water and then isopropyl alcohol after each fillet.

4. Place approximately 200 cc of dry ice in a clean stainless steel blender canister, then fill the canister approximately ½ with fish tissue. A new **clean** (see section **9.5.13)** canister is used for each fish.

5. The tissue and dry ice are ground into a fine powder.

6. The ground tissue is placed on clean aluminum foil.

7. If all of the fillet cubes cannot be ground at the same time , the ground tissue is placed in a clean stainless steel bowl and mixed throughly after the entire fillet is ground. The stainless steel bowl is cleaned following procedures outlined in section **9.5.13.** first with tap water, then deionized water followed with isopropyl alcohol.

8. After mixing, tissue to be analyzed for metals is placed in a 50 ml conical tube. The lab sample number and the letters "mets" are placed on the tube. Place the letters "WPC" on the tube. Caps are loosely placed on the tubes to allow the dry ice to sublimate.

9. After mixing, tissue to be analyzed for mercury is placed in a 50 ml conical tube. The lab sample number and the letters "Hg" are placed on the tube. Place the letters "WPC" on the tube. Caps are loosely placed on the tubes to allow the dry ice to sublimate.

10. If organic analysis is being performed, wrap all remaining tissue in clean aluminum foil. Wrap the aluminum foil with lab tape and write the lab sample number and the word "pesticides" on the tape. Write the letters "WPC" on the tape.

11. The samples are placed in a freezer until transport to the lab for analysis.

12. Tighten the caps on the conical tubes before delivery to the lab.

1.1.13 Cleaning and Sterilization Procedures

After each fish or shellfish sample is processed, the canister and other utensils need to be thoroughly cleaned and sterilized. Each sample should be processed with clean, dry equipment. The procedure for cleaning processing equipment following the grinding procedure is:

1. Each canister should be placed under **hot** running tapwater to allow the remaining powder to break free from the blade assembly and canister walls.

2. The canister should be scrubbed thoroughly with a brush inside and out.

3. Then the canister should be rinsed with deionized water and followed by a rinse with isopropyl alcohol and allowed to dry before use.

4. All knives, lids, bowls, spoons, etc ; should be cleaned following the same procedure. Scrub with a brush under **hot** running tapwater, rinse with deionized water and follow with isopropyl alcohol. Allow drying before use on the next sample.

1.2 Shellfish Collection

1.2.1 Oyster Sampling Procedure

Oysters are collected from the mid-intertidal portion of endemic reefs. Oysters are collected by hand using screwdrivers and hammers where necessary to break them free from clumps

1. In general, collect a minimum of 20 -30 legally harvestable (75 mm or greater) specimens from each station in order to produce 200 grams of shell liquor and meat.

2. Clean oysters in ambient water and place on wet ice in labeled coolers. Label should

include station location and date.

1.2.2 Crab Sampling Procedure

Crabs are collected with baited commercial-style crab pots.

1. Bait traps with whole gut-slit shad or other fish.

2. Attach a float to each crab pot.

3. Deploy traps overnight at each station.

4. Remove legally harvestable (127 mm carapace width) blue crabs from trap as soon as possible the next morning. Approximately 20 crabs are collected from each station.

5. Place crabs on wet ice in a labeled cooler for transport to the lab. Label should include station location and date.

1.2.3 Oyster Work-up Procedure

1. Assign a collection number to each station of oysters. The first two numbers of the collection number will be the year the oysters were collected. The last three numbers of the collection number will be the order in which the oysters are worked-up. If the oysters are the 500th sample worked up in 1998 the lab number will be 98500. Record collection number in log book.

2. Record station name and number, collectors, and date of collection in log book.

3. Discard any gaping oysters

4. Shuck oysters at the Aquatic Biology Lab and weigh composite tissue on platform scales. Record composite weight of tissue in log book. Transfer the tissue with forceps that have been cleaned and rinsed first with deionized water and then isopropyl alcohol.

5. Transfer the tissue to clean aluminum foil.

6. Wrap lab tape around aluminum foil package of oysters and place sample number on tape.

7. Place oyster tissue in freezer until ready for processing.

1.2.4 Crab Work-up Procedure

Approximately twenty crabs are included in the composite sample to obtain the 100 g of somatic tissue needed from each station.

1. Assign a collection number to each station of crabs and record sample number in log

book. The first two numbers of the collection number will be the year the oysters were collected. The last three numbers are the order in which the crabs were worked-up. If the crabs are the 500th sample worked-up in 1998 the collection number will be 98500. Record collection number in log book.

2. Record station number, date of collection, and collectors name in log book.

3. Obtain tissue by removing the claws, carapace, and hepatopancreatic material using stainless steel scissors and forceps rinsed in deionized water and isopropyl alcohol each time the station changes.

4. The body is broken in half and the exposed tissue is extracted from the shell with cleaned stainless steel scissors and forceps. Caution is taken to avoid contamination of tissue and instruments with any residual hepatopancreatic material.

5. Weigh composite tissue on platform scales and record weight in log book.

6. Place tissue in clean aluminum foil. Place lab tape around aluminum foil, and write collection number on tape.

7. Place tissue in freezer until ready for processing.

1.2.5 Oyster And Crab Processing Procedure

Processing should be performed as soon as possible after the oysters and crabs have been worked-up.

1. Assign a lab sample number to each container of oysters or crab. The lab sample number is a 10 digit number. The first six numbers of the lab sample number will be the date processing of that tissue began. The last four numbers will be the order the sample was processed beginning with the number 1000. The 10th sample processed on April 01, 1998 would be assigned lab number 0401981009.

2. Remove samples from refrigerator as needed.

3. Place frozen tissue on a clean chopping board and cut into approximately 10 mm cubes using a stainless steel knife and hammer.

4. Place approximately 200cc of dry ice in a clean(see section **9.5.13)** stainless steel blender canister, then fill the canister approximately ½ with tissue.

5. The tissue and dry ice are ground into a fine powder.

6. The ground tissue is placed on clean aluminum foil.

7. If all of the tissue cannot be ground at the same time, the ground tissue is placed in a clean stainless steel bowl and mixed throughly after all tissue is ground.

8. After mixing, tissue to be analyzed for mercury is placed in a 50 conical tube. The lab sample number and the letters "Hg" and "WPC" are placed on the tube. Caps are loosely placed on tubes to allow the dry ice to sublimate.

9. After mixing, tissue to be analyzed for metals is placed in a 50 ml conical tube. The lab sample number and the letters "mets" and "WPC" are placed on the tube. Caps are loosely placed on the tubes to allow the dry ice to sublimate.

10. All remaining tissue is placed in clean aluminum foil. Wrap the aluminum foil with lab tape and write the lab sample number, the word "pesticides", and letters "WPC" on the tape.

11. The samples are placed in a freezer until transport to the lab for analysis.

12. Tighten the caps on the conical tubes before delivery to the lab.

1.3 Alligator processing

All Alligator samples will be processed for mercury , metals (cadmium, chromium, copper, lead, nickel, and zinc), and pesticides. Alligator meat is provided by SCDNR and all log book information may not be provided for each sample.

1. Enter all available information in the log book. This information is provided by the alligator trappers and may include sex, length, weight, date taken, and location. Record the SCDNR tag number also.

2. Assign a lab number to each portion of alligator meat. The lab number is a 10 digit number. The first six numbers of the lab sample number will be the date processing of that meat began. The last four numbers will be the order the sample was processed beginning with the number 1000. The 10th samples processed on a start date of May 15, 98 would be assigned a lab sample number 0515981009.

3.Remove tissue samples from freezer as needed to prevent thawing of samples.

4. Place meat on a clean chopping board and cut into approximately 10mm cubes with a stainless steel knife and hammer..

5. Place approximately 200cc of dry ice in a clean (see section **9.5.13)** stainless steel blender canister, then fill the canister approximately ½ with fish tissue.

6. The tissue and dry ice are ground into a fine powder.

7. The ground tissue is placed on clean aluminum foil.

8. If all of the meat cannot be ground at the same time, the ground tissue is placed in a clean stainless steel bowl and mixed throughly after the entire sample is ground.

9. After mixing, tissue to be analyzed for metals is placed in a 50 ml conical tube. The lab sample number and the letters "mets" and "WPC" are placed on the tube. Caps are loosely

placed on the tubes to allow the dry ice to sublimate.

10. After mixing, tissue to be analyzed for mercury is placed in a 50 ml conical tube. The lab sample number and the letters “Hg” and “WPC” are placed on the tube. Caps are loosely placed on the tubes to allow dry ice to sublimate.

11. All remaining tissue is wrapped in clean aluminum foil. Wrap the aluminum foil with lab tape and write the lab sample number and the word “pesticide” and letters “WPC” on the tape.

12. The samples are placed in a freezer until transport to the SCDHEC Lab for analysis.

13. Tighten the caps on the conical tubes before delivery to the lab.

APPENDIX 1

Fish Tissue Log Sheets

APPENDIX 2

Inorganic Analysis Fish Tissue Data Sheet

APPENDIX 3

Organic Analysis Fish Tissue Data Sheet

Appendix 3: Access Analytical, Inc. Standard Operating Procedure for Fish Tissue Analysis

ACCESS ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

FOR

Fish Tissue Analysis

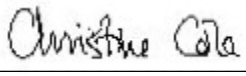
SOP Revision 1.0

SOP ID: AXS - SOP – SP1 -

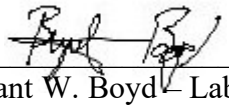
Effective Date: 8.8.2019

APPROVAL SIGNATURES:

Ashley B. Amick – President Date: 11/4/2019



Christine A. Cole – Quality Assurance Director Date: 11/4/2019



Bryant W. Boyd – Laboratory Director Date: 11/4/2019

Scope and Application

This SOP is for a specific project preparing fish tissue for analysis of fluoride and uranium from fish fillets and whole fish. The fish will be prepared and testing for fluoride at Access Analytical. An equivalent portion of fish will be analyzed by another lab for uranium according to their SOP.

Summary of Method

Fish samples are ground and blended to make a homogenized, representative sample of fillet and whole fish. Half the sample is analyzed by another lab for uranium by their SOP. Access Analytical will analyze the other representative sample for fluoride. This is done by weighing a homogenized portion of the sample, adding the sample to the IC eluent (matrix match), stirring vigorously to allow the soluble fluoride to become miscible into the eluent, the analyzing the settled eluent on the IC.

NOTE: We attempted several methods as there is no defined method for fluoride solid analysis in the industry standards. Unlike metals that are digested in acid, adding any mineral acids to "digest" would interfere with most detection methods of anions. In addition, most standards simply say to "prepare the sample" without any direction.
There are no commercially available substances to matrix match our samples for blanks, LFB, etc. or to test the optimal way to recover the fluoride. To overcome this issue, we made our own "standards" to determine the best method for recovery. A fresh caught fish was filleted and divided in two portions. One portion was kept as the reference, and the other portion was soaked in a 10 ppm fluoride solution and stored at room temperature (fortified fish fillet). The goal was to use osmosis to force a higher concentration of fluoride into the tissue to ensure we had some fluoride in the sample. We used these samples to determine the best route of extraction. A summary of our findings:

1. We heated a portion of fish at 100 ^{o}C covered with 6 N NaOH to trap any fluoride that could boil off. That sample was then incinerated at 500 ^{o}C to reduce the sample to ash that would then be analyzed. This did not work; we hypothesize there is too much oil in the fish which made the sample explode in the furnace, which hurt the recovery of the sample.
   The goal of heating the sample was to decompose the sample into small, ash-like parts. The result of heating the sample with the sodium hydroxide trap did not change the composition to be much different than cutting it up, we did not pursue the heat any further.
2. According to Standard Methods, complicated matrices can be distilled using sulfuric acid and condensing tubes. We purchased the distillation apparatus and distilled test samples. However, the carryover was high in sulfate, which caused issues when we ran the samples on the IC. See Image 1.0 for the apparatus.

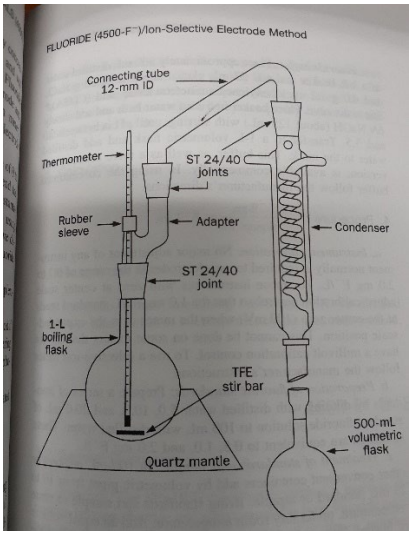


Image 1.0 Distillation Apparatus per SM 4500.

After careful consideration, input from other labs, and knowing the high solubility of fluoride ions, we decided that agitation of small pieces of sample would give the best reading of fluoride.

Definitions

Blank: An aliquot of reagent water analyzed exactly like a sample to make sure there is no contaminates in the column or system

Lab Fortified Blank: An aliquot of reagent water or other blank matrices to which known quantities of method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

Continuing Calibration Blank: An aliquot of reagent water analyzed exactly like a sample to make sure there is no carryover one sample to the next from sample retention in the column

Continuing Calibration Verification (CCV): An aliquot of reagent water with known quantities of method analytes added in the laboratory that are analyzed exactly like a sample. Recoveries are calculated to make sure there is no carryover one sample to the next from sample retention in the column and ensuring the analysis run continues to be in control.

Laboratory Fortified Matrix Spike: An aliquot of the eluent from the “extraction” to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

Laboratory Fortified Matrix Spike Duplicate: A duplicated aliquot of the eluent from the "extraction" laboratory fortified matrix spike to check for precision of the instrument and matrix recoveries.

Interferences

Any anion that is only weakly retained on the column may elute in the retention time window of fluoride and potentially interfere. Precision and accuracy information must be obtained for each sample matrix to account for this type of interference.

Safety Apparatus and Materials

Wear safety glasses, gloves and lab coat. Wash hands well after handling chemicals and avoid skin contact.

Instruments and Equipment

Blender: Waring 500-Watt Bar Blender: Stainless steel blades are sufficient as the only metal tested will be uranium, which is not found in stainless steel so it will not be a source of contamination.
PTFE Cutting board
Kitchen knife
125 mL Erlenmeyer Flasks
Stir bars
Stir plates
Ion Chromatograph and all related components

Reagents and Consumable Materials

Heavy Duty Aluminum foil: to cover cutting board for each fillet
Autosampler vials with caps
Syringe filters (0.45 microns)
10 mL Syringes
Eluent (SNIPs sodium carbonate/sodium bicarbonate) Lot 012819
Solutions from multielement standards

Sample Preservation and Handling

Samples were received at the lab filleted and frozen.

Calibration Procedures and Reagent Standardizations

Calibrate per SOP for Ion Chromatography requirements.

Test Procedure

Fish Tissue preparation:

Fish arrived already filleted and ready for processing.

1. Assign sample ID for each fish.
2. Determine if the fish is scaled or not. If the fish is already filleted, leave it blank.
3. Record the weight of each sample.
4. Using a sharp knife, each sample is cut into 2.5 cm cubes to aid in grinding.
5. Add fish to blender. Blend until a paste-like texture.
    a. If the fish needs to be blended in batches, do the following to make sure the sample composite is as homogenous as possible:
        i. Put all batches of blended fish into mixing container.
        ii. Divide the sample into quarters.
        iii. Opposite quarters are mixed together by hand.
        iv. Then mix the two halves together.
        v. Repeat the blending of batches and hand mixing at minimum of 2 more times. If the sample does not look uniform in texture, repeat until it does and then repeat blending and mixing process 2 more times.
6. Remove the blended fish. Mix well.
7. Divide the sample in about half. Record the weight of sample for Access Analytical. Then weight the other half and record as the Sub Lab weight.
8. Pack the sample for storage/shipment.
9. Repeat for all samples, cleaning the knife and blender between each sample. Use a new sheet of foil on the cutting board between each sample.
10. Some samples would not blend, such as small fillets. Those samples were divided in half without blending.

Fluoride Analysis:

Fluoride:

1. Tare the balance with a disposable weigh boat.
2. Record Fish ID.
3. Weigh out fish and record the weight.
4. Repeat for all the samples.
5. Transfer the fish into labeled 125 mL Erlenmeyer Flask.
6. Add 100 mL of IC eluent to each flask.
7. Stir on stir plate for 2 hours.
8. Let the solids settle overnight.
9. Run the supernatant on the IC, making dilutions as need.
10. Calculate the weight in fish by:

$$100\ mL * \frac{IC\ reading\ mg}{L} * \frac{1\ L}{1000\ mL} = \frac{mg}{weight\ of\ fish\ g} * \frac{1000\ g}{kg}$$

Approved Analytical Methods and References

APHA (2012) *Standard Methods for the Examination of Water and Wastewater 22nd Edition*. 4500-F- B. Primary Distillation Step and 4500 F-C. ISE Method. 4-84 – 4-87. Washington, DC/ Port City Press.

Environmental Protection Agency (2000). *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories: Volume 1: Fish Sampling and Analysis Third Edition.* (EPA 823- B-00-007). Washington, DC. Office of Water. 7-12 – 7-15.

Malde, M., Bjorvatn, K., Julshamn., K. Food Chemistry 73 (2001) *Determination of Fluoride in Food by the Use of Alkali Fusion and Fluoride Ion-Selective Electrode*. Elsevier.

Quality Control

The balance must be checked with ASTM weights prior before use. The points are: 0.100 mg and 100 mg. A continuing check must be performed on the balance using the 1.0 g check after every 20 samples weighed.

Samples run on IC were calibrated to 0.200 ppm solution, continuing calibration verification, blanks, as well as matrix spikes and matrix spike duplicates. The spike and spike duplicates were unsuccessful, however the continuing calibration verification passed, so there was no carryover from one sample to another.

Lab Fortified Blank: 90-110% recovery
CCV: 90-110% recovery
Blanks: <0.06 ppm
MS: 80-120%
MSD: RPD less than 10%

Examples Forms

N/A

Revision Notes

ORGINAL VERSION:	**DATE CREATED:**	**SOP AUTHOR:**	**NOTES:**
1.0	8.8.19	C. Cole	Started SOP but was developed as more information was determined on the process for preparation and recovery.

REVISION #:	**DATE REVISED:**	**REVISED BY:**	**CHANGES MADE:**

Appendix 4: Determination of Metals in Water, Soils and Wastes by ICP-OES by EPA SW-846 Method 6010D and Prep Methods 3010A/3050B

AES, Inc.
3080 Presidential Dr.
Atlanta, GA 30340

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DOCUMENT CONTROL NUMBER: Rev. 15

DOCUMENT DISTRIBUTION NUMBER: IA-13002

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Technical Director: Dana B. Till Date: 10/9/2023

Laboratory Manager: Ryan Sall Date: 10/9/2023

Quality Assurance Manager: Date: 10/9/2023

Department Supervisor: Date: 10/9/2023

STANDARD OPERATING PROCEDURE FOR THE
DETERMINATION OF METALS IN WATER, SOILS AND WASTES BY ICP-OES
BY EPA SW-846 METHOD 6010D AND PREP METHODS 3010A/3050B

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma-optical emission spectrometry (ICP-OES) is a spectrometric technique used to determine trace elements, including metals, in aqueous solutions. In ICP-OES, a sample solution is aspirated (i.e., nebulized) continuously into an inductively coupled, argon-plasma discharge, where analytes of interest are converted to excited-state, gas-phase atoms or ions. As the excited-state atoms or ions return to their ground state, they emit energy in the form of light at wavelengths that are characteristic of each specific element. The intensity of the energy emitted at the chosen wavelength is proportional to the amount (concentration) of that element in the analyzed sample. Thus, by determining which wavelengths are emitted by a sample and their respective intensities, the elemental composition of the given sample relative to a reference standard may be quantified.

1.2 All matrices, excluding filtered groundwater samples but including groundwater aqueous samples, TCLP and EP extracts, industrial and organic wastes, and solid samples, require acid digestion prior to analysis. Groundwater samples that have been pre-filtered and acid-preserved will not need acid digestion. Results are analytically reported as "dissolved metals". Samples which are not digested must either use an internal standard or be matrix matched with the standards. Refer to the procedure section for appropriate digestion procedures.

1.3 Table 2-1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 7-3 lists the recommended analytical wavelengths for the elements in a clean aqueous matrix. Laboratory determined detection limits are listed in the QA Manual, Section 5. The instrument detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements and matrices other than those listed in Table 7-3 may be analyzed by this method if acceptable performance at the concentrations of interest is demonstrated.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, aqueous and solid samples must be acidified, solubilized, or digested using appropriate sample preparation procedures as mandated by the EPA.

2.2 The analysis described in this procedure involves multi-elemental determinations by ICP-OES using simultaneous and/or sequential instrumentation. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by radio-frequency, inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored at specific wavelengths by a photosensitive device. A background correction technique is required to compensate for the variable background contribution to the determination of the analytes.

2.3 The following analytes can be analyzed by this method:

Table 2-1
Metallic Analytes Analyzed by ICP-OES

Aluminum	Antimony	Arsenic
Barium	Beryllium	Boron
Cadmium	Calcium	Chromium
Cobalt	Copper	Iron
Lead	Magnesium	Manganese
Phosphorus	Molybdenum	Nickel
Silica	Potassium	Selenium
Strontium	Silver	Sodium
Sulfur	Titanium	Thallium
Tin	Vanadium	Zinc

3.0 INTERFERENCES

3.1 Spectral interferences can arise from background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap from the molecular spectra of the same target element, or optical spectral-line overlaps between target elements.

