GENERAL PLAN OF OPERATIONS

APPENDIX 1

INTEGRATED MONITORING PLAN

November 2019

Prepared by:

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Acronyms

AAC	Alaska Administrative Code
ABA	Acid Base Accounting
ADEC	Alaska Department of Environmental Conservation
ADF&G	Alaska Department of Fish & Game
ADNR	Alaska Department of Natural Resources
ADC	Atmospheric Depositional Container
AP	Acid Generation Potential
ARD	Acid Rock Drainage
AWQS	Alaska Water Quality Standards
BMP	Best Management Practices
CFR	Code of Federal Regulations
EPA	Environmental Protection Agency
EPT	Aquatic insects (Ephemeroptera, Plecoptera, and Trichoptera)
FB	Field Blank
FL	Fork Length (tip of snout to the end of middle caudal fin rays)
Forest Service	U.S. Department of Agriculture Forest Service
FWMP	Fresh Water Monitoring Plan
GPO	General Plan of Operations
HGCMC	Hecla Greens Creek Mining Company
HDPE	High Density Polyethylene
ICP	Inductively Coupled Plasma Analysis
IDL	Instrument Detection Limit
IMP	Integrated Monitoring Plan
LCS	Laboratory Control Sample
MD	Method Blank
MDL	Method Detection Limit
MLE	Maximum Likelihood Estimate
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MW	Monitoring Well
NEPA	National Environmental Policy Act
NP	Acid Neutralization Potential
NNP	Net Neutralization Potential
QAPP	Quality Assurance Project Plan
TDF	Tailings Disposal Facility
TDR	Time Domain Reflectometry
RTM	Real Time Monitoring
SOW	Statement of Work
UGM	Undifferentiated Glacial and Marine
USEPA	U.S. Environmental Protection Agency

Units of Measure

mm	millimeter
cm	centimeter
m	meter
km	kilometer
m^2	meter squared
m^3	meter cubed
mg	miligram
µg	microgram
ml	milliliter

1 Introduction

Hecla Greens Creek Mining Company (HGCMC) prepared this *Integrated Monitoring Plan (IMP)* to meet the operational needs of the site while addressing the goals and objectives of the federal and state regulatory agencies. This Plan was developed to meet the requirements of the Alaska Department of Environmental Conservation (ADEC) in accordance with AS 46.03.010 et. seq. and 18 AAC 60.015 et. seq. and 18 AAC 80.005 et. seq. and the U.S. Forest Service (Forest Service) implementation of 40 CFR § 1505.3 to ensure monitoring requirements identified in the National Environmental Policy Act (NEPA) documents that relate to HGCMC are met.

The Greens Creek Mine is owned and operated by HGCMC, a wholly-owned subsidiary of Hecla Mining Company, Inc. The Greens Creek Mine is located near Hawk Inlet on northern Admiralty Island, in the Tongass National Forest, approximately 18 miles southwest of Juneau, Alaska (Figure 1-1). The mine site is situated partly within the Admiralty Island National Monument, and completely within the municipal boundaries of the City and Borough of Juneau. The mine site is comprised of federal and patented mining claims. The Greens Creek mine facilities are located within the Greens Creek, Zinc Creek, Tributary Creek, and Cannery Creek watersheds.

The Forest Service has issued special use permits/leases for various aspects of the operations. In addition, HGCMC holds 17 patented mining claims (7,300 acres), 645 unpatented mining claims (12,200 acres) in the area, and 17 acres in Hawk Inlet under a warranty deed with Bristol Resources, Inc.

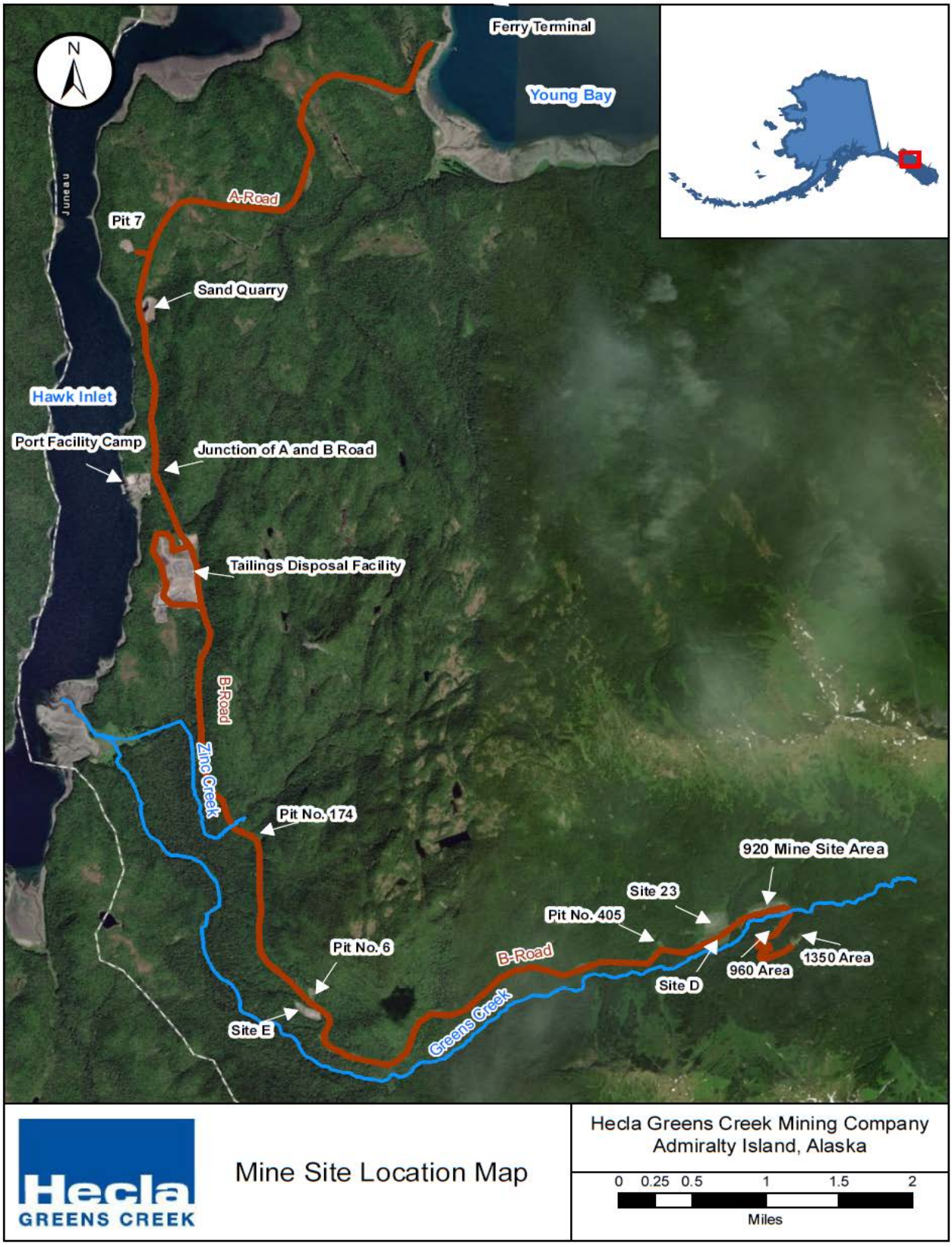
The Greens Creek Mine has been in operation since 1989, with only a short temporary cessation of operations due to low metal prices from April 1993 until July 1996. HGCMC produces three concentrates containing four payable metals (silver, zinc, lead, and gold) for shipping to smelters around the world.

1.1 Purpose

It is the goal of HGCMC to operate the mine and milling processes in a manner that will ensure the protection of the environment. This monitoring plan will assist HGCMC in the establishment and refinement of operating procedures to ensure the long-term protection of land, wildlife, and water resources. Periodic updates of the monitoring plan will coincide with regulatory changes, five-year environmental audit reviews, process modifications, or anomalies noted as a result of monitoring and sampling.

This IMP and the associated *Quality Assurance Project Plan (QAPP) of the General Plan of Operations Appendix 1 Integrated Monitoring Plan* (Appendix 1.A), are an intricate part of the environmental and operational management system for the Greens Creek Mine. The overall operation and each process component have specific management plans, which share common elements with this monitoring plan.

FIGURE 1-1: GREENS CREEK PROJECT LOCATION



To minimize duplication of information and rationale for specific monitoring and sampling requirements, the reviewer needs to reference the following site analytical reports and management plans:

- *Greens Creek Mine Reclamation and Closure Plan, GPO Appendix 14, April 2019*
- *Greens Creek Mine Tailings Disposal Facility Management Plan, GPO Appendix 3, April 2019*
- *Greens Creek Mine Waste Rock Management Plan, GPO Appendix 11, April 2019*
- *Greens Creek Mine Standard Operating Procedure, Construction Rock Environmental Characterization, March 2010*
- *Greens Creek Mine 2010 Site Water Balance, February 2010*
- *Greens Creek Mine Site 23/D Hydrogeology and Geochemistry Analysis, March 2004*

1.2 General Information

Location: (Mine Portal) Latitude 58° 04'58" North, Longitude 134° 37'57" West
Name of Facility: Hecla Greens Creek Mining Company – Greens Creek Mine
Type of Facility: Underground Silver, Lead, Zinc, and Gold Mine and Milling Operation

Corporate Information:
Business Name: Hecla Greens Creek Mining Company
PO Box 32199
Juneau, Alaska 99803
Telephone: (907) 789-8100

General Manager: Brian Erickson

Hecla Greens Creek Mining Company is a wholly-owned subsidiary of:
Hecla Mining Company
6500 N. Mineral Drive, Suite 200
Coeur d'Alene, Idaho 83815

Designated Contact Person for Regulatory Issues:
Name: Christopher Wallace
Title: Environmental Affairs Manager
Telephone: (907) 790-8473

1.3 Objectives

Compliance monitoring is undertaken to verify that the project operates within permit limitations thereby minimizing the impact on the environment during operations and post-closure. The objective of this document is to provide HGCMC and state/federal regulators with a clear and concise plan that lists monitoring and sampling criteria for surface water and groundwater quality, geochemical characterization of materials, geotechnical stability of structures, and aquatic biological resources present at the site. The relevant procedural information for sample collection, sample analysis, data analysis, and reporting are contained in Appendix 1.A.

1.4 Summary of Monitoring

This IMP presents the elements of HGCMC's monitoring and sampling program that have been initiated for operations. The monitoring and sampling areas cover critical aspects of the project's infrastructure, including Hawk Inlet facilities, Tailings Disposal Facility (TDF), waste rock sites, inactive rock quarries, and mill site. This document will be updated as needed, based on regulatory changes, periodic reviews, process modifications, and the results of monitoring which indicate that further attention may be warranted.

Table 1-1 presents a summary of the water quality monitoring, biological monitoring, geochemical characterization, and geotechnical monitoring activities performed during the period of active mining operations. More detailed information on monitoring at each facility and component is provided in subsequent sections. Compliance monitoring of wastewater and stormwater discharges, air emissions and other resources, such as Hawk Inlet monitoring, are addressed under specific permits and not included in this document.

TABLE 1-1: SUMMARY OF MONITORING ACTIVITIES

Facility	Component	Method	Media	Parameters	Frequency
Project Area	Water Quality Compliance	Fresh Water Monitoring Program	Surface Water	Suite P or Q	Monthly
			Ground Water	Suite Q	Quarterly
	Aquatic Community Health	Biological Monitoring	Fish, macro-invertebrates, periphyton	Metals, abundance, diversity	Annually
	Const. Rock Characterization	ABA, ICP *	Rock	NNP, metals	As needed
Tailings Disposal Facility	Internal Water Quality Monitoring	Water sampling	Ground Water	Suite C1 or C2	Quarterly
			Pore Water	Suite L1	Annually
	Tailings Characterization	ABA, ICP	Tailings	NNP, metals	Annually
	Stability	Visual inspection	TDF surface	Checklist	Monthly
		Compaction	Tailings	% moisture, density	Quarterly
		Wells, piezometers	GW, pore water	Water level, pressure	Monthly
	Fugitive Dust	ADC *	Dust	mass, Pb, Zn	Bi-weekly
Site 23	Internal Water Quality Monitoring	Water sampling	Ground Water, Drains	Suite C1 or C2	Quarterly, Some sites Annually
	Waste Rock Characterization	ABA, ICP	Waste Rock	NNP, metals	Quarterly
	Stability	Visual inspection	Site 23 surface	Checklist	Monthly
		Survey	hubs, inclinometers	movement	Semi-Annually
		Wells, piezometers	GW, pore water	Water level, pressure	Monthly
Inactive Waste Rock Sites & Quarries	Internal Water Quality Monitoring	Water sampling	Surface Water	Suite C1	Annually or Semi-Annually
	Material Characterization	ABA, ICP	Rock	NNP, metals	Once every five years
	Stability	Visual inspection	Area	cracks, sloughs	Quarterly
Dam Systems	Geotechnical Stability	Visual inspection	Embankments, spillway	ADNR-Dam Safety Checklist	Monthly
		Survey	Monuments	movement	Quarterly or Semi-Annually

* ABA – Acid-Base Accounting
ICP – Inductively Coupled Plasma
ADC – Atmospheric Deposition Container

2 Fresh Water Monitoring Program

2.1 Project Background

Monitoring and sampling surface and groundwater resources is an integral part of the environmental protection measures at the project.

The Hecla Greens Creek Mining Company (HGCMC) Fresh Water Monitoring Program (FWMP), in conjunction with the Quality Assurance Project Plan (QAPP) (Appendix 1.A), documents the methods and procedures for sample collection, laboratory analysis, data management, and information utilization necessary to ensure that monitoring requirements are fulfilled. Both surface water and groundwater monitoring are included. The FWMP and QAPP are to be reviewed and updated as needed to ensure the best use of resources, appropriate quality of data, and use of the results in management decisions.

Before 1995, freshwater monitoring at the Greens Creek Mine was conducted under two documents; the Greens Creek Fresh Water Monitoring Operations Manual 1988; and the draft General Plan of Operations (GPO), Appendix 1 (June 1992). These documents were revised and combined into the 1995 Fresh Water Monitoring Program. The purpose of the 1995 revision was to update the information goals for monitoring, and the standard procedures for sample collection, laboratory analysis, data handling, data analysis, and information utilization. Information goals are specific quantitative and qualitative statements describing the information expectations of the monitoring program. Information utilization is defined as how the information derived from data analysis is reported and applied to management decisions.

The 2000 revision of the FWMP was a result of a Greens Creek sponsored interagency regulatory review of the Greens Creek Mine. The Project Team consisted of representatives from the Greens Creek Mine and several State and Federal regulatory agencies, including the State of Alaska Department of Natural Resources (ADNR), Environmental Protection Agency (EPA), United States Forest Service (Forest Service), United States Fish and Wildlife Service (USFWS), State of Alaska Department of Fish and Game (ADFG), State Attorney General Office (AGO) and State of Alaska Department of Environmental Conservation (ADEC). The purpose of the review was to allow the State and Federal agencies having jurisdiction over the mine to ascertain overall compliance with existing authorizations and environmental laws and to implement corrective action, if needed; amend existing authorizations or plans, if necessary; and process any new authorizations required to provide for confidence in regulatory compliance and environmental effectiveness of the Greens Creek programs. The revision incorporated changes requested and approved by the participating regulatory agencies and Greens Creek Mine.

This 2019 revision was undertaken in conjunction with the renewal of the Waste Management Permit. An environmental audit of Greens Creek Mine was required as part of the permit's renewal. HDR Engineering, Inc. conducted the audit and submitted a final report in January 2019. Recommendations from the audit have been incorporated into the IMP and QAPP.

2.2 Actions for Compliance Monitoring Directives

Implement the revised FWMP.

Conduct annual reviews of information goals, analytical data, statistical analyses, and sampling frequencies to ensure that information utilization needs are met.

Apply the information derived from data analysis and interpretation to management decisions.

2.3 Data Quality Objectives (DQOs)

DQOs are quantitative and qualitative objectives for the quality of the data used. DQOs define the quality of services requested from the laboratory and are used in the quality assurance (QA) review by comparing the quality control (QC) data against the DQOs to qualify the data as entirely usable, estimated, or rejected as unusable. Refer to the QAPP (Appendix 1.A) for additional detail on the DQOs.

2.3.1 Qualitative DQOs

Qualitative DQOs are established for representativeness and comparability.

Representativeness is a determination of how well the sample represents environmental conditions. It is addressed by monitoring site selection and sample collection and handling protocols. Requirements for blank analyses and QA reviews of blank data verify that samples have not been contaminated in the sampling or analytical processes.

Comparability is a determination of how well data from different sources compare to each other. It is addressed by ensuring appropriate method detection limits are achieved, and QC measures and QA data reviews are performed to verify that the data are of known and acceptable quality.

2.3.2 Quantitative DQOs

Quantitative DQOs are established for method detection limits (MDLs), minimum levels (MLs), precision, accuracy, and completeness.

MLs are established for each analyte at 90% of the Alaska Water Quality Standards (AWQS) with one exception: the ML for chromium will be the same as for chromium VI. Waters monitored under this plan are protected for all uses, and the most protective standard is applicable (18 AAC 70.020(1)). Of concern for these waters is protection for the growth and propagation of freshwater fish, shellfish, other aquatic life, and wildlife (18 AAC 70.020(1)(c)).

For those analytes having a hardness dependent AWQS, the hardness value used to calculate the standard for determining the ML was based on the 25th percentile of the measured hardness at surface water and groundwater sampling sites over the previous five years. Surface water and groundwater hardness values were summarized independently for the 25th percentile determination. Table 3 in the QAPP (Appendix 1.A) shows the MLs for each analyte evaluated by this plan.

MDLs are calculated based on the ML using certain information developed by EPA (EPA 821-B-95-002, April 1995). For the purposes of this plan, the MDL=ML÷3.18, rounded up to the same number of significant digits as the AWQS for that analyte. Table 3 of the QAPP (Appendix 1.A) shows the MDLs for each analyte evaluated by this plan.

Precision is a measure of the ability to replicate analysis and is expressed as the relative percent difference (RPD). The RPD criterion for water samples is ±20% and is only applicable when the analyte concentration is more than five times the instrument detection limit (IDL), and if the native amount is not greater than four times the spiked amount.

Accuracy is a measure of how close the analytical result is to the actual concentration of the analyte and is expressed as percent recovery (%R). The Matrix Spike/Matrix Spike Duplicate (MS/MSD) criteria are 75-125 %R for all metals. The criteria are only applicable for MS/MSD analyses if the native amount is not greater than four times the spiked amount. The accuracy limits for the Laboratory Control Sample (LCS) are method dependent, e.g. 90-110 %R for Inductively Coupled Plasma Analysis (ICP).