3.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined through measurements obtained adjacent to the analyte wavelength peak. Spectral scans of samples or single-element solutions in the analyte regions may indicate when the use of alternate wavelengths is desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements obtained on both sides of the wavelength peak, or by measured emission obtained only on one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular), or otherwise adequately corrected to reflect the same change in background intensity as that which occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

3.1.2 To determine the appropriate location for off-line background correction, the area adjacent to the wavelength on either side must be scanned, so that the apparent emission intensity from all other method analytes may be recorded. This spectral information must be documented and kept on file. The location selected for the background correction must be either free of off-line interelement spectral interference or a computer routine must be used for the automatic correction of all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby

spectral interference effects from all method analytes and common elements, and provide for their automatic correction on all analyses. Tests to determine spectral interference must be performed using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single-element solutions are sufficient; however, for analytes such as iron that may be found at high concentrations, a more appropriate test would be to use a concentration near the upper limit of the analytical range.

3.1.3 Overlaps from the molecular spectra of the same target element may be avoided through the use of an alternate wavelength for quantitation. Interelement spectra overlaps are typically compensated through the use of equations that correct for interelement contributions. Instruments that use equations for interelement correction necessitate that interfering element(s) are analyzed at the same time as the target element(s) of interest. When operative and uncorrected, interelement interferences will produce false positive or positively biased determinations. However, if the interference affects the point selected for background correction, the resulting overcorrection will cause a negative bias. More extensive information on interfering effects at various wavelengths and resolutions is available in reference wavelength tables and books. Analysts may apply interelement-correction equations determined on their instruments with tested concentration ranges to compensate (off-line or on-line) for the effects of interfering elements. Selected potential spectral interferences observed for the recommended wavelengths are given in Table 3-2. For multivariate calibration methods that employ whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the calibration algorithm. The interferences listed in Table 3-2 are those that occur between method analytes. Only interferences of a direct overlap nature are shown. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

3.1.4 When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false positive analyte concentrations) arising from 100 mg/L of the interference element. For example, if As is to be determined at 193.696 nm in a sample containing approximately 10 mg/L of Al, according to Table 3-2, 100 mg/L of Al will yield a false positive signal equivalent to an As concentration of approximately 1.3 mg/L. Correspondingly, the presence of 10 mg/L of Al will result in a false positive signal for As equivalent to approximately 0.13 mg/L. The analyst is cautioned that alternate instruments may exhibit somewhat different levels of interference than those shown in Table 3-2. The interference effects must, therefore, be evaluated for each individual instrument since the intensities will vary.

3.1.5 Interelement corrections will vary for the same emission line among instruments because of differences in resolution. Such differences are determined by the grating, entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data.

Analysts should continually note that some samples may contain uncommon elements that could contribute spectral interferences.

3.1.6 The interelement effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height, and argon flow rate). When using the recommended wavelengths, the analyst must determine and document for each wavelength the effect from referenced interferences (Table 3-2) as well as any other suspected interference that may be specific to the instrument or matrix. The analyst should utilize a computer routine for the automatic correction on all analyses.

3.1.7 The accuracy of interelement corrections and/or the absence of interferences must be verified daily through the analysis of mixed element spectral interference check (SIC) solutions and at least every 6 months through the analysis of single element SIC solutions.

3.2 Physical interferences are effects associated with the sample nebulization and transport processes.

3.2.1 Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or acid concentrations. If physical interferences are present, they must be reduced through sample dilution, the use of a peristaltic pump, the use of an internal standard, or the use of a high-solids nebulizer.

3.2.2 Another problem that can occur when high concentrations of dissolved solids are present is salt buildup at the tip of the nebulizer, thus affecting aerosol flow rate and resulting in instrumental drift. Salt buildup can be controlled through wetting the argon prior to nebulization, use of a tip washer, use of a high-solids nebulizer, or sample dilution. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance. This may be accomplished with the use of mass flow controllers. The tests described in Section 8 will help determine if a physical interference is present.

3.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. However, if observed, can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), buffering of the sample through by matrix-matching, and standard-addition procedures. Chemical interferences are highly dependent on matrix type and analyte.

3.4 Memory interferences result when analytes in a previous sample contribute to the intensity signals measured in a subsequent sample.

3.4.1 Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray

chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples.

3.4.2 The possibility of memory interferences should be considered within an analytical run. When recognized, suitable rinse times should be established to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. The estimation may be made by aspirating a standard containing the element(s) of interest at a concentration level that is ten times the typical or expected amount, or at the upper limit of the linear range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by the analysis of the rinse blank at a series of designated intervals. The length of the rinse time necessary for reducing the analyte signal(s) to less than or equal to the IDL should be noted.

3.4.3 A rinse period of at least 60 seconds should be used between samples and standards until a more suitable rinse time is established. If memory interference is determined to be present, the sample must be reanalyzed following use of the newly established rinse period.

3.5 Table 3-2 indicates potential interferences and analyte concentration equivalents that arise from interference at the 100 mg/L level. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

Table 3-1
Analyte Elemental Concentrations Used For Interference Measurements in Table 3-2

Analytes	(mg/L)		Analytes	(mg/L)
Aluminum	10		Manganese	1
Arsenic	10		Molybdenum	10
Boron	10		Sodium	10
Barium	1		Nickel	10
Beryllium	1		Lead	10
Calcium	1		Antimony	10
Cadmium	10		Selenium	10
Cobalt	1		Silicon	1
Chromium	1		Thallium	10
Copper	1		Vanadium	1
Iron	1		Zinc	10
Magnesium	1			

Table 3-2
POTENTIAL INTERFERENCES AND ANALYTE CONCENTRATION EQUIVALENTS (mg/L) ARISING FROM INTERFERENCE AT THE 100 mg/L LEVEL

Analyte	Wavelength (nm)	Interferent									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	-	-	-	-	-	-	0.21	-	-	1.4
Antimony	206.833	0.47	-	2.9	-	0.08	-	-	-	0.25	0.45
Arsenic	193.696	1.3	-	0.44	-	-	-	-	-	-	1.1
Barium	455.403	-	-	-	-	-	-	-	-	-	-
Beryllium	313.042	-	-	-	-	-	-	-	-	0.04	0.05
Cadmium	226.502	-	-	-	-	0.03	-	-	0.02	-	-
Calcium	317.933	-	-	0.08	-	0.01	0.01	0.04	-	0.03	0.03
Chromium	267.716	-	-	-	-	0.003	-	0.04	-	-	0.04
Cobalt	228.616	-	-	0.03	-	0.005	-	-	0.03	0.15	-
Copper	324.754	-	-	-	-	0.003	-	-	-	0.05	0.02
Iron	259.940	-	-	-	-	-	-	0.12	-	-	-
Lead	220.353	0.17	-	-	-	-	-	-	-	-	-
Magnesium	279.079	-	0.02	0.11	-	0.13	-	0.25	-	0.07	0.12
Manganese	257.610	0.005	-	0.01	-	0.002	0.002	-	-	-	-
Molybdenum	202.030	0.05	-	-	-	0.03	-	-	-	-	-
Nickel	231.604	-	-	-	-	-	-	-	-	-	-
Selenium	196.026	0.23	-	-	-	0.09	-	-	-	-	-
Sodium	588.995	-	-	-	-	-	-	-	-	0.08	-
Thallium	190.864	0.30	-	-	-	-	-	-	-	-	-
Vanadium	292.402	-	-	0.05	-	0.005	-	-	-	0.02	-
Zinc	213.856	-	-	-	0.14	-	-	-	0.29	-	-

Dashes indicate that no interference was observed even when interferents were introduced at the following levels:
Al - 1000 mg/L Mg - 1000 mg/L Ca - 1000 mg/L Mn - 200 mg/L Cr - 200 mg/L
Ti - 200 mg/L Cu - 200 mg/L V - 200 mg/L Fe - 1000 mg/L

Interferences will be affected by background and wavelength choice and other interferences may be present.

3.6 Other sources of interference include sample contamination. Dust in the laboratory environment, impurities in reagents, and impurities on laboratory apparatus that the sample comes in contact with are all sources of potential contamination. Sample

containers can introduce either positive or negative errors in the measurement of trace elements by (a) contributing contaminants through leaching or surface desorption and (b) by depleting concentrations through adsorption

3.6.1 Aqua Regia (HNO3+HCl) may be used to remove organic deposits from glassware; however, the analyst should be cautioned that the glassware must be thoroughly rinsed with water to remove the last traces of acid.

3.6.2 If it can be documented through an active analytical quality control program using spiked samples and reagent blanks that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

4.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

4.1 For the determination of trace elements, contamination and loss are of prime concern. In this regard, the collection and treatment of the sample prior to analysis require particular attention. Laboratory glassware, including the sample bottle (whether polyethylene, polypropylene, or FEP-fluorocarbon), should be thoroughly washed with detergent and tap water and rinsed with (1+1) nitric acid, tap water, (1+1) hydrochloric acid, tap water, and finally deionized water.

4.2 Before collection of the sample, a decision must be made regarding the type of metal analysis that is desired; that is dissolved, suspended, or total, so the appropriate preservation and pretreatment steps may be accomplished. Filtration, acid preservation, etc. are to be performed at the time the sample is collected, or as soon as possible thereafter.

4.2.1 For the determination of dissolved elements, the sample must be filtered through 0.45µm membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus is recommended to avoid possible contamination). Use the first 50 – 100 mL of sample to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) HNO_3 to a pH $\leq$ 2. Normally, 3 mL of (1+1) acid per liter should be sufficient to preserve the sample.

4.2.2 For the determination of suspended elements, a measured volume of unpreserved sample must be filtered through a 0.45µm membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservation is required.

4.2.3 For the determination of total elements, the sample is preserved with (1+1) HNO_3 to a pH $\leq$ 2. Normally, 3 mL of (1+1) acid per liter should be sufficient to preserve the sample.

4.3 All samples must be analyzed within 180 days of sampling.

5.0 REAGENTS AND STANDARDS

5.1 Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. Re-distilled acids are acceptable.

5.1.1 Hydrochloric Acid, concentrated (sp.gr. 1.19).

5.1.2 Hydrochloric Acid, (1:1): Add 500mL concentrated HCl to 400mL deionized water and dilute to 1 liter.

5.1.3 Nitric Acid, concentrated (sp.gr. 1.41)

5.1.4 Nitric Acid, (1:1): Add 500mL concentrated HNO_3 to 400mL deionized water and dilute to 1 liter.

5.2 Hydrogen Peroxide, 30%. Purchase commercially prepared solution.

5.3 Deionized water, 18 megaohm or greater.

5.4 Single element standard solutions. Purchased from the vendor as certified solution at concentration of 1000 mg/L or other suitable concentration.

5.5 Primary Multi-element Standard 1 (AESI-CAL-1) and Secondary Multi-element Standard 1 (AESI-CAL-1QC). Purchased as certified solutions from Inorganic Ventures. Two independently prepared lot numbers are required. Concentrations are as follows:

Table 5-1
Primary and Secondary Multi-element Standard 1 Concentrations

Concentration (mg/L)	Metal Ion
10	Ag
100	As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Tl, V, Zn
1000	Al, Ca, Fe, K, Mg, Na

5.6 Primary Multi-element Standard 2 (VAR-CAL-1) and Secondary Multi-element Standard 2 (VAR-QC-1). Purchased as certified solutions from Inorganic Ventures. Two independently prepared lot numbers are required. Concentrations are as follows:

Table 5-2
Primary and Secondary Multi-element Standard 2 Concentrations

Concentration (mg/L)	Metal Ion
100	Mo, Sb, Sn, Ti

5.7 Initial calibration 1 (ICAL1) standard 1 and CRI Standard 1 (AESI-CAL-2). Purchased as certified solution from Inorganic Ventures. Concentrations are as follows:

Table 5-3
Primary Multi-element Standard 1 Concentrations-CRI/ICAL1

Concentration (mg/L)	Metal Ion
2	Sb
5	Mo, Sn, Ti

5.8 Initial calibration 1 (ICAL1) standard 2 and CRI Standard 2 (AESI-CAL-3). Purchased as certified solution from Inorganic Ventures. Concentrations are as follows:

Table 5-4
Primary Multi-element Standard 2 Concentrations-CRI/ICAL1

Concentration (mg/L)	Metal Ion
0.5	Ag, Be, Cd, Mn
1	Ba, Co, Cr, Cu, Pb, V
2	Ni, Se, Tl, Zn
5	As
10	Ca, Fe, Mg
20	Al
50	K
100	Na

5.9 Interferent Check Standard (ICL500-6). Purchased as certified solution from Environmental Express. Concentrations are as follows:

Table 5-5
Interferent Check Standard Concentrations

Concentration (mg/L)	Metal Ion
2000	Fe
5000	Al, Ca, Mg

5.10 Laboratory Control Multi-element Spike Standard (LCS; AESI-CAL-4A). Purchased as certified solution from Inorganic Ventures. Concentrations are 100 mg/L for B, Li, P, Si, Sr, and S.

5.11 Initial calibration standard 2 (ICAL2). Prepare by adding 5mL each of the IV primary multi-element standards, 0.5mL each of the B, P, and S, single element standards, 0.05mL of the Sr single element standards, 40mL of concentrated nitric acid, and 50mL of concentrated hydrochloric acid to a 1000mL volumetric flask and diluting to volume with deionized water. The resulting concentrations are as follows:

Table 5-6
ICAL 2 Standard Concentrations

Concentration (mg/L)	Metal Ion
5	Al, Ca, Fe, K, Mg, Na
0.5	As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, P, Se, Sb, Sn, Sr, S, Tl, Ti, V, Zn
0.05	Ag

5.12 Initial calibration standard 3 (ICAL3), Mid Level verification standard 1 (CRI-Mid level), and continuing calibration verification standard (CCV). Prepare by adding 10mL each of the IV primary multi-element standards, 1mL each of the B, P, and S, single element standards, 0.1mL of the Sr single element standards, 40mL of concentrated nitric acid, and 50mL of concentrated hydrochloric acid to a 1000mL volumetric flask and diluting to volume with deionized water. The resulting concentrations are as follows:

Table 5-7
ICAL 3, CRI-Mid Level, and CCV Standards Concentrations

Concentration (mg/L)	Metal Ion
10	Al, Ca, Fe, K, Mg, Na
1	As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, P, Se, Sb, Sn, Sr, S, Tl, Ti, V, Zn
0.1	Ag

5.13 Initial calibration standard 4 (ICAL4). Prepare by adding 20mL of the IV primary source multi-element standards, 2mL each of the B, P, and S, single element standards, 0.2mL of the Sr single element standards, 40mL of concentrated nitric acid, and 50mL of concentrated hydrochloric acid to a 1000mL volumetric flask, and diluting to volume with deionized water. The concentrations of the metals are as follows:

Table 5-8
ICAL4 Standard Concentrations

Concentration (mg/L)	Metal Ion
20	Al, Ca, Fe, K, Mg, Na
2	As, B, Ba, Be,Cd,Co,Cr,Cu,Mn,Mo,Ni, Pb, Se,Sb,Sn, Sr,Tl,Ti,V,Zn
0.2	Ag

5.14 Initial calibration standard 5 (ICAL5) and check standard (CKSTD). Prepare by adding 30mL of the IV primary source multi-element standards, 3mL each of the B, P, and S, single element standards, 0.3mL of the Sr single element standards, 40mL of concentrated nitric acid, and 50mL of concentrated hydrochloric acid to a 1000mL volumetric flask, and diluting to volume with deionized water. The concentrations of the metals are as follows:

Table 5-9
ICAL5 and CKSTD Standards Concentrations

Concentration (mg/L)	Metal Ion
30	Al, Ca, Fe, K, Mg, Na
3	As, B, Ba, Be,Cd,Co,Cr,Cu,Mn,Mo,Ni, Pb, Se,Sb,Sn, Sr,Tl,Ti,V,Zn
0.3	Ag

5.15 Working interference check standard (ICSA) or Spectral Interference Check (SIC). Prepare by adding 50mL of the ICS standard, 40mL of concentrated nitric acid, and 50mL of concentrated hydrochloric acid to a 1L volumetric flask and diluting to volume with deionized water. The concentrations of metals in this standard are 250 mg/L for aluminum, calcium, and magnesium, and 100 mg/L for iron.

5.16 Secondary working interference check standard (ICSAB) or Spectral Interference Check (SIC). Prepare by adding 50mL of the ICS standard, 5mL each of the IV multi-element standards, 0.5mL each of the primary single-element standards for B, S, Sr, P, 40mLof concentrated nitric acid, and 50mL of concentrated hydrochloric acid to a 1000mL volumetric flask and diluting to volume with deionized water. The concentrations of metals in the standard are as follows:

Table 5-10
ICSAB Standard Concentrations

Concentration (mg/l)	Metal Ion
105	Fe
255	Al, Ca, Mg
5.0	K, Na
0.5	As, B, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, P, Se, Sb, Sn, Sr, S, Ti, Tl, V, Zn
0.05	Ag

5.17 CRI Standards.

5.17.1 The CRI is prepared by adding 1mL each of the IV CRI standards, 0.05mL of the Laboratory Control Multi-element Spike Standard, 4mL of concentrated nitric acid and 5mL of concentrated hydrochloric acid to a 100mL volumetric flask and diluting to volume with deionized water. The resulting metal concentrations are as follows:

Table 5-11
CRI Standard Concentrations

CRI Concentration (mg/L)	Metal Ion
0.005	Ag, Be, Cd, Mn
0.01	Ba, Co, Cr, Cu, Pb, V
0.02	Li, Ni, Sb, Se, Tl, Zn
0.05	As, B, Mo, P, S, Sn, Sr, Ti
0.1	Ca, Fe, Mg, Si
0.2	Al
0.5	K
1.0	Na

5.17.2 CRI-2. This standard is made daily from the CRI standard in 5.14.1. Dilute 5 mL of CRI standard with 4%/5% Acid in 5.15 to a 10 mL final volume. The resulting metals concentrations are as follows:

Table 5-11A
CRI-2 Concentrations

CRI Concentration (mg/L)	Metal Ion
0.0025	Ag, Be, Cd, Mn
0.005	Ba, Co, Cr, Cu, Pb, V
0.01	Ni, Sb, Se, Tl, Zn
0.025	As, Mo, Sn, Ti, B, Sr, S, P
0.05	Ca, Fe, Mg
0.1	Al
0.25	K
0.5	Na

5.17.3 CRI-5. This standard is made daily from the CRI standard in 5.14.1. Dilute 2 mL of CRI standard with 4%/5% Acid in 5.15 to a 10 mL final volume. The resulting metals concentrations are as follows:

Table 5-11B
CRI-5 Standard Concentrations

CRI Concentration (mg/L)	Metal Ion
0.001	Ag, Be, Cd, Mn
0.002	Ba, Co, Cr, Cu, Pb, V
0.004	Ni, Sb, Se, Tl, Zn
0.01	As, Mo, Sn, Ti, B, Sr, S, P
0.02	Ca, Fe, Mg
0.004	Al
0.1	K
0.2	Na

5.18 Linear Calibration Range Standards.

5.18.1 Linear Calibration Range Standard 1 (LCR-1). Prepare by adding 20mL each of the IV primary source multi-element standards, 4mL of concentrated nitric acid, and 5mL of concentrated hydrochloric acid to a 100mL volumetric flask and diluting to volume with deionized water. The resulting concentrations are as follows:

Table 5-12
Linear Calibration Range Standard 1 Concentrations

Concentration (mg/L)	Metal Ion
200	Al, Ca, Fe, K, Mg, Na
20	As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Ti, Tl, V, Zn
2	Ag

5.18.2 Linear Calibration Range Standard 2 (LCR-2). Prepare by adding 10mL each of the IV primary source multi-element standards, 4mL of concentrated nitric acid, and 5mL of concentrated hydrochloric acid to a 100mL volumetric flask and diluting to volume with deionized water. The resulting concentrations are as follows:

Table 5-13
Linear Calibration Range Standard 2 Concentrations

Concentration (mg/L)	Metal Ion
100	Al, Ca, Fe, K, Mg, Na
10	As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Ti, Tl, V, Zn
1	Ag

5.18.3 Linear Calibration Range Standard 3 (LCR-3). Prepare by adding 5mL each of the IV primary source multi-element standards, 4mL of concentrated nitric acid, and 5mL of concentrated hydrochloric acid to a 100mL volumetric flask and diluting to volume with deionized water. The resulting concentrations are as follows:

Table 5-14
Linear Calibration Range Standard 3 Concentrations

Concentration (mg/L)	Metal Ion
50	Al, Ca, Fe, K, Mg, Na
5	As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Sn, Ti, Tl, V, Zn
0.5	Ag

5.18.4 Linear Calibration Range Lead/Inhouse Standard (LCR-Pb/Inhouse). Prepare by adding 20mL of the Pb single-element standard, 10mL of the P, B, and S single-element standards, 1mL of the Sr single-element standard, 4mL of concentrated nitric acid, and 5mL of concentrated hydrochloric acid to a 100mL volumetric flask and diluting to volume with deionized water. The resulting concentration is 200 mg/L for Pb, 100 mg/L for B, P, S, and 10 mg/L for Sr.

5.19 Post-Digestion Spike (PDS). Prepare by adding 20mL of the IV multi-element standards, 5mL of primary source standards for B, Sr, and S, and 10mL of primary source P standard, 40mL of HNO3, and 50mL of HCl to a 1L volumetric flask and diluting to volume with DI water.

5.20 Standards for elements not present in the Multielement mixes, such as Li and Si are prepared by diluting single element stock standards to the desired concentrations per project requirements.

5.20.1 Lithium and Silicon

NOTE: Lithium is not used nor analyzed for with SW6010D.

5.20.1.1 CRI-Mid Level – Prepare by adding 0.5mL of primary VHG Labs Li standard, 1.25mL of primary Fisher Scientific Si standard, 20mL of HNO3, and 25mL of HCl to a 500mL volumetric flask and diluting to volume with DI water.

5.20.1.2 ICAL/CKSTD – Prepare by adding 2mL of primary VHG Labs Li standard, 5mL of primary Fisher Scientific Si standard, 40mL of HNO3 , and 50mL of HCl to a 1L volumetric flask and diluting to volume with DI water.

5.20.1.3 ICV – Prepare by adding 1mL of secondary Environmental Express Li standard, 1mL of secondary VHG Labs Si standard, 40 mL of HNO3 , and 50mL of HCl to a 1L volumetric flask and diluting to volume with DI water.

5.20.1.4 CRI – Prepare by adding 0.02mL of Li primary VHG Labs Li, 0.1mL of Si primary Fisher Scientific Si, 40 mL of HNO3 , and 50mL of HCl to a 1L volumetric flask and diluting to volume with DI water.

5.20.1.5 ICSAB – Prepare by adding 0.25mL of VHG Labs Li standard, 0.25mL of Fisher Scientific Si standard, 2.5mL of the ICS standard, 20mL of HNO3, and 25mL of HCl to a 500mL volumetric flask and diluting to volume with DI water.

5.20.1.6 CCV – Prepare by adding 1mL of primary VHG Labs Li standard, 1mL of Fisher Scientific Si standard, 40mL of HNO3, and 50mL of HCl to a 1L volumetric flask and diluting to volume with DI water.

5.20.1.7 PDS – Prepare by adding 0.5mL of VHG Labs Li standard, 2.5mL of Fisher Scientific Si standard, 20mL of HNO3, and 25mL of HCl to a 500mL volumetric flask and diluting to volume with DI water.

5.20.1.8 LCR – Prepare by adding 2.5mL of VHG Labs Li standard, 50mL of primary source Si standard, 20mL of HNO_3, and 25mL of HCl to a 500mL volumetric flask and diluting to volume with DI water.

5.21 Three types of blanks are required for the analysis. The calibration blank is used to establish the analytical curve, while the reagent blank (method blank) is used to correct for possible contamination resulting from varying amounts of the acids used in sample processing and the rinse blank is used to flush the instrument between samples when necessary to reduce memory interferences.

5.21.1 The calibration blank is prepared by diluting 40 ml concentrated HNO_3 and 50 ml concentrated HCl to 1000 ml using deionized water.

5.21.2 The reagent blank (method blank) must contain all the reagents, in the same volumes, used in processing the samples. The reagent blank (method blank) must be carried through the complete procedure and contain the same acid concentration as the sample solution used for analysis.

5.21.3 The Rinse Blank is prepared by diluting 40 ml concentrated HNO_3 and 50 ml concentrated HCl to 1000 ml using deionized water.

5.22 LCS CRM for 6010B_WIPE/WIPE_MET_ICP_P or WIPE_INHOUSE_MET/WIPE_INHOUSE_P
Use 0.125g of Sigma Aldrich CRM Trace Metals – Loamy Clay 1 (CRM052-50G). This will give a final value of 10 total ug for Lead meeting the AIHA, LLC-LAP, requirement.

5.23 Recommended Vendor List. The standards used for this method are purchased using the catalog numbers and vendors indicated below.

Table 5-15
ICP Standards and Chemicals

Standard Name	Vendor Name	Concentration	Catalog Number
Primary Multi-element Standard 1	Inorganic Ventures	Various	AESI-CAL-1
Primary Multi-element Standard 2	Inorganic Ventures	Various	VAR-CAL-1
Secondary Multi-element Standard 1	Inorganic Ventures	Various	AESI-CAL-1 QC (Separate Lot # from primary)
Secondary Multi-element Standard 2	Inorganic Ventures	Various	VAR-QC-1 (Separate Lot # from primary)
CRI Standard 1	Inorganic Ventures	Various	AESI-CAL-2
CRI Standard 2	Inorganic Ventures	Various	AESI-CAL-3
Laboratory Control Multi-element Spike Standard (LCS)	Inorganic Ventures	Various	AESI-CAL-4A
Interferent Check Standard (ICS or SIC)	Environmental Express	Varied	ICL500-6
Primary Boron Standard	Environmental Express*	1000 µg/mL	HP10007-4
Secondary Boron Standard	VHG Labs*	1000 µg/mL	PBW-100
Primary Lithium Standard	VHG Labs*	1000 µg/ml	PLIN-100

Secondary Lithium Standard	Environmental Express*	1000 µg/mL	HP100029
Primary Phosphorus Standard	Agilent Technologies*	1000 µg/mL	ICP-015
Secondary Phosphorus Standard	Environmental Express*	1000 µg/mL	HP100039-1
Primary Silicon(Silica-Si) Standard	Agilent Technologies *	1000 µg/mL	ICP-014
Secondary Silicon(Silica-Si) Standard	Ricca *	1000 µg/mL	RV010050-100N
Primary Strontium Standard	Agilent Technologies*	1000 µg/mL	ICP-038
Secondary Strontium Standard	Ricca*	1000 µg/mL	RV010317-100N
Primary Sulfur Standard	Environmental Express*	1000 µg/mL	HP100054-5
Secondary Sulfur Standard	VWR Scientific*	1000 µg/mL	BDH82026-048
Hydrochloric Acid	Fisher Scientific*	Concentrated	A144C-212
Nitric Acid	Fisher Scientific*	Concentrated	A200S-212
Hydrogen Peroxide	Fisher Scientific*	30% Solution	H325-4
CRM Trace Metals-Loamy Clay 1 CRM052-50G	Sigma Aldrich	Various	CRM052-50G

***Single element standards and acids may be purchased from alternate sources as needed.**

6.0 APPARATUS AND MATERIALS

6.22 Inductively Coupled Plasma-Optical Emission Spectrometer. Agilent 5100 instrument and software.

6.23 Argon gas supply. Analytical grade or better.

6.24 Block digester

6.25 Plastic digestion vessels - 70-ml disposable with graduations.

6.26 General Glassware. Pipettes, volumetric flasks, funnels, etc.

7.0 PROCEDURE

7.22 Preparation of digestion log form and digestion log in LIMS.

7.22.1 Each day the section supervisor prepares a backlog. The log lists samples included in batches that have not been completed and closed in LIMS. If a sample is on this list and it has been digested or analyzed, check LIMS to verify that the batch has been closed. Samples are listed on the backlog in the order of due date.

7.22.1.1 Any samples that are received with a “rush” status will have a chain of custody delivered to the prep department by sample receiving.

7.22.1.2 Prepare a written digestion log using the metals digestion logbook that is kept in the digestion area of the laboratory. The digestion log is written after the samples have been added to the prep batch but before you being the digestion process. The following entries must be made in the log:

7.22.1.2.1 Date and time the batch is opened or the date and time the sample(s) is placed on the hot block.

7.22.1.2.2 All samples included in the digestion batch.

7.22.1.2.3 Volume or weight of samples digested.

7.22.1.2.4 Date and time the digestion is completed.

7.22.1.2.5 Digestion procedure employed.

7.22.1.2.6 The initials of the digestion analyst(s).

7.22.1.2.7 Laboratory number of all reagents used, including spiking standard and acids.

7.22.1.2.8 Volume of spiking standard and acids used.

7.22.1.2.9 Final volume of all digestates.

7.22.1.2.10 Date and time the batch is closed.

7.22.1.2.11 Initials of all spike witnesses. Note that the witness MUST actually witness the spiking operation.

7.22.1.3 Prepare an electronic extraction sheet in LIMS using the following procedure.

7.22.1.3.1 Open a Prep Batch in LIMS by double clicking the “Prep” box.

7.22.1.3.2 To create a new form, click the “add” box. The prep start date, start time, and batch number are automatically created by the LIMS. The start date and time can be updated as needed.

7.22.1.3.3 Select the appropriate Prep Code (i.e. 3010A, 3050B_S, etc.) from the pull down list. The LIMS will automatically assign a MB and LCS sample to the prep list. If an LCSD is desired, click on the space below the sample name LCS and enter the information.

7.22.1.3.4 Enter the technician’s name from the pull down menu.

7.22.1.3.5 Click “Load Samps” and then “User” tab to obtain a list of samples that need preparation by that method. Select the samples to be included in the batch for desired prep method.

7.22.1.3.6 The samples can be individually selected by highlighting the sample and clicking the "→" arrow or all samples can be selected by clicking the "⇒" arrow. Then select "OK".

7.22.1.3.7 Samples that have been selected for re-analysis can be manually added to the sample list following the procedure outlined in section 7.1.1.3.3.

7.22.1.3.8 "Save" the batch by clicking on a previous batch number on the list and then return to the newly created batch.

7.22.1.3.9 Close the batch by entering the date and time.

7.22.2 Table 7-1 indicates the type of samples that comprise an analytical batch. Note: NELAC requirements specify that the maximum number of client samples in a prep batch cannot exceed 20. Further, a prep batch cannot be left "open", i.e. samples added, for a period that exceeds 24 hours.

Table 7-1
Samples in a NELAC Batch

Method Blank (MB)
Laboratory Control Sample (LCS)
Laboratory Control Sample Duplicate (LCSD) (only needed if no MSD)
Matrix Spike/Matrix Spike Duplicate (MS/MSD) (If supplied by client)
Dilution Test Sample (DL)
Post Digestion Spike Sample (PDS)
Client Samples (up to 20 per batch)

This method requires the analysis of additional samples not required by NELAC. See the information in Table 7-2, ICP Run Sequence.

7.23 Sample preparation

7.23.1 Sample preparation for the Determination of Dissolved Metals is described in detail in Table 7-8.

7.23.2 Sample preparation for the Determination of Total Recoverable Metals in Water Samples by Method 3010A is described in detail in Table 7-5.

7.23.3 Sample preparation for the Determination of Total Metals in Soil Samples by Method 3050B is described in detail Table 7-6.

7.23.4 Sample preparation for the Determination of Total Metals in Waste Samples by Method 3050B is described in detail Table 7-7.

7.23.5 Sample preparation for the determination of Total Metals in Wipe Samples is described in Table 7-9.

7.24 Instrument Operation for the Agilent 5100.

7.24.1 Software

7.24.1.1 Choose ICPExpert ICON on the desktop. Select '*Worksheet*'. Choose '*New*'. Then *'Sequence'*. Select worksheet type for the following analysis:
- 7.24.1.1.1 Regular Metals- 200.7_6010.vwst
- 7.24.1.1.2 Silicon- Silicon2.vwst
- 7.24.1.1.3 Inhouse (Boron, Sulfur, Strontium, Phosphorous) - INHOUSE.vwst
- 7.24.1.1.4 Lithium- Lithium.vwst

7.24.1.2 Name file with this format: MMDDYY (LETTER of analysis) (i.e. July 13, 2009; 1st worksheet = 071309A). Press '*Save*'.

7.24.1.3 Press 12th ICON from the left (or shift F4) to turn plasma on.

7.24.1.4 Check flow of sample & waste tubing. If not seen, speed the flow rate up by pressing the gas pump button with a solid blue line.

7.24.1.5 Let the instrument warm up for 30 mins. Make calibration standards if not already made.

7.24.2 TORCH ALIGNMENT

7.24.2.1 After 30mins; perform a torch alignment if the torch was replaced. If not then proceed to "Starting Calibration & Analysis".

7.24.2.2 Select Autosampler from the tool bar

7.24.2.3 Under move probe, change rack to 2 and tube to 12

7.24.2.4 Click the Go To button (make sure tuning solution is in position 12 and uncapped).

7.24.2.5 After a few minutes (2-4), go to the '***Window***' tab of the worksheet, then '***Instrument***' tab then ***'Torch Align'*** tab.

7.24.2.6 Press the ***'Torch Scan'*** button. Once the curve has started on a downward slop, press ***'Stop'*** and change to the other orientation (i.e. Horizontal, Vertical) and repeat procedure.

7.24.2.7 Remove sample probe from the tuning solution and return to the rinse solution by selecting rinse from the autosampler page. The desired range for each orientation is as follows: Horizontal (close to 1), Vertical (close to 0).

7.24.3 STARTING CALIBRATION & ANALYSIS

7.24.3.1 Type in the loading list if samples are present. If not proceed to calibrating.
- 7.24.3.1.1 Switch to the ***'Sequence'*** tab of the worksheet.
- 7.24.3.1.2 Begin typing from 1:1. (Note: to make the loading list longer, Go to ***'Sequence Editor'*** and change sample count to ~120 or whatever desired

7.24.4 SETTING UP INSTRUMENT TO SHUT-DOWN AT THE END OF THE ANALYSIS

7.24.4.1 If running samples overnight go to sequence, sequence parameters, and select plasma off/pump off. This will turn plasma off after analysis.

7.24.4.2 If loading more than 60 samples, go to ***'Autosampler Setup,'*** click on Tray #3, and indicate the 'Rack Type' as 60 x 25ml. Press ***OK*** to update changes. Switch to the Analysis tab when finished typing in the loading list & highlight all samples to be analyzed by scrolling down left column.

7.24.4.3 Press the Green arrow button to start the calibration. Make sure all calibration standards are passing; if not, re-calibrate.

Table 7-2
ICP Run Sequence

1. Check Standard
2. ICV
3. ICB
4. LLV
5. MLV
6. SIC-A
7. SIC-B
8. Up to 10 samples (could include batch QC and/or client samples)
9. CCV
10. CCB
11. Repeat 8-10 as needed

Table 7-3
Wavelengths for ICP Agilent Spectrometer

Element	Agilent 5100 Wavelength
Aluminum	308.215
Arsenic	193.696
Antimony	217.582
Barium	413.064
Beryllium	313.107
Boron	249.678
Cadmium	228.802
Calcium	373.690
Chromium	267.716
Cobalt	230.786
Copper	327.395
Iron	238.204
Lead	220.353
Magnesium	279.800
Manganese	257.610
Molybdenum	204.598
Nickel	221.648
Phosphorus	213.618
Potassium	766.491
Selenium	196.026
Silica	288.158
Silver	328.068
Sodium	589.592
Strontium	216.596
Sulfur	181.972
Tin	189.927
Titanium	337.280
Thallium	351.923
Vanadium	311.837
Zinc	206.200

Table 7-4
Instrument Quality Control Requirements for ICP Analysis

Quality Control Sample	Acceptable Result
Check Standard	90-110% recovery
ICV	90-110% recovery
ICB	$\leq$ 1/2 LLOQ
LLV	80-120% recovery
MLV	90-110% recovery
SIC-A	Unspiked elements < ± LLOQ
SIC-B	Unspiked elements < ± LLOQ
CCV	90-110% recovery
CCB	$\leq$ LLOQ or Project specific requirements in LIMS

Table 7-5
3010A Prep Checklist
3010A

____ **:** PRINT (check) BACKLOG REPORT FOR THE PREP AND EACH TEST CODE
(3010A: 1311_BOTTLE, 1311_M, 1312_B, 6010B_W_T, 6010B_B_W_T, 6010B_PO4_W_T 6010B_SI_W, 6010B_SR_W_T, 6010B_TAL_W_T, INHOUSE_M_W and SM2340B (if sample is on this report, it may need to go on 200.7)

____ : PICK A SAMPLE FOR QC (homogeneous is ideal), (1311_M for a batch with only 1311_M)

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO (Use the blank that has been tumbled with TCLP samples) (QC, then order of backlog) Write samples in the log book from the back log.

____ : ADD SAMPLE TO DIGESTION VESSEL AND RECORD VOLUME.
If the sample does not look like drinking water, then evaluate the sample matrix.
-Pour 1ml into test tube and add 1ml DI H2O to check water solubility. If insoluble, see supervisor before proceeding.
-Add 1ml HNO3 to check reaction. If strong or violent reaction occurs, see supervisor before proceeding.

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, prep code, date/time, technician (your name), click loadsamps, select user, single arrow samples into selected column, check sample IDs, OK, add DUP & MS/MSD, check volumes. All 3010A batches will need DL & PDS. PRINT A COPY OF THE BATCH

____ : ADD 50 mL of DI water to LCS & MB vessels from DI wash bottle. USE 10 mL SAMPLE FOR TCLP.

____ : SPIKE LCS, MS, & MSD with 0.50 mL EACH OF THE 2 MULTI-ELEMENT STANDARDS. (WITNESS NEEDED)

____ : ADD 2.5 mL conc. HCL & 2.0 mL conc. HNO_3, cover the vessels with watch glasses and heat for 40-60min.

____ : REMOVE FROM HEAT AND LET COOL. BRING TO A FINAL VOLUME OF 50 mL

____ : ***SHAKE WELL***

____ : LINE UP SAMPLES IN ORDER THAT THEY APPEAR IN LOG

____ : DELIVER SAMPLES TO THE METALS LAB

NOTE: Method SW3010A states that the final acid concentration in the digestate should be no more than 10%. 2.5 mL of conc. HCl equals 5% and 2.0 mL of conc. HNO_3 equals 4% of the 50 mL final volume.

Table 7-6
3050B_S Prep Checklist
3050B_S

____ **:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE
(3050B_S: 6010B_S, 6010_A9_S, 6010B_PO4_S, 6010B_S_CLP, 6010B_TAL_S, INHOUSE_M)

____ : PICK A SAMPLE FOR QC (original, DUP, MS/MSD) HOMOGENIZE SAMPLE

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO (QC, then order of backlog)

____ : ADD SAMPLE TO DIGESTION VESSEL AND RECORD WEIGHT (1.0-2.0g)

For the sample chosen for MS/MSD or DUP, the initial weights used for the un-spiked sample, matrix spike and matrix spike duplicate, or unspiked duplicate if applicable, must be within 5% of each other.

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, prep code, date/time, technician (your name), click loadsamps, select user, single arrow samples into selected column, check sample IDs, OK, add DUP & MS/MSD, check weight. All 3050B_S batches will need DL & PDS. PRINT A COPY OF THE BATCH

____ : SPIKE LCS & MS with 0.5 mL of multi-element standard. (WITNESS NEEDED)

____ : ADD 2.0 mL conc. HNO_3, COVER THE VESSELS WITH WATCH GLASSES, THEN HEAT FOR 10 MIN

____ : ADD 4.0 mL conc. HNO_3, THEN HEAT FOR 30 MIN

____ : REMOVE from heat, add 2-3 mL DI H_2O, let cool, ADD 1 DROPPER OF 30% H_2O_2, and HEAT FOR 10 MIN.

____ : REMOVE from heat, and let cool

____ : ADD 5 mL HCL, HEAT UNTIL VOLUME drops to 5 mL or 1 HOUR

____ : ***SHAKE WELL***

____ : REMOVE FROM HEAT AND LET COOL. BRING TO A FINAL VOLUME OF 50 mL

Batch Info.

____ **:** Does it have BACKLOG REPORT FOR THE PREP AND EACH TEST CODE

____ : Is the SAMPLE FOR QC (dup, ms) adequate

____ : ARE the SAMPLES IN ORDER AND CHECK PREP AND TEST INFO

____ : Check SAMPLE final volume in DIGESTION VESSEL, does it match Recorded VOLUME

____ : CHECK prep log against BATCH IN THE LIMS (sample prep, prep code, date/time, technician, check sample IDs, check volumes) or Printed COPY OF THE BATCH

Table 7-7
3050B_X Prep Checklist
3050B_X

1) For Samples that are a soil/oil waste mixture or appear to be 90 to 100% Oily matrix:

___ : Print the backlog reports for **3050B_X** and **6010B_X**
-check for comments from Project manager

___ : Pick a sample for QC (original, MS/MSD) and homogenize sample

___ : Put samples in order and check prep and test info (QC then order of backlog). Use crucible for ashing sample. Record the sample ID on the crucible bottom using pencil, not Sharpie. Using a Sharpie marker can burn off during ashing. Sometimes pencil will also burn off. Draw diagram of samples to help with identification of samples.

___ : Add 1g to the crucible and record the measurement

For the sample chosen for MS/MSD, the initial weights used for the un-spiked sample, matrix spike and matrix spike duplicate must be within 5% of each other.

___ : Ash the sample by placing into a cold muffle furnace and set temperature to 300-350°C. Let bake for 3-4 hrs. or until sample is fully turned to ash. *** WARNING**- Do NOT open oven if smoke is coming out!

___ : Remove with tongs, let cool

___ : Add **2.0 mL of conc. HNO_3 and 1.5 mL conc. HCl**, cover the crucible with a watch glass, then heat for 30 min.

___ : Remove from heat and let cool. Transfer to a plastic digestion vessel, rinse crucible 2-3 times with DI Water.

___ : Add **2-4 mL of conc. HNO_3 and 3-5 ml conc. HCl**. Cover the crucible with a watch glass, and heat again until ash is dissolved. NOTE: The process may have to be repeated until ash is dissolved. Record the total amount of conc. HNO_3 and HCl used to dissolve sample ash.

___ : Prepare clean, snap cap vials for MB and LCS.

___ : Spike LCS, MS, MSD with 0.5 mL of multi-element Standard (need witness).

___ : Add the same amount of conc. HNO_3 and conc. HCl to MB and LCS that was required to dissolve sample ash.

___ : Digest in hot block ~ 30 to 45 minutes.

___ : Remove from heat and let cool. Bring samples to a final volume of 50 mL with DI Water.

2) For Samples that are a liquid waste matrix containing an oily layer:

___ : Print the backlog reports for **3050B_X** and **6010B_X**
-check for comments from Project manager

___ : Pick a sample for QC (original, MS/MSD) and homogenize sample

___ : Put samples in order and check prep and test info (QC then order of backlog)

___ : Add 1g to a digestion vessel and record the weight in the logbook.

For the sample chosen for MS/MSD, the initial weights used for the un-spiked sample, matrix spike and matrix spike duplicate must be within 5% of each other.

____ : SPIKE LCS, MS, & MSD with 0.5 mL of multi-element standard. (WITNESS NEEDED)

____ : ADD 2.0 mL conc. HNO_3, COVER THE VESSELS WITH WATCH GLASSES, THEN HEAT FOR 10 MIN

____ : ADD 4.0 mL conc. HNO_3, THEN HEAT FOR 30-35 MIN

____ : REMOVE from heat, let cool, ADD 1 DROPPER OF 30% H_2O_2 to remove organic material. Digest and check for reaction. Add another dropper of 30% H_2O_2. Digest again and check for reaction. If reaction is strong, then add another dropper of 30% H_2O_2. Digest until reaction stops.