Completeness is a measure of how many planned analyses for all analytes resulted in usable data, defined as all data that is not rejected and is expressed in percent (%). The completeness criterion is 95% for a water year, which is October 1st through September 30th.

2.4 Monitoring Sites

HGCMC has designated freshwater monitoring sites including those utilized in the FWMP. Once a site is established it is never changed and remains a site even if it becomes inactive. If a site is obliterated by construction or moved, the original site number becomes inactive, and the new monitoring location is given a unique site number.

Monitoring can be discontinued, and a site becomes inactive for a variety of reasons. These include if the site is destroyed due to construction or natural phenomenon, was stopped at some time in the past prior to the 2014 FWMP revision or deemed no longer necessary by the regulatory agencies and HGCMC.

2.4.1 Description and Location of Fresh Water Monitoring Sites

Table 2-1 lists all surface, and groundwater monitoring sites in the current FWMP contains a brief location description and coordinates. Figure 2-1 depicts the approximate locations of compliance monitoring sites. These sites are considered "active." They have been determined to meet the analytical and informational needs necessary for comparison and interpretation of previous data to those of the current conditions at the site. Other sites that were previously required for monitoring are called "inactive" and are not discussed here. Details of the inactive sites can be found in previous FWMPs and annual reports.

One new surface water monitoring site has been added to replace a site that was abandoned due to natural phenomena. Greens Creek upstream of the mining activities meandered and carved a new channel segment that bypasses the Upper Greens Creek Site 48. A new site was established at the confluence of Greens Creek with the new channel and is designated Site 63. This is documented in an Alaska Department of Fish and Game memorandum dated September 7, 2018.

TABLE 2-1: ACTIVE MONITORING SITE LOCATIONS

Site #	Site Name	Location	Latitude	Longitude
6	Middle Greens Creek	The site is on Greens Creek downstream of the mine and mill. It is about 15 m upstream of the confluence of Bruin Creek.	58°04'47.424" N	134°38'25.849" W
9	Tributary Creek	The site is on Tributary Creek, about 800 m downstream of the TDF, and about 500 m upstream of the confluence with Zinc Creek.	58°06'22.040" N	134°44'44.100" W
13	Upper East Mine Drainage	Small drainage to the East from the 1350 adit. The site is below a former waste rock storage area.	58°04'47.685" N	134°37'39.951" W
27	MW-2S	The site is an 2.4 m deep well completed in the peat/sand unit. It is in muskeg about 60 m south of the TDF.	58°06'48.546" N	134°44'38.365" W
29	MW-3S	The site is a 4.6 m deep well completed in the peat/sand unit. It is in muskeg about 50 m west of the TDF.	58°06'59.860" N	134°44'51.821" W
32	MW-5S	This site is a shallow well completed in the peat/sand unit. It is in the Muskeg about 50 m west of the TDF.	58°06'57.732" N	134°44'51.225' W
46	Lower Bruin Creek	The site is on Bruin Creek downstream of waste rock areas 23 and D. It is about 20 m upstream of Greens Creek.	58°04'46.450" N	134°38'32.580" W
49	Upper Bruin Creek	The site is on Bruin Creek upstream of waste rock area 23.	58°05'04.070" N	134°38'30.410" W
54	Greens Creek below D-Pond	The site is on Greens Creek downstream of waste rock areas 23 and D. It is about 20 m upstream of the confluence of Gallagher Creek.	58°04'41.681" N	134°38'46.529" W
57	MW-23-00-3	The site is a 20.7 m deep well completed in gravel and clay. It is upgradient of waste rock area 23.	58°04'59.933" N	134°38'39.881" W
60	Lower Althea Drainage	The site is on a small drainage about 150 m downstream of Pond 7.	58°04'41.770" N	134°45'08.432" W
61	Greens Creek Floodplain	The site is a surface water site ~ 40 m west of D Pond in the floodplain.	58°04'43.480" N	134°38'52.910" W
62	Greens Creek Lower than 54	The site is on Greens Creek downstream of waste rock areas 23 and D. It is about 250 m downstream of Site 54.	58°04'38.650" N	134°39'06.000" W
63	Upper Greens Creek	The site is on Greens Creek upstream of all mining activities. It is about 250 m upstream from the Greens Creek bridge at the 920 portal.	58°04'57.720" N	134°37'42.240" W
609	Lower Further Drainage	This site is on small drainage about 200 m downstream of the TDF.	58°07'05.707" N	134°45'06.332" W
711	Greens Creek above Site E	This site is on Greens Creek about 50 m upstream of Site E.	58°04'08.425" N	134°43'27.181" W
712	Greens Creek below Site E	This site is on Green Creek about 200 m downstream of Site E.	58°04'13.858" N	134°43'42.438" W

Site #	Site Name	Location	Latitude	Longitude
37	Cannery Creek Upper	Located on Cannery Creek about 50 m upstream of the B-Road		
1923	Cannery Creek Lower	Located on Cannery Creek downstream of the B-Road		
TBD	TDF Well			
TBD	TDF Well			
TBD	TDF Well			

2.4.2 Monitoring Sites

FIGURE 2-1: FRESH WATER MONITORING SITE LOCATION MAP

2.5 Monitoring

2.5.1 Site Selection

A primary criterion for selecting a monitoring site is that it must meet the DQO for representativeness. A monitoring site must be in the appropriate location so that collected data is representative of the facility or condition (i.e., natural background) it is intended to monitor. This is determined based upon an annual review, analysis, and interpretation of collected data.

The current FWMP sites listed in Table 2-1 have been demonstrated to be representative for monitoring potential water quality impacts from the mine operations, while also maintaining an efficient monitoring program. The addition and activation of any new sites would be associated with either a facility expansion and the need to establish proper up-gradient and down-gradient compliance points or in response to a statistically significant change in water quality at an existing site and the need to better characterize the nature and extent of the change. Changes to FWMP monitoring sites must be approved by the regulatory agencies.

2.5.2 Frequency Selection

Monitoring frequency is determined based upon results of previous data analysis, planned future uses of data, and changes in mine operations. The frequency will be sufficient to detect any seasonal trends. For new monitoring sites, quarterly or monthly sampling will be sustained until sufficient samples are taken to conduct statistical trend analyses. Exceptions can be made based on site accessibility and hazards, such as brown bear activity. Unexpected events may also affect monitoring frequency.

2.5.3 Analytical Parameters for Fresh Water Monitoring

The suite of analytical parameters for samples collected at a given site in a given sample period is based upon an annual review of the information goals. The suite of analytical parameters is selected to meet those informational needs based on results from previous analysis.

Surface water sample analytical Suite P (Table 2-2: Suite P (Surface Water)) contains the shortest list of critical analytes developed over the course of the mine life. The listed parameters generally characterize constituents of concern at surface water monitoring sites.

A more comprehensive analytical profile is used for groundwater analysis and periodically used for surface water, typically during months of low flows. Suite Q (Table 2-3) analytical profile contains additional dissolved metals associated with the Greens Creek orebody or waste rock that are important indicators for groundwater and surface water quality during periods of low flow.

TABLE 2-2: SUITE P (SURFACE WATER)

Analytical Parameters	
Conductivity	pH
Temperature	Sulfate
Total Alkalinity	Hardness
Dissolved Metals	
Arsenic	Lead
Cadmium	Mercury
Copper	Zinc

TABLE 2-3: SUITE Q (GROUND AND SURFACE WATER)

Analytical Parameters	
Conductivity	pH
Temperature	Sulfate
Total Alkalinity	Hardness
Dissolved Metals	
Arsenic	Mercury
Barium	Nickel
Cadmium	Selenium
Copper	Silver
Chromium	Zinc
Lead	

2.5.4 Fresh Water Quality Monitoring Schedule

The frequency of sampling surface and groundwater sites has been developed over the life of the operation with numerous adjustments as the program has continuously been re-evaluated and refined. Table 2-4: Fresh Water Monitoring Schedule provides a general overview of annual surface and groundwater sampling.

TABLE 2-4: FRESH WATER MONITORING SCHEDULE

Site	Site Name	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec
006FMS	Middle Greens Creek	Q		P		P		P		P		P	
009FMS	Tributary Creek-Lower	Q		Q		Q		Q		Q		Q	
013FMS	Mine Adit Discharge East					Q		Q		Q		Q	
027FMG	Monitoring Well 2S					Q		Q		Q		Q	
029FMG	Monitoring Well 3S					Q		Q		Q		Q	
032FMG	Monitoring Well 5S					Q		Q		Q		Q	
046FMS	Lower Bruin Creek	Q				P				P		Q	
063FMS	Upper Greens Creek	Q		P		P		P		P		P	
049FMS	Control Site Upper Bruin Creek	Q				P				P		Q	
054FMS	Greens Creek below D-Pond	Q		P		P		P		P		P	
057FMG	Monitoring Well -23-00-03	Q				Q				Q		Q	
060FMS	Althea Creek - Lower					Q		Q		Q		Q	
061FMS	Greens Creek Floodplain	Q		P		P		P		P		P	
062FMS	Greens Creek Lower Than 54	Q		P		P		P		P		P	
609FMS	Further Creek Lower					Q		Q		Q		Q	
711FMS	Greens Creek Above Site E					Q				P			
712FMS	Greens Creek Below Site E					Q				P			
37FMS	Cannery Creek Upper	Q		P		P		P		P		Q	
1923FMS	Cannery Creek Lower	Q		P		P		P		P		Q	
TBD	TDF Well					Q		Q		Q		Q	

Site	Site Name	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec
TBD	TDF Well					Q		Q		Q		Q	
TBD	TDF Well					Q		Q		Q		Q	

KEY:
P= Suite P
Q= Suite Q

2.6 Sample Collection

Following the current monitoring schedule in Section 2.4.4, water samples are collected using protocols designed to minimize bias from systematic and/or erratic contamination introduced during sample collection. Procedures for the collection of surface water and groundwater samples are provided in the QAPP (Appendix 1.A).

2.7 Sample Documentation, Packaging, and Shipping

All FWMP samples are collected by HGCMC personnel, packaged, and transported off Admiralty Island for laboratory analyses. Information on the protocols for documentation, packaging, and shipping of samples is provided in the QAPP (Appendix 1.A).

2.8 Sample Analyses

Independent laboratories will be used for water sample analyses. A written statement of work (SOW) defining contractual requirements, DQOs, and data deliverables for the FWMP will be prepared and sent to any laboratory selected to conduct water quality analyses. Laboratories will also be periodically audited.

2.8.1 Scope of Work for Analyses

A written SOW shall be provided to the selected laboratory(s) giving direction on the analytical work to be furnished, which includes the following.

- The anticipated number of samples, including QC samples, the analytes to be monitored, and the DQOs that must be met will be stated.

- The laboratory shall notify HGCMC immediately if any sample is lost due to a lab accident. This prompt notification allows HGCMC the option of re-sampling to replace the sample or taking additional samples to confirm the unusual result.

- Water quality sample analyses shall be performed within holding times and using the approved methods listed in 40 CFR § 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.

- The laboratory shall be responsible for biological sample preparation. This includes the final cleaning of benthic macroinvertebrate samples of the debris before analysis and rinsing

periphyton samples with DI water before analysis.

- The laboratory shall provide their latest comprehensive MDL study, done in accordance with 40 CFR § 136 Appendix B, to the third party conducting the QA review and will provide updates as they are done.

- Field Blank (FB) samples shall be analyzed for the same suite of analytes as the sample collected at the site where the FB was collected.

- For every sample group, a method blank (MB) shall be analyzed for each analyte scheduled for analysis in that sample group.

- For every sample group, a laboratory control standard shall be analyzed that is traceable to different source standards than the ones used for calibrations. The LCS will have a concentration for each required metal at its MDL level or, for those analytes whose MDL is outside the range of the calibration curve, at a concentration appropriate to the curve. Duplicate analysis of this LCS will also be performed.

- For every sample group matrix, spike/matrix spike duplicate (MS/MSD) analyses shall be performed for all the metals scheduled for that group. The laboratory will select the site on which MS/MSD analyses are performed and rotate it monthly to ensure all sites are included. In the laboratory the sample from the selected site will be split into thirds and two of them spiked accordingly. At least one fraction will be spiked, and the laboratory will select that fraction. The spiking level should result in concentrations at or above the AWQS for each metal.

- The laboratory shall keep the complete set of raw data for the samples including sample preparation logs and instrument calibration information in easily accessible files for a period of at least 6 months

- The laboratory shall notify HGCMC immediately upon any change in certification status, personnel, equipment, or any other aspect of laboratory operations that may adversely impact the integrity of the samples or the attainment of DQOs for the analytical results.

2.8.2 Scope of Work for Data Deliverables

The written SOW provided to the selected laboratory(s) shall give direction on the data deliverables to be continuously been in a report to HGCMC, on laboratory letterhead, within 45 days of sample receipt, with the following information:

- Document the date samples were received by the laboratory, whether the shipping container was received with the seal intact, and if all samples listed on the sample inventory sheet were present.

- Document whether inductively coupled plasma (ICP) was used and if raw data were generated before inter-element and background corrections were applied.

- Document any problems, QC criteria exceedances, holding time exceedances, and observations affecting sample integrity and provide a detailed description.

- Provide a statement of authenticity and certification of the data with the date the report was generated and dated signature of the lab manager.

- Document the results of all sample analyses, including blind duplicates submitted at HGCMC's discretion, with HGCMC sample numbers and their corresponding laboratory number(s), date received, analyses performed (analyte and dissolved, total, or total recoverable fraction), analytical result, IDL, MDL, ML, and unit of measurement for each analyte.

- Document the results of the MB and FB analyses for each analyte.

- Document the results of the LCS analyses including the calculated %R for each analyte, and the RPD of the LCS results for each analyte.

- Document the results of the MS/MSD analyses including the calculated %R for each analyte, and the RPD of the MS and MSD results for each analyte.

- Document all analyses not meeting holding times, MDLs, or the precision and accuracy control limits by flagging them in the analytical report and provide definitions for the flags.

- Provide a compatible electronic file with the analytical results in a format compatible with the Environmental Management Database System, to reduce errors and labor required for data entry in the HGCMC database.

2.9 Quality Assurance

Data used for decision making are to be of known and acceptable quality. All data are reviewed by a qualified QA reviewer to determine if the DQOs have been met. A qualified QA reviewer has no bias about the data quality and can evaluate the possible impacts on data comparability introduced by the use of multiple laboratories in the analysis of samples. As a result of the QA review, data may be qualified as estimated or rejected for failure to meet the DQOs.

The requirements for field and laboratory quality control measures and methods for data verification and validation are provided in the QAPP (Appendix 1.A).

2.10 Reporting

Data specification and collection provide the foundation of a monitoring system. Review, evaluation, and reporting the data is the next essential step. Information users base decisions on the monitoring results and contents of reports.

2.10.1 Purpose of Reports

Documentation and communication of information resulting from data evaluation is the purpose of reports.

- Defined, periodic, HGCMC reports document the following:
  a) The monitoring activities.

  b) The information gained in the monitoring process.

  c) The results of information evaluation.

- Reports communicate information that is used as follows.
  a) To provide the basis for management decisions.

  b) To provide the basis for assessing the effectiveness and efficiency of the FWMP.

2.10.2 Responsibility for Reports

HGCMC is responsible for the preparation and distribution of the reports specified in this section.

2.10.3 Distribution of Reports

The reports specified in this section are to be distributed in electronic format to the Forest Service, and ADEC.

2.10.4 Reports of Exceptions

The purpose of a report of exception is to communicate changes or unanticipated problems and resulting actions. Exceptions are very short-term temporary conditions not requiring an FWMP modification. An example is the taking of additional samples for a short period of time to verify an unusual result. The report also documents the event for the historical record.

The content of a report of exception varies depending on the exception. The information provided should be clear and fully explained.

Reports of exception are made as needed and may be either an emergency or not an emergency. Emergencies are events with actual or potential significant resource damage. A report for an emergency such as a chemical spill affecting freshwater is distributed as soon as possible.

Events that are unanticipated and unscheduled but do not appear to cause or have the potential of causing significant resource damage are not time-critical. They may be reported along with the next scheduled report.

2.10.5 Biannual Reports

The purpose of the biannual reports is to provide information which the ADEC, Forest Service, and HGCMC use to determine the following:

a) If any changes to the monitoring schedule are needed.

b) If any other changes to the FWMP are needed including any aspects of monitoring, evaluation, or reporting.

c) If any changes in best management practices (BMPs) are needed.

The content of the biannual reports covers activities during the preceding 6 months. The reports will cover January 1 to June 30 and July 1 to December 31, and include the following items:

a) A table of contents.

b) A list of interventions (procedural changes, natural phenomena, and mine operation changes) that could possibly affect data during the reporting period and any effects detected from visual data analyses.

c) A list of any negotiated FWMP or mine BMP modifications that were made including changes to the monitoring schedule and the problems they address.

d) A list of company and agency personnel who were involved in the FWMP during the reporting period and their function or job title.

e) A list of proposed program modifications including proposed revisions to the monitoring schedule, and discussion/rationale for proposed changes based on data analysis.

f) The data analyses required for each individual monitoring site include the following:

(1) An interpretive report of the conclusions drawn from the data analyses including comparisons to previous years' data, baseline data, and background data.

(2) A clarification of what data were used in the analyses and identifying any data which was not included such as data that was qualified as rejected by the QA reviewer or confirmed as an outlier based on the outlier analyses and re-sampling performed by HGCMC.

The evaluation and handling of potential outliers will be performed using the guidance found in the EPA document "Guidance for Data Quality Assessment", EPA/600/R-96/084. Section 4.4 of the EPA document provides guidance on identifying potential outliers, choosing the proper statistical test, evaluating the results and documenting the process.

The first step is to review the data to determine whether any of the points may be potential outliers. Graphical representations are the most common method. Once potential outliers are found, the data must undergo a statistical test designed to detect outliers. The statistical test chosen must be applicable to the distribution type of the data set and the number of potential outliers in the data set.

At this point, the results of the statistical outlier test must be evaluated fully to determine whether the potential outliers are a true outlier or simply an extreme value that may be part of the data set's distribution. No data points should ever be excluded solely based upon statistical testing. Any potential outliers identified by proper statistical testing must be verified. The verification of outliers must include scientific support that the data point is truly an outlier. If further checking does not suggest the point is an outlier, the results of the statistical test cannot be used to label the point as an outlier. If the support is found the data point may be identified as an outlier.

The data analysis performed on the data set to which the outlier belongs must be performed once with the outlier included and again with the outlier excluded. The results are then to be reviewed to determine the impact on the data analysis with regards to the contribution of the outlier data points.

The final step for outlier designation is documentation. The rationale for the choice of the outlier test must be given, along with the results. Then, the supporting scientific facts must be given to demonstrate the outlier is not just a statistical anomaly but was, in fact, a true outlier. Finally, the impact on the outlier data point had on the statistical processing of the data must be given.

(3) A list of qualified data from the QA review reports.

(4) A chronological list by the site of all data collected during the reporting period that exceeds AWQS.

(5) A comparison of medians will be made. Data outliers shall not be used in the data set used for median comparisons. Values between the MDL and ML will be used. A notation will be included in the report that states which values used in the median comparison fall between the MDL and ML. Data values below the MDL shall be assigned a value of zero for the purposes of median comparisons. A description of applicable median comparisons follows.

Analytical results must be statistically compared to determine whether concentration changes have occurred in a geographic situation or over time. Since nearly all data is

not from a normally distributed population, it is necessary to compare the medians between the data sets. Although the initial step involves difference testing of the medians, several additional steps are taken to fully evaluate the meaning of that difference testing

The first step is an analysis of variance-based upon the ranked data. Ranking must be used due to the nonparametric distributions. The results of the analysis of variance are evaluated to estimate what level of significance is attached to the difference testing of the means. The significance level is then compared to the project objectives to ascertain whether the two data sets differ. This significance level must receive equal attention as did the result of the difference testing.

Multiple comparisons testing is then performed so that the indications given in the earlier median testing and significance testing are confirmed. If the multiple comparisons testing does not support the conclusions of the earlier testing, then further examination is needed to rule out the possibility that false indications were given. If the multiple comparisons testing confirms the other testing, then there is greater confidence the original results are indicative of site conditions. The multiple comparison methods chosen must be sufficiently robust to either confirm or countermand the simpler one-on-one testing.

(6) X-Y graphs of the analytes specified and a trend analysis if indicated by visual inspection of the graphs. The scale shall be appropriate to conduct visual trend analyses, i.e., each scale will be as confined as possible based on each data range. AWQS criteria will be displayed on the graphs. Data outliers shall not be displayed on the x-y graphs. Data qualified by the QA contractor shall be labeled as such on the x-y graphs. Data values below the MDL shall be assigned a value of zero for the purposes of the x-y graphs.
Any indeterminate trend (may or may not be a trend) shall be verified using statistical trend analysis. Data outliers shall not be used in the statistical trend analysis. Data values below the MDL shall be assigned a value of zero for the purposes of the trend analysis. Trend analyses must be performed on the data sets such that the appropriate level of confidence is achieved. This level is based upon the traditional false positive/false negative rate (related to α) that can be tolerated. Also, the statistical test chosen must be powerful enough to conclude whether a trend is present or not. In other words, the test cannot be so weak that no conclusion is reached, even on data where clear trends are evident.

Also, the test must be selected, and the test parameters are chosen such that the distribution of the data is either properly matched or is non-parametric. If the data are tested and proven to be normally distributed, then normal statistical tests shall be utilized. If the data distributions cannot be matched, then non-parametric testing is needed.

Once these two issues are resolved, the statistical test must be able to handle a seasonality component. The first step in the process is to choose a proper technique to determine whether the data have a seasonality component. If they do, the trend test must have a seasonality parameter to adjust for this component in the data.

Further, the data set must contain enough data within the periodicity of the season to allow for this testing. This means that a seasonality component cannot be identified unless there are frequent enough data points within each season to allow for this conclusion to be reached. An example would be that a seasonal component of about 6 months (one wet and one dry season per the calendar year) cannot be tested if the data were only obtained quarterly or semi-annually unless independent proof of the seasonal component can be provided.

2.10.6 Data Management

This section documents information storage, access, and archive practices for both hardcopy and electronic information.

2.10.7 Reports

- Access to records is controlled by the remoteness of the location and the limited access to mine premises.
- All incoming original hardcopy laboratory reports and associated QA review reports are filed chronologically at the mine.
- Electronic copies of HGCMC's reports are stored on a local server, which is backed up and maintained by the information technology department.
- Original hardcopies never leave the premises. They are photocopied as needed for distribution and satisfying information requests.
- Hardcopy reports may be archived 6 years after the date of creation. They may be moved to a less accessible location provided the previous five years of hardcopy are kept readily accessible.

2.10.8 Electronic Data

- A relational database containing all the FWMP data is maintained by HGCMC at the mine. Copies or partial copies of the database may be distributed to others as needed to facilitate data analysis.

- Data security is maintained by limiting access rights to the database files through network login IDs and passwords. Passwords are changed as needed.

- Laboratory data are electronically imported or manually entered into the HGCMC database. Associated qualifiers are manually entered after the QA review report is finalized and received by HGCMC.

- Personnel will be trained in reading the datasheets, electronic data transfer, and using the database before data entry is performed.

- All data (100%) entered into the database manually, and a sample (10%) of the data imported into the database electronically, are verified against the hardcopy before the data are used for analysis.

- Data produced before January 1989 may be archived to maintain processing speed and reduce the size of the backups.

- If data is archived it must be reloaded before database upgrades or enhancements are made to ensure it remains accessible and compatible. After the changes are completed it may be archived again.

- Changes to the database structure or utilities may be needed as a result of changes to the FWMP, data analysis protocols, or other reasons. A log of database changes, enhancements, problems, and fixes is kept to aid in troubleshooting.

2.11 Program Audits

Program audits provide an evaluation of the efficiency and effectiveness of the QA functions of the FWMP. This feedback loop provides the information needed for continuous improvement of the FWMP. The audit procedures below evaluate how well the information goals and DQO's are being met.

2.11.1 Responsibilities

HGCMC has the primary responsibility for ensuring that the data are of known and acceptable quality and the FWMP has been implemented as designed and thus has primary audit responsibility.

The Forest Service and ADEC have regulatory oversight responsibility and may perform independent audits on a random and/or as-needed basis. Other agencies may also perform audits.

2.11.2 Data Acquisition Audits

A review of the data collection system will evaluate whether the QC procedures in the FWMP are being followed and if documentation of these activities is sufficient to establish the quality of the information collected. Findings may be used to make improvements to the FWMP or to initiate corrective action by HGCMC for lapses in execution or documentation.

- HGCMC will perform one audit per year. The results of this audit will be included in the applicable report.

- The laboratory and QA review reports for a randomly selected month in conjunction with the FWMP and the current monitoring schedule are reviewed for the following determinations:

  a) The completeness of the laboratory data versus what was planned in the monitoring schedule and if the correct analytical fractions were analyzed.

  b) Whether or not analyses were performed within holding times.

c) Whether or not a QA review of the data was performed, and the amount of data qualified as estimated or rejected.

2.11.3 Data Management Audits

A review of data management evaluates whether the procedures for data management in the FWMP are being followed and if data integrity is being maintained. If lapses in data management are found corrective action will be taken by HGCMC and documentation kept on file at the mine site.

- HGCMC will perform one audit per year. The results of this audit will be included in the applicable report.
- The data management specifications of the FWMP are reviewed for the following determinations:

a) Whether all reports were received within the specified time and copies forwarded as required.

b) Whether hardcopy and electronic data are stored such that unauthorized access is minimized.

c) Whether or not laboratory data have been QA reviewed and qualified if necessary, which is documented with a report.

d) Whether laboratory reports and QA review report originals are in the files where expected.

e) Whether the laboratory data with appropriate qualifiers have been accurately entered into the database.

f) Whether the statistical analysis of the data is being appropriately performed and reports are found in the files where expected.

g) Whether the FWMP has been reviewed and updated as needed.

h) Whether previous copies of updated versions of the FWMP are retained and found in the files where expected.

2.11.4 Laboratory Audits

A review of the laboratory's facility, equipment, personnel, organization, and management will evaluate the data reliability the laboratory can produce. The laboratory as a system is verified against the documentation provided in their QA manual, their MDLs, and the SOW defining the services to be provided to HGCMC. A complete and thorough audit may be done through contractual services. HGCMC

may choose to accept the results of a third-party audit done for other purposes, such as drinking water certification or national accreditation programs such as A2LA, instead of performing their own audit.

- Laboratory audits should be performed at least every five years.
- Guidelines for laboratory audits are available from the USEPA or ASTM Standard Practice E548. The basic elements are summarized below.

  a) Organization:
  Well Organized
  Duties/Responsibilities Clearly Defined
  Supervision/Inspection/Audit/Self-Appraisal Program

  b) Staff:
  Technical Competence Qualifications Documented
  Training/Maintenance/Upgrading of Competence Sufficient Supervision
  Adequate Number of Staff

  c) Equipment:
  Adequate in Kind and Quality Maintained

  d) Calibration/Reference Standards

  e) Test Methods/Standard Operating Procedures

  f) Environment/Facilities:
  Space
  Physical/Chemical Control Housekeeping

  g) Samples:
  Handling
  Storage
  Integrity/Chain of Custody

  h) Analytical Reports and Record-Keeping

  i) QA program with specified QC activities

- A copy of the letter of certification or accreditation may be used as the documentation of an audit. Otherwise, the auditor will prepare a report listing the items reviewed and the conclusions of the review with any recommendations. Copies will be provided to the Forest Service and HGCMC and kept on file at the mine site.

3 Internal Monitoring of Mine Waste Rock

The Greens Creek Mine has one active waste rock facility (Site 23) and multiple inactive waste rock sites. Characterization and monitoring of active and inactive mine waste rock sites is ongoing and will continue over the active life of the mine. Classification and segregation of characterized waste rock provide the basis for ongoing management at active and inactive sites. Geochemical characterization and geotechnical stability monitoring of Site 23 is required by the Waste Management Permit.

The geochemical characterization programs for the Greens Creek Mine are well established. Waste rock from the mine is visually and geochemically characterized and managed accordingly. Representative samples for the characterization of mine waste are based on operational and geological records identifying materials mined.

Material characterization is performed using one of the established analytical procedures: multi-element ICP analysis, and Acid-Base Accounting (ABA) using the Modified Sobek Method to determine acid Neutralization Potential (NP), Acid generation Potential (AP) and Net Neutralization Potential (NNP). These analytical tools are used to accurately classify the material and its potential to affect water quality.

Sites, where characterized materials have been placed for either permanent or temporary disposal, are monitored for water quality. The water quality monitoring is an internal monitoring program and not part of the FWMP. The sampling is of contact water (i.e., pore water, leachate or seepage) within the waste rock facility boundaries and is therefore not expected to be compliant with AWQS. The objective of the monitoring is to track water quality trends to support predictions regarding geochemical weathering processes and effects on water quality. The results from the internal monitoring may be used to refine facility-specific management plans or reclamation plans.

The following subsections provide an overview of the monitoring schedules and type of characterization testing for active and inactive sites.

3.1 General Classification of Mine Waste Rock

Due to its variable geochemical properties and acid generation potential, mine waste rock is managed based on the following classification system. The waste rock classification by an experienced geologist at the underground blast face or muck pile is based on visual characteristics as verified through analytical testing.

Waste Rock Types:

- **Class 1:** This material has a Net Neutralization Potential (NNP) greater than 100 tons calcium carbonate ($CaCO_3$)/1000 tons. No special handling is required.
- **Class 2:** This material has an NNP value between 100 and -100 tons $CaCO_3$/1000 tons and is placed at least two feet from the final pile surface.
- **Class 3:** This material has an NNP value between -100 and -300 tons $CaCO_3$/1000 tons and is placed at least two feet from the final pile surface.
- **Class 4:** This material has an NNP value of less than -300 tons $CaCO_3$/1000 tons and is kept underground as fill.

Waste rock at Greens Creek has two general conditions; fresh waste rock from the mine and weathered waste rock from inactive waste rock sites. New waste rock is generally alkaline (pH 7-9). Weathered waste rock from inactive sites is either near neutral (pH 6-8) or acidic (pH <6).

3.2 Characterization and Monitoring of Rock

The schedule for the monitoring and analytical testing of the active waste rock site, inactive waste rock sites, and rock used in the construction of facilities are listed in Table 3-1: Monitoring: Active / Inactive Waste Rock Sites & Quarries. Analytical suites are listed in Table 3-2: Analytical Suites: Water Quality Monitoring.

TABLE 3-1: MONITORING: ACTIVE / INACTIVE WASTE ROCK SITES & QUARRIES

Site Name	Monitoring Type	Parameters	Frequency	Responsibility
Site 23 (Active)	site	visual inspections	monthly	SOps, Env
	groundwater	C1 or C2	annually	Env
	water levels (wells, piezometers)	depth to water, pressures	semi-annually, some sites monthly or quarterly	Env, SOps
	leachate - drains	C1 or C2, flow	quarterly	Env
	rock characterization	ABA, ICP (metals), paste pH	quarterly	Env
	rock characterization – in situ *	ABA, ICP (metals), paste pH	once every 5 years	Env
	survey hubs, inclinometers	stability, movement	semi-annually	SOps
	material placement	tons, cubic yards	daily, monthly	Mine; SOps
Inactive Waste Rock	site	visual inspections	quarterly	Env
	surface water	C1 or C2	annually	Env
	rock characterization – in situ *	ABA, ICP (metals), paste pH	once every 5 years	Env
Construction Rock**	rock characterization	ABA, ICP (metals), paste pH	as necessary – prior to use	SOps

KEY:

* Paste pH, ABA, multi-element ICP from outer pile slope or quarry wall at least every five years, at a depth deep enough to encounter Class 2/Class 3 waste rock if less than five feet.

** Five samples per lithologic unit should be considered the minimum number of samples necessary to represent a potential source area or volume of rock less than 100,000 tons. For larger tonnages collect at least 10 samples per 100,000 tons of rock produced in an individual campaign or over multiple years.

ABA = Acid Base Accounting determines NP, AP, NNP
ICP= Multi-element Inductively Coupled Plasma
VI = Visual Inspection
LY = Lysimeter (Suite L1)
SOps = HGCMC Surface Operations Department
Env = HGCMC Environmental Department
Mine = HGCMC Mine Operations Department

TABLE 3-2: ANALYTICAL SUITES: INTERNAL WATER QUALITY MONITORING

Sampling Suite C1	Parameters		
	Arsenic	Zinc	Alkalinity
	Barium	Antimony	Silica
	Cadmium	Mercury	Chloride
	Chromium	Aluminum (total)	Sulfate
	Copper	Calcium	Orthophosphate
	Iron	Magnesium	Thiosulfate
	Lead	Sodium	Total Dissolved Solids
	Manganese	Potassium	Total Suspended Solids
	Molybdenum	Hardness	Bicarbonate
	Nickel	DOC	Alkalinity
	Silver	Thallium	Acidity
	Selenium	Ammonia, TKN	

Sampling Suite C2	Parameters		
	Arsenic	Thallium	Alkalinity
	Cadmium	Nickel	Acidity
	Chromium	Zinc	Chloride
	Copper	Calcium	Sulfate
	Iron	Magnesium	Total Dissolved Solids
	Lead	Sodium	Total Suspended Solids
	Manganese	Potassium	

Sampling Suite L1	Parameters		
	Aluminum	Manganese	Sodium
	Arsenic	Magnesium	Potassium
	Barium	Molybdenum	DOC
	Cadmium	Nickel	Ammonia
	Calcium	Silver	Chloride
	Chromium	Zinc	Sulfate
	Copper	Antimony	Thiosulfate
	Lead	Selenium	Orthophosphate

All metals are dissolved unless otherwise noted

3.2.1 Mine Waste Rock Characterization and Monitoring

Greens Creek uses the numerical system described in Section 3.1 for production waste rock classification and placement. Waste rock classification is based on rock type and pyrite content. Interpretation of development and exploration drilling information allows mine geologists and engineers to estimate the quantities of argillite and phyllite anticipated during mining. Where practical the mine plan tries to minimize development in high pyrite rock, although mining potentially acid-producing rock is unavoidable. Production geologists visually inspect the active mining face and muck piles to determine the waste rock lithology and pyrite content, estimate the NNP value and assign the material a Classification Number. Chip samples of the material are periodically collected for ABA analysis. The ABA results help document the types of material produced and validates the visual classification system.

Waste rock disposal management follows the following criteria:[1]

- Mixing of Class 2 and Class 3 is allowed to avoid physical discontinuities in the waste rock dump;
- Priority use of Class 1 is of higher beneficial use at Site 23 and the TDF area as an outer slope encapsulating layer;
- Place Class 1 as a 0.61 m thick layer at Site 23 and the TDF.

3.2.2 Site 23 Characterization and Monitoring

Class 1, 2, and 3 waste rock are brought to the active waste rock Site 23 by underground haul trucks and placed in stockpiles. The designated placement zones linked to the three classes of rock are marked on the active lift area prior to placement of waste rock and are sampled quarterly. Quantities of Class 1 and Class 2/3 waste rock placed at Site 23 will be tracked and included in the quarterly reports to ADEC, as required by the Waste Management Permit.

Active Areas:

- Two composite samples from each stockpile of Class 1 and Class 2/3 quarterly for ABA. Samples are collected from the top 30.5 cm within active placement areas.
- Outer side slopes will be sampled at least every five years, at a depth deep enough to encounter Class 2/3 waste rock if less than five feet. Samples will be analyzed for ABA, paste pH, and ICP metals.
- Groundwater/leachate samples will be collected quarterly from the finger drains and curtain drains when the flow is greater than 1 liter per minute (Suite C1 or C2).
- Groundwater wells (EDMS Site #: 50, 51, 326, 1263) will be sampled annually (Suite C1).
- Site 23 will be visually monitored for signs of damage or potential damage from settlement, ponding, leakage, instability, frost action, erosion, thawing of the waste, or operations at the site. Monitoring will be performed weekly and documented monthly as required by the Waste Management Permit.

3.2.3 Inactive Waste Rock Sites Characterization and Monitoring

Water quality monitoring is conducted at several inactive waste rock dump sites on a semi-annual or annual basis. Geochemical samples are taken once every five years and analyzed for ABA, paste pH, and ICP metals. This monitoring is conducted until the waste rock is removed, the site is reclaimed, and stabilized. Once all the material is removed from an inactive waste rock site, that site can be removed from the sampling program.

Site E is an example of an inactive waste rock site. It is located 4.6 miles up the B Road between the Hawk Inlet port facility and the 920-mill site. Approximately 279,000 m^3 of waste rock and glacial till were placed at the site from 1988 to 1994. Waste rock removal from the site and co-disposal of the material with tailings at the tailings facility is expected to significantly improve water quality in the small drainages

[1] ADEC approved this change to the Greens Creek disposal method in a letter dated May 13, 2004.

between Site E and Greens Creek, while also improving pore water chemistry and geotechnical stability of the TDF.

- The frequency of monitoring surface water is dependent upon the yearly activity at the site; greater activity results in increased monitoring frequency. Minimally sites are monitored annually.
- Outer side slopes of the exposed waste rock will be sampled at least every five years. Samples will be analyzed for ABA, paste pH, and ICP metals. The location of each sample will be recorded on a map.