____ : REMOVE FROM HEAT AND LET COOL.

NOTE: Sometimes an oily residue will be noticed in the vial. If this happens, sample will be transferred to a clean, snap cap vial prior to digestion. Filter if necessary with Whatman 40 filters.

____ : ADD 2-3 mL OF DI WATER AND 5.0 mL OF HCl.

____ : DIGEST FOR 40 MIN.

____ : REMOVE FROM HEAT AND LET COOL.

____ : BRING TO A FINAL VOLUME OF 50 mL.

Table 7-8
SAMP_FILT Prep Checklist (For Dissolved Metals)
SAMP_FILT

____ **:** PRINT (check) BACKLOG REPORT FOR THE PREP AND EACH TEST CODE
(SAMP_FILT: 6010B_W_D, 6010B_B_W_D, 6010B_PO4_W_D, 6010B_SR_W_D, 6010B_TAL_W_D, 200.7_W_D, 200.7_B_W_D, 200.7_PO4_W_D, 200.7_SI_W_D, 200.7_SR_W_D, 200.8_D, 6020_W_D, 6020_A1_W_D, 6020_A2_W_D, 6020_A9_W_D, 245.1_W_D)

____ : PICK A SAMPLE FOR QC (MS&MSD) (homogeneous is ideal)

____ : BATCH SAMPLE IN THE LIMS (sample prep, add, prep code, date/time, technician (your name), click loadsamps, select user, single arrow samples into selected column, check sample IDs, OK, add MS/MSD, check volumes). PRINT A COPY OF THE BATCH

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO. Write samples in the log book from the back log.

____ : FILTER 50 mL SAMPLE THROUGH 0.45 µm SYRINGE FILTERS TO DIGESTION VESSEL. (IF THE SAMPLE HAS A BAD MATRIX WHICH MAKES IT DIFFICULT TO FILTER, YOU CAN REDUCE THE VOLUME FILTERED to 25 mL)

____ **IF SAMPLE IS CLOUDY OR SEDIMENT IS PRESENT AFTER FILTRATION, OR IF SAMPLES CANNOT BE SYRINGE FILTERED DUE TO HIGH SOLIDS CONTENT, SEE SUPERVISOR BEFORE CONTINUING. DOCUMENT ALL SAMPLING DIFFICULTIES IN THE LOG BOOK COMMENTS.**

____ : FILTER 50 mL SAMPLE TO MS AND MSD VESSELS FOR THE QC SAMPLE.

____ : FILTER 50 mL of DI WATER THROUGH SYRINGE FILTERS to LCS & MB VESSELS.

FOR ALL 200.8 AND 6020 TEST CODE PREPS:

____ : ADD 1.0 mL OF **OMNITRACE HNO_3/ 50 mL sample** TO EACH SAMPLE AND QC AND SHAKE WELL.

____ : SPIKE LCS, MS, & MSD with 0.5 mL of **10** ppm multi-element standard and/or single element standard as required by select list (WITNESS NEEDED). **SHAKE WELL**

FOR ALL 200.7 AND 6010 TEST CODE PREPS:

____ : ADD 0.5 mL OF HNO_3 **AND** 0.5 mL HCL/ **50 mL sample** TO EACH SAMPLE AND QC AND SHAKE WELL.

____ : SPIKE LCS, MS, & MSD with 0.5 mL of **100** ppm multi-element standard and/or single element standard as required by select list (WITNESS NEEDED). **SHAKE WELL**

IF THE SAMPLES ARE FIELD FILTERED:

____ : ADD 50 mL SAMPLE TO DIGESTION VESSEL.

____ : ADD 50 mL SAMPLE TO MS AND MSD VESSELS FOR THE QC SAMPLE.

____ : ADD 50 mL Filtered DI WATER TO LCS & MB VESSELS EACH. MB AND LCS MUST BE FILTERED PRIOR TO SPIKING.

____ : ACIDIFY FOR EITHER 200.7/6010 TEST CODES OR 200.8/6020 TEST CODES AS ABOVE

____ : SPIKE LCS, MS, & MSD FOR EITHER 200.7/6010 TEST CODES OR 200.8/6020 TEST CODES AS ABOVE

____ : ***SHAKE WELL***

____ : LINE UP SAMPLES IN ORDER THAT THEY APPEAR IN LOG

Table 7-9
WIPE_MET_ICP_P Prep Checklist
WIPE_MET_ICP_P

____ **:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (6010B_WIPE).

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO.

____ : BATCH SAMPLES IN THE LIMS, PRINT BATCH.

____ : PREPARE MB BY USING GHOST WIPE.

____ : PREPARE LCS & LCSD (WITNESS NEEDED) BY USING A BLANK GHOST WIPE AND **ADDING 0.125g of Sigma Aldrich CRM Trace Metals – Loamy Clay 1 (CRM052-50G) soil reference material. Record AES number in logbook of soil reference material.**

____ : PREPARE DIGESTED CRA (**REQUIRED FOR LEAD ONLY**) (WITNESS NEEDED) BY ADDING **2.0 mL** OF **10 mg/L** LEAD CRA STANDARD TO A BLANK WIPE IN A DIGESTION TUBE.

____ : ADD 5 mL CONC. HNO_3. WIPE MUST BE COMPLETELY COVERED WITH DIGESTION SOLUTION; ADD DI WATER IF NECESSARY.

____ : HEAT FOR AT LEAST 10 MIN FOR GHOST WIPES, OR UNTIL SAMPLE HAS THE CONSISTENCY OF MASHED POTATOES, WHICHEVER IS LONGER.

____ : REMOVE FROM HEAT AND COOL FOR APPROX. 5 MINUTES.

____ : ADD 6 mL 30% H_2O_2.

____ : HEAT FOR AT LEAST 10 MIN

____ : ADD 5 mL HCl.

____ : HEAT FOR AT LEAST 10 MIN.

____ : REMOVE FROM HEAT AND LET COOL. BRING TO A FINAL VOLUME OF **100 mL.**

____ : ***SHAKE WELL***

Table 7-10
WIPE_INHOUSE_MET Prep Checklist
WIPE_INHOUSE_P

____ **:** PRINT BACKLOG REPORT FOR THE PREP AND EACH TEST CODE (WIPE_INHOUSE_MET).

____ : PUT SAMPLES IN ORDER AND CHECK PREP AND TEST INFO.

____ : BATCH SAMPLES IN THE LIMS, PRINT BATCH.

____ : PREPARE MB BY USING GHOST WIPE.

____ : PREPARE LCS & LCSD (WITNESS NEEDED) BY USING A BLANK GHOST WIPE AND **ADDING 0.125g of Sigma Aldrich CRM Trace Metals – Loamy Clay 1 (CRM052-50G) soil reference material. Record AES number in logbook of soil reference material.**

____ : ADD 5 mL CONC. HIGH PURITY HNO_3.

____ : ADD DI WATER TO COMPLETELY COVER WIPE WITH THE DIGESTION SOLUTION.

____ : HEAT FOR AT LEAST 30 MIN FOR GHOST WIPES, OR UNTIL SAMPLE HAS THE CONSISTENCY OF MASHED POTATOES, WHICHEVER IS LONGER.

____ : REMOVE FROM HEAT AND COOL FOR APPROX. 10 MINUTES.

____ : ADD 6 mL 30% H_2O_2. THEN HEAT FOR AT LEAST 20 MIN.

____ : REMOVE FROM HEAT AND COOL FOR APPROX. 10 MINUTES.

____ : ADD 5 mL CONC. HIGH PURITY HCl. THEN HEAT FOR AT LEAST 10 MIN.

____ : REMOVE FROM HEAT AND LET COOL.

____ : FILTER SAMPLE AND RINSE CONTAINER AT LEAST 3 TIMES.

____ : BRING TO A FINAL VOLUME OF **100 mL** WITH DI WATER.

____ : ***SHAKE WELL***

8.0 QUALITY ASSURANCE REQUIREMENTS

8.1 Each person using this procedure is required to comply with the formal quality control program specified by AES. The minimum requirements of this program consist of an initial demonstration of capability, and the periodic analysis of laboratory reagent blanks, fortified blanks, and other laboratory solutions as a continuing check on performance. The laboratory, through the analyst, is required to maintain performance records that define the quality of the data that are generated. Detailed quality assurance procedures can be found in SOP# QA-01000, "Quality Assurance Manual," Section 5. Subsequent sections define portions of the quality control program.

8.1.1 Demonstration of Capability (DOC). Each analyst must demonstrate proficiency for each method performed by performing Initial Demonstration of Capability (IDOC) prior to unsupervised analysis of analytical samples and Continuing Demonstration of Capability (CDOC) at least annually. Detailed descriptions of IDOC and CDOC requirements and acceptance limits can be found in Section 5 of SOP#QA-01000, "Quality Assurance Manual".

8.1.2 Lower Limit of Quantitation Check Standard (LLOQ). The laboratory should establish the LLOQ as the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. The LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be ± 35% of the true value and RSD should be ≤ 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias. Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks, Ottawa sand, diatomaceous earth, etc.) or a representative sample matrix (free of target compounds). Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

8.1.3 Instrument Detection Limits (IDLs). IDLs are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation, however, it should be understood that the lower limit of quantitation needs to be verified according to the guidance in Sec. 8.1.2. IDLs in µg/L can be estimated as the mean of the blank result plus three times the standard deviation of 10 replicate analyses of the reagent blank solution. (Use zero for the mean if the mean is negative). Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be

determined at least once using new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project. IDLs should be established at minimum on an annual basis, for each matrix type analyzed and for each preparatory/determinative method combination used. An instrument log book should be kept with the dates and information pertaining to each IDL performed.

8.1.4 Linear Dynamic Range or Upper Quantitation Limit (UQL). The linear range establishes the highest concentration that may be reported without diluting the sample. Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. The standard must recover within 10% of the true value, and if successful, establishes the linear range. The linear range standards must be analyzed in the same instrument run as the calibration they are associated with (i.e., on a daily basis) but may be analyzed anywhere within that run. If a linear range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range. Once established, any raw sample values over the established UQL must be diluted and reanalyzed or qualified as an estimated value.

8.1.5 This is accomplished is accomplished through a 5-point calibration curve (calibration blank, ICAL 1, ICAL 2, ICAL3, ICAL4, ICAL5) and a 2-point calibration curve (Li/Si only). For a curve to be acceptable the correlation coefficient must be $\geq$0.995 and the %Error must be less than 30% for the low level and 20% for all other levels. ICB (Initial Calibration Blank) is analyzed after the calibration (or after the ICV) and all target analytes must be less than the Reporting Limit. The verification of the curve is also performed using a second source standard Initial Calibration Verification (ICV/QCS), acceptance criteria $\pm$5%. Continuing Calibration Verification standards (CCVs) are analyzed to after every 10 samples and at the end of each analysis batch to check the validity of the calibration curve, acceptance criteria $\pm$10%. Sensitivity Check is accomplished through the analysis of the CRI in Section 8.1.13. CCB (Continuing Calibration Blank) is analyzed immediately after each CCV (after every 10 samples, and at the end of each analysis batch run) sample and all target analytes must be less than the Reporting Limit.

8.1.6 Initial Calibration Verification (ICV). An ICV standard must be analyzed after establishment of each calibration curve. The ICV is a standard prepared from a different source than the initial calibration standards. It is analyzed at approximately the mid-level of the calibration and serves as a check that the initial calibration standards are at the correct concentrations. The acceptance range is 90-110% of the true value.

8.1.7 Initial Calibration blank (ICB) - If a multi-level calibration is used, an ICB is analyzed immediately after the calibration (or after the ICV) and must not contain target analytes above half the LLOQ. If the ICB consistently has target analyte concentrations greater than half the LLOQ, the LLOQ should be re-evaluated.

8.1.8 Low-level verification (LLV or CRI) - For a multi-point calibration, the low level standard should quantitate to within 80-120% of the true value.

8.1.9 Mid-level verification (MLV or CRI-Mid Level) - For a multi-point calibration, the mid-level standard should quantitate to within 90-110% of the true value.

8.1.10 Spectral interference checks (SIC). Two types of SIC checks are used. Individual element SIC checks are performed when the instrument is initially setup and at least once every 6 months thereafter. The mixed element SIC solution is used daily to check that the instrument is free from interference from elements typically observed in high concentration and to check that any interference corrections applied are still valid.

8.1.10.1 Single element interference checks – Single element SIC solutions are used to evaluate possible spectral interferences. At a minimum, single element SIC checks must be performed for the 23 elements listed in Table 5-9. The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be less than two times the LLOQ. The concentration of the SIC checks becomes the highest concentration allowed in a sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements. The single element SIC checks are performed when the instrument is setup and at least once every 6 months thereafter.

8.1.10.2 Mixed element interference checks – The mixed element SIC solutions are used daily to check that the instrument is free from interference from elements typically observed in high concentration and to check that any interference corrections applied are still valid. Known and documented contaminants from the single element SIC solutions are subtracted from the observed values in the mixed element SIC check. The mixed element SIC solutions are analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than ± the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, or alternatively the LLOQ may be raised to twice the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions. These may be present up to the concentration documented plus the LLOQ.

8.1.11 Continuing Calibration Verification (CCV). Verify the ongoing validity of the calibration curve after every 10 samples, and at the end of each analysis batch run, through the analysis of a CCV standard. For the curve to be considered valid the analysis result of the CCV standard must be within ± 10% of the true value. If

the calibration cannot be verified, sample analysis must be discontinued, the cause of the problem determined, and the instrument recalibrated. All samples following the last acceptable CCV standard must be reanalyzed.

8.1.12 Continuing Calibration Blank (CCB). A CCB must be analyzed immediately after each CCV sample. Value must be less than the PQL for all target analytes. Verify the ongoing validity of the calibration curve after every 10 samples, and at the end of each analysis batch run, through the analysis of a CCB. For the curve to be considered valid the analysis result of the CCB must not contain target analytes above the LLOQ or less than Project specific requirements in LIMS.

8.1.13 Method blank. For each batch of 20 or fewer client samples analyzed, at least one method blank must be carried throughout the entire sample preparation and instrument determination process. The importance of the method blank is to aid in identifying when and/or if sample contamination is occurring. The method blank is considered to be acceptable if it does not contain the target analytes at concentration levels that exceed the LLOQ or less than Project specific requirements in LIMS. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e. targets are not present in samples or sample concentrations are ≥ 10x the blank). Other criteria may be used depending on the needs of the project. The laboratory should not subtract the results of the method blank from those of any associated samples. If the method blank fails to meet the necessary acceptance criteria, it should be reanalyzed once. If still unacceptable, then all samples associated with the method blank must be re-prepared and re-analyzed, along with all other appropriate analysis batch QC samples. If the method blank results do not meet the acceptance criteria and reanalysis is not practical, then the laboratory should report the sample results along with the method blank results, and provide a discussion of the potential impact of the contamination on the sample results. Further corrective actions for failing QC and/or acceptance criteria must be handled in accordance with Section 8.2.

8.1.14 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD). At least one LCS should be prepared and analyzed with each batch of analytical samples processed, at a minimum frequency of one LCS per every 20 samples. The LCS is spiked DI water and must be carried through all preparation and analysis steps used for analytical samples. The LCS should be spiked at the same levels and using the same spiking materials as the corresponding MS/MSD. When the results of the MS analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can acceptably perform the analysis in a clean matrix. LCS acceptance limits should be set at ± 20%. If the result of an LCS does not meet the established acceptance criteria, it should be re-analyzed once. If still unacceptable, then all samples associated with the LCS must be re-prepared and re-analyzed, along with all other appropriate analysis batch QC samples. The LCSD is only analyzed in the absence of an MSD. The recovery of the analytes must be within ± 20%. RPD value when compared to the LCS must be ≤ 20%. LCS/LCSD recovery or RPD outside control limits must be handled in accordance with Section 8.2.

8.1.15 Matrix Spike and Matrix Spike Duplicate Samples (MS/MSD). Documenting the effect of the matrix should include the analysis of at least one matrix spike/matrix spike duplicate (MS/MSD) pair for each batch of samples processed, at a minimum frequency of one pair per every 20 samples. An MS/MSD pair is used to document the bias and precision of a method in a given sample matrix. MS/MSD samples should be spiked with each target element at the project-specific action levels, or, when lacking project-specific action levels, between the low- and midlevel standards, as appropriate. MS/MSD acceptance limits should be set at ± 25% recovery and ≤ 20% RPD. If the bias and precision indicators in an analytical batch fail to meet the acceptance criteria, then the interference tests listed in Sections 8.1.16 and/or 8.1.17 should be performed. Recovery or RPD outside control limits must be handled in accordance with Section 8.2.

8.1.16 Dilution Test (DL). If the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 25 times greater than the LLOQ), an analysis of a 1:5 dilution should agree to within ± 20% of the original determination. If not, then a chemical or physical interference effect should be suspected. The matrix spike is often a good choice of sample for the dilution test, since reasonable concentrations of most analytes are present. Elements that fail the dilution test are reported as estimated values.

8.1.17 Post-Digestion Matrix Spike (PDS). If a high concentration sample is not available for performing the dilution test, then a post-digestion MS should be performed. The test only needs to be performed for the specific elements that failed original matrix spike limits, and only if the spike concentration added was greater than the concentration determined in the unspiked sample. Following preparation, which may include, but is not limited to, pre-filtration, digestion, dilution and filtration, an aliquot, or dilution thereof, should be obtained from the final aqueous, unspiked-analytical sample, and spiked with a known quantity of target elements. The spike addition should be based on the indigenous concentration of each element of interest in the sample. The recovery of the post-digestion MS should fall within a ± 25% acceptance range, relative to the known true value, or otherwise within the laboratory-derived acceptance limits. If the post-digestion MS recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values.

8.1.18 MDL Requirement. Method Detection Limit, MDL. The MDL is established according to AES MDL SOP QC-05013, current revision, which is based upon procedures outlined in 40 CFR Part 136 Appendix B Revision 2. Ongoing MDL data is collected on a quarterly basis by the analysis of a minimum of two spiked samples and two method blank samples prepared and analyzed on different calendar days for each instrument. At least once every 13 months, the Ongoing Annual Verification is performed, evaluated, and documented according to evaluation criteria outlined in AES MDL SOP QC-05013, current revision. Current laboratory MDLs are located in LIMS.

8.1.19 Interference Check Sample (ICS). The ICS is used to verify inter-element and background correction factors. Analyze the interference check sample at the beginning, end, and at periodic intervals throughout the sample run. Results

should fall within the established control limits of ± 20%. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

8.1.20 Contract Required Detection Limit (CRI). The CRI sample is designed to verify that samples with concentrations at or near the laboratory derived Reporting Limit can be readily measured on a daily basis. The results are acceptable if the measured concentration of each analyte is within the 70-130% recovery range; other range is 50-150% for Al, Sb, Ba, Ca, Fe, Pb, Mg, K, Tl.

8.2 Out of Control Conditions and Corrective Actions. Contingencies for handling out-of-control or unacceptable data are included in SOP# QA-01000, "Quality Assurance Manual," in Sections 5.8 and 13.0. Included are tables that detail corrective actions for failing QC and/or acceptance criteria.

8.3 Documentation of data. Document and record all analytical sequence, standard preparation, instrument maintenance, and any procedure deviations in appropriate logbooks.

9.0 HEALTH AND SAFETY REQUIREMENTS

9.1 Health and Safety: Safety glasses and latex type gloves must be worn at all times when dealing with any chemicals, samples, or reagents. A lab coat is also highly recommended. Close-toed shoes and clothing that covers the legs (no shorts or dresses) must be worn any time an analyst is working in the laboratory.

9.2 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a health hazard and exposure should be kept as low as reasonably possible. All health and safety concerns for any chemicals are listed in the Safety Data Sheets (SDS) provided by the supplier or manufacturer of these chemicals. A copy of any SDS is available for review at any time.

9.3 The exhaust hoods should be maintained in a clean condition. Avoid getting Kimwipes or other paper inside the hood stack. This is not only a fire hazard but can also decrease the flow within the hood.

9.4 Acids should be handled with care. Always add acids and caustic solutions to water.

10.0 DATA REPORTING

10.1 The LIMS system automatically calculates the data based upon factors that are set up for each test category.

10.2 Current laboratory MDLs and Reporting Limits (RLs) are located in LIMS.

10.3 Calculations are performed by LIMS as follows:

10.3.6 Aqueous Samples:

$$\textbf{mg/L} = \frac{\textbf{Inst. Result (µg/mL) x Final Vol. (mL) x DF}}{\textbf{Volume Digested (mL)}}$$

10.3.7 Soil Samples:

$$\textbf{mg/Kg} = \frac{\textbf{Inst. Result (µg/mL) x Final Vol. (mL) x DF}}{\textbf{Initial Sample wt (g) (x decimal \%Solids if dry wt basis)}}$$

10.3.8 Surface Wipe Samples:

$$\textbf{µg, Total} = \frac{\textbf{Inst. Result (µg/mL) x Final Vol. (mL) x DF}}{\textbf{1 (entire wipe digested)}}$$

10.3.9 Data import to LIMS for Agilent 5100:

10.3.9.1 Data export to excel spreadsheet from software.

10.3.9.1.1.1 Go to ***'File'***. Click on Export. Say Yes to Overwrite. Click on Excel spreadsheet shortcut on the taskbar. Click on the running man icon and enter test code (i.e. 6010B_W_T).

10.3.9.1.1.2 When the next screen appears, check the desired data to import. Always delete rows 2, 3, 4 and the other rows from other data that will not be imported. Verify that the sample name, sample type, and test code are correct. Change ***'Samp ID'*** column to Abbreviations (i.e. Continuing Calibration Verification= CCV) & ***'Samp Type ID'*** for each sample.

10.3.9.1.1.3 Click on the red diamond icon, click ***'Yes'*** to command.

10.3.9.1.1.4 Click 'File' and 'Save As'.

10.3.9.1.1.5 Change 'Save as type:' to 'CSV (Comma delimited)'. Change 'File name:' to 'LimsdataYYMMDD' or other appropriate name. Change 'Save in:' folder to \\Aes_server\I\import\Instrument and click 'Save'.