3.2.4 Construction Rock Characterization

All construction rock currently used on-site outside of containment is shipped in from quarries not associated with the Greens Creek operation. Construction rock originating from offsite is sampled by personnel from the surface operations, environmental or geology departments (or consultants) who are familiar with acid rock drainage (ARD) and metals leaching principles. The number of samples required depends on the compositional variability of the rock and the amount of rock or aggregate to be quarried:

- Five (5) samples per lithologic unit are considered the minimum number of samples necessary to represent a potential source area or volume of rock less than 100,000 tons.
- At least 10 samples per 100,000 tons of rock or greater produced in an individual campaign or over multiple years are collected.

Samples of non-weathered rock are to be collected from outcrops or through drilling and should represent the range of compositional variability of the source area. Five to ten pounds of rock per sample is generally sufficient for routine geochemical characterization. The sample may be a composite of several pieces of rock from an area or zone representing a single rock type. Composites of mixed rock types should be avoided.

Depending on the intended use of the rock and the results of the ABA and ICP analyses, additional testing may be warranted. Additional tests may include:

- Whole-rock assay for major and trace elements reported as oxides;
- Mineral content determined by X-Ray diffraction;
- Abrasion tests to determine rock durability;
- Kinetic leach tests (40-week humidity cell) to determine the potential for metals and sulfate mobility.

4 Internal Monitoring of Tailings

The Internal Monitoring of Tailings describes monitoring within the tailings pile area, in contrast to the compliance monitoring (under the Fresh Water Monitoring Program) at peripheral facility boundary sites. As such, data generated by the Internal Monitoring Plan effort are not for compliance purposes but provide a continuing perspective on in-pile geochemical processes.

There are three principal issues that affect potential ARD and metal leaching from the Greens Creek tailings facility including the setting and design of the individual facility, the operation of the facility, and reclamation and closure. Aspects of the facility design, operation, and closure that serve to minimize ARD and metal leaching risk are described in Tailings, Appendix 3 and the Reclamation Plan, Appendix 14 of the General Plan of Operations.

4.1 Monitoring Objectives

Monitoring is conducted to confirm the following:

- The site is constructed according to the approved construction plans;
- The site is maintained in a stable condition over the short and long term;
- Water management system components are effective and maintained as designed;
- Geochemical and hydrologic processes are defined and meet expectations with respect to limiting oxidation and leaching and minimizing the effects on the receiving environment; and,
- The effectiveness of Best Management Practices to control fugitive dust from escaping the facility.

Inspections and monitoring for the tailings facility, including water levels, water quality and geochemical testing of the tailings and production rock, are described in this section and summarized in Table 4-1.

4.2 Tailings Characterization and Monitoring

During the period the mine is active samples of mill tailings are collected prior to transport to the TDF and post-placement samples are collected at the TDF. These samples are analyzed for ABA.

- When operating, samples are collected daily from the mill tailings filter press. From these daily samples, a monthly composite sampled is obtained for analyses.
- Six (6) samples are collected annually from active placement areas.

Every five years until final closure of the tailings facility, older tailings are sampled to determine the NP and AP values as a proactive measure to further characterize the TDF material. The intent of this sampling is to monitor the consumption of the buffering capacity of tailings.

4.3 Other Monitoring

See Table 4-1 for a summary of monitoring activities for the tailings facility. Visual observations and material sampling are used to ensure that the construction of the facility is according to approved construction plans. Visual observations and routine maintenance ensure that the water management system is functioning as designed. Water quality data, flow, and level monitoring, material sampling and information from site meteorology stations are used to define geochemical and hydrologic processes

occurring at the site. This information is evaluated with respect to design expectations, and modifications are made, if necessary, to minimize effects on the receiving environment in the short and long term.

The number and location of water samples collected each year may vary due to the constantly changing conditions within this active facility. Efforts are made to extend and protect monitoring wells as the height of the tailings pile increases, but occasionally wells get damaged or destroyed. Suction lysimeters buried within the pile can also lose their functionality due to the deterioration of the tubing over time. New suction lysimeters are installed as the pile grows. The number of wells and lysimeters located within the tailings facility ensures that sufficient data can be collected to satisfy the monitoring objectives.

TABLE 4-1: SUMMARY OF MONITORING AND SAMPLING ACTIVITY – TAILINGS FACILITY

Monitoring Type	Parameters	Frequency	Responsibility
site	visual inspections	daily, monthly	SOps, Env
material placement	tons cubic yards	daily (load counts) monthly (survey)	SOps
groundwater	C1 or C2	annually	Env
water levels (wells, piezometers)	depth to water, pressures	semi-annually, some sites monthly or quarterly	Env, SOps
drains, wet wells	C1 or C2, flow	quarterly	Env (WQ) SOps (flow)
suction lysimeters	L1	annually	Env
tailings characterization	ABA: ICP (metals), paste pH	monthly annually	Mill Env
tailings characterization – *in situ*	ABA, ICP (metals), paste pH	once every 5 years	Env
compaction	percent moisture, wet density	quarterly or annually	SOps

KEY:
ADC = atmospheric deposition container
ABA determines AP, NP, and NNP
C1, C2, and L1 sampling suite parameters are listed in Table 3-2
SOps = HGCMC Surface Operations Department
Env = HGCMC Environmental Department
Mill = HGCMC Mill Operations Department
* The frequency of monitoring is dependent upon the season and the ambient conditions

5 Fugitive Dust Monitoring

The control of fugitive dust from the tailings facility is a required mitigation measure in the 2013 Final Environmental Impact Statement and Record of Decision for the Tailings Disposal Facility expansion. The monitoring of fugitive dust emissions is a requirement of the Waste Management Permit. Deposition of dust to the west, south, and southwest of the tailings facility is believed to be the source of elevated (above background) lead concentrations that have been recorded in Tributary Creek.

Monitoring conducted between 2011 and 2019, visual observations, and operational experience indicate that dust loss from the tailings pile occurs when dry, windy conditions persist at the site. These conditions typically occur for short periods between mid-November and late March when high-pressure systems produce cold, dry weather and strong northerly winds. Table 5-1 summarizes the fugitive dust monitoring program associated with the TDF.

TABLE 5-1: SUMMARY OF FUGITIVE DUST MONITORING

Monitoring Type	Parameters	Frequency	Responsibility
fugitive dust – ADC	total deposition mg/m^2/day, lead deposition µg/m^2/day	weekly or bi-weekly or monthly	Env
fugitive dust – real time	mg/m^3	continuous - averaged 15min interval	Env
fugitive dust – visual	presence of dust	Daily	SOps

KEY:
ADC = atmospheric deposition container

5.1 Atmospheric Depositional Containers

Atmospheric depositional container (ADC) monitoring is used to determine long term temporal changes in the fugitive dust load, along with spatial distribution of the dust. The ADC program implemented at the TDF is an adaptation of the American Society for Testing and Materials (ASTM) D1379 Standard Test Method for Collection and Measurement of Dustfall (Settleable Particulate Matter). ADCs are a basic and rugged passive accumulator of windblown dust and the data is used to supplement other monitoring data. Though crude and non-specific this methodology is useful in the study of long-term trends.

5.1.1 Description of Sample Locations

HGCMC will monitor fugitive dust emissions with six ADCs deployed to the south, southwest, and west of the TDF (Figure 5-1). These six sites were chosen because the primary area of placement from 2020 through 2024 will be in the southern extent of the TDF, and that the predominant wind direction is from the north and northeast. Two of these sites have been monitored since 2011 and another site since 2015.

TABLE 5-2: ADC SAMPLE SITES

Site	Direction relative to active placement (2020 through 2024)	Status
1901	northwest	Sampled since 2011
1902	west	Sampled since 2011
1904	south	Sampled since 2015
2101	southwest	New site (2020)
2102	southwest	New site (2020)
2103	south	New site (2020)

5.1.2 Sample Collection and Laboratory Analysis

ADCs will be collected and replaced with clean ADCs once a month (28 days + 7) from April 1st to October 31st and weekly (7 days + 2) or biweekly (14 days + 4), atmospheric condition-dependent, from November 1st to March 31st. Any ADC can be collected sooner than the specified minimum frequency but will not exceed the maximum frequency specified.

ADCs are filtered through a pre-weighed 90 mm filter with 1.5-micron pore size. The filters are dried and weighed to determine the total mass of material on the filter. About once a quarter, the filters are sent to an independent laboratory for total lead analysis.

5.1.3 Data Analyses and Reporting

Results from the monitoring equate to the amount of material that passes through the opening of the ADC over the sampling period. This information is used to calculate the average daily lead deposition rate.

$$D_{Pb} = W/A/P \ \mu g/(m^2/day)$$

where:

A = collection area, the cross-sectional area of the inside diameter of the top of the container, m^2,

W = particulate Pb mass from the laboratory analysis of the filter, µg, and

P = length of the sampling period, days.

The results from the ADC monitoring will be evaluated in context with the meteorological data and surface operations activities. Visual and statistical temporal analyses will be conducted using the data collected, and if statistically significant negative changes are identified and corroborated by other monitoring data, additional dust control measures will be implemented as defined in the dust mitigation plan. Biannually the monitoring data and analysis will be included in the reporting to the ADEC required under the WMP. Biennially HGCMC will propose revisions to the fugitive dust mitigation measures if WQS exceedances continue to occur at Site 9 Tributary Creek.

5.2 Real-Time Monitoring

The monitoring described under Section 5.1 Atmospheric Depositional Containers is used to determine the long-term effectiveness of the fugitive dust controls. To facilitate the short-term evaluation of the mitigation measures HGCMC plans to install a (perhaps two) real-time monitor (RTM) to the south of the

TDF (Figure 5-1). This monitor will measure the volume of dust in a cubic meter of air and the data will be trended in real-time for supervisorial review.

5.2.1 Description of Sample Location

Long term monitoring (2011-2018) shows that the deposition to the south and southwest of the facility is normally the highest. HGCMC plans to install the RTM to the south of the facility, proximal to Site 1904 which is situated at the head of the Tributary watershed. If a second monitor is installed the planned location is adjacent to the Pond 10 pumphouse.

5.2.2 Data Collection and Analysis

Monitoring data will be collected and trended on a continuous basis. For analysis purposes, the data will be averaged over a fifteen-minute interval. Short-term temporal analysis of the data will be conducted in context of fugitive dust mitigation measures implemented on a daily/weekly basis. Furthermore, the data will be compared to ADC data collected over the same period.

5.2.3 Reporting

Graphs of the RTM data will be prepared and submitted biannually. These graphs will also include markers indicating when fugitive dust mitigation activities were implemented. When capable a statistical analysis will be conducted for dusting periods in which additional measures were implemented. This will allow for an evaluation of the control under consistent meteorological conditions.

5.3 Visual Monitoring and Reporting

Daily the tailings disposal facility operator will make and record their observations with regards to fugitive dust at the TDF throughout the day shift. This will include observations at the beginning of the shift as to signs of dusting from the previous night. These observations by the nature of tailings placement will be limited to daylight hours (~6.5 hours for the shortest day of the year).

It is expected that these observations will correlate well with the RTM measurements. Therefore, during periods of the day when it is dark or the operator is not present the RTM will be used to signal the need for additional mitigation controls. HGCMC will include in the biannual reporting a summary of these observations as compared to the RTM.

FIGURE 5-1: DUST MONITORING LOCATIONS

6 Biological Monitoring

The role of biological monitoring is to ensure the continued use of Greens Creek and its tributaries by fish and other aquatic species and to document the continued health of all levels of the biological community: primary productivity, invertebrate communities, and fish. Biological monitoring will also detect early changes to the aquatic community that may result from changes in water chemistry, either through surface or groundwater inputs to the system.

Results from biological monitoring are compared to baseline conditions, or if baseline data are unavailable, to a reference site that is unaffected by the mine. There were few baseline studies conducted before the development of the Greens Creek Mine using current state-of-the-art protocols. The existing biological monitoring program is designed to compare present conditions to future conditions, with consideration given to any previous monitoring. HGCMC contracts with the ADF&G for the monitoring and reporting for this activity. This document serves as the quality assurance plan for biological monitoring.

6.1 Elements of the Biological Monitoring Program

The biological monitoring program for the Greens Creek Mine addresses the following factors:

1. Abundance and condition of juvenile fish;
2. Whole-body concentrations of Cd, Cu, Hg, Pb, Se, Ag, and Zn in juvenile fish;
3. Periphyton biomass, estimated by chlorophyll-a concentrations;
4. Abundance and community structure of benthic invertebrates;

6.2 Summary for Biological Monitoring

Table 6-1 summarizes the sites to be sampled, factors sampled at each site, and sampling frequency.

TABLE 6-1: SUMMARY OF BIOLOGICAL MONITORING SITES

Site Name	Monitoring Objective	Compare to:	Frequency	Factors	Time to Sample
Upper Greens Creek (Site #63)	Routine, control		Annually	FA, FM, P, MI	July
Greens Creek Below D-Pond (Site #54)	Routine, treatment	Control	Annually	FA, FM, P, MI	July
Tributary Creek (Site #9)	Baseline	Change over time	Annually	FA, FM, P, MI	July

KEY:
WQ - water quality
FA - fish abundance
FM - fish metals content
P - periphyton biomass
MI - macroinvertebrate abundance, community
Baseline - the conditions at the beginning of the biological monitoring program

TABLE 6-2: SUITE R (BIOLOGICAL MONITORING PARAMETERS)

Juvenile Fish	Periphyton	Aquatic Invertebrates
1. Relative abundance and condition. 2. Subsample from each sample site will be analyzed for whole-body concentrations of; • Cadmium, • Copper, • Mercury (added in 2012) • Lead, • Selenium, • Silver • Zinc. (Metals are to be reported as total per dried weight of tissue). 3. The laboratory shall also report the percent moisture of the samples so that wet weight values can be calculated. 4. Water temperature will be measured.	1. Samples will be collected for estimates of Chlorophylls a, b, and c.	1. Samples will be collected to determine abundance and community structure.

Biological monitoring parameters identified in Suite R further augments the surface and groundwater monitoring and sampling program to accurately track the viability of the aquatic environment in Greens Creek and its tributaries.

6.2.1 Description of Sample Locations

Upper Greens Creek: FWMP Site 63

Site 63 is located upstream of all mine and mill facilities, except for exploratory drilling, and serves as the control reach for comparing data collected downstream at Site 54. Site 63 is at approximately 260 m elevation, and about 0.7 km upstream from the concrete weir in Greens Creek, which blocks upstream fish passage.

Greens Creek below D-Pond: FWMP Site 54

Site 54 is located approximately 25 m downstream of production rock storage areas 23 and D and monitored to detect potential effects from the rock storage areas and treatment ponds, in addition to the mine, mill and shop facilities upstream. Site 54 is at about 225 m elevation and 0.4 km downstream of Site 6.

Tributary Creek: FWMP Site 9

This site was previously monitored for water quality under the former Fresh Water Monitoring Plan (FWMP) from 1981 through 1993. It was reactivated in 2001 for inclusion in the biological monitoring program. Site 9 is located 1.2 km downstream of the dry-stack tailings facility at about 25 km elevation and is monitored to detect potential effects from the tailings facility. This is the closest free-flowing stream reach suitable for biomonitoring to the TDF. As these disposal facilities were situated on the hydrographic divide, there is no comparable upstream site.

6.3 Periphyton Biomass

6.3.1 Rationale

Many fish species are highly migratory, and their presence or absence does not adequately describe the health of a specific reach of stream. Periphyton, or attached algae, is sensitive to changes in water quality. Their abundance confirms that productivity is occurring at specific locations within a water body. Algae generally have short life cycles; therefore monitoring biomass provides an ideal indicator to detect short-term effects (Barbour et al. 1999).

6.3.2 Sample Collection and Laboratory Analysis

The protocol for collecting and analyzing stream periphyton is derived from the Freshwater Biological Sampling Manual, Resources Inventory Committee, Province of British Columbia (1997), Alaska Department of Fish and Game (1998), and Barbour et al (1999). Periphyton sampling should not occur in-stream near minnow traps that are soaking as this violates the conditions necessary for depletion trapping.

Ten rocks are collected from the streambed of the creek in each study reach for sampling. A 5 by 5 cm square of high-density foam is placed on each rock; material around the foam square is removed by scrubbing with a toothbrush and then rinsed away using a spray bottle containing stream water. The foam square is removed and the isolated area scrubbed with a toothbrush. Loosened periphyton is rinsed onto a 1 µm (47 mm diameter) glass fiber filter attached to a vacuum pump. After extracting as much water as possible from the sample on the glass fiber filter, approximately 1 ml saturated $MgCO_3$ is added to the filter to prevent acidification and conversion of chlorophyll to phaeophytin. The glass fiber filter is wrapped in a large paper filter to absorb additional water, and placed in a sealed, labeled plastic bag with desiccant. The samples are frozen on-site in a light-proof cooler with additional desiccant and transported to laboratory for analysis. Samples are kept frozen until laboratory analyses are conducted by Division of Habitat staff.

Periphyton sampling at Site 9 will occur after fish sampling to avoid disturbing juvenile fish, though biologists must work carefully to avoid disturbing stream substrate which could affect periphyton results. Alternatively, samples could be collected upstream or downstream of the fish sample reach.
Laboratory analysis requires the extraction of chlorophyll pigments and measurement of chlorophyll concentrations on a fluorometer or spectrophotometer. Measurements on a spectrophotometer require a centrifuge. Laboratory analysis follows established protocol (USEPA and standard methods).

6.4 Benthic Macroinvertebrate Density and Richness

6.4.1 Rationale

Benthic macroinvertebrates classified in the Orders Ephemeroptera (mayflies), Plecoptera (stoneflies), and Trichoptera (caddis flies), collectively known as EPT taxa, are sensitive to changes in water quality and an important food source for fish. Most benthic macroinvertebrates have a complex one-year (or more) life cycle and limited mobility, therefore, benthic macroinvertebrates provide an ideal indicator to detect short-term and long-term effects within local aquatic communities (Barbour et al. 1999). An abundant and diverse group of EPT taxa indicate a healthy local aquatic community and results can be used to assess overall stream health with other local studies (e.g. periphyton biomass).

6.4.2 Sample Collection and Laboratory Analysis

Eight benthic macroinvertebrate samples are to be collected from each site using methods modified from Barbour et al (1999). More than eight can be collected to improve calculated mean densities. There is flexibility with respect to which invertebrate sampling equipment is used as long as is it consistent with the methods described in Barbour et al. (1999) (e.g. Surber or Hess sampler). In the past, samples were collected from each site with a Hess sampler using a random sample design. Samples are to be collected exclusively from riffle habitats where the greatest amount of taxonomic richness and density are usually observed. This sample design eliminates the variability from sampling pools or other habitats where pollution-sensitive taxa are less likely to be present. The sample collection methods should be standardized throughout the year by having one biologist collect all invertebrate samples, spend the same amount of time collecting each sample (e.g. 5 minutes), and dig to the same depth at each sample site (10-15 cm).

For sample collection, the Hess sampler is pushed into the stream bottom, encompassing 0.086 m^2 of the substrate, to define the sample site. The substrate is manually disturbed and rocks are brushed within the sample area and then removed. Fine gravels are disturbed to about 10–15 cm depth to collect buried individuals. Macroinvertebrates are collected using a 363 μm mesh net, then relocated to a pre-labeled 500 mL Nalgene® bottle and preserved in 80% denatured ethanol and shipped to the laboratory for processing. Macroinvertebrate samples are later sorted from debris and identified to the lowest practical taxonomic level by a taxonomist.

Macroinvertebrate sampling at Site 9 should occur after fish sampling to avoid disturbing juvenile fish distribution. Alternatively, samples could be collected upstream or downstream of the fish sample reach.

6.5 Juvenile Fish Populations

6.5.1 Rationale

Salmonids are highly migratory, predators, and good indicators of long-term effects and habitat conditions (Barbour et al. 1999), therefore monitoring fish populations affords another biological level to detect change within the aquatic community and assess overall stream health.

6.5.2 Sample Collection

Fish populations are sampled using a modification of a three-pass removal method described by the Forest Service (Bryant 2000). Fish are collected using 0.635 cm square mesh galvanized Gee's minnow traps baited with salmon roe that was previously treated with Betadine® disinfectant solution. Approximately 25 minnow traps are deployed within each sample reach; the final number of traps used are dependent on stream conditions and habitat availability during field sampling. Natural features such as shallow riffles or small waterfalls are used to help define the upper and lower reach boundaries, in order to minimize fish migration into the sample reach, where possible. To assist with meeting the closed-reach assumption of the three-pass removal method, baited "block" traps are also set upstream and downstream of each sample reach to capture potential migrants.

Sample reaches are identified by aluminum tree tags and flagging set during previous years' sampling. There may be slight variation in reach lengths between sites, depending on available habitat for minnow trapping. The target length for each sample reach is 50 m.

Minnow traps are placed throughout each sample reach focusing on pools, undercut banks, bank alcoves, under root-wads or logjams, and other habitats where fish are likely to be captured. In higher velocity sites, rocks are placed in the traps to increase trap weight and provide cover for fish. In each fish sample reach, the traps are set for about 1.5 hours, and then retrieved and captured fish are identified to species, measured to FL, and placed in a mesh holding bag in the stream.

Block traps are set for the entire 1.5 hours sampling period. Fish captured in block traps are counted and identified to species, but not included in further analyses. Ten Dolly Varden from the first trapping period at each site are to be retained for laboratory analysis of whole-body metals concentrations. Fish not retained for the metals analyses are returned to the stream reach immediately after sampling is completed.

Each salmonid captured is weighed to investigate the mean fish condition between sites and years for each species.

6.5.3 Data Analyses and Presentation

Juvenile fish abundance shall be reported as the number of fish, by species, captured during a single pass (1.5 hour) depletion trapping event, following established methods.

6.6 Metals Concentrations in Juvenile Fish

6.6.1 Rationale

Monitoring whole-body metals concentrations in juvenile fish assesses metal loading in aquatic communities near the Greens Creek mine. Current year data are compared to previous years' data to detect change over time and water quality data can be compared as well to examine relationships. Weber, Scannell, and Ott (2001) documented metals accumulation in juvenile fish tissues within two months of migration into mineralized tributaries, therefore results can detect both short-term and long-term changes in tissue metal concentrations.

6.6.2 Sample Collection and Laboratory Analysis

Ten juvenile Dolly Varden within the size range 85–125 mm FL are captured in the minnow traps collected from each site for whole-body metals analyses. The specified size range improves the likelihood of sampling only resident fish, assuming the age of fish in that size class is 2–3 year Dolly Varden that have not migrated to sea. Sample fish are measured to FL, individually packed in clean, pre-labeled bags and frozen on-site until transport to the laboratory. Biologists handling the fish wear VWR Certiclean Class 100 Nitrile gloves to reduce the risk of metal contamination.

At the laboratory, the fish are weighed without removal from the bags, and correction made for the weight of the bag. The fish are submitted to a private analytical laboratory (Columbia Analytical Services,

Inc. in Kelso, Washington), where they are digested, dried, and analyzed for silver (Ag), arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), lead (Pb), selenium (Se), and zinc (Zn) on a dry-weight basis, with percent total solids also reported.

6.6.3 Reporting

- Periphyton Biomass

  Periphyton samples will be analyzed on either a fluorometer or a spectrophotometer.

  Chlorophylls a, b, and c will be calculated from samples measured on the spectrophotometer.

  Periphyton biomass will be reported as mg chlorophyll-a / m^2 of the stream substrate. Comparisons will be made among the control and treatment sample sites using appropriate statistical methods. Data will be presented graphically, and the data values will be contained in appendices to biomonitoring reports.

- Benthic Macroinvertebrates

Data compilation and analyses for benthic samples should follow the protocol of the Alaska Stream Condition Index (Major and Barbour 1999), as described below and with modifications.

List of Metrics:

<u>Abundance Measures</u>

Total invertebrates counted per subsample

Total aquatic invertebrates per subsample

Total terrestrial invertebrates per subsample

Estimated total aquatic invertebrates per sample

Estimated total terrestrial invertebrates per sample

% sample terrestrial

% sample aquatic

<u>Taxonomic Richness Measures</u>

Total aquatic taxa Average taxa/sample

No. of Ephemeroptera taxa

No. of Plecoptera taxa

No. of Trichoptera taxa

Community Measures (estimate of total sample)

Est. number Ephemeroptera

Est. number Plecoptera

Est. number Diptera

Percent Ephemeroptera

Percent Plecoptera

Percent Diptera

Richness Measures

Composition Measures

% EPT

% Chironomidae

% Dominant Taxon

The metrics are calculated from the data collected and recorded on the laboratory bench sheet after the laboratory identification and analysis.

- Abundance of Rearing Fish

Analysis of fish population estimates should include a graphical display of fish abundance trends at all bio-monitoring sites, and a statistical comparison of means (or medians) between populations at control and treatment sites.

Data analysis should include graphical displays of annual fish population trends by species for each bio-monitoring site. Graphs displaying species/length distribution by year should also be provided.

Potential change in juvenile fish abundance between the Greens Creek control and treatment bio-sites will be analyzed. The results of this analysis should be compared with similar statistics for water quality, metals content, periphyton biomass, macroinvertebrate indices and toxicity collected at these monitoring sites for the same time periods. This information will be used to evaluate and document potential cause-effect relations between changes in water quality, and aquatic biota abundance, distribution and community structure.

- Metals Concentrations in Rearing Fish

The median, maximum, and minimum concentrations of each metal will be reported for each sampling site. Comparisons will be made among sampling sites. Metals concentrations also will be compared to metals concentrations in whole-body juvenile fish of similar species from

other regions of Alaska (e.g. Weber Scannell et al., 1995, 1998, 2000b; Snyder-Conn et al. 1992, 1993).

7 Geotechnical Monitoring and Inspections

The Greens Creek Mine has a TDF, two waste rock areas, and three registered dams that require geotechnical monitoring for stability and structural integrity. The TDF and waste rock sites (Site 23 and Site D) are monitored for potential movement and long-term stability as part of the general plan of operations, standard operating procedures, and as required by the Waste Management Permit. The Pond 7 Dam (AK00307) and Pond 10 Dam (AK00316), referred to collectively as the Pond 7/10 Dam System, are monitored in accordance with the Pond 7/10 Operations and Maintenance Program manual. Likewise, the Sand Pit Dam (AK00317) is monitored in accordance with the Sand Pit Dam Operations and Maintenance Program manual. The respective Operation and Maintenance Program manuals have been reviewed and approved by the ADNR-Dam Safety and Construction Unit. The routine monitoring and inspection provisions pertinent to geotechnical stability that are listed in the Operation and Maintenance Program manuals are listed within this IMP, but to ensure full compliance with all provisions of the Dam Safety Permits, they should be referenced.

Table 7-1 provides a summary of monitoring and inspection requirements to verify the geotechnical stability of specific waste rock sites, the TDF, and certified embankments. Monitoring activities include visual inspections, pneumatic piezometers, vibrating wire piezometers, inclinometers, standpipe water level, and survey monuments. Water monitoring and sampling for these facilities are covered in Section 3.2 and 4.1.

TABLE 7-1: GEOTECHNICAL MONITORING AND INSPECTION

Site Name	Daily	Weekly	Monthly	Quarterly	Semi-Annual	Annually	Other	Responsibility
Site 23/D	SI	VI	VI, PP	SL	IC	SM		SOps, Env
TDF	SI VW	VI	VI, SL		IC			SOps, Env
Pond 7/10 Dam System	SI SP VW	VI	VI		SM		PSI 3-yr	SOps, Env
Sand Pit Dam			VI SM	SM (after 7/19)			PSI 5-yr	SOps, Env

KEY:
PSI = Periodic Safety Inspection
IC = Inclinometer
PP = pneumatic piezometers
SI = Safety Inspection
SM = Survey Monument – embedded in concrete
SL = Stand Pipe Water Level
SP = Seepage return flow rate
VI = Visual Inspection
VW = Vibrating wire piezometer (recorded on data logger)
SOps = HGCMC Surface Operations Department
Env = HGCMC Environmental Department

8 Quality Assurance/Quality Control Program

The *Quality Assurance Project Plan (QAPP) of the General Plan of Operations Appendix 1 Integrated Monitoring Plan* (Greens Creek 2014) found in Appendix 1.A presents the rationale and technical requirements for the monitoring and methodologies that are presently used at the site to further improve site-wide monitoring.

9 References

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Appendix 1.A
Quality Assurance Project Plan (QAPP) of the General Plan of Operations Appendix 1 Integrated Monitoring Plan

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List of Appendices

Acronyms

AAC	Alaska Administrative Code
ABA	Acid Base accounting
ADEC	Alaska Department of Environmental Conservation
ADNR	Alaska Department of Natural Resources
AGO	Attorney General's Office
ANILCA	Alaska National Interest Land Conservation Act
APDES	Alaska Pollution Discharge Elimination System
ASTM	Alaska Sampling and Testing Methods
AWQS	Alaska Water Quality Standards
CFR	Code of Federal Regulations
CH	Clean Hands
CV	Casing Volume
CWA	Clean Water Act
DH	Dirty Hands
DI	Deionized
DOW	ADEC, Division of Water
DQO	Data Quality Objective
DW	Depth to Water
EA	Environmental Assessment
EPA	Environmental Protection Agency
EIS	Environmental Impact Statement
FWMP	Fresh Water Monitoring Plan
HGCMC	Hecla Greens Creek Mining Company
ICP	Inductively Coupled Plasma Analysis
IWMMP	Integrated Waste Management and Monitoring Plan
IDL	Instrument detection limit
LCS	Laboratory Control Standard
MAG	Management Information Goals
MDL	Method Detection Limit
MIG	Monitoring Information Goals
MQO	Measurement Quality Objective
ND	Non-Detect
NEPA	National Environmental Policy Act
NIST	National Institute of Standards and Technology
NP	Acid Neutralization Potential
NNP	Net Neutralization Potential
PQL	Practical Quantification Limit

QA	Quality Assurance
QAP	Quality Assurance Plan
QAPP	Quality Assurance Project Plan
QC	Quality Control
QMP	Quality Management Practice
RL	Reporting Limit
RPD	Relative Percent Difference
RIG	Regulatory Information Goals
RS	Reference Standard
SIG	Statistical Information Goals
SOP	Standard Operating Procedure
SRM	Standard Reference Material
TD	Total Depth
TDF	Tailings Disposal Facility
TDR	Time Domain Reflectometry
USDOT	United States Department of Transportation
XRD	X-Ray Diffraction

Units of Measure

ac	acre
cm	centimeter
in	inch(es)
ft	feet/foot
km	kilometer
m	meter
mm	millimeter
m^2	meter squared
ml	milliliter
µg	microgram
oz/st	troy ounces per short ton
ppm	parts per million
st	short ton
yd^3	cubic yards

A PROJECT MANAGEMENT ELEMENTS

A.1 Title and Approvals

Title: Quality Assurance Project Plan (QAPP) of the General Plan of Operations Appendix 1 Integrated Monitoring Plan (IMP)

Name: Keith Malone
General Manager, Vice President

Organization Name:

Hecla Greens Creek Mining Company

Signature: _____ **Date**: _____

Name: Christopher Wallace
Project QA Officer

Organization Name:

Hecla Greens Creek Mining Company

Signature: _____ **Date**: _____

Name: Tim Pilon
ADEC DOW Project Manager
Organization
ADEC DOW-Wastewater D/C Auth Pgms

Signature: _____ **Date**: _____

Name: Doug Kolwaite
ADEC, QA Officer

Organization Name:

ADEC DOW WQSAR Program

Signature: _____ **Date**: _____

A.2 DISTRIBUTION LIST

This list includes the names and addresses of those who receive copies of the approved QAPP and subsequent revisions.

Table 1 Distribution List

NAME	POSITION	AGENCY/ COMPANY	DIVISION/ BRANCH/SECTION	CONTACT INFORMATION
Keith Malone	Project Manager	HGCMC	VP and General Manager - Greens Creek Mine	Phone: (907) 789-8137 Email: kmalone@hecla-mining.com
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Sue Weber	Senior Project Manager	ACZ	Sample analysis	Phone: (970) 879-6590 Email: suew@acz.com
Cameron Sell	Data Manager	HGCMC	Environmental Engineer – Greens Creek Mine	Phone: (907) 790-8457 Email: csell@hecla-mining.com
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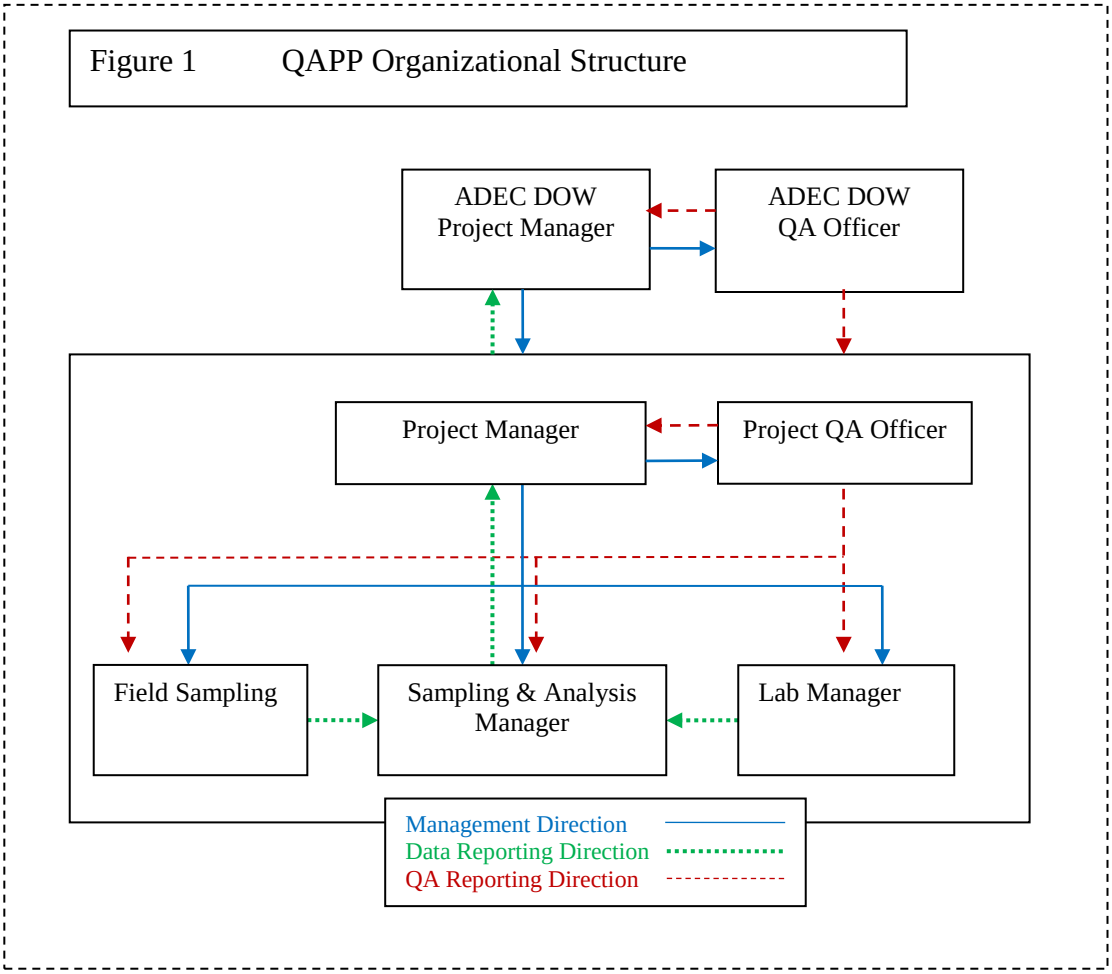
Doug Kolwaite	ADEC QA Officer	ADEC	Division of Water/ WQSAR/QA	Phone: (907) 465-5305 Email: doug.kolwaite@alaska.gov

A.3 PROJECT/TASK ORGANIZATION

Duties and responsibilities of key individuals are listed below and summarized in Figure 1:

- Project Manager – Vice President and General Manager of the Hecla Greens Creek Mining Company.
- Project QA Officer – Environmental Affairs Manager responsible for permitting, regulatory compliance, and oversight of all aspects of implementing the Quality Assurance Project Plan (QAPP) and Field Procedures Manual.
- Sampling & Analysis Manager – This individual will maintain the quality of field activities, sample collection, sample handling, laboratory analysis and data analysis, and document the quality of data at each processing level. The manager identifies major aspects of the project requiring specific quality control and demonstrates that quality control is a major focus for this project.
- Data Manager – This individual identifies the procedures to be used to verify that sample and field monitoring data is accurately entered and available for analysis.
- Laboratory Manager – Responsible for the overall review and approval of contracted laboratory analytical work, responding to sample result inquiries and method specific details.
- ADEC Project Manager - Responsible for overall technical and contractual management of the project. For Permit related monitoring projects, responsible for ensuring the permit complies with permit required water quality monitoring as specified in the approved QAPP.
- ADEC Quality Assurance Officer - Responsible for QA review and approval of plan and oversight of QA activities ensuring collected data meets project's stated data quality goals.