10.3.9.1.1.6 Close Excel & open LIMS and the AES System.

10.3.9.2 **Data Export to LIMS from Excels spreadsheet.**

10.3.9.2.1 In LIMS, click on '*Data Entry*'.

10.3.9.2.2 Click '*Add*'.

10.3.9.2.3 Fill out the following lines: 1) Instrument ID, 2) Run Start Date, 3) Analyst

10.3.9.2.4 Go to AES System and choose the 'Data Import' tab.

10.3.9.2.5 Choose InstrumentID and Import Format.

10.3.9.2.6 Click on the 'RunNo ~ RunID' matching the one you created in LIMS.

10.3.9.2.7 Click 'txt.csv Import', choose the previously saved file (\\Aes_server\l\import\Instrument\LimsdataYYMMDD), and click 'Open'. After importing has completed, you will see a list of imported samples. Click 'OK'. If there were no errors during importing, you will see the message 'Data is imported to LIMS'. Click 'OK'.

10.3.9.3 **SAVE DATA**

10.3.9.3.1 Save both raw data & run log on desktop.

10.3.9.3.2 Go to the ***'File'*** tab- ***'Report Settings'***- *Style- Rack loading guide*- ***Print*** (to Adobe PDF) - ***OK***. Save as MMDDYY A, B, C, etc. _Run Log.pdf. (Note: Include in the naming before run log Lithium, Silicon, or Inhouse if it is that type of analysis).

10.3.9.3.3 Add a note to the beginning of the Run Log including the analyst who loaded the instrument, the analyst who checked the loading list, and the MET #s of the instrument calibration and QC standards.

10.3.9.3.4 Click 'Save' to update changes and close the Run Log.

10.3.9.3.5 Go to Report Settings window- click on ***'All Data'*** from *'Style'* section- Select '**QC Solutions**' from '*Content*' section. Print (to Adobe PDF) - OK. Save as MMDDYY A, B, C, etc. _Raw Data.pdf and close (Note: Include Lithium, Silicon, or Inhouse in the naming before raw data if it is that type of analysis).

10.3.9.4 **Copy and Paste Raw Data & Run Log to Portal Folder.**

10.3.9.4.1 Click on the 'Metals on Lab' Icon on the desktop-'instrument'- Current Year- Month of Analysis.

10.3.9.4.2 Create a new folder. Rename as Instrument_YYMMDD A, B, C, etc. Copy and paste both raw data and run log into the folder.

11.0 FILE MAINTENANCE

11.1 All data are printed or scanned to pdf files and electronically stored on the Portal Server.

11.2 Electronic data is periodically transferred to a server computer and then placed into long term storage by writing the information onto portable hard drives. Two copies are made. One copy is stored on the laboratory premises, while the other copy is taken offsite by the company President.

12.0 INSTRUMENT MAINTENANCE

12.1 Instrument logbooks. Instrument logbooks must be completed any time maintenance is performed on the instrument. This includes routine maintenance such as changing or cleaning injection port liners, trimming column ends, and in the case of the GC/MS, cleaning the source. It also includes any non-routine maintenance that may be performed such as the replacement of motors in tower autosamplers.

Each instrument logbook must have a cover page that includes the following information, for example:

Equipment name. Example: GC-5
Manufacturers name. Example: Hewlett Packard 6890 GC
Serial Number. Example: 13226589A
Date Received. Example: 11/01/00
Date Placed into Service. Example: 11/05/00

12.2 Routine Maintenance: Typical routine maintenance consists of keeping the system clean and insuring that all generated QC remains acceptable.

12.2.6 Routine Maintenance of the Agilent 5100.

12.2.6.1 **VISUAL INSPECTION**
Spray Chamber, Nebulizer, Torch, Cone, Pump Tubing (sample & waste), and Exhaust. Check the maintenance logbook to verify if the tubing & torch need to be replaced. Rule of Thumb- tubing/ torch are always replaced every other day; however, they can be changed daily if the torch is dirty or tubing has been clamped overnight.

12.2.6.2 **To change the torch**:
12.2.6.3 Open plasma door.
12.2.6.4 Remove all connections from torch.
12.2.6.5 Press down & twist latch to the left.
12.2.6.6 Remove the torch towards the left. Place the torch in concentrated HCL for cleaning for a period not to exceed 24 hours. Remove from acid, rinse with deionized water, dry with paper towel, and store.
12.2.6.7 Remove the cone by unscrewing counterclockwise.
12.2.6.8 Clean cone in hot/warm tap water with scouring pad.
12.2.6.9 Dry cone with paper towel.
12.2.6.10 Put cone back first. (Note: Can only fit one way, rotate until screws fit.)
12.2.6.11 Place torch back in plasma compartment and the connections to it. The tube closest to the gas outlet is placed in the nearest hole.
12.2.6.12 Close plasma compartment.
12.2.6.13 Change sample & waste tubing. Remove one end first and replace with new tubing. Follow along the path and replace the other ends.
12.2.6.14 Clamp tubing down.
12.2.6.15 A torch alignment is performed any time the torch is replaced. Refer to section 7.3.2 for instructions on how to perform a torch alignment.
12.2.6.16 Clean nebulizer as needed by squeezing HNO_3 through it.
12.2.6.17 Spray chamber is cleaned as needed by sonicating with Triton X-100

Note: The software has a help menu that can be referred to for additional assistance.

12.3 Non-routine maintenance. This usually includes problems such as the inability to save files to disk or operational activities such as an autosampler that will not function. If this type of problem occurs, contact the department manager for assistance. Record the problem and resolution steps in the logbook.

13.0 METHOD PERFORMANCE

13.1 Method performance data can be found in the referenced methods.

13.2 Laboratory specific method performance data is referenced in Section 17 of this SOP.

14.0 POLLUTION MANAGEMENT

14.1 All laboratory analysis generates wastes. Some wastes can be hazardous such as acidic wastes, alkaline wastes, metal bearing wastes, and organic wastes.

14.2 Some wastes are generated because of the test procedure such as organic extractions and acid digestions.

14.3 The following procedures should be adhered to when disposing of hazardous wastes.

14.3.6 Wastes with pH levels above 12 or less than 4 should be neutralized prior to disposal.

14.3.7 Wastes with other pH levels may be directly discharged into the sinks.

14.3.8 SOP# HS-03005 Waste Disposal and SOP# SR-09002 Sample Receiving further discuss methods for disposal of samples and waste materials.

14.4 When disposing of laboratory wastes, the waste disposal log must be completed. To complete this log, supply the following information.

Sample Number
Method of disposal and treatment prior to disposal
Date of sample disposal
Name of person performing the disposal duty

15.0 DEFINITIONS

15.1 Primary Grade –A dry chemical that has been dried at 250°C for 4 hours cooled and stored in a desiccator.

15.2 LCS - Laboratory Control Sample. A known amount of sought for analyte is added to distilled water or clean soil and the concentration measured after all procedures are applied to the sample. The resulting determined concentration must fall within test specified limits.

15.3 DI water- Deionized water

15.4 RSD – Relative Standard Deviation

15.5 MS- Matrix Spike. Procedure where a known amount of sought for analyte is added to a sample and the resulting concentration measured. The recovery is defined as the measured result of the spiked sample less the concentration of the same analyte in the unspiked sample multiplied by 100 percent.

15.6 MSD- Matrix Spike Duplicate.

15.7 CCV - Continuing calibration verification standard.

15.8 ICV – Initial calibration verification standard. This standard must be prepared from a second source than that used for the calibration curve. That is, it must be from a different manufacturer or lot than the calibration standard.

15.9 LCSD - Laboratory Control Sample Duplicate

15.10 Dissolved - Those elements which will pass thorough a 0.45 µm membrane filter.

15.11 Total Recoverable or Total Metals – The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

15.12 Spectral Interference Check (SIC) Samples **–** Formerly known as interference check standards ICSA and ICSAB. These are solutions containing high concentrations of interfering elements that can be used to verify the absence of spectral interferences.

15.13 Linear Dynamic Range – The concentration range over which the analytical curve remains linear.

15.14 Interelement Correction Factor (IEC) – The mathematical formula for the correction of a positive or negative bias on a target analyte due to spectral overlap.

15.15 Reagent blank - A volume of deionized water containing the same acid matrix as the calibration standards carried through the entire analytical scheme.

15.16 Calibration blank - A volume of deionized water acidified with HNO_3 and HCl.

15.17 Quality control sample - A solution obtained from an outside source having known concentration values to be used to verify the calibration standards.

15.18 Calibration standards - A series of known standard solutions used by the analyst for calibration of the instrument (i.e. preparation of the analytical curve).

15.19 DOC – Demonstration of capability. Can be initial demonstration of capability (IDOC) or continuing demonstration of capability (CDOC).

15.20 IDP – Initial demonstration of performance. See DOC.

15.21 LLV – Low-level verification standard.

15.22 MLV – Mid-level verification standard.

16.0 REFERENCES

16.1 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 6010D, Revision 5, July 2018.

16.2 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3010A, Revision 1, July 1992.

16.3 Test Methods for the Evaluating Solid Waste, Physical Chemical Methods, Method 3010A and 3050B, Revision 2, December 1996.

16.4 NIOSH Manual of Analytical Methods, Fourth Edition, Method 7303, http://www.cdc.gov/niosh/docs/2003-154/default.html.

16.5 LabNotes Volume 26 Number 2 December 2015, Newsletter of the Laboratory Certification Program, Wisconsin DNR Laboratory Certification Program, Rick Mealy Editor, http://dnr.wi.gov/regulations/labcert/.

17.0 VALIDATION DATA

17.1 Method validation data in the form of MDL study data when applicable is available at AES Portal Server: http://portal/Quality Assurance/**MDL.**

17.2 Method validation data in the form of IDOC/CDOC study data when applicable is available at AES Portal Server: http://portal/Technical Management/**Demonstrations of Capability and SOP Sign Forms.**

18.0 SOP REVISION HISTORY

Revision Date	Revision #	Summary of and Reason for Changes/Updates	Responsible for Revision
1/20/2003	7	Update	Greg Jones
12/16/2005	8	Update	Greg Jones
10/6/2008	9A	SC/MUR Update	Dana Till
9/9/2009	9	Revision number was not updated with the revision. Labeled previous revision, 10/6/2008, as Revision 9A for clarification. Update to Sections 3.1.8, Tables 7-3 through 7-8, 8.1.6, 8.1.8, 8.1.9, 8.1.11.1, and 8.1.11.3.	Dana Till
5/25/2016	10	Updates to Sections 1.1, 3.1, 3.1.3, 3.1.7, 3.2.1, 3.4.2, 3.4.3, 3.5, 5.5, 5.12, 5.13, 5.14, 5.15, 5.16, 6.1, 7.1.1, 7.1.1.3, 7.3.1, 8.1.2	Melanie Riffle

		through 8.1.17, 10.2, 10.3.4, 15.12, 15.19, 15.20, 15.21, 15.22, and 16.0. Updates to Tables 3-2, 5-1, 5-2, 5-3, 5.4, 5-5, 5-6, 5-8, 5-9, 5-10, 5-11, 7-1, 7-2, 7-3, 7-4, 7-5, 7-6, 7-8, and 7-9.	
7/31/2018	11	Updates to Sections 8.1.18, 8.2, and Table 7-10.	Dana Till
12/18/2020	12	Updates to Sections 5.10, 5.11, 5.12, 513, 5.14.1.2, 5.15.1, 5.16, 517..1, 5.17.1.7, 5.17.2, 8.1.5, 8.1.9, Table 5-6, Table 5-7, Table 5-9, Table 5-11, Table 5-12, Table 5-13, Table 5-14, Table 5-15, and Table 7-10. (NELAC assessment)	Tanyika Allen Dana Till
6/30/2022	13	Updates to Sections 5.7, 5.8, 5.11, 5.12, 5.13, 5.14, 5.15, 5.16.1, 5.16.2, 5.16.3, 5.17.1, 5.17.2, 5.17.3, 5.17.4, 5.19.1, 5.21, 8.1.5, and Tables 5-3, 5-4, 5-6, 5-7, 5-10A, 5-10B, 5-11, 5-12, 5-13, 5-14, 7-9, 7-10.	Tanyika Allen Dana Till
9/9/2022	14	NELAC Assessment Updates to Sections	Tanyika Allen Dana Till
10/9/2023	15	SC Renewal update to Sections 5.21 and 5.21.3. Editorial update to 16.1 on 11/6/2023.	Dana Till

APPENDIX I
EXAMPLE DATA REVIEW CHECKLIST ICP-OES SW6010D

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ICP-OES 6010 DATA REVIEW CHECKLIST

Batch ID:_____ LIMS Run ID:_____

QA ANALYST

INSTRUMENT STARTUP AND CALIBRATION

NA ___ : Perform instrument maintenance per maintenance schedule.
NA ___ : Fill out instrument maintenance logbook daily.
___ ___ : Print run log and have another analyst check racks and signoff (racks **MUST** be checked before any data is reported.
NA ___ : Let instrument warm-up for 30 minutes
___ ___ : Calibrate instrument per SOP
NA ___ : While instrument is calibrating, review data not processed from the previous day.
NA ___ : Gather all samples requiring dilution or reanalysis.
NA ___ : Under the Backlog Report tab, get and review the backlog by clicking on the "Backlog Report" button.
NA ___ : Review the Backlog Report, paying attention to TAT and due dates.
___ ___ : After calibration is complete, evaluate the calibration.
___ ___ : Evaluate CRIs. Make sure all analytes and %RSDs pass on the regular CRI. Make sure As and its %RSD passes on the CRI-5. Make sure all analytes for the CRI-Mid Level passes within 10% of the true value.
___ ___ : Evaluate ICSA. Recoveries for spiked analytes must pass within 30% of the true value. All unspiked analytes must be between ± the PQL.
___ ___ : Evaluate ICSAB. Recoveries for spiked analytes must pass within 20% of the true value for all regular metals and 30% of the true value for all minerals.
___ ___ : Evaluate LCRs. Recoveries must pass within 10% of the true value.

GETTING SAMPLES

___ ___ : Print the Prep Batch Report that corresponds to the appropriate batch to retrieve.
___ ___ : Make sure all digestate vessels present are on the Prep Batch Report and are recorded in the Sample Prep Logbook. If any digestate is missing, or a sample is not recorded in the Logbook or listed on the Prep Batch Report, then let the Prep Supervisor, Section Supervisor, and/or Department Manager know.
___ ___ : Compare information on printed Prep Batch Report to Sample Prep Logbook to make sure information is complete and consistent.
___ ___ : Check that Sample IDs on digestate vessels match those in the Sample Prep Logbook and the Prep Batch Report.
___ ___ : Check that Sample Prep Logbook page is completely filled out. **Pay attention to missing information such as Spike witness, Temperature, Time, Acid ID Numbers, etc.**
___ ___ : Check that Final and Initial Volumes are entered correctly. **NOTE: If default volumes are not used, make sure there is an explanation in the comment section of the Prep logbook and in the comment section of the prep batch report in LIMS.**
___ ___ : Perform a visual observation of samples. Make note of any abnormalities including color and presence of solids, etc. Make sure they are recorded in comment section of logbook and Prep Batch Report.
___ ___ : Check to make sure there are no write-overs on the Prep Logbook page. All mistakes must have only one line drawn through them, with the date and initials of the person correcting the mistake beside the cross out.
___ ___ : Sign and Date Prep Logbook Page.
___ ___ : Scan Prep Logbook Page and Prep Batch Report into one pdf file.
___ ___ : Save pdf file as Prep Batch Number_Test Method. If the Prep Logbook Page being scanned is for Add-on samples, **DO NOT SAVE OVER** the original pdf file. Name the new pdf file as Prep Batch Number_Test Method-2, and so forth for each additional scan.
___ ___ : Place pdf file in the Metals-on-Lab -> 4-Logbook Pages folder.

ICP-OES 6010 Data Review Checklist_Rev0_2019.doc
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LOADING SAMPLES

___ ___ : After instrument passes all calibration QC requirements, load samples that require dilution or reanalysis from previous runs
___ ___ : Before a new batch is loaded, use the AES system to perform the following initial checks
___ ___ : Under the Batch-Data Review screen, enter in the BatchID and click on "Get Data" button.
___ ___ : Make sure that the pop-up screen says "Prep dates are OK". If anything else appears, then investigate the cause, then have the Prep Supervisor, the Section Supervisor, or the Department Manager fix the issue.
___ ___ : Review the "Comments" and "Comments1" columns for any issues of concern (i.e. bad sample matrix or PQL changes).
___ ___ : Review the "ClientSampID" column for any Blank samples (ie Field Blank, Trip Blank, Equipment Blank, Rinse Blank, Media Blank, Matrix Blank, or Blank). Make note of any blanks for review during data review.
___ ___ : Under the Sample Info tab, make note of all Testcodes selected for the samples within the Batch.
___ ___ : Under the Select List tab, make note of all analytes selected for the samples under each testcode.
NOTE: for TAL testcodes and testcodes with only one analyte, the select list may not be present; therefore, the testcode/select list will not show up under the Select List tab. The testcode will only show up under the Sample Info tab. This is because when these testcodes are selected, every analyte in its list needs to be reported.
___ ___ : Under the MDL/PQL check tab, select the appropriate testcode from the drop down menu.
___ ___ : Then click on the "PQL Check" button, and check for and make note of any PQL changes for the appropriate samples.
___ ___ : Repeat the MDL/PQL check process for each testcode in the batch.
___ ___ : Then check the CRIs in the daily calibration to make sure all analytes and %RSDs for the lowered PQLs pass. **DO NOT LOAD SAMPLES ON THE INSTRUMENT UNTIL ALL OF THE APPROPRIATE CRIs PASS.**
___ ___ : Type in the loading list for all batches to be analyzed. Begin with batches that have rush samples due that day, then batches with late samples, and lastly batches with standard turnaround samples.
___ ___ : Print out loading list run log. Get loading list checked before exporting/importing any samples. Checked loading list must be signed and dated by the person checking the loading list.
___ ___ : Scan and upload checked and signed loading list run log to the Run Log folder.
___ ___ : During the workday, evaluate CCV/CCBs as they are being analyzed. **DO NOT LOAD SAMPLES ON THE INSTRUMENT IF THE CCV/CCBs ARE FAILING FOR REQUIRED ANALYTES.**

EXPORTING/IMPORTING DATA

___ ___ : Scan and upload checked and signed loading list run log to the Run Log folder.
___ ___ : Print out raw data to pdf.
___ ___ : Add note for standards information, MET numbers, who loaded samples, and who checked the loading list to the raw data pdf.
___ ___ : Create a run file in LIMS.
___ ___ : Upload the raw data pdf to the appropriate run folder.
___ ___ : Under the Batch-Data Review screen, enter in the BatchID and click on "Get Data" button.
___ ___ : Make sure that the pop-up screen says "Prep dates are OK". If anything else appears, then investigate the cause, then have the Prep Supervisor, the Section Supervisor, or the Department Manager fix the issue.
___ ___ : Export data from the instrument.
___ ___ : Open the Data_Prep excel file, and set up the batch of samples per Method requirements and Company policy.
___ ___ : Make sure all LCRs are imported with each batch run.
___ ___ : Make sure closing QC (ICSA, ICSAB, CCV, CCB for all Air, paint and wipe test codes and CCV, CCB for all 6010 method test codes) is imported.
___ ___ : Export data into LIMS using the AES New System: Data Import tab.

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LIMS "MAIN" RUN SCREEN

___ ___ : Double click each SampleID to verify that all Sample IDS and Test Codes have been properly assigned per Backlog Report.
___ ___ : Check that all instrument QC has been run at the required frequencies.
___ ___ : Check that all Sample Types have been properly assigned.
___ ___ : Check that all samples are linked to the correct Prep Batch by comparing the batch number to what is listed in the "Batch ID" column.
___ ___ : Check the Analysis Date/Time column for any samples that were analyzed at midnight. **NOTE: LIMS does not recognize any samples that have an analysis time of 12:00 am. LIMS removes the time; therefore, the time has to be manually updated to include the analysis time down to the seconds based on the raw data.**
___ ___ : Make sure all dilution factors have been entered in the "DF" column. Make sure to avoid entering dilution factors in the "DLNo" column.
___ ___ : Enter in all the Blank references for the initial instrument QC.
___ ___ : Enter in all the Blank references for the CCVs.
___ ___ : Enter in all the Blank references for the closing instrument QC.
___ ___ : Make sure that the Blank references for all samples and Batch QC is correct.
___ ___ : Make sure that the Spike references for all Batch QC is correct.
___ ___ : Make sure that the RPD references for all Batch QC is correct.
___ ___ : Make sure that the Spike and RPD references for all Batch QC that was diluted is correct.

LIMS "DATA" SCREEN

___ ___ : In LIMS, click on the "Calc SEQ" button.
___ ___ : Under the Run-Data Review screen, enter in the RunNo and click on the "Get Data" button.
___ ___ : Under the "Select List check" tab, click on the "Merge Rpt" button. **NOTE: If any samples are in the batch but not uploaded in the run yet or is in the run but does not require that run's test code, then all of the analytes will show up as being selected. In either of these two instances, the select list must be manually merged together by using the "Select List" tab under the Batch-Data Review screen.**
___ ___ : Under the "Rec/Qual" tab, click on the "Select All" button. **NOTE: QCs are the default samples to be updated. If any of the client samples need to be update, then click on the "Samples" button to select them.**
___ ___ : Under the "Rec/Qual" tab, click on the "Update checked QCs according to Merged rpt" button. **NOTE: If samples in the run have already been QA'ed, then their select lists will not be updated. The samples have to be un-QA'ed for the select lists to be updated.**
___ ___ : Under the "RawVal/PQL" tab, click on the appropriate button to select each successive set of 10 samples. Any samples pass the 60th will have to be manually selected. **NOTE: Only 10 samples can be selected at a time, whether automatically or manually selected.**
___ ___ : Once a set of 10 samples have been select, then click on the "Show Checked Seq" button.
___ ___ : Scroll across the screen to check each sample to see if any of the analytes are more negative than the PQL. **NOTE: The word "negative" will appear in red for any analyte that is more negative than the PQL. However, the ICB, CCBs, ICSAs, and MBLKs will have to be manually checked for any analytes that have lowered PQLs.**
___ ___ : If the run is a water batch (excluding TCLP and SPLPs), then the total results must be compared to the dissolved results. The results check can be performed on either the dissolved run or the total run.
___ ___ : Under the "T/D" tab, select each sample to check individually. **NOTE: Only one sample/analysis can be selected to compare at a time. The results for the last sample checked in the column is what will appear. For example, if 10 samples are in the run. If you want to compare sample three's results, then samples four through 10 cannot be selected.**
___ ___ : Once a sample is selected, click on the "Find Total/Dissolved Samples" button.
___ ___ : If the sample does not require both total and dissolved analyses, then the test code and SampID fields will appear/remain empty.