Figure 1 QAPP Organizational Structure



A.4 BACKGROUND AND PROJECT OBJECTIVES

A.4.1 Problem Definition

The Greens Creek Mine is a lead, zinc, silver and gold mine and mill located on the northwest portion of Admiralty Island, approximately 18 miles southwest of Juneau, Alaska. The facility has been in operation since 1989, with one temporary cessation of operations from 1993 to 1996. The mine's current production rate is 2,200 to 2,400 tons of ore per day. Major site facilities include the underground mine, mill, waste rock storage areas, dry tailing disposal facility, port facilities, and roads connecting these components. The facilities are located within the Greens Creek, Zinc Creek, Tributary Creek and Cannery Creek drainages, which flow into Hawk Inlet.

Routine monitoring is performed as described in the Greens Creek Mine Integrated Monitoring Plan (IMP) to fulfill monitoring requirements defined in the mine's Environmental Impact Statements (EIS), Records of Decision, Environmental Assessments (EA) and ADEC Waste Management Permit. The data generated from monitoring activities must be of appropriate quantity and quality to satisfy the project objectives.

A.4.2 Project Objective(s)

The objectives of the QAPP are:

- Ensure that monitoring requirements in the National Environmental Policy Act (NEPA) documents that relate to HGCMC are met. 40 CFR § 1505.3 states that agencies may provide for monitoring to assure their decisions are carried out.
- Ensure that Alaska Water Quality Standards (AWQS) are met. The State of Alaska, Department of Environmental Conservation has promulgated water quality standards to protect all uses of a water body.
- Ensure the intent of the Clean Water Act (CWA) is met. While this plan does not address discharges authorized by the mine's discharge permit under the CWA, some procedures described in this plan are similar to those described in 40 CFR § 136. This CFR referenced document describes guidelines that were established for test procedures for the analysis of pollutants discharged under Section 402 Alaska Pollution Discharge Elimination System (APDES) and Section 401 (State Certification) of the CWA.
- Ensure monitoring of surface water and groundwater and corrective actions will be in accordance with State regulations 18 AAC 60.820 – 18 AAC 60.860.
- Ensure test procedures for the analysis of water samples shall conform to the parameters, methods and procedures in the IMP and in 18 AAC 60.820 – 18 AAC 60.860.
- Ensure that the intent of the Alaska National Interest Land Conservation Act (ANILCA) is met.
- Evaluate the effectiveness of the IMP and QAPP annually.
- Collect information for specific reclamation needs and additional resource protection requirements as needed.
- Ensure the economic efficiency of the IMP and QAPP.
- Add and/or delete monitoring sites as needed; and modify schedules, protocols and methods as needed to ensure that all the goals of the IMP and QAPP are met.

This QAPP will be used to maintain the quality of field activities, sample collection, sample handling, laboratory analysis and data analysis, and to document the quality of data at each processing level. The QA/QC program identifies major aspects of the project requiring specific quality control and demonstrates that quality control is a major focus for this project.

A.5 PROJECT / TASK DESCRIPTION and SCHEDULE

A.5.1 Project Description

The Greens Creek Mine Integrated Monitoring Plan (IMP) documents the required material characterization, stability, freshwater samples, and biological samples which are collected at the prescribed frequency to ensure that the monitoring requirements defined in the mine's Environmental Impact Statements (EIS), Records of Decision, Environmental Assessments (EA) and ADEC Waste Management Permit are fulfilled. The IMP will be periodically reviewed and updated as necessary to coincide with regulatory changes, five-year environmental audit reviews, process modifications, or anomalies noted as a result of monitoring and sampling. Refer to the most current agency approved

version of the IMP for a detailed description of monitoring. Table 2 shown below provides a general overview of monitoring activities.

A.5.2 Project Implementation Schedule

Table 2 Project Implementation Schedule

Product	Media	Sampling Site	Parameters	Frequency	Time Frame
QAPP Preparation					
Monitoring	Surface Water	Project Area	Water quality, flow	Monthly/ Quarterly	Year-round
	Groundwater	Millsite, Site 23/D, and TDF	Water quality, static water level	Quarterly/ Annually	Year-round
	Tailings Characterization	Mill Tailings	ABA*	Monthly, Annually	Year-round
	Waste Rock Characterization Dump Stability Drains	Site 23	ABA* Inclinometer Water Quality	Quarterly Semi-annually Quarterly/Monthly	Year-round
	Bio-monitoring	Greens Creek and Tributary Creek	WQ, FA, FM, P, MI**	Annually	July
Lab Analysis	All Media	All sites		Analyses within sample holding time requirements	Year-round
Field Audit	Audit of field monitoring operations	All sites		< 30 days of project start-up	1/project
Reports	All Media	All sites		Quarterly Annually	May, Sep., Nov. April 15

* ABA - Acid Base Accounting

** WQ - water quality, FA-fish abundance and distribution, FM-fish metals content, P-periphyton biomass, MI-macroinvertebrate abundance

A.6 DATA QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

A.6.1 Data Quality Objectives (DQOs)

Data Quality Objectives (DQOs) are qualitative and quantitative statements that:

- Clarify the monitoring objectives; and,
- Define the appropriate type of data needed.

The IMP describes the various types of monitoring performed throughout the project area, locations and frequency of monitoring (where applicable), and the data generated from the monitoring. Each type of monitoring has different DQOs based on the purpose for, and intended application of, the data.

The project's overall DQOs are to collect appropriate data to:

- Determine if water resources are protective of the applicable AWQS at compliance monitoring locations and identify water quality trends;
- Document the condition and long-term health of aquatic biological resources;
- Demonstrate that mine waste rock and tailings facilities are being managed in accordance with approved plans and permits; and,
- Determine if facility specific management and reclamation plans are adequate to protect the environment during operations and post-closure.

A.6.2 Measurement Quality Objectives (MQOs)

Measurement Quality Objectives (MQOs) are a subset of DQOs. MQOs are derived from the monitoring project's DQOs. MQOs are designed to evaluate and control various phases (sampling, preparation, and analysis) of the measurement process to ensure that total measurement uncertainty is within the range prescribed by the project's DQOs. MQOs define the acceptable quality (data validity) of field and laboratory data for the project. MQOs are defined in terms of the following data quality indicators:

- Detectability
- Precision
- Bias/Accuracy
- Completeness
- Representativeness
- Comparability

Detectability - is the ability of the method to reliably measure a pollutant concentration above background. ADECs Division of Water uses two components to define detectability: method detection limit (MDL) and practical quantification limit (PQL) or reporting limit (RL).

- The MDL is the minimum value which the instrument can discern above background but no certainty to the accuracy of the measured value. For field measurements the manufacturer's listed instrument detection limit (IDL) can be used.
- The PQL or RL is the minimum value that can be reported with confidence (usually some multiple of the MDL).

Sample data measured below the MDL is reported as ND or non-detect. Sample data measured ≥ MDL but ≤ PQL or RL is reported as estimated data. Sample data measured above the PQL or RL is reported as reliable data unless otherwise qualified per the specific sample analysis.

The detectability criterion is addressed by specifying to the analytical laboratory the analytical methods and associated MDL and PQL required for each type of monitoring. For water quality monitoring the MDL and PQL are based on the applicable AWQS.

Precision - is a measure of the ability to replicate an analysis and is expressed as the relative percent difference (RPD). The RPD criterion for water samples is ±20% and is only applicable when the analyte concentration is more than 5 times the IDL, and as long as the native amount is not greater than 4 times the spiked amount. The RPD criterion for biological samples is ±35% due to the greater degree of variability in samples.

Bias (Accuracy) - is a measure of confidence that describes how close a measurement is to its "true" value and is expressed as %R. Methods to determine and assess accuracy of field and laboratory measurements include instrument calibrations and various types of QC checks (e.g., sample split measurements, sample spike recoveries, matrix spike duplicates, continuing calibration verification checks, internal standards, external standards, and sample blank measurements). Bias/Accuracy is usually assessed using the following formula:

$$Accuracy = \frac{MeasuredValue}{TrueValue} \times 100$$

The Matrix Spike/Matrix Spike Duplicate (MS/MSD) criteria are 75-125 %R for all metals. The criteria are only applicable for MS/MSD analyses as long as the native amount is not greater than 4 times the spiked amount. The accuracy limits for the Laboratory Control Sample (LCS) are method dependent, e.g. 90-110 %R for Inductively Coupled Plasma-Mass Spectrometry (ICP-MS).

Completeness - is a measure of the percentage of valid samples collected and analyzed to yield sufficient information to make informed decisions with statistical confidence. Project completeness is determined for each pollutant parameter using the following formula:

$$\frac{T - (I+NC)}{T} \times (100\%) = \text{Completeness}$$

Where T = Total number of expected sample measurements.
I = Number of invalid sample measured results.
NC = Number of sample measurements not produced (e.g. spilled sample, etc).

The Fresh Water Monitoring Program (FWMP) is the only monitoring program for which completeness is a stated DQO. The completeness criterion is 95% for a water year (October 1 – September 30).

Representativeness - assigns what parameters to sample for, where to sample, type of sample (grab, continuous, composite, etc.) and frequency of sample collection. The IMP specifies these criteria for each type of monitoring.

Comparability - is a measure that shows how data can be compared to other data collected by using standardized methods of sampling and analysis. HGCMC utilizes standardized methods for the

collection and analysis of water quality samples to ensure comparability of data generated. Metals concentrations in water samples are measured in the dissolved fraction (filtered samples) to limit potential variability caused by mineralized sediments in surface and ground water. This enables comparison of surface water and ground water data, from both the internal monitoring program for tailings and waste rock sites and the FWMP compliance monitoring sites, to help explain water quality trends or data anomalies. Data collected from the FWMP sites is compared to the applicable AWQS.

Different laboratories are used for analysis of FWMP samples and samples from the internal monitoring of tailings and waste rock sites. HGCMC frequently collects split samples and submits them to both laboratories for analyses of the same constituents. This QA/QC check of the laboratories validates the comparability of the data.

The Measurement Quality Objectives for the FWMP compliance monitoring are shown in Table 3. The laboratory may achieve lower MDLs than specified but not higher.

Table 3 Measurement Quality Objectives (MQOs)

Analyte	Method	MDL[1]	PQL[2]	AWQS[3]	Precision[5,7]	Accuracy[6,7]	Complete
Total Alkalinity, mg/L	2320	1.0	18	20	+/- 20 RPD	75-125 %R	95%
Hardness, mg/L	2340B	1.0	None	None	+/- 15 RPD	75-125 %R	95%
Conductivity, µmhos/cm	2510	10	None	None	+/- 10%	+/- 10%	95%
pH, s.u.	4500-H+			6.5 - 8.5	+/- 0.2	+/- 0.1	95%
Arsenic, diss., µg/L	1638m	2	9	10	+/- 20 RPD	75-125 %R	95%
Barium, diss., µg/L	1638m	280	900	1000	+/- 20 RPD	75-125 %R	95%
Cadmium, diss., µg/L	1638m	0.15 / 0.11	0.47 / 0.34	0.52 / 0.38	+/- 20 RPD	75-125 %R	95%
Chromium, diss., µg/L	1638m	3.1	9.9	100	+/- 20 RPD	75-125 %R	95%
Copper, diss., µg/L	1638m	1.4 / 1.0	4.6 / 3.2	5.1 / 3.6	+/- 20 RPD	75-125 %R	95%
Lead, diss., µg/L	1638m	0.25 / 0.15	0.81 / 0.49	0.90 / 0.54	+/- 20 RPD	75-125 %R	95%
Mercury, diss., µg/L	1631e	0.0003	0.011	0.012	+/- 20 RPD	75-125 %R	95%
Nickel, diss., µg/L	1638m	12.7 / 9.4	40.4 / 30.0	44.9 / 33.3	+/- 20 RPD	75-125 %R	95%
Selenium, diss., µg/L	1638m	1.42	4.5	5.0	+/- 20 RPD	75-125 %R	95%
Silver, diss., µg/L	1638m	0.21 / 0.10	0.66 / 0.33	0.73/0.37[4]	+/- 20 RPD	75-125 %R	95%
Sulfate, mg/L	M300.0-IC	70	225	250	+/- 20 RPD	75-125 %R	95%
Zinc, diss., µg/L	1638m	12.9 / 9.3	41.0 / 29.4	45.6 / 32.7	+/- 20 RPD	75-125 %R	95%

1. MDL=PQL÷3.18, rounded up to the same number of significant digits as the AWQS for that analyte. If AWQS for this constituent is hardness dependent, two numbers are listed. First number listed is for surface water sites, the second is for groundwater sites.
2. PQL based on AWQS x 0.9. If AWQS for this constituent is hardness dependent, two numbers are listed. First number listed is for surface water sites, the second is for groundwater sites.
3. If AWQS is hardness dependent, two numbers are listed for the purposes of calculating the MDL and PQL. First number listed is based on a hardness value of 37 to represent the 25th percentile of surface water hardness values, the second number listed is based on a hardness value of 25 to represent the 25th percentile of groundwater hardness values. AWQS is for chronic conditions unless otherwise noted. The actual hardness dependent AWQS for that constituent will depend on the actual hardness of the sample, not on the number that appears in this table.
4. AWQS is a 24 hour average (acute).
5. The precision DQO is only applicable when the analyte concentration is more than 5 times the IDL.
6. Listed accuracy is for MS/MSD only. The accuracy DQO for the LCS QC sample is method dependent.
7. The precision and accuracy DQOs for MS/MSD analyses are only applicable as long as the native amount is not greater than 4 times the spiked amount.

A.7 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

All personnel collecting samples will be trained in protocols currently used for collection of water quality, geochemical characterization of materials, geotechnical stability of structures, and aquatic biological samples. Written record must be made for training of all new personnel in either field notes/notebook or sampling sheets. Training of personnel collecting samples will be provided and documented by senior staff of HGCMC.

Contracted laboratories performing analytical work must have the requisite knowledge and skills in execution of the analytical methods being requested. Information on laboratory staff competence is usually provided in each lab's Quality Management Plan (QMP) and/or Quality Assurance Project Plan (QAPP). The QMP for PNNL Marine Sciences Laboratory (FWMP) is included as Appendix 1.A.C, and the QAP for ACZ Analytical Laboratories, Inc. (internal monitoring) is included as Appendix 1.A.D.

Table 4 Training Requirements

Specialized Training/Certification	Field Staff	Lab Staff	Monitoring Supervisor	Lab Supervisor	Project QA Officer
Safety training	X	X	X	X	X
Water sampling techniques	X		X		X
Instrument calibration and QC activities for field measurements	X		X		X
Instrument calibration and QC activities for laboratory measurements		X		X	X
QA principles			X	X	X
QA for water monitoring systems			X		X
Chain of Custody procedures for samples and data	X	X	X	X	X
Handling and Shipping of Hazardous Goods	X	X	X	X	X
EPA Approved Field Measurement Method Training	X		X		X
Specific EPA Approved Lab Analytical Method Training		X		X	X

A.8 DOCUMENTS AND RECORDS

A.8.1 Documentation of Measurements, Sampling, and Inspections

For each measurement or sample taken, the following information is recorded:

- Place, date, and time of inspection, observation, measurement, or sampling;
- Person(s) who inspected, observed, measured, or sampled;
- Dates the analyses were performed and by which analytical facility;
- Analytical techniques or methods used;
- Accuracy of the analytical method (detection limits); and,
- Results of all required analysis.

Chain of Custody forms accompany all samples to assure sample holding times and handling procedures are met throughout the sample and analytical process.

A.8.2 Retention of Records

During operation, closure, and reclamation all records of monitoring activities and results, calibrations, and maintenance are retained for a period of at least three years from the date that the permit expires and as long as necessary to comply with applicable laws.

A.8.3 Monitoring Reports and Submission Schedules

The ADEC Waste Management Permit requires submission of quarterly reports summarizing inspection and monitoring results. Reports for the first three calendar quarters are due within 60 days after the end of the quarter. These reports are submitted to ADEC to specifically satisfy the reporting requirements of the Waste Management Permit, with courtesy copies provided to the USFS and ADNR. The quarterly reports address the following:

- Summaries of inspections and monitoring results;
- Analytical results for monitoring performed at the FWMP compliance sites during the corresponding quarter, with comparisons to historical data;
- Quantities and disposition of tailings and waste rock; and,
- Summary of water flow and management monitoring and meteorological data during the quarter.

The report for the fourth calendar quarter will be submitted by April 15 of the following year and serve as an Annual Report. The Annual Report will satisfy the reporting requirements of the ADEC, USFS and ADNR. In addition to the information provided in the quarterly reports, the Annual Report will address the following:

- Geochemical monitoring of tailings, waste rock and construction rock;
- Geotechnical stability monitoring of the tailings disposal facility, Site 23 and Pond 7/10;
- Internal water quality monitoring of the tailings disposal facility and Site 23;
- Monitoring of fugitive dust from the tailings disposal facility; and,
- An assessment of the adequacy of the reclamation surety bond.

All work associated with the annual aquatic bio-monitoring is performed by an independent outside entity with expertise in that field. This includes data collection, analysis, interpretation of results, and preparation of a technical report. Currently, the aquatic bio-monitoring is performed by the Alaska Department of Fish and Game, Division of Habitat, under annual contract to HGCMC. The technical report on the bio-monitoring is submitted by April 15 of the following year.

In addition to the quarterly and annual reports, Waste Management Permit stipulates conditions which require notification to ADEC not later than 5:00 p.m. of the next regular work day. These conditions include:

- Wildlife casualties associated with facility activities;
- When a statistically significant increase in a constituent concentration above a WQS is discovered at a surface or ground water monitoring location; or,
- Any non-compliance with a permit condition.

If a statistically significant increase in a constituent concentration above a WQS or a non-compliance condition is discovered, HGCMC shall:

- Determine the extent of the exceedance or non-compliance;
- In consultation with ADEC and documented in writing, implement a plan to restore compliance and determine the cause of the exceedance or non-compliance;
- Submit to ADEC, within seven working days after an exceedance or non-compliance is verified by HGCMC, a plan for corrective actions to prevent adverse environmental impacts and avoid future exceedances of a similar nature; and,
- Implement the corrective action plan as approved by ADEC.

Below is a table of all documents and records that will be produced and their disposition, including location and retention time.

Table 5 Project Documents and Records

Categories	Record/Document Types	Location	Retention Time
Site Information	Network Description	On Site	
	Site characterization file		
	Site maps		
	Site pictures		
Environmental Data Operations	QA Project Plan		
	Field Method SOPs		
	Field Notebooks		
	Sample collection/measurement records		
	Sample Handling & Custody Records		
	Chemical labels, SDS sheets		
	Inspection/Maintenance Records		
Raw Data	Lab data (sample, QC and calibration) including data entry forms		3 years after permit expires
Data Reporting	Discharge Monitoring Reports (DMRs, for permitted facility)		3 years after permit expires
	Progress reports		
	Project data/summary reports		
	Lab analysis reports		
	Investigation summary (CATS)		
	Inspection Report		
Data Management	Data management plans/flowcharts		
	Data algorithms		
Quality Assurance	Control charts		
	Data quality assessments		
	DMRQA and PE samples		3 years after permit expires
	Site audits		
	Lab audits		
	QA reports/corrective action reports		
	Response		
	Performance Evaluation Samples		

B DATA GENERATION AND ACQUISITION

B.1 SAMPLING

See the Integrated Monitoring Plan for specific sampling processes and designs.