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___ ___ : If the sample requires both total and dissolved analyses, then the counterpart test code and SampID will appear in the appropriate fields.
___ ___ : Click on the "Find Run/Sequence" button.
___ ___ : If the counterpart test code has not been uploaded into LIMS, then only one SeqNo and RunNo line will appear. This is the SeqNo and RunNo for the sample that you have selected.
___ ___ : If the counterpart test code has been uploaded into LIMS, then more than one SeqNo and RunNo line will appear. This is the SeqNo and RunNo for the sample that you have selected and all of the analyses of its counterpart testcode that have been uploaded into LIMS.
___ ___ : Click on the "Show Checked Sequences" button to show the results of the total and dissolved samples. NOTE: The sample/analysis originally selected will be the first "CalcVal Seq" column results. Its counterpart testcode analyses will appear next.
___ ___ : The analytes selected to report will have an "x" next to them. Compare results of the total and dissolved samples. The total results should be higher than the dissolved results.
___ ___ : If the dissolved results are higher than the total results, then repour and reanalyze both digestates under the same calibration.
___ ___ : If the dissolved results are still higher than the total results, then reanalyze both samples directly from the bottle under the same calibration.
___ ___ : If the dissolved results are still higher than the total results, then the results have been confirmed, and a comment needs to be added to both analytical runs.
___ ___ : In LIMS, under the drop-down menu, select "Rpt".
___ ___ : Check for any S or B flags on the CKSTD, ICV, CRI, ICSAB (except minerals), CRI-2, CRI-5, CRI-MID LEVEL, LCR, LCR-2, LCR-3, LCR-Pb, CCVs, LCS/LCSD, MS/MSD, PDS. If there are any flags, then investigate the possible causes. Look out for the wrong samp type being select, the wrong Blk, Spk, and/or RPD reference number entered, the wrong Batch number entered, and/or the wrong Spk amount entered or used.
___ ___ : Check for any R flags on the LCSD, MSD, DL, and DUP (when present).
___ ___ : Check for any H flags on the samples and/or Batch QC. NOTE: Determine whether the H-flag is valid. Look out for missing collection date/time, incorrect collection date/time, incorrect prep start/end date, incorrect analysis date, and/or mistaken H-flag in the prep batch's "PQual" column.
___ ___ : Check for any E flags on the Batch QC and/or samples, and/or if the samples are over the daily LCRs. If samples are E-flagged but less than the LCR, then adjust the high limit accordingly under the "UQL" column. If samples are greater than the daily LCRs, then turn off the analyte and dilute the sample and/or Batch QC. NOTE: E-flags on Instrument QC is fine.
___ ___ : Check for and remove any * flags on the instrument or Batch QC.
___ ___ : Check for and remove any * flags on client samples that also have J flags. If the client sample doesn't have both flags, then do not remove the * flag.
___ ___ : Check %RSDs for every target analyte of every sample that is above the PQL. If no target analyte is above the PQL, then check the %RSDs on the minerals (only check values that are above 1.0 mg/L) to verify that the sample was actually analyzed. If the %RSDs are greater than 15% for Air, Paint, and Wipe samples and 20% for all other samples, then reanalyzed the sample.
___ ___ : Check at least two analytes for two different samples to verify that readings have been properly entered/downloaded. **NOTE: If values are manually entered, then all analytes need to be verified.**
___ ___ : Verify at least two different samples' final results by hand calculation.

Aqueous Samples: $\text{mg/L} = \frac{\text{Inst. Result (µg/mL) x Final Vol. (mL) x DF}}{\text{Volume Digested (mL)}}$

Soil Samples: $\text{mg/Kg} = \frac{\text{Inst. Result (µg/mL) x Final Vol. (mL) x DF}}{\text{Initial Sample wt (g) (x decimal \%Solids if dry wt basis)}}$

Wipe Samples: $\text{µg, Total} = \frac{\text{Inst. Result (µg/mL) x Final Vol. (mL) x DF}}{\text{1 (or dimensions used)}}$

Paint Samples: $\text{wt\%} = \frac{\text{Inst. Result (µg/mL) x Final Vol. (mL) x DF}}{\text{Initial Sample wt (g)}}$

Air Samples: $\text{µg, Total} = \text{Inst. Result (µg/mL) x Final Vol. (mL) x DF}$

AES, Inc.
3080 Presidential Drive
Atlanta, GA 30340

1 (entire filter digested)

___ ___ : Using the AES New System: DataControl tab, check for any samples that may be double-reporting.
___ ___ : Under the InstrumentID drop-down menu, select the appropriate instrument.
___ ___ : Select the appropriate RunNo ~ RunID.
___ ___ : If any samples have been downloaded into LIMS more than once, then the sampleID will appear blue in color.
___ ___ : If any samples have been downloaded into LIMS more than once and are double-reporting, then the SeqNo will appear red in color.
___ ___ : Click on the blank column next to the appropriate sample.
___ ___ : Click on the Duplicate control button to view the sample results.
___ ___ : Note which analytes are double-reporting and correct them.
___ ___ : Compare all results to make sure that values match/are similar between any original and reanalysis and/or dilutions.
___ ___ : Write CARs for all S, R, B, and H flags and all ERLs.
___ ___ : Upload run to the portal server using the Data Review System for Data Inserters.
___ ___ : Click off run to allow for the run to be properly validated.
___ NA : Close CARs and/or narrate workorders for all S, R, B, and H flags and all ERLs.
___ NA : Notify PM, Lab Manager, PM Director, Department Manager, and Section Supervisor of any CARs resulting in re-extract and/or due date exceedance.

Analyst's Signature: _____

ICP-OES 6010 Data Review Checklist_Rev0_2019.doc
Page 5 of 5

Appendix 5: Analytical Reports

ACCESS ANALYTICAL, INC.

ANALYTICAL REPORT

CLIENT

SCDHEC
2600 Bull Street
Columbia SC 29201

ATTENTION
Taylor Shearer

PROJECT ID
Gills Creek Watershed Lead in Fish Study

LABORATORY REPORT NUMBER
2405X07

DATE
June 05, 2024

Primary Data Review By

Chris Pafford
Project Manager, EETSE-Atlanta

Secondary Data Review By

Ashley Amick
Project Manager, Access Analytical
aamick@axs-inc.com

PLEASE NOTE:

- **Unless otherwise noted, all analysis on this report performed at Eurofins Environment Testing Southeast-Atlanta, LLC (EETSE-Atlanta), 3080 Presidential Drive, Atlanta, GA 30340.**
- **EETSE-Atlanta is SCDHEC certified laboratory # 98016, NCDENR certified lab # 562, GA certified lab # FL-E87582, NELAP certified laboratory # E87582**
- **AIHA-LAP,LLC Laboratory ID:100671 for Industrial Hygiene samples (Organics, Metals, PCM Asbestos, Gravimetric), Environmental Lead (Paint, Soil, Dust Wipes, Air), and Environmental Microbiology (Fungal) Direct Examination.**
- Local support services for this project are provided by Access Analytical, Inc. All questions regarding this report should be directed to your local Access Analytical representative at 803.781.4243 or toll fee at 883.315.4243

Access Lab Report #: _____

Sub Lab (if applicable): _____ / Sub Report #: _____

ACCESS ANALYTICAL, INC.

Access Analytical, Inc. -- Irmo
15 Thames Valley Rd. ~ Irmo, SC 29063
Phone: 803-781-4243 / Fax: 803-781-4303 / Web: www.axs-inc.com
SCDHEC Lab Certification # 32571 / NELAP # E871145

Chain of Custody Record

2405x07

Client:	SCDHEC			Preservatives (see codes):	0		
Attn:	Taylor Shearer			Bottle Types (see codes):	P		
Address:	2600 Bull Street						
City:	Columbia	State:	SC	29201			
Phone:	803-898-1538	Fax:					
Email:	shearetv@dhec.sc.gov						
Project Name:	Gills Creek Watershed Lead in Fish Study						
Sampled By (Signature):	Taylor Shearer						

Preservation Codes / Bottle Types:

***Preservative Codes:**
0 = None, **1** = HCL, **2** = HNO3, **3** = H_2SO_4, **4** = NaOH, **5** = $Na_2S_2O_3$, **6** = Method 5035 set w/ $NaHSO_4$ & CH_3OH, **7** = NaOH/ZnOAC, **8** = H_3PO_4, **9** = cooled to ≤6°C, **10** = cooled to ≤10°C, **11** = Amm.Cl, **12** = Ascorbic Acid / HCL, **13** = EDA

***Matrix Codes:**
GW = ground water, **WW** = waste water, **DW** = drinking water, **SW** = surface/storm water, **S** = soil, **SL** = sludge, **A** = air, **IW** = industrial waste, **O** = other (specify in comments section)

***Program Area Codes:**
CWA = Clean Water Act (for wastewaters), **SDWA** = Safe Drinking Water Act (for drinking water), **SHW** = Solid and Hazardous Wastes (for soils, ground waters and waste samples)

***Container Type:** G = Glass, P = Plastic

Lab ID:	Sample Name:	Date Collected:	Time Collected:	G=Grab C=Comp	Matrix (see codes)	Program Area (see codes)	Total # Containers	† REQUESTED LAB ANALYSIS: † Total Lead in Tissue	Notes / Comments
	FF-01	05-13-24	0700	G	O*	n/a	1	# Containers per Test >> 1	*Fish Tissue Samples
	FF-02	05-13-24	0700	G	O*	n/a	1	# Containers per Test >> 1	
	FF-03	05-13-24	0700	G	O*	n/a	1	# Containers per Test >> 1	
	FF-04	05-13-24	0700	G	O*	n/a	1	# Containers per Test >> 1	
	FF-05	05-13-24	0700	G	O*	n/a	1	# Containers per Test >> 1	
	FF-06	05-13-24	0700	G	O*	n/a	1	# Containers per Test >> 1	
	FF-07	05-13-24	0700	G	O*	n/a	1	# Containers per Test >> 1	
	FF-08	05-13-24	0800	G	O*	n/a	1	# Containers per Test >> 1	
	FF-09	05-13-24	0800	G	O*	n/a	1	# Containers per Test >> 1	
	FF-10	05-13-24	0800	G	O*	n/a	1	# Containers per Test >> 1	
	FF-11	05-13-24	0800	G	O*	n/a	1	# Containers per Test >> 1	
	FF-12	05-13-24	0800	G	O*	n/a	1	# Containers per Test >> 1	
	FF-13	05-13-24	0800	G	O*	n/a	1	# Containers per Test >> 1	

Turnaround Time Requested:		Project Location:		Relinquished By:	Received By:	Date:	Time (24hr):	Samples Received on Ice:
Standard		SC	X	Taylor Shearer	Krystan Sanders	5/29/24	1038	___Y ___N ___N/A
Rush *		NC		Krystan Sanders		5/29	5:11PM	___Y ___N ___N/A
*Date Required		Other (Specify):						___Y ___N ___N/A
Rush data emailed/faxed by end of business day on date required. Standard TAT is 7-10 business days.				Received in lab by:		5/29	9:36PM	___Y ___N ___N/A

Chain of Custody Page 1 of 4

Sample Temp. Upon Receipt in Lab: 1.8 (°C)

White Copy: Lab original / Canary Copy: File Copy / Pink Copy: Client Copy

NOTE: Relinquishing samples via this Chain of Custody document constitutes client acceptance of Access Analytical terms and conditions.

Access Lab Report #: _____

Sub Lab (if applicable): _____ / Sub Report #: _____

Access Analytical, Inc.

Access Analytical, Inc. -- Irmo
15 Thames Valley Rd. ~ Irmo, SC 29063
Phone: 803-781-4243 / Fax: 803-781-4303 / Web: www.axs-inc.com
SCDHEC Lab Certification # 32571 / NELAP # E871145

Chain of Custody Record

2405 X07

Client:	SCDHEC
Attn:	Taylor Shearer
Address:	2600 Bull Street
City:	Columbia
State:	SC
	29201
Phone:	803-898-1538
Fax:	
Email:	sheartv@dhec.sc.gov
Project Name:	Gills Creek Watershed Lead in Fish Study
Sampled By (Signature):	Taylor

Preservatives (see codes): 0

Bottle Types (see codes): P

t REQUESTED LAB ANALYSIS: t

Total Lead in Tissue

Preservation Codes / Bottle Types:

***Preservative Codes:**
0 = None, **1** = HCL, **2** = HNO3, **3** = H_2SO_4, **4** = NaOH, **5** = $Na_2S_2O_3$, **6** = Method 5035 set w/ $NaHSO_4$ & CH_3OH, **7** = NaOH/ZnOAC, **8** = H_3PO_4, **9** = cooled to ≤6°C, **10** = cooled to ≤10°C, **11** = Amm.Cl, **12** = Ascorbic Acid / HCL, **13** = EDA

***Matrix Codes:**
GW = ground water, **WW** = waste water, **DW** = drinking water, **SW** = surface/storm water, **S** = soil, **SL** = sludge, **A** = air, **IW** = industrial waste, **O** = other (specify in comments section)

***Program Area Codes:**
CWA = Clean Water Act (for wastewaters), **SDWA** = Safe Drinking Water Act (for drinking water), **SHW** = Solid and Hazardous Wastes (for soils, ground waters and waste samples)

***Container Type:** G = Glass, P = Plastic

Lab ID:	Sample Name:	Date Collected:	Time Collected:	G=Grab C=Comp	Matrix (see codes)	Program Area (see codes)	Total # Containers	# Containers per Test >>	Total Lead in Tissue	Notes / Comments
	FF-14	05-13-24	0800	G	O*	n/a	1	# Containers per Test >>	1	*Fish Tissue Samples
	FF-15	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FF-16	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FF-17	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FF-18	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FF-19	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FF-20	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	

Turnaround Time Requested:		Project Location:		Relinquished By:	Received By:	Date:	Time (24hr):	Samples Received on Ice:
Standard		SC	X	Taylor	Kristian Anderson	5/24	1038	___Y ___N ___N/A
Rush *		NC		Kristian Anderson		5/29	5:11PM	___Y ___N ___N/A
*Date Required		Other (Specify):						___Y ___N ___N/A
Rush data emailed/faxed by end of business day on date required. Standard TAT is 7-10 business days.				Received in lab by:	courier	5/29	9:36PM	___Y ___N ___N/A

Chain of Custody Page 2 of 4

Sample Temp. Upon Receipt in Lab: 1.8 (°C)

White Copy: Lab original / Canary Copy: File Copy / Pink Copy: Client Copy

NOTE: Relinquishing samples via this Chain of Custody document constitutes client acceptance of Access Analytical terms and conditions.

Access Lab Report #: _____

Sub Lab (if applicable): _____ / Sub Report #: _____

ACCESS ANALYTICAL, INC.

Access Analytical, Inc. -- Irmo
15 Thames Valley Rd. ~ Irmo, SC 29063
Phone: 803-781-4243 / Fax: 803-781-4303 / Web: www.axs-inc.com
SCDHEC Lab Certification # 32571 / NELAP # E871145

Chain of Custody Record

2405X07

Client:	SCDHEC			Preservatives (see codes):	0		
Attn:	Taylor Shearer			Bottle Types (see codes):	P		
Address:	2600 Bull Street						
City:	Columbia	State:	SC	29201			
Phone:	803-898-1538	Fax:					
Email:	shearetv@dhec.sc.gov						
Project Name:	Gills Creek Watershed Lead in Fish Study						
Sampled By (Signature):							

Preservation Codes / Bottle Types:

***Preservative Codes:**
0 = None, **1** = HCL, **2** = HNO3, **3** = H_2SO_4, **4** = NaOH, **5** = $Na_2S_2O_3$, **6** = Method 5035 set w/ $NaHSO_4$ & CH_3OH, **7** = NaOH/ZnOAC, **8** = H_3PO_4, **9** = cooled to ≤6°C, **10** = cooled to ≤10°C, **11** = Amm.Cl, **12** = Ascorbic Acid / HCL, **13** = EDA

***Matrix Codes:**
GW = ground water, **WW** = waste water, **DW** = drinking water, **SW** = surface/storm water, **S** = soil, **SL** = sludge, **A** = air, **IW** = industrial waste, **O** = other (specify in comments section)

***Program Area Codes:**
CWA = Clean Water Act (for wastewaters), **SDWA** = Safe Drinking Water Act (for drinking water), **SHW** = Solid and Hazardous Wastes (for soils, ground waters and waste samples)

***Container Type:** G = Glass, P = Plastic

Lab ID:	Sample Name:	Date Collected:	Time Collected:	G=Grab C=Comp	Matrix (see codes)	Program Area (see codes)	Total # Containers	† REQUESTED LAB ANALYSIS: †	Total Lead in Tissue	Notes / Comments
	FW-01	05-13-24	0700	G	O*	n/a	1	# Containers per Test >>	1	*Fish Tissue Samples
	FW-02	05-13-24	0700	G	O*	n/a	1	# Containers per Test >>	1	Whole body composits
	FW-03	05-13-24	0700	G	O*	n/a	1	# Containers per Test >>	1	
	FW-04	05-13-24	0700	G	O*	n/a	1	# Containers per Test >>	1	
	FW-05	05-13-24	0700	G	O*	n/a	1	# Containers per Test >>	1	
	FW-06	05-13-24	0700	G	O*	n/a	1	# Containers per Test >>	1	
	FW-07	05-13-24	0700	G	O*	n/a	1	# Containers per Test >>	1	
	FW-08	05-13-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FW-09	05-13-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FW-10	05-13-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FW-11	05-13-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FW-12	05-13-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FW-13	05-13-24	0800	G	O*	n/a	1	# Containers per Test >>	1	

Turnaround Time Requested:		Project Location:		Relinquished By:	Received By:	Date:	Time (24hr):	Samples Received on Ice:
Standard		SC	X		Trystan Sanders	5/21/24	1038	___Y ___N ___N/A
Rush *		NC		Trystan Sanders		5/29	5:11PM	___Y ___N ___N/A
*Date Required		Other (Specify):						___Y ___N ___N/A
Rush data emailed/faxed by end of business day on date required. Standard TAT is 7-10 business days.				Received in lab by: _____ courier		5/29	9:36PM	___Y ___N ___N/A

Chain of Custody Page 3 of 4

Sample Temp. Upon Receipt in Lab: 1.8 (°C)

White Copy: Lab original / Canary Copy: File Copy / Pink Copy: Client Copy

NOTE: Relinquishing samples via this Chain of Custody document constitutes client acceptance of Access Analytical terms and conditions.

Access Lab Report #: _____

Sub Lab (if applicable): _____ / Sub Report #: _____

Access Analytical, Inc.

Access Analytical, Inc. -- Irmo
15 Thames Valley Rd. ~ Irmo, SC 29063
Phone: 803-781-4243 / Fax: 803-781-4303 / Web: www.axs-inc.com
SCDHEC Lab Certification # 32571 / NELAP # E871145

Chain of Custody Record

2405X07

Client:	SCDHEC			Preservatives (see codes):	0		
Attn:	Taylor Shearer			Bottle Types (see codes):	P		
Address:	2600 Bull Street						
City:	Columbia	State:	SC	29201			
Phone:	803-898-1538	Fax:					
Email:	shearetv@dhec.sc.gov						
Project Name:	Gills Creek Watershed Lead in Fish Study						
Sampled By (Signature):							

Preservation Codes / Bottle Types:

***Preservative Codes:**
0 = None, **1** = HCL, **2** = HNO3, **3** = H_2SO_4, **4** = NaOH, **5** = $Na_2S_2O_3$, **6** = Method 5035 set w/ $NaHSO_4$ & CH_3OH, **7** = NaOH/ZnOAC, **8** = H_3PO_4, **9** = cooled to ≤6°C, **10** = cooled to ≤10°C, **11** = Amm.Cl, **12** = Ascorbic Acid / HCL, **13** = EDA

***Matrix Codes:**
GW = ground water, **WW** = waste water, **DW** = drinking water, **SW** = surface/storm water, **S** = soil, **SL** = sludge, **A** = air, **IW** = industrial waste, **O** = other (specify in comments section)

***Program Area Codes:**
CWA = Clean Water Act (for wastewaters), **SDWA** = Safe Drinking Water Act (for drinking water), **SHW** = Solid and Hazardous Wastes (for soils, ground waters and waste samples)

***Container Type:** G = Glass, P = Plastic

Lab ID:	Sample Name:	Date Collected:	Time Collected:	G=Grab C=Comp	Matrix (see codes)	Program Area (see codes)	Total # Containers	t REQUESTED LAB ANALYSIS: t	Total Lead in Tissue	Notes / Comments
	FW-14	05-13-24	~~0700~~ 0800	G	O*	n/a	1	# Containers per Test >>	1	*Fish Tissue Samples
	FW-15	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	Whole body composits
	FW-16	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FW-17	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FW-18	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FW-19	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
	FW-20	05-15-24	0800	G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	
				G	O*	n/a	1	# Containers per Test >>	1	

Turnaround Time Requested:		Project Location:		Relinquished By:	Received By:	Date:	Time (24hr):	Samples Received on Ice:
Standard		SC	X	[signature]	[signature]	5/21/24	1038	___Y ___N ___N/A
Rush *		NC		[signature]	[signature]	5/29	5:11PM	___Y ___N ___N/A
*Date Required		Other (Specify):						___Y ___N ___N/A
Rush data emailed/faxed by end of business day on date required. Standard TAT is 7-10 business days.				Received in lab by: [signature] courier		5/29	9:36PM	___Y ___N ___N/A
Chain of Custody Page 4 of 4						Sample Temp. Upon Receipt in Lab: 1.8 (°C)		

White Copy: Lab original / Canary Copy: File Copy / Pink Copy: Client Copy

NOTE: Relinquishing samples via this Chain of Custody document constitutes client acceptance of Access Analytical terms and conditions.

Client:	SCDHEC	**Client Sample ID:**	FF-01
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-001	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.248	0.605	mg/Kg	377035	1	05/31/2024 13:31	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-02
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-002	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.240	0.586	mg/Kg	377035	1	05/31/2024 13:34	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-03
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-003	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.246	0.601	mg/Kg	377035	1	05/31/2024 13:43	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-04
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-004	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.229	0.559	mg/Kg	377035	1	05/31/2024 13:45	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-05
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-005	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.268	0.653	mg/Kg	377035	1	05/31/2024 13:48	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-06
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-006	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.241	0.588	mg/Kg	377035	1	05/31/2024 13:51	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-07
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-007	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.270	0.659	mg/Kg	377035	1	05/31/2024 13:54	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-08
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-008	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.237	0.577	mg/Kg	377035	1	05/31/2024 13:57	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-09
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-009	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.247	0.602	mg/Kg	377035	1	05/31/2024 14:00	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-10
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-010	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.305	0.745	mg/Kg	377035	1	05/31/2024 14:03	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-11
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-011	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.279	0.680	mg/Kg	377035	1	05/31/2024 14:06	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-12
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-012	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.256	0.624	mg/Kg	377035	1	05/31/2024 14:09	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-13
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-013	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.258	0.629	mg/Kg	377035	1	05/31/2024 14:18	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-14
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-014	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.254	0.619	mg/Kg	377035	1	05/31/2024 14:28	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-15
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-015	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.236	0.576	mg/Kg	377035	1	05/31/2024 13:16	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-16
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-016	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.232	0.565	mg/Kg	377035	1	05/31/2024 14:31	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-17
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-017	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.247	0.602	mg/Kg	377035	1	05/31/2024 14:34	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-18
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-018	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.222	0.540	mg/Kg	377035	1	05/31/2024 14:37	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-19
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-019	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.251	0.612	mg/Kg	377035	1	05/31/2024 14:40	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FF-20
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-020	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.238	0.581	mg/Kg	377035	1	05/31/2024 14:43	RR

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-01
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-021	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.280	0.682	mg/Kg	377036	1	05/31/2024 17:05	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-02
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-022	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.338	0.824	mg/Kg	377036	1	05/31/2024 17:26	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-03
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-023	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.334	0.815	mg/Kg	377036	1	05/31/2024 17:29	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-04
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-024	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.290	0.706	mg/Kg	377036	1	05/31/2024 17:32	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-05
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-025	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.222	0.542	mg/Kg	377036	1	05/31/2024 17:35	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-06
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-026	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.326	0.795	mg/Kg	377036	1	05/31/2024 17:38	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-07
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 7:00:00 AM
Lab ID:	2405X07-027	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.304	0.742	mg/Kg	377036	1	05/31/2024 17:42	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-08
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-028	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.245	0.598	mg/Kg	377036	1	05/31/2024 17:45	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-09
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-029	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.260	0.634	mg/Kg	377036	1	05/31/2024 17:48	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-10
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-030	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.273	0.667	mg/Kg	377036	1	05/31/2024 17:51	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-11
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-031	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.329	0.804	mg/Kg	377036	1	05/31/2024 17:54	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-12
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-032	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.285	0.695	mg/Kg	377036	1	05/31/2024 18:03	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-13
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-033	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.283	0.690	mg/Kg	377036	1	05/31/2024 18:06	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-14
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-034	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	0.419	J	0.264	0.644	mg/Kg	377036	1	05/31/2024 18:09	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-15
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-035	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.272	0.662	mg/Kg	377036	1	05/31/2024 18:12	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-16
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-036	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.272	0.664	mg/Kg	377036	1	05/31/2024 18:15	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-17
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-037	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.337	0.822	mg/Kg	377036	1	05/31/2024 18:19	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-18
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-038	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.320	0.780	mg/Kg	377036	1	05/31/2024 18:22	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-19
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-039	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.268	0.654	mg/Kg	377036	1	05/31/2024 18:25	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

Client:	SCDHEC	**Client Sample ID:**	FW-20
Project Name:	Gills Creek Watershed Lead in Fish Study	**Collection Date:**	5/13/2024 8:00:00 AM
Lab ID:	2405X07-040	**Matrix:**	Solid

Analyses	Result	Qual	MDL	Reporting Limit	Units	BatchID	DF	Date Analyzed	Analyst
METALS, TOTAL SW6010D (SW3050B)									
Lead	BRL		0.260	0.634	mg/Kg	377036	1	05/31/2024 18:28	DS

Qualifiers:

*	Value exceeds maximum contaminant level	E	Estimated value above quantitation range
BRL	Not detected at MDL	S	Spike Recovery outside limits due to matrix
H	Holding times for preparation or analysis exceeded	J	Estimated value detected below Reporting Limit
N	Analyte not NELAC certified	>	Greater than Result value
B	Analyte detected in the associated method blank	<	Less than Result value
F	Analyzed in the lab which is a deviation from the method	Narr	See case narrative

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SAMPLE/COOLER RECEIPT CHECKLIST

1. Client Name: **Access Analytical, Inc** AES Work Order Number: **2405X07**

2. Carrier: FedEx ☐ UPS ☐ USPS ☐ Client ☐ Courier ☑ Other _____

		Yes	No	N/A	Details	Comments
3.	Shipping container/cooler received in good condition?	●	○	○	damaged ☐ leaking ☐ other ☐	
4.	Custody seals present on shipping container?	○	●	○		
5.	Custody seals intact on shipping container?	○	○	●		
6.	Cooler temperature(s) within limits of 0-6°C? [See item 12 for temperature recordings.]	○	○	●		
7.	Chain of Custody (COC) present?	●	○	○		
8.	Chain of Custody signed, dated, and timed when relinquished and received?	●	○	○		
9.	Sampler name and/or signature on COC?	●	○	○		
10.	Were all samples received within holding time?	●	○	○		
11.	TAT marked on the COC?	●	○	○	If no TAT indicated, proceeded with standard TAT per Terms & Conditions.	

12. Cooler 1 Temperature 1.8 °C Cooler 2 Temperature _____ °C Cooler 3 Temperature _____ °C Cooler 4 Temperature _____ °C
Cooler 5 Temperature _____ °C Cooler 6 Temperature _____ °C Cooler 7 Temperature _____ °C Cooler 8 Temperature _____ °C

13. Comments: _____

I certify that I have completed sections 1-13 (dated initials). RW 5/30/24

		Yes	No	N/A	Details	Comments
14.	Temperature blanks present?	○	○	●		
15.	Were sample containers intact upon receipt?	●	○	○		
16.	Custody seals present on sample containers?	○	●	○		
17.	Custody seals intact on sample containers?	○	○	●		
18.	Do sample container labels match the COC?	●	○	○	incomplete info ☐ illegible ☐ no label ☐ other ☐	
19.	Are analyses requested indicated on the COC?	●	○	○		
20.	Were all of the samples listed on the COC received?	●	○	○	samples received but not listed on COC ☐ samples listed on COC not received ☐	
21.	Was the sample collection date/time noted?	●	○	○		
22.	Did we receive sufficient sample volume for indicated analyses?	●	○	○		
23.	Were samples received in appropriate containers?	●	○	○		
24.	Were VOA samples received without headspace (< 1/4" bubble)?	○	○	●		
25.	Were trip blanks submitted?	○	○	●	listed on COC ☐ not listed on COC ☐	

26. Comments: _____

I certify that I have completed sections 14-26 (dated initials). RW 5/30/24

This section only applies to samples where pH can be checked at Sample Receipt.

		Yes	No	N/A	Details	Comments
27.	Have containers needing chemical preservation been checked?*	○	○	●		
28.	Containers meet preservation guidelines?	○	○	●		
29.	Was pH adjusted at Sample Receipt?	○	○	●		

***Note: Certain analyses require chemical preservation but must be checked in the laboratory and not upon Sample Receipt such as Coliforms, VOCs and Oil & Grease/TPH. This also excludes metals by EPA 200.7, 200.8 and 245.1 which will be verified between 16 and 24 hours after preservation.**

I certify that I have completed sections 27-29(dated initials). RW 5/30/24

Client: SCDHEC
Project Name: Gills Creek Watershed Lead in Fish Study
Workorder: 2405X07

ANALYTICAL QC SUMMARY REPORT

BatchID: 377035

Sample ID: **MB-377035** Client ID: Units: **mg/Kg** Prep Date: **05/31/2024** Run No: **546424**
SampleType: **MBLK** TestCode: **METALS, TOTAL SW6010D** BatchID: **377035** Analysis Date: **05/31/2024** Seq No: **13048115**

Analyte	Result	RPT Limit	SPK value	SPK Ref Val	%REC	Low Limit	High Limit	RPD Ref Val	%RPD	RPD Limit	Qual
Lead	BRL	1.00									

Sample ID: **LCS-377035** Client ID: Units: **mg/Kg** Prep Date: **05/31/2024** Run No: **546424**
SampleType: **LCS** TestCode: **METALS, TOTAL SW6010D** BatchID: **377035** Analysis Date: **05/31/2024** Seq No: **13048117**

Analyte	Result	RPT Limit	SPK value	SPK Ref Val	%REC	Low Limit	High Limit	RPD Ref Val	%RPD	RPD Limit	Qual
Lead	44.07	5.00	50.00		88.1	80	120				

Sample ID: **2405X07-015AMS** Client ID: **FF-15** Units: **mg/Kg** Prep Date: **05/31/2024** Run No: **546424**
SampleType: **MS** TestCode: **METALS, TOTAL SW6010D** BatchID: **377035** Analysis Date: **05/31/2024** Seq No: **13048122**

Analyte	Result	RPT Limit	SPK value	SPK Ref Val	%REC	Low Limit	High Limit	RPD Ref Val	%RPD	RPD Limit	Qual
Lead	24.19	2.88	28.80		84.0	75	125				

Sample ID: **2405X07-015AMSD** Client ID: **FF-15** Units: **mg/Kg** Prep Date: **05/31/2024** Run No: **546424**
SampleType: **MSD** TestCode: **METALS, TOTAL SW6010D** BatchID: **377035** Analysis Date: **05/31/2024** Seq No: **13048124**

Analyte	Result	RPT Limit	SPK value	SPK Ref Val	%REC	Low Limit	High Limit	RPD Ref Val	%RPD	RPD Limit	Qual
Lead	24.19	2.88	28.84		83.9	75	125	24.19	0.011	20	

Qualifiers:

>	Greater than Result value	<	Less than Result value	B	Analyte detected in the associated method blank
BRL	Below reporting limit	E	Estimated (value above quantitation range)	H	Holding times for preparation or analysis exceeded
J	Estimated value detected below Reporting Limit	N	Analyte not NELAC certified	R	RPD outside limits due to matrix
Rpt Lim	Reporting Limit	S	Spike Recovery outside limits due to matrix		

Client: SCDHEC
Project Name: Gills Creek Watershed Lead in Fish Study
Workorder: 2405X07

ANALYTICAL QC SUMMARY REPORT

BatchID: 377036

Sample ID: **MB-377036** Client ID: Units: **mg/Kg** Prep Date: **05/31/2024** Run No: **546467**
SampleType: **MBLK** TestCode: **METALS, TOTAL SW6010D** BatchID: **377036** Analysis Date: **05/31/2024** Seq No: **13049673**

Analyte	Result	RPT Limit	SPK value	SPK Ref Val	%REC	Low Limit	High Limit	RPD Ref Val	%RPD	RPD Limit	Qual
Lead	BRL	5.00									

Sample ID: **LCS-377036** Client ID: Units: **mg/Kg** Prep Date: **05/31/2024** Run No: **546467**
SampleType: **LCS** TestCode: **METALS, TOTAL SW6010D** BatchID: **377036** Analysis Date: **05/31/2024** Seq No: **13049674**

Analyte	Result	RPT Limit	SPK value	SPK Ref Val	%REC	Low Limit	High Limit	RPD Ref Val	%RPD	RPD Limit	Qual
Lead	51.01	5.00	50.00		102	80	120				

Sample ID: **2405X07-021AMS** Client ID: **FW-01** Units: **mg/Kg** Prep Date: **05/31/2024** Run No: **546467**
SampleType: **MS** TestCode: **METALS, TOTAL SW6010D** BatchID: **377036** Analysis Date: **05/31/2024** Seq No: **13049676**

Analyte	Result	RPT Limit	SPK value	SPK Ref Val	%REC	Low Limit	High Limit	RPD Ref Val	%RPD	RPD Limit	Qual
Lead	34.07	3.41	34.12		99.9	75	125				

Sample ID: **2405X07-021AMSD** Client ID: **FW-01** Units: **mg/Kg** Prep Date: **05/31/2024** Run No: **546467**
SampleType: **MSD** TestCode: **METALS, TOTAL SW6010D** BatchID: **377036** Analysis Date: **05/31/2024** Seq No: **13049677**

Analyte	Result	RPT Limit	SPK value	SPK Ref Val	%REC	Low Limit	High Limit	RPD Ref Val	%RPD	RPD Limit	Qual
Lead	34.04	3.41	34.12		99.8	75	125	34.07	0.103	20	

Qualifiers:

>	Greater than Result value	<	Less than Result value	B	Analyte detected in the associated method blank
BRL	Below reporting limit	E	Estimated (value above quantitation range)	H	Holding times for preparation or analysis exceeded
J	Estimated value detected below Reporting Limit	N	Analyte not NELAC certified	R	RPD outside limits due to matrix
Rpt Lim	Reporting Limit	S	Spike Recovery outside limits due to matrix		

End of Report

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ANALYTICAL REPORT

PREPARED FOR

SC-DES
Attn: Taylor Shearer

Generated 12/19/2024 3:42:53 PM Revision 1

JOB DESCRIPTION

Gills Creek Watershed Lead in Fish Study
SCDHEC

JOB NUMBER

705-13852-1

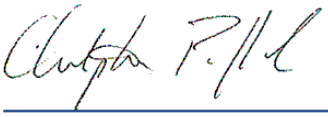
Eurofins Atlanta
3080 Presidential Dr
Atlanta GA 30340

See page two for job notes and contact information.

Job Notes

The test results in this report relate only to the samples as received by the laboratory and will meet all requirements of the methodology, with any exceptions noted. This report shall not be reproduced except in full, without the express written approval of the laboratory. All questions should be directed to the Eurofins Environment Testing Southeast, LLC Project Manager.

Authorization



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Revision 1

Authorized for release by
Christopher Pafford, Customer Service Manager
christopher.pafford@et.eurofinsus.com
(770)457-8177

Table of Contents

Definitions/Glossary

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Glossary

Abbreviation	These commonly used abbreviations may or may not be present in this report.
☼	Listed under the "D" column to designate that the result is reported on a dry weight basis
%R	Percent Recovery
CFL	Contains Free Liquid
CFU	Colony Forming Unit
CNF	Contains No Free Liquid
DER	Duplicate Error Ratio (normalized absolute difference)
Dil Fac	Dilution Factor
DL	Detection Limit (DoD/DOE)
DL, RA, RE, IN	Indicates a Dilution, Re-analysis, Re-extraction, or additional Initial metals/anion analysis of the sample
DLC	Decision Level Concentration (Radiochemistry)
EDL	Estimated Detection Limit (Dioxin)
LOD	Limit of Detection (DoD/DOE)
LOQ	Limit of Quantitation (DoD/DOE)
MCL	EPA recommended "Maximum Contaminant Level"
MDA	Minimum Detectable Activity (Radiochemistry)
MDC	Minimum Detectable Concentration (Radiochemistry)
MDL	Method Detection Limit
ML	Minimum Level (Dioxin)
MPN	Most Probable Number
MQL	Method Quantitation Limit
NC	Not Calculated
ND	Not Detected at the reporting limit (or MDL or EDL if shown)
NEG	Negative / Absent
POS	Positive / Present
PQL	Practical Quantitation Limit
PRES	Presumptive
QC	Quality Control
RER	Relative Error Ratio (Radiochemistry)
RL	Reporting Limit or Requested Limit (Radiochemistry)
RPD	Relative Percent Difference, a measure of the relative difference between two points
TEF	Toxicity Equivalent Factor (Dioxin)
TEQ	Toxicity Equivalent Quotient (Dioxin)
TNTC	Too Numerous To Count

Access Lab Report #: _____

Sub Lab (if applicable): _____ / Sub Report #: _____

Access Analytical, Inc. -- Irmo
15 Thames Valley Rd. ~ Irmo, SC 29063
Phone: 803-781-4243 / Fax: 803-781-4303 / Web: www.axs-inc.com
SCDHEC Lab Certification # 32571 / NELAP # E871145

Chain of Custody Record

Client:	SCDES
Attn:	Taylor Shearer
Address:	2600 Bull Street
City:	Columbia
State:	SC
	29201
Phone:	803-898-1538
Fax:	
Email:	Taylor.shearer@des.sc.gov
Project Name:	Gills Creek Watershed Lead in Fish Study
Sampled By (Signature):	

Preservatives (see codes): 0

Bottle Types (see codes): P

Preservation Codes / Bottle Types:

***Preservative Codes:**
0 = None, **1** = HCL, **2** = HNO3, **3** = H_2SO_4, **4** = NaOH, **5** = $Na_2S_2O_3$, **6** = Method 5035 set w/ $NaHSO_4$ & CH_3OH, **7** = NaOH/ZnOAC, **8** = H_3PO_4, **9** = cooled to ≤6°C, **10** = cooled to ≤10°C, **11** = Amm.Cl, **12** = Ascorbic Acid / HCL, **13** = EDA

***Matrix Codes:**
GW = ground water, **WW** = waste water, **DW** = drinking water, **SW** = surface/storm water, **S** = soil, **SL** = sludge, **A** = air, **IW** = industrial waste, **O** = other (specify in comments section)

***Program Area Codes:**
CWA = Clean Water Act (for wastewaters), **SDWA** = Safe Drinking Water Act (for drinking water), **SHW** = Solid and Hazardous Wastes (for soils, ground waters and waste samples)

***Container Type:** G = Glass, P = Plastic

Lab ID:	Sample Name:	Date Collected:	Time Collected:	G=Grab C=Comp	Matrix (see codes)	Program Area (see codes)	Total # Containers	# Containers per Test >>	REQUESTED LAB ANALYSIS: Total Lead in Tissue
	FF-21	10-31-24	0900	G	O*	n/a	1	# Containers per Test >>	1
	FF-22	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FF-23	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FF-24	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FF-25	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FF-26	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FF-27	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FF-28	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
				G	O*	n/a	1	# Containers per Test >>	1
				G	O*	n/a	1	# Containers per Test >>	1
				G	O*	n/a	1	# Containers per Test >>	1
				G	O*	n/a	1	# Containers per Test >>	1
				G	O*	n/a	1	# Containers per Test >>	1

Notes / Comments

*Fish Tissue Samples [Fillets]

705-13852 COC

Turnaround Time Requested:

- Standard
- Rush * (*Rush data emailed/faxed by end of business day on date required. Standard TAT is 7-10 business days.)
- *Date Required

Project Location: SC X, NC, Other (Specify):

Relinquished By:	Received By:	Date:	Time (24hr):	Samples Received on Ice:
		11/13/24	1137	___Y ___N ___N/A
		11/26	4:19PM	___Y ___N ___N/A
				___Y ___N ___N/A
		11/26	9:00PM	___Y ___N ___N/A

Received in lab by: _____

Sample Temp. Upon Receipt in Lab: 0.9 (°C) 1.0°C

Chain of Custody Page _____ of _____

White Copy: Lab original / Canary Copy: File Copy / Pink Copy: Client Copy

NOTE: Relinquishing samples via this Chain of Custody document constitutes client acceptance of Access Analytical terms and conditions.

Job ID: 705-13852-1 Eurofins Atlanta

Job Narrative
705-13852-1

REVISION

The report being provided is a revision of the original report sent on 12/9/2024. The report (revision 1) is being revised due to update formatter to include MDL J flags per client request..

Analytical test results meet all requirements of the associated regulatory program listed on the Accreditation/Certification Summary Page unless otherwise noted under the individual analysis. Data qualifiers and/or narrative comments are included to explain any exceptions, if applicable.

- Matrix QC may not be reported if insufficient sample is provided or site-specific QC samples were not submitted. In these situations, to demonstrate precision and accuracy at a batch level, a LCS/LCSD may be performed, unless otherwise specified in the method.
- Surrogate and/or isotope dilution analyte recoveries (if applicable) which are outside of the QC window are confirmed unless attributed to a dilution or otherwise noted in the narrative.

Regulated compliance samples (e.g. SDWA, NPDES) must comply with the associated agency requirements/permits.

Receipt
The samples were received on 11/26/2024 9:00 PM. Unless otherwise noted below, the samples arrived in good condition, and, where required, properly preserved and on ice. The temperatures of the 2 coolers at receipt time were 0.9°C and 1.0°C.

Metals
No additional analytical or quality issues were noted, other than those described above or in the Definitions/ Glossary page.