Water samples are collected using protocols designed to minimize bias from systematic and/or erratic contamination introduced during sample collection. Water quality protocols are performance based and were developed from prior HGCMC sampling protocols incorporating selected procedures from EPA and U.S. Geological Survey methods. These protocols are applicable to the analytes being monitored, and the MDLs and MLs required assuring appropriate comparisons to AWQS. While these water quality sampling protocols are not required to be used, they are recommended. If other water quality sampling protocols are used, they should be based on proven methodologies such that the required MDLs and MLs can be achieved without experiencing false positive constituent levels due to introduced contamination.

B.2 SAMPLING METHOD REQUIREMENTS

B.2.1 Sampling Containers

The following applies to water samples collected under the FWMP:

- Sample containers are supplied by the laboratory conducting the analyses (PNNL Marine Sciences Laboratory).
- Sample containers will be pre-cleaned and pre-labeled at the laboratory prior to shipment to HGCMC. Filters and tubing used in the sample collection are also provided by the laboratory. They will be stored in a dry, dust free environment to avoid contamination on the outside of the bottles that could be inadvertently transferred to the sample during collection.
- Each bottle for trace metal analyses is placed within its own set of double re-sealable bags. Each bottle for the measurement of general wet chemistry analytes is placed within a single re-sealable bag. The individually bagged bottles for each site are placed together into a large clear plastic bag designated for that site.
- If a pre-cleaned bottle becomes uncapped during shipment or storage it will be returned to the laboratory and not used.
- Containers are supplied without chemical preservative. Collected samples are delivered to the laboratory within 24 hours and proper chemical preservation is performed at the laboratory.

The following applies to water samples collected under the internal monitoring program:

- Sample containers are supplied by the laboratory conducting the analyses (ACZ Laboratories, Inc.).
- Sample containers requiring chemical preservation will be pre-preserved at the laboratory prior to shipment to HGCMC. They will be stored in a dry, dust free environment to avoid contamination on the outside of the bottles that could be inadvertently transferred to the sample during collection.
- HGCMC is responsible for procuring filters and tubing that are certified as appropriate for use in the collection of environmental samples.
- HGCMC may print and affix the appropriate labels to the containers.
- All bottles for each site will be placed together into a large clear plastic bag.

The table below lists specific analyte/method criteria for parameter holding times and preservation methods. For parameters not listed in this table, see 40 CFR 136.6 for EPA-approved preservation methods and containers. 40 CFR 136.6 is available at: http://www.gpoaccess.gov/cfr/index.html

Table 6 Preservation and Holding Times for the Analysis of Samples

Analyte		Matrix	Container	Volume	Sample Preparation	Maximum Holding Time
Hardness		Water	poly	500 mL	0.2% HNO_3	180 days
pH		Water	poly	500 mL	Field filter; unpreserved	24 hours[1]
Conductivity		Water	poly	500 mL	Field filter; unpreserved	14 days
Bicarbonate		Water	poly	500 mL	Field filter; unpreserved	14 days
Alkalinity		Water	poly	500 mL	Field Filter; unpreserved	14 days
Ca, Mg, Na, K		Water	poly	250 mL	Field Filter, 0.2% HNO_3	180 days
Sulfate, chloride		Water	poly	60 mL	Field Filter; unpreserved	28 days
Nitrate-Nitrite		Water	poly	250 mL	Unfiltered; H_2SO_4 to pH < 2	28 days
Hardness		Water	poly	100 mL	HNO_3 to pH < 2; < 6°C	180 days
Mercury Total		Water	poly	250 mL	Unfiltered; 0.5% HCL	90 days
Dissolved Metals	Silver Ag	Water	poly	500 mL	Field Filter, 0.2% HNO_3	180 days
	Arsenic As	Water	poly	500 mL	Field Filter, 0.2% HNO_3	180 days
	Barium Ba	Water	poly	500 mL	Field Filter, 0.2% HNO_3	180 days
	Cadmium Cd	Water	poly	500 mL	Field Filter, 0.2% HNO_3	180 days
	Chromium Cr	Water	poly	500 mL	Field Filter, 0.2% HNO_3	180 days
	Copper Cu	Water	poly	500 mL	Field Filter, 0.2% HNO_3	180 days
	Nickel Ni	Water	poly	500 mL	Field Filter, 0.2% HNO_3	180 days
	Lead Pb	Water	poly	500 mL	Field Filter, 0.2% HNO_3	180 days
	Selenium Se	Water	poly	500 mL	Field Filter, 0.2% HNO_3	180 days
	Zinc Zn	Water	poly	500 mL	Field Filter, 0.2% HNO_3	180 days

[1]From sample receipt

B.2.2 Sampling Methods

B.2.2.1 General Procedures

All personnel collecting samples will be trained in protocols currently used for collection of water quality samples. For all FWMP sampling, and whenever possible for internal monitoring, sampling will be done by teams of at least two trained people. Two people provide additional safety and overall efficiency while collecting samples in the field.

The following procedures apply regardless of the site type (ground water or surface water). Contamination will be minimized by paying strict attention to the work being done, awareness of potential contaminant sources, and minimizing atmospheric dust and debris from roads, vehicles, sampling locations, and the general environment.

a) Assemble all requisite supplies for the samples scheduled to be collected that day, place them

in the vehicle, and drive to the sample location(s) parking a safe distance away when the sample site is near a roadway to minimize contamination by airborne particulate.

b) Open the storage cooler and remove the appropriate site bag containing the sample bottles and any QC sample bottle(s) scheduled for that site. Gather all ancillary supplies in a heavy clear plastic bag or cooler.

c) Walk to the sampling location and set up to take samples.

d) At each site samplers involved in collecting samples will put on a new set of clean gloves. Only disposable, non-powdered latex gloves will be used during sample collection. The sampler will take extra care to ensure their gloves remain clean throughout the sample collection process. If there are any doubts the glove(s) will be replaced immediately.

B.2.2.2 Surface Water Sample Collection Procedures

At each location the following information is to be recorded in a field log book: sample team, date, time, site name, sample ID, analytical suite, field parameters (pH, conductivity and temperature), flow measurement or estimate (if practicable), weather conditions, and any other information that will aid in the interpretation of the data.

Samples are collected facing upstream to minimize the potential for contamination by disturbed bottom sediments.

For each unpreserved sample bottle to be filled when conditions exist to completely submerge the sample bottle without disturbing sediments:

- Completely submerge the bottle and remove the cap. Hold the cap so that the liner is facing upstream allowing flushing of the cap interior, and partially fill the bottle.
- While the bottle is still submerged, replace the cap and remove the bottle from the water.
- Shake the bottle several times and empty the bottle downstream and/or away from the site.
- After two more rinses, submerge the bottle entirely allowing the bottle to completely fill with sample leaving as little air space as possible.
- Replace the cap and place the sample in the plastic site bag.

For collecting samples in pre-preserved bottles, samples that require filtering in the field, or from sites where conditions do not exist to completely submerge the bottle without disturbing sediments, either:

- Utilize a clean, triple rinsed, and appropriately sized sample bottle as a transfer device to fill the required sample bottle; or,
- Use a peristaltic pump with new, clean tubing to draw directly from the stream exercising care to not disturb sediments.

B.2.2.3 Ground Water Sample Procedures

Ground water samples are collected using a variety of methods that are based on the depth of the well, and whether it is artesian/flowing, has a rapid recharge rate, or a slow recharge rate. Artesian/flowing wells do not require purging prior to sample collection. Wells that recharge rapidly are purged to remove a minimum of three (3) casing volumes. Wells that recharge slowly are purged using low flow pumping techniques to minimize drawdown of the water column. For wells where pumping is not possible, a disposable bailer is used for purging and sample collection. In all cases, wells are allowed to

freely flow, or are purged, until the pH, conductivity, and temperature have stabilized, or until the well runs dry.

At each location the following information is to be recorded in a field log book: sample team, date, time, site name, sample ID, static water level (before purging), total depth, purge volume, analytical suite, field parameters (pH, conductivity and temperature), flow measurement or estimate (if artesian), weather conditions, and any other information that will aid in the interpretation of the data.

After properly purging the well, groundwater samples are collected as follows:

- *If using an electric pump:* Attach a length of new tubing to the well's discharge tubing and the pump. Operate the pump and flush the tubing prior to sample collection. Both team members are careful not to touch the end of the tubing, or to let it touch anything.
- *If using a manual bailer:* Retrieve water from the well by slowly lowering the bailer into the well, minimizing the suspension of sediment if present. When practicable, retrieve and discard at least one bailer volume to rinse the bailer prior to sample collection. Pour/dispense water from the bailer into the sample bottles.
- Unpreserved sample bottles will be rinsed with water from the well by partially filling the bottle, replacing the cap, shaking vigorously to also rinse the cap, and emptying the bottle away from the site. Repeat two more times to triple rinse each bottle prior to sample collection.
- Pre-preserved bottles are not rinsed prior to sample collection.
- Collect the samples by holding the bottles under the end of the tubing or bailer, avoiding contact between the bottle and tubing or bailer. Secure the cap and place the sample bottle into the inner bag (if applicable) and re-seal it.

B.2.3 Sample Bottle Labeling

Each sample container requires a label large enough to record the information needed to readily identify the sample. The information recorded on each label will include the project name, sample point, date/time collected, filtered or unfiltered, preservation, and sampler's initials. Permanent waterproof ink or permanent marker should be used for all labeling purpose. The following are general guidelines to bottle labeling:

1 Put on a pair of clean gloves (new gloves should be used for each sample set).

2 Pull the sample set out, and fill out the necessary sections (site, date, time, and sampler) on the label, for each sample bottle.

3 To maintain consistent record keeping and to aid in efficient computer data processing, it is important to record the exact sample station identification on the sample label, corresponding to sample points contained in the IMP.

B.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

B.3.1 Sample Custody Procedures

All water quality samples are collected by HGCMC personnel, packaged, and transported off Admiralty Island for laboratory analyses. This section describes the steps necessary to properly document the sample shipment, package the samples for shipment, and to arrange for and coordinate shipment of the samples from the mine site to the laboratory.

A chain of custody form and a bill of lading are filled out for each sample shipment. A copy of each is kept by sampling personnel to properly document and track the sample shipment. Example chain of custody forms are provided in Appendix 1.A.B. Documentation will be filed at the HGCMC mine site.

A bill of lading is completed for the shipping carrier to be used. HGCMC has accounts with Alaska Airlines Gold Streak Service and Federal Express. The carrier used is based on their ability to deliver samples to the laboratory's location, and the carrier's flight schedule. The account number is put on the bill of lading.

The samples and documentation are inspected and reviewed for accuracy, completeness, and legibility. The reviewer by initialing the chain of custody form documents the review as complete. The items to be reviewed are as follows:

1 The monitoring schedule is referenced to ensure all sample bottles including the QC samples are present.
2 The sample bottle labels and the chain of custody are reviewed.
3 The bill of lading is reviewed to ensure the correct delivery address.

B.3.2 Sample Packaging and Shipping Requirements

Packaging, marking, labeling, and shipping of samples will comply with all regulations promulgated by the U.S. Department of Transportation (USDOT) in 49 CFR 171-177. Staff should receive the necessary training for shipping samples or consult with the sub-contracted laboratory for shipping instructions.

Packaging

For the testing laboratory to generate valid test results, the integrity of field samples must be intact upon receipt at the laboratory. Protocols ensuring proper integrity of field samples from the time of collection to the time of receipt at the testing lab include:

- packing samples to prevent breakage or leakage;
- immediately cooling and maintaining unpreserved samples at <6°C;
- delivering samples to the lab in a time frame that allows analysis within the parameters' recommended holding times (see Table 6); and
- confirming the receipt and integrity of field samples with documentation generated by the shipper and the testing lab.

Packaging the samples is facilitated by the laboratory shipping empty bottle sets in the coolers that will be used for shipping the samples back to the laboratory. Coolers protect the sample containers, and provide the necessary environmental conditions (cleanliness, temperature, etc.) during transport. Blue Ice or frozen water in appropriate containers is used to maintain a temperature of <6°C within the coolers

during sample shipment to the laboratory, and it is HGCMC's responsibility to freeze Blue Ice or water-filled containers prior to use. Below is a checklist of procedures for packaging water samples for shipment:

1 In a clean place without removing bottles from their resealable bag(s) ensure each sample bottle lid is tight, the bottle is properly labeled, and the cooler is clean to help minimize any contamination.

2 Ensure each sample bottle for metals analyses is within a set of double resealable bags, each sample bottle for the measurement of physical analytes is within a single resealable bag, and both are within the large clear heavy plastic bag designated for each site.

3 Place all site bags into the cooler. Set the bottles snugly in the cooler using clean packing material as necessary to prevent the sample bottles from moving within the cooler during transportation.

4 Place sufficient previously frozen Blue Ice or water filled bottles in the cooler with the samples to maintain the cooler temperature at <6°C during transportation.

5 Copy the chain of custody form, seal the original in a resealable plastic bag, and place the bag within the cooler. Retain the copy for HGCMC's files.

6 Place strapping tape around the cooler as necessary to ensure the lid does not open during transportation and to confirm the cooler has not been tampered with during transportation. Tape should be applied over the cooler lid lock mechanism if present.

7 Secure the shipping label to the top of the cooler.

8 Transport the cooler to a secure storage area or to the shipping agent.

Schedule of Shipment

Shipment of samples is coordinated between sampling personnel, laboratory personnel, and the transportation carrier(s) to be used. Samples are shipped expeditiously to the laboratory and should arrive in less than 2 days from the sample collection date. Holding time limitations must be considered when decisions are made regarding sampling and shipping times.

Notes:

- Sample shipments are not scheduled when it would result in expected delivery on weekends or holidays. Samples must be unpacked, logged, and preserved immediately upon receipt at the laboratory.
- Shipments are scheduled in consideration of the ability to get samples to town in time to meet the carrier's flight schedule. The carrier's schedule is checked beforehand for changes due to holidays or other reasons which could result in delayed delivery.
- The sample cooler(s) is brought to the drop-off point or common carrier in town and a copy of the bill of lading is returned to the mine for filing.
- A copy of the bill of lading is faxed to the Laboratory or they are called with the air bill number confirming to them the expected shipment and delivery time.

B.4 QUALITY CONTROL REQUIREMENTS

Quality Control (QC) is the overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the monitoring project's data quality objectives.

B.4.1 Field Quality Control Measures

Quality Control measures in the field include but are not limited to:

- Proper cleaning of sample containers and sampling equipment.
- Maintenance, cleaning and calibration of field equipment/ kits per the manufacturers and/or laboratory's specifications, and field Standard Operating Procedures (SOPs).
- Chemical reagents and standard reference materials are used prior to expiration dates.
- Proper field sample collection and analysis techniques.
- Correct sample labeling and data entry.
- Proper sample handling and shipping/transport techniques.
- QA/QC Samples (should generally be equal to 15% of the total field and/or lab measurements or at least 1/sampling event, whichever is greater), including:
    - Field Blank (to the laboratory) samples
    - Field Replicate samples
    - Field Replicate measurements

B.4.2 Laboratory Quality Control (QC) Measures

Consistency in the use of fundamental laboratory techniques and practices over time is essential for creating a useful, reliable, and technically defensible database of analytical test results. Monitoring shall be conducted in accordance with EPA-approved analytical procedures and in compliance with 40 CFR Part 136, Guidelines Establishing Test Procedures for Analysis of Pollutants.

Quality Control in laboratories includes the following:

Calibration - Initial calibration ensures that the instruments are set up and adjusted properly to generate acceptable quantitative and qualitative test results. Initial instrument calibration procedures for most analyses require a minimum of three calibration standards and a blank. The associated calibration curve is required to have a linearity of 0.995 to be acceptable for sample analysis for most methods. Verifying the calibration ensures that acceptable sample test results are initially and continually produced throughout the analytical test run. Calibration verification (CV) standards are analyzed after completion of initial calibration and at required frequencies (typically every 10 to 20 samples) during and at completion of analytical testing. CV test results must meet acceptance criteria (typically 90-110% recovery for most methods) in order to generate valid sample test results.

Blanks - Calibration blanks and preparation blanks or method blanks are used to monitor the background associated with the analysis and preparation procedures. Blanks are required to be analyzed as a component of the initial instrument calibration and/or at a minimum frequency of 5% of the test sample quantity analyzed during each test run or one per batch, whichever is greater. Test results on all blanks must meet acceptance criteria (typically +/- reporting limit) in order to generate valid sample test results.

Laboratory Control Standards (LCS) - Analysis of LCSs are used to monitor the overall performance of the laboratory, including both sample preparation and analysis procedures. A certified SRM also

referred to as a reference standard (RS), is typically used as an LCS in most analytical laboratories. SRMs must be analyzed for all applicable test methods at a minimum frequency of 5% of the test sample quantity analyzed during each test run or one per batch, whichever is greater. Test results on all LCSs are required to meet acceptance criteria for accuracy (typically 75-125% recovery) in order for sample test results to be valid.

Matrix Spikes - These are used to monitor analytical performance with regard to accuracy within a specific sample matrix. Analysis of matrix spikes for all applicable methods is required at a minimum frequency of 5% of the test sample quantity analyzed during each test run or one per batch, whichever is greater. Test results on all matrix spikes are required to meet acceptance criteria for accuracy (typically 75-125% recovery) in order for sample test results to be valid.

Duplicates - Duplicates are used to monitor analytical performance with regard to precision. Analysis of sample duplicates for all applicable methods is required at a minimum frequency of 5% of the test sample quantity analyzed during each test run or one per batch, whichever is greater. Test results on all sample duplicates are required to meet acceptance criteria for precision (typically $\leq$20-25% RPD) in order for sample test results to be valid.

Sample Analysis – Sample analysis must be performed within the recommended holding times for each parameter tested (See Table 6). Sample preparation and analysis must correctly follow prescribed methodology. Reported test results must be derived from data that falls within the calibration range for each test parameter.

Contracted laboratories will provide analytical results after verification and validation by the laboratory QA Officer. The laboratory must provide all relevant QC information with its summary of data results so that the Project QA Officer or his/her designee can perform field data verification and validation, and review the laboratory reports. It is understood that Synectics is contracted to HGCMC to conduct the lab data review for the FWMP. The Project QA Officer or his/her designee (Synectics) reviews these data to ensure that the required QC measurement criteria have been met. If a QC concern is identified in the review process, the Project Sampling & Analysis Manager and Project QA Officer will seek additional information from the contracted laboratory to resolve the issue and take appropriate corrective action/s.