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Client Sample ID: FF-21
Date Collected: 10/31/24 09:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13852-1
Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.2	0.25	mg/Kg		12/05/24 08:34	12/05/24 14:23	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Client Sample ID: FF-22

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13852-2

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.3	0.27	mg/Kg		12/05/24 08:34	12/05/24 14:26	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Client Sample ID: FF-23

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13852-3

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.1	0.24	mg/Kg		12/05/24 08:34	12/05/24 14:28	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Client Sample ID: FF-24
Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13852-4
Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.1	0.23	mg/Kg		12/05/24 08:34	12/05/24 14:31	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Client Sample ID: FF-25

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13852-5

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.4	0.29	mg/Kg		12/05/24 08:34	12/05/24 14:02	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Client Sample ID: FF-26

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13852-6

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.2	0.24	mg/Kg		12/05/24 08:34	12/05/24 14:34	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Client Sample ID: FF-27

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13852-7

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.1	0.22	mg/Kg		12/05/24 08:34	12/05/24 14:39	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Client Sample ID: FF-28
Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13852-8
Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.2	0.25	mg/Kg		12/05/24 08:34	12/05/24 14:42	1

Detection Summary

Client: Access Analytical Services
Job ID: 705-13852-1
Project/Site: Gills Creek Watershed Lead in Fish Study
SDG: SCDHEC

Client Sample ID: FF-21 — Lab Sample ID: 705-13852-1

No Detections.

Client Sample ID: FF-22 — Lab Sample ID: 705-13852-2

No Detections.

Client Sample ID: FF-23 — Lab Sample ID: 705-13852-3

No Detections.

Client Sample ID: FF-24 — Lab Sample ID: 705-13852-4

No Detections.

Client Sample ID: FF-25 — Lab Sample ID: 705-13852-5

No Detections.

Client Sample ID: FF-26 — Lab Sample ID: 705-13852-6

No Detections.

Client Sample ID: FF-27 — Lab Sample ID: 705-13852-7

No Detections.

Client Sample ID: FF-28 — Lab Sample ID: 705-13852-8

No Detections.

This Detection Summary does not include radiochemical test results.

Login Sample Receipt Checklist

Client: Access Analytical Services

Job Number: 705-13852-1
SDG Number: SCDHEC

Login Number: 13852
List Number: 1
Creator: Torres, Dominique

List Source: Eurofins Atlanta

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	True	
The cooler's custody seal, if present, is intact.	True	
Sample custody seals, if present, are intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time (excluding tests with immediate HTs)	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	True	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	N/A	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

Lab Chronicle

Client: Access Analytical Services
Job ID: 705-13852-1
Project/Site: Gills Creek Watershed Lead in Fish Study
SDG: SCDHEC

Client Sample ID: FF-21
Lab Sample ID: 705-13852-1
Date Collected: 10/31/24 09:00
Matrix: Solid
Date Received: 11/26/24 21:00

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25070	SA	EET ATL	12/05/24 08:34
Total/NA	Analysis	6010D		1	25349	DAB	EET ATL	12/05/24 14:23

Client Sample ID: FF-22
Lab Sample ID: 705-13852-2
Date Collected: 11/05/24 08:00
Matrix: Solid
Date Received: 11/26/24 21:00

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25070	SA	EET ATL	12/05/24 08:34
Total/NA	Analysis	6010D		1	25349	DAB	EET ATL	12/05/24 14:26

Client Sample ID: FF-23
Lab Sample ID: 705-13852-3
Date Collected: 11/05/24 08:00
Matrix: Solid
Date Received: 11/26/24 21:00

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25070	SA	EET ATL	12/05/24 08:34
Total/NA	Analysis	6010D		1	25349	DAB	EET ATL	12/05/24 14:28

Client Sample ID: FF-24
Lab Sample ID: 705-13852-4
Date Collected: 11/05/24 08:00
Matrix: Solid
Date Received: 11/26/24 21:00

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25070	SA	EET ATL	12/05/24 08:34
Total/NA	Analysis	6010D		1	25349	DAB	EET ATL	12/05/24 14:31

Client Sample ID: FF-25
Lab Sample ID: 705-13852-5
Date Collected: 11/05/24 08:00
Matrix: Solid
Date Received: 11/26/24 21:00

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25070	SA	EET ATL	12/05/24 08:34
Total/NA	Analysis	6010D		1	25349	DAB	EET ATL	12/05/24 14:02

Client Sample ID: FF-26
Lab Sample ID: 705-13852-6
Date Collected: 11/05/24 08:00
Matrix: Solid
Date Received: 11/26/24 21:00

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25070	SA	EET ATL	12/05/24 08:34
Total/NA	Analysis	6010D		1	25349	DAB	EET ATL	12/05/24 14:34

Lab Chronicle

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Client Sample ID: FF-27

Lab Sample ID: 705-13852-7

Date Collected: 11/05/24 08:00
Matrix: Solid
Date Received: 11/26/24 21:00

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25070	SA	EET ATL	12/05/24 08:34
Total/NA	Analysis	6010D		1	25349	DAB	EET ATL	12/05/24 14:39

Client Sample ID: FF-28

Lab Sample ID: 705-13852-8

Date Collected: 11/05/24 08:00
Matrix: Solid
Date Received: 11/26/24 21:00

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25070	SA	EET ATL	12/05/24 08:34
Total/NA	Analysis	6010D		1	25349	DAB	EET ATL	12/05/24 14:42

Laboratory References:

EET ATL = Eurofins Atlanta, 3080 Presidential Dr, Atlanta, GA 30340, TEL (770)457-8177

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13852-1
SDG: SCDHEC

Method: 6010D - Metals (ICP)

Lab Sample ID: MB 705-25070/1-A
Matrix: Solid
Analysis Batch: 25349

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 25070

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		2.0	0.41	mg/Kg		12/05/24 08:34	12/05/24 13:56	1

Lab Sample ID: LCS 705-25070/2-A
Matrix: Solid
Analysis Batch: 25349

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 25070

Analyte			Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec Limits		
Lead			50.0	48.3		mg/Kg		97	80 - 120		

Lab Sample ID: 705-13852-5 MS
Matrix: Solid
Analysis Batch: 25349

Client Sample ID: FF-25
Prep Type: Total/NA
Prep Batch: 25070

Analyte	Sample Result	Sample Qualifier	Spike Added	MS Result	MS Qualifier	Unit	D	%Rec	%Rec Limits		
Lead	ND		35.1	32.5		mg/Kg		93	75 - 125		

Lab Sample ID: 705-13852-5 MSD
Matrix: Solid
Analysis Batch: 25349

Client Sample ID: FF-25
Prep Type: Total/NA
Prep Batch: 25070

Analyte	Sample Result	Sample Qualifier	Spike Added	MSD Result	MSD Qualifier	Unit	D	%Rec	%Rec Limits	RPD	RPD Limit
Lead	ND		35.1	32.1		mg/Kg		91	75 - 125	1	20

Accreditation/Certification Summary

Client: Access Analytical Services
Job ID: 705-13852-1
Project/Site: Gills Creek Watershed Lead in Fish Study
SDG: SCDHEC

Laboratory: Eurofins Atlanta

Unless otherwise noted, all analytes for this laboratory were covered under each accreditation/certification below.

Authority	Program	Identification Number	Expiration Date
South Carolina	State	98016	06-30-25

The following analytes are included in this report, but the laboratory is not certified by the governing authority. This list may include analytes for which the agency does not offer certification.

Analysis Method	Prep Method	Matrix	Analyte
6010D	3050B	Solid	Lead

END OF REPORT

eurofins | Environment Testing

ANALYTICAL REPORT

PREPARED FOR

SC-DES
Attn: Taylor Shearer

Generated 12/19/2024 5:08:56 PM Revision 1

JOB DESCRIPTION

Gills Creek Watershed Lead in Fish Study
SCDHEC

JOB NUMBER

705-13856-1

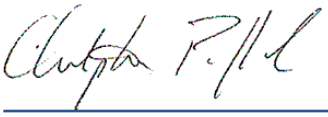
Eurofins Atlanta
3080 Presidential Dr
Atlanta GA 30340

See page two for job notes and contact information.

Job Notes

The test results in this report relate only to the samples as received by the laboratory and will meet all requirements of the methodology, with any exceptions noted. This report shall not be reproduced except in full, without the express written approval of the laboratory. All questions should be directed to the Eurofins Environment Testing Southeast, LLC Project Manager.

Authorization



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12/19/2024 5:08:56 PM
Revision 1

Authorized for release by
Christopher Pafford, Customer Service Manager
christopher.pafford@et.eurofinsus.com
(770)457-8177

Table of Contents

Definitions/Glossary

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Qualifiers

Metals

Qualifier	Qualifier Description
J	Result is less than the RL but greater than or equal to the MDL and the concentration is an approximate value.

Glossary

Abbreviation	These commonly used abbreviations may or may not be present in this report.
☼	Listed under the "D" column to designate that the result is reported on a dry weight basis
%R	Percent Recovery
CFL	Contains Free Liquid
CFU	Colony Forming Unit
CNF	Contains No Free Liquid
DER	Duplicate Error Ratio (normalized absolute difference)
Dil Fac	Dilution Factor
DL	Detection Limit (DoD/DOE)
DL, RA, RE, IN	Indicates a Dilution, Re-analysis, Re-extraction, or additional Initial metals/anion analysis of the sample
DLC	Decision Level Concentration (Radiochemistry)
EDL	Estimated Detection Limit (Dioxin)
LOD	Limit of Detection (DoD/DOE)
LOQ	Limit of Quantitation (DoD/DOE)
MCL	EPA recommended "Maximum Contaminant Level"
MDA	Minimum Detectable Activity (Radiochemistry)
MDC	Minimum Detectable Concentration (Radiochemistry)
MDL	Method Detection Limit
ML	Minimum Level (Dioxin)
MPN	Most Probable Number
MQL	Method Quantitation Limit
NC	Not Calculated
ND	Not Detected at the reporting limit (or MDL or EDL if shown)
NEG	Negative / Absent
POS	Positive / Present
PQL	Practical Quantitation Limit
PRES	Presumptive
QC	Quality Control
RER	Relative Error Ratio (Radiochemistry)
RL	Reporting Limit or Requested Limit (Radiochemistry)
RPD	Relative Percent Difference, a measure of the relative difference between two points
TEF	Toxicity Equivalent Factor (Dioxin)
TEQ	Toxicity Equivalent Quotient (Dioxin)
TNTC	Too Numerous To Count

Access Lab Report #: _____

Sub Lab (if applicable): _____ / Sub Report #: _____

Access Analytical, Inc. -- Irmo
15 Thames Valley Rd. ~ Irmo, SC 29063
Phone: 803-781-4243 / Fax: 803-781-4303 / Web: www.axs-inc.com
SCDHEC Lab Certification # 32571 / NELAP # E871145

Chain of Custody Record

Client:	SCDES
Attn:	Taylor Shearer
Address:	2600 Bull Street
City:	Columbia
State:	SC
	29201
Phone:	803-898-1538
Fax:	
Email:	Taylor.shearer@des.sc.gov
Project Name:	Gills Creek Watershed Lead in Fish Study
Sampled By (Signature):	

Preservation Codes / Bottle Types:

***Preservative Codes:**
0 = None, **1** = HCL, **2** = HNO3, **3** = H_2SO_4, **4** = NaOH, **5** = $Na_2S_2O_3$, **6** = Method 5035 set w/ $NaHSO_4$ & CH_3OH, **7** = NaOH/ZnOAC, **8** = H_3PO_4, **9** = cooled to ≤6°C, **10** = cooled to ≤10°C, **11** = Amm.Cl, **12** = Ascorbic Acid / HCL, **13** = EDA

***Matrix Codes:**
GW = ground water, **WW** = waste water, **DW** = drinking water, **SW** = surface/storm water, **S** = soil, **SL** = sludge, **A** = air, **IW** = industrial waste, **O** = other (specify in comments section)

***Program Area Codes:**
CWA = Clean Water Act (for wastewaters), **SDWA** = Safe Drinking Water Act (for drinking water), **SHW** = Solid and Hazardous Wastes (for soils, ground waters and waste samples)

***Container Type:** G = Glass, P = Plastic

Lab ID:	Sample Name:	Date Collected:	Time Collected:	G=Grab C=Comp	Matrix (see codes)	Program Area (see codes)	Total # Containers	REQUESTED LAB ANALYSIS: t	Total Lead in Tissue
	FW-21	10-31-24	0900	G	O*	n/a	1	# Containers per Test >>	1
	FW-22	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FW-23	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FW-24	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FW-25	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FW-26	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FW-27	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
	FW-28	11-05-24	0800	G	O*	n/a	1	# Containers per Test >>	1
				G	O*	n/a	1	# Containers per Test >>	1
				G	O*	n/a	1	# Containers per Test >>	1
				G	O*	n/a	1	# Containers per Test >>	1
				G	O*	n/a	1	# Containers per Test >>	1
				G	O*	n/a	1	# Containers per Test >>	1

Preservatives (see codes): 0
Bottle Types (see codes): P

Notes / Comments

***Fish Tissue Samples**

*Composite of whole fish

705-13856 COC

Turnaround Time Requested:		Project Location:		Relinquished By:	Received By:	Date:	Time (24hr):	Samples Received on Ice:
Standard		SC	X			11/13/24	1137	Y N N/A
Rush *		NC				11/26	4:14PM	Y N N/A
*Date Required		Other (Specify):						Y N N/A
						11/26	9:00PM	Y N N/A

*Rush data emailed/faxed by end of business day on date required. Standard TAT is 7-10 business days.

Chain of Custody Page _____ of _____

Received in lab by: _____

Sample Temp. Upon Receipt in Lab: 0.9 (°C) 1.0 °C

White Copy: Lab original / Canary Copy: File Copy / Pink Copy: Client Copy

NOTE: Relinquishing samples via this Chain of Custody document constitutes client acceptance of Access Analytical terms and conditions.

Job ID: 705-13856-1 Eurofins Atlanta

Job Narrative
705-13856-1

REVISION

The report being provided is a revision of the original report sent on 12/10/2024. The report (revision 1) is being revised due to update formatter to include MDL J flagged data per client request.

Analytical test results meet all requirements of the associated regulatory program listed on the Accreditation/Certification Summary Page unless otherwise noted under the individual analysis. Data qualifiers and/or narrative comments are included to explain any exceptions, if applicable.

- Matrix QC may not be reported if insufficient sample is provided or site-specific QC samples were not submitted. In these situations, to demonstrate precision and accuracy at a batch level, a LCS/LCSD may be performed, unless otherwise specified in the method.
- Surrogate and/or isotope dilution analyte recoveries (if applicable) which are outside of the QC window are confirmed unless attributed to a dilution or otherwise noted in the narrative.

Regulated compliance samples (e.g. SDWA, NPDES) must comply with the associated agency requirements/permits.

Receipt
The samples were received on 11/26/2024 9:00 PM. Unless otherwise noted below, the samples arrived in good condition, and, where required, properly preserved and on ice. The temperatures of the 2 coolers at receipt time were 0.9°C and 1.0°C.

Metals
No additional analytical or quality issues were noted, other than those described above or in the Definitions/ Glossary page.

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-21
Date Collected: 10/31/24 09:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-1
Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	0.36	J	1.2	0.24	mg/Kg		12/04/24 14:15	12/05/24 14:54	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-22

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-2

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.4	0.28	mg/Kg		12/04/24 14:15	12/05/24 14:57	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-23

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-3

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.5	0.31	mg/Kg		12/04/24 14:15	12/05/24 15:00	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-24

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-4

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.3	0.26	mg/Kg		12/04/24 14:15	12/05/24 15:03	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-25

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-5

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.2	0.25	mg/Kg		12/04/24 14:15	12/09/24 09:51	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-26

Date Collected: 11/05/24 00:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-6

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.4	0.28	mg/Kg		12/04/24 14:15	12/09/24 09:54	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-27

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-7

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.2	0.25	mg/Kg		12/04/24 14:15	12/09/24 09:57	1

Client Sample Results

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-28

Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-8

Matrix: Solid

Method: SW846 6010D - Metals (ICP)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		1.3	0.26	mg/Kg		12/04/24 14:15	12/09/24 10:06	1

Detection Summary

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-21 — Lab Sample ID: 705-13856-1

Analyte	Result	Qualifier	RL	MDL	Unit	Dil Fac	D	Method	Prep Type
Lead	0.36	J	1.2	0.24	mg/Kg	1		6010D	Total/NA

Client Sample ID: FW-22 — Lab Sample ID: 705-13856-2

No Detections.

Client Sample ID: FW-23 — Lab Sample ID: 705-13856-3

No Detections.

Client Sample ID: FW-24 — Lab Sample ID: 705-13856-4

No Detections.

Client Sample ID: FW-25 — Lab Sample ID: 705-13856-5

No Detections.

Client Sample ID: FW-26 — Lab Sample ID: 705-13856-6

No Detections.

Client Sample ID: FW-27 — Lab Sample ID: 705-13856-7

No Detections.

Client Sample ID: FW-28 — Lab Sample ID: 705-13856-8

No Detections.

This Detection Summary does not include radiochemical test results.

Eurofins Atlanta

Login Sample Receipt Checklist

Client: Access Analytical Services

Job Number: 705-13856-1
SDG Number: SCDHEC

Login Number: 13856
List Number: 1
Creator: Torres, Dominique

List Source: Eurofins Atlanta

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	N/A	
The cooler's custody seal, if present, is intact.	N/A	
Sample custody seals, if present, are intact.	N/A	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time (excluding tests with immediate HTs)	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	N/A	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	N/A	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

Lab Chronicle

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-21
Date Collected: 10/31/24 09:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-1
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25064	BR	EET ATL	12/04/24 14:15
Total/NA	Analysis	6010D		1	25364	KB	EET ATL	12/05/24 14:54

Client Sample ID: FW-22
Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-2
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25064	BR	EET ATL	12/04/24 14:15
Total/NA	Analysis	6010D		1	25364	KB	EET ATL	12/05/24 14:57

Client Sample ID: FW-23
Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-3
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25064	BR	EET ATL	12/04/24 14:15
Total/NA	Analysis	6010D		1	25364	KB	EET ATL	12/05/24 15:00

Client Sample ID: FW-24
Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-4
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25064	BR	EET ATL	12/04/24 14:15
Total/NA	Analysis	6010D		1	25364	KB	EET ATL	12/05/24 15:03

Client Sample ID: FW-25
Date Collected: 11/05/24 08:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-5
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25064	BR	EET ATL	12/04/24 14:15
Total/NA	Analysis	6010D		1	25775	DAB	EET ATL	12/09/24 09:51

Client Sample ID: FW-26
Date Collected: 11/05/24 00:00
Date Received: 11/26/24 21:00

Lab Sample ID: 705-13856-6
Matrix: Solid

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25064	BR	EET ATL	12/04/24 14:15
Total/NA	Analysis	6010D		1	25775	DAB	EET ATL	12/09/24 09:54

Lab Chronicle

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Client Sample ID: FW-27

Lab Sample ID: 705-13856-7

Date Collected: 11/05/24 08:00
Matrix: Solid
Date Received: 11/26/24 21:00

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25064	BR	EET ATL	12/04/24 14:15
Total/NA	Analysis	6010D		1	25775	DAB	EET ATL	12/09/24 09:57

Client Sample ID: FW-28

Lab Sample ID: 705-13856-8

Date Collected: 11/05/24 08:00
Matrix: Solid
Date Received: 11/26/24 21:00

Prep Type	Batch Type	Batch Method	Run	Dilution Factor	Batch Number	Analyst	Lab	Prepared or Analyzed
Total/NA	Prep	3050B			25064	BR	EET ATL	12/04/24 14:15
Total/NA	Analysis	6010D		1	25775	DAB	EET ATL	12/09/24 10:06

Laboratory References:

EET ATL = Eurofins Atlanta, 3080 Presidential Dr, Atlanta, GA 30340, TEL (770)457-8177

Client: Access Analytical Services
Project/Site: Gills Creek Watershed Lead in Fish Study

Job ID: 705-13856-1
SDG: SCDHEC

Method: 6010D - Metals (ICP)

Lab Sample ID: MB 705-25064/1-A
Matrix: Solid
Analysis Batch: 25364

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 25064

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Lead	ND		2.0	0.41	mg/Kg		12/04/24 14:15	12/05/24 14:09	1

Lab Sample ID: LCS 705-25064/2-A
Matrix: Solid
Analysis Batch: 25364

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 25064

Analyte			Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec Limits		
Lead			50.0	48.8		mg/Kg		98	80 - 120		

Lab Sample ID: 705-13954-B-1-B MS
Matrix: Solid
Analysis Batch: 25364

Client Sample ID: Matrix Spike
Prep Type: Total/NA
Prep Batch: 25064

Analyte	Sample Result	Sample Qualifier	Spike Added	MS Result	MS Qualifier	Unit	D	%Rec	%Rec Limits		
Lead	48		29.9	80.0		mg/Kg		105	75 - 125		

Lab Sample ID: 705-13954-B-1-C MSD
Matrix: Solid
Analysis Batch: 25364

Client Sample ID: Matrix Spike Duplicate
Prep Type: Total/NA
Prep Batch: 25064

Analyte	Sample Result	Sample Qualifier	Spike Added	MSD Result	MSD Qualifier	Unit	D	%Rec	%Rec Limits	RPD	RPD Limit
Lead	48		30.0	79.7		mg/Kg		104	75 - 125	0	20

Accreditation/Certification Summary

Client: Access Analytical Services
Job ID: 705-13856-1
Project/Site: Gills Creek Watershed Lead in Fish Study
SDG: SCDHEC

Laboratory: Eurofins Atlanta

Unless otherwise noted, all analytes for this laboratory were covered under each accreditation/certification below.

Authority	Program	Identification Number	Expiration Date
South Carolina	State	98016	06-30-25

The following analytes are included in this report, but the laboratory is not certified by the governing authority. This list may include analytes for which the agency does not offer certification.

Analysis Method	Prep Method	Matrix	Analyte
6010D	3050B	Solid	Lead

END OF REPORT