Table 7 Laboratory Quality Control Samples

Field/Lab Quality Control Sample	Measurement Parameter	Frequency	QC Acceptance Criteria Limits
Lab Blank	All parameters	1:20	<5x MDL
Lab Fortified Blank	All ICP-MS (1638) and ICP-OES (200.7) Metals and Hardness	1:20	75-125%
	Mercury, Alkalinity, pH and Conductivity	NA	NA
Initial Calibration Verification Check Standard	All parameters	1 at beginning of analytical run	±10%
Continuing Calibration Verification Check Standard	All parameters	1:10	±15%
Matrix Spike/Matrix Spike Duplicate	All ICP-MS (1638) and ICP-OES (200.7) Metals and Hardness	1:20	75-125%
	Mercury, Alkalinity, pH and Conductivity	NA	NA
Lab Duplicate Sample	All parameters	1:20	RPD <20%
External QC Check Standard	All parameters	1:20	75-125%

B.5 EQUIPMENT TESTING, INSPECTION AND MAINTENANCE REQUIREMENTS

This section describes the procedures and criteria used to verify that all instruments and equipment are acceptable for use.

Field equipment used for sample collection and field measurements requires a program of control, calibration, adjustment, and maintenance. Portable water quality instruments in good working order are used for the field measurement of a standard set of field parameters summarized in Table 8. Note: The make and model of these instruments may vary over time.

Table 8 Field Testing Equipment

Equipment	Parameter
Solinst model 101 Depth to Water Tape	Water Level (groundwater wells)
Global Water FP111 Flow Probe	Stream Flow
Hydrolab Quanta Multi-Probe System	pH Water temperature Dissolved oxygen Oxidation/reduction potential (ORP/Eh) Electrical conductivity Turbidity
Hach 2100P Portable Turbidimeter	Turbidity
Oakton pH/Con 10 Series	pH Electrical Conductivity Water Temperature
YSI EC 300	Electrical Conductivity
YSI 30	Electrical Conductivity
Solinst Model 408 Double Valve Pump Solinst Model 464 Pump Control Unit	Groundwater

All field measurement data are recorded in field log books then input into an electronic database. Field crews may use field instrumentation and equipment maintained at the project site and/or instrumentation and equipment brought in from off-site.

Calibration, Operational Checks, Maintenance, and Record Keeping

Monitoring staff will document that required acceptance testing, inspection and maintenance have been performed. Records of this documentation should be kept with the instrument/equipment kit in bound logbooks or data sheets.

Field instrument preparations, calibration, and/or operational checks typically are performed at the beginning of each day's sampling activities. These tasks are performed following instrument manufacturer's recommended procedures or the procedures contained in this manual. A check of field instrument calibration is conducted initially (before sampling), at the completion of the day's field measurements, and as needed throughout the day, to establish and document that instruments are operated within specified tolerances.

Documentation of calibration measurements for field instruments must be completed every day prior to use and recorded in a field note book. Standards used for instrument calibration, operational checks, and calibration verification must be in accordance with applicable criteria such as the National Institute of Standards Technology (NIST), American Society for Testing and Materials (ASTM) standards, or other accepted procedures outlined in the instrument manufacturer's specifications.

Prior to use, maintenance procedures must be conducted on field instruments failing to meet acceptable operating specifications during calibration and calibration verification procedures. A record should be

maintained of field instruments' make(s)/model(s), status of parts needed, working status, deficiencies (if any), instrument maintenance records, and any additional pertinent information.

Contracted and sub-contracted laboratories will follow the testing, inspection and maintenance procedures required by EPA Clean Water Act approved methods and as stated in the respective laboratory's QAP and SOPs.

Field Instrument Handling Procedures

The Greens Creek site location is subject to varying climatic conditions over the course of a typical calendar year. During the fall, winter, and spring months, air temperatures may be below freezing for extended periods of time. Electrodes used for measuring pH, oxidation reduction potential (ORP/Eh), dissolved oxygen, and conductivity may be ruined or rendered inoperable if allowed to freeze. Procedures must be followed to protect field instrumentation from freezing out in the field during water quality monitoring events. Prior to beginning field activities:

- select a cooler/insulating box of adequate size to hold all of the field instruments and associated equipment needed for performing field measurements
- equip the inside of a cooler with padding such as "bubble wrap" (sample protection)
- when freezing conditions occur, add an adequate amount of a heat source to the cooler (heat packs/hand warmers or other) to maintain temperatures above freezing inside the cooler while in the field.

Field Equipment and Instrument Decontamination Procedures

All sample collection equipment and field instrumentation that comes into contact with a sample must be decontaminated following sampling. Decontamination procedures differ depending on the instrument or equipment, as described below:

- For the water level meter, portable submersible pump, and peristaltic pump, the following procedure should be followed:

    1 rinse in water
    2 wash with an anionic detergent
    3 rinse in deionized water (DI)
    4 air dry
    5 dispose of cleaning agent at the proper waste facility.

The purpose of the water and detergent wash is to remove particulate matter and other potential contaminants. The purpose of the final DI rinse is to remove detergent and any residual contaminants.

- Hydrolab Quanta (refer to the Hydrolab Procedures Manual):

    1 thoroughly rinse all probes three times with tap water

    2 place in storage/transport cup, which should have ¼ inch of tap water or pH 4 buffer (if preferred), before traveling to the new site or for short-term storage.

If traveling to another site for sampling:

    1 rinse the probe with site water at new location, to remove any residual water from the previous site.

2 between sites place probe in the transfer cup.

Using deionized water for storage purposes causes the pH probe to malfunction and require immediate replacement. Between sampling locations, the steps outlined above are recommended.

If the Quanta Multi-purpose probes appear to contain deposits or contaminants that cannot be removed from the rinse steps above, and a "drift" in parameter readout is observed, the Quanta meter can be sent into the nearest vendor for repair, or the simple cleaning methods described below can be done weekly or as needed for removing stubborn deposits:

1 Spray probes with the over-the-counter cleaning agent, "Scrubbing Bubbles," making sure that the lenses are sprayed over well, OR use Alconox solution.

2 Allow bubbles to sit for a couple of minutes.

3 Using the small tube brush is included in the maintenance kit; carefully scrub around all the probes to remove debris and build-up.

4 Rinse well with tap water, making sure to remove all the suds.

5 Dispose of any diluted cleaning agents and water at the proper waste facility.

B.6 INSPECTION / ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Field staff are responsible for ensuring that supplies and consumables (e.g., standard materials and solutions, filters, pumps, tubing, sample bottles, glassware, reagents, calibration standards, electronic data storage media, etc.) are inspected and accepted for use in the monitoring project.

All reagents, calibration standards, and kit chemicals are to be inspected to ensure that expiration dates have not been exceeded prior to use in the monitoring project. No standard solutions, buffers, or other chemical additives should be used if the expiration date has passed. It is the responsibility of the sampling manager or his/her designee to keep appropriate records, such as logbook entries or checklists, to verify the inspection/acceptance of supplies and consumables, and restock these supplies and consumables when necessary.

All sample collection devices and equipment will be appropriately cleaned prior to use in the monitoring project. All sample containers, tubing, filters, etc. provided by a laboratory or by commercial vendor, will be certified clean for the analyses of interest. Contracted and sub-contracted laboratories will follow procedures in their laboratory's Quality Assurance Plan (QAP) and SOPs for inspection/acceptance of supplies and consumables.

B.7 DATA MANAGEMENT

The success of a monitoring project relies on data and their interpretation. It is critical that data be available to users and that these data are:

- Of known quality;
- Reliable;
- Aggregated in a manner consistent with their prime use; and
- Accessible to a variety of users.

Quality Assurance/Quality Control (QA/QC) of data management begins with the raw data and ends with a defensible report, preferably through the computerized messaging of raw data.

Data management encompasses and traces the path of the data from their generation to their final use or storage (e.g., from field measurements and sample collection/recording through transfer of data to computers (laptops, data acquisition systems, etc.), laboratory analysis, data validation/verification, QA assessments and reporting of data of known quality to the respective ADEC Division of Water Program Office). It also includes/discusses the control mechanism for detecting and correcting errors.

Various people are responsible for separate or discrete parts of the data management process:

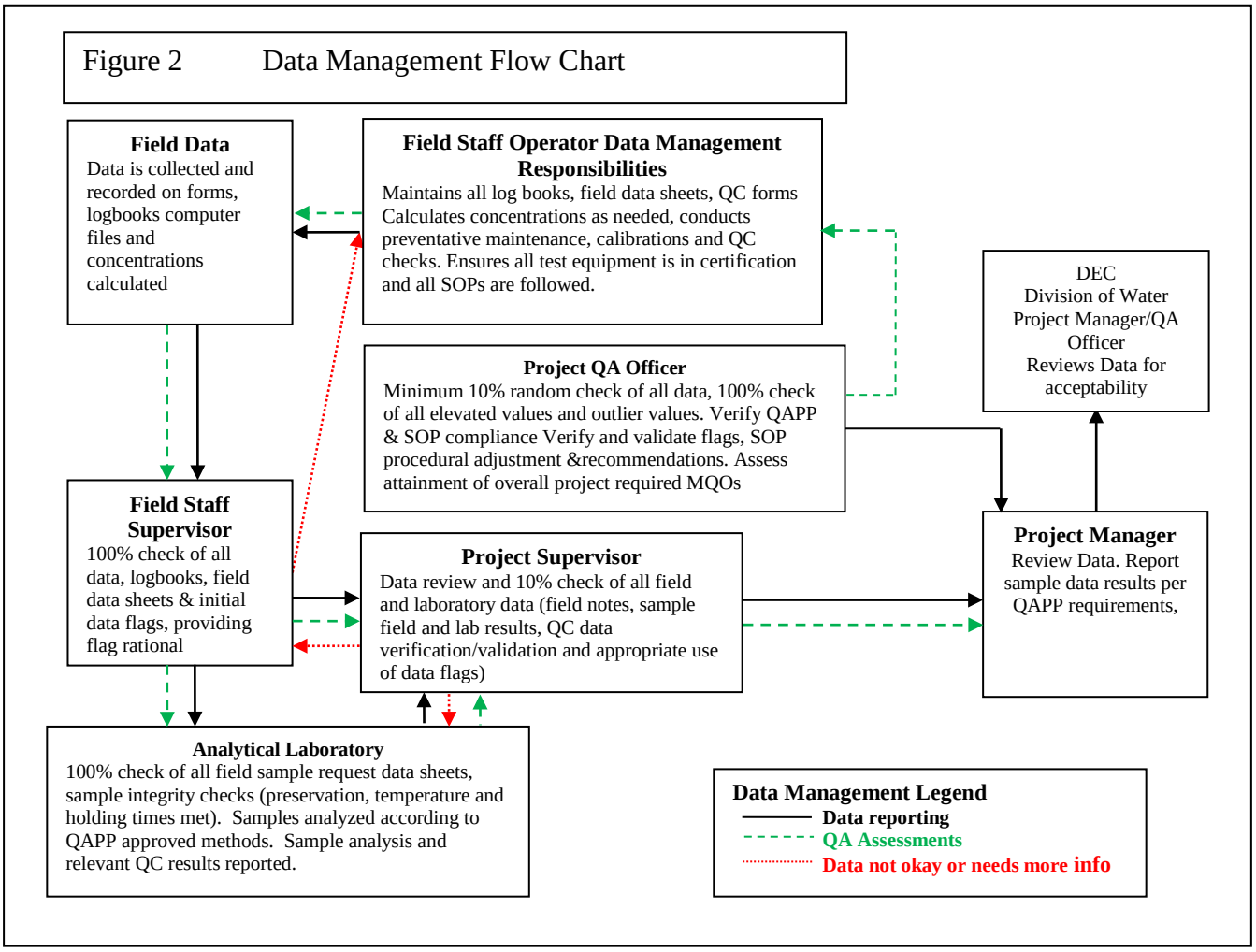
- The field samplers are responsible for field measurements/sample collection and recording of data and subsequent shipment of samples to laboratories for analyses. They assemble data files, which includes raw data, calibration information and certificates, QC checks (routine checks), data flags, sampler comments and meta data where available. These files are assembled and forwarded for secondary data review by the sampling supervisor.
- Laboratories are responsible to comply with the data quality objectives specified in the QAPP and as specified in the laboratory QAP and method specific SOPs. Validated sample laboratory data results are reported to the sampling coordinator/supervisor/project supervisor.
- Secondary reviewers (lab manager/sampling & analysis manager/project QA officer) are responsible for the review, verification and validation of field and laboratory data, and reporting validated data to the Project Manager.
- The Project QA Officer is responsible for performing routine independent reviews of data to ensure the monitoring projects data quality objectives are being met. Findings and recommended corrective actions (as appropriate) are reported directly to project management.
- The Project QA Officer is responsible for final data certification.
- ADEC DOW Project Manager/QA Officer/AQS data entry staff conducts a final review (tertiary review) and submits the validated data to STORET, AQMS, ICI-APDES, DROPS as appropriate.

The Data Management Flow Chart at the end of this section provides a visual summary description of the data flow/management process for environmental data collected in support of ADEC's Division of Water decision making processes.

Data Storage and Retention

A relational database containing all water quality data is maintained by HGCMC at the mine. Copies or partial copies of the database may be distributed to others as needed to facilitate data analysis. Data security is maintained by limiting access rights to the database files through network login IDs and passwords. Passwords are changed as needed. Laboratory data are electronically imported or manually entered into the HGCMC database. Associated qualifiers are manually entered after the QA review report is finalized and received by HGCMC. All data (100%) entered into the database manually, and a sample (5%) of the data imported into the database electronically, are verified against the hardcopy before the data are used for analysis. Laboratory Records will be retained by the contract laboratory for a minimum of five years. Project records will be retained by HGCMC at the mine site through final reclamation.

Figure 2 Data Management Flow Chart



C ASSESSMENT AND OVERSIGHT

C.1 ASSESSMENTS AND RESPONSE ACTIONS

The following QA assessment activities are provided to serve as a guideline of activities to be performed by the Project's QA Officer or his/her designee to evaluate the overall monitoring system (data collection, analysis, and reporting).

Field Assessments (each pollutant)

- Precision (replicate) sample measurements. Project should have minimum of 1 paired measurements/sampling event or 5% of project samples, whichever is greater. Replicate measurements should be evenly spaced over project timeline. Precision criteria to be specified in the project's Measurement Quality Objectives (MQO) table, see section A.6.2.

Field samples collected for subsequent laboratory analysis (each pollutant)

- Field blank samples for each analyte to be measured. Project should have minimum of 1 field blank measurement/sampling event or 5% of project samples, whichever is greater.
- Sample splits (one split sent to lab analyzing project samples, other split sent to a reference lab).
- Matrix spike duplicates (MSD) (assesses total measurement bias for project – both precision & accuracy). Frequency of MSDs usually specified by analytical method. Accuracy and precision of criteria for each pollutant and analytical method to be specified in the project's MQO table see section A.6.2.

Note: It is the responsibility of the laboratory to enroll itself in these blind PT studies with the results mailed/emailed directly to the ADEC DOW Water Quality Assurance Office and the Monitoring Project's QA Officer. Routine laboratory performance in the blind PT sample studies will be used to assess overall laboratory data quality as well as monitoring project data quality.

On-Site Assessments

- Inspection of field monitoring operations for compliance with QAPP requirements.
- Laboratory Audit (if concerns arise regarding laboratory data quality)
- Audit of project field measurement data results.

Project Data Assessments

- Audits of Monitoring Data for reproducibility of results from recalculation/reconstruction of field/lab unprocessed data.
- Calculation of monitoring project's overall achieved precision, accuracy and data completeness compared to QAPP defined precision, accuracy and data completeness goals.

C.2 REVISIONS TO QAPP

Annually the QAPP will be reviewed and revised as needed. Minor revisions may be made without formal comment. Such minor revisions may include changes to identified project staff (but not lead project staff: QA project officer, project manager, sampling manager, contracted laboratories), QAPP distribution list and/or minor editorial changes.

Revisions to the QAPP that affect stated monitoring Data Quality Objectives, Method Quality Objectives, method specific data validation *"critical"* criteria and/or inclusion of new monitoring methods must solicit input and pre-approval by ADEC DOW QA Officer/ADEC Project Management before being implemented.

C.3 QA REPORTS TO MANAGEMENT

The following table describes assessment types, frequency, content, responsible individual/s, and distribution of assessment reports to management and other recipients and actions to be taken.

Table 9 QA Reports to Management

QA Report Type	Contents	Presentation Method	Report Issued by	Reporting Frequency	
				As Required	Year
On-site Field Inspection Audit Report	Description of audit results, audit methods and standards/equipment used and any recommendations	Written text and tables, charts, graphs displaying results	Project QA Officer/auditor	✔	
Field Split Sample Report	Evaluation/comparison of result of split sample results from different laboratories, audit method.	Written text and tables, charts, graphs displaying results	Project QA Officer/auditor	✔	
On-site Laboratory Audit Report	Description of audit results, audit methods and standards/equipment used and any recommendations	Written text and tables, charts, graphs displaying results	Project QA Officer/auditor	✔	
3rd Party PT (DMRQA, etc.) Audit Report	Description of audit results, methods of analysis and any recommendations	Written text and charts, graphs displaying results	Project QA Officer/auditor	✔	✔
Corrective Action Recommendation	Description of problem(s); recommended action(s) required; time frame for feedback on resolution of problem(s)	Written text/table	Project QA Officer/auditor	✔	
Response to Corrective Action Report	Description of problem(s), description/date corrective action(s) implemented and/or scheduled to be implemented	Written text/table	Project Manager overseeing sampling and analysis	✔	
Data Quality Audit	Independent review and recalculation of sample collection/analysis (including calculations, etc.) to determine sample result. Summary of data audit results; findings; and any recommendations	Written text and charts, graphs displaying results	Project QA Officer	✔	
Quality Assurance Report to Management	Project executive summary: data completeness, precision, bias/accuracy	Written text and charts, graphs displaying results	Project QA Officer	✔	✔

D DATA VALIDATION AND USABILITY

D.1 DATA REVIEW, VERIFICATION AND VALIDATION REQUIREMENTS

D.1.1 Data Validation

Data validation means determining if data satisfy QAPP defined user requirements; that is, that the data refer back to the overall data quality objectives. Data validation is an analyte and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of a specific data set to ensure that the reported data values meet the quality goals of the environmental data operations (method specific data validation criteria). It is important that the data reviewers be familiar with the specific methods and QA/QC requirements associated with the Greens Creek project in order to properly review and validate associated analytical data. Water quality monitoring data is used for establishing baseline conditions, predicting water quality at various project facilities, and developing water quality discharge limitations. For these reasons, and because the data may also be the basis for future closure and reclamation decisions and strategies, it is critical that sample analyses and associated data meet method requirements and project specifications.

D.1.2 Data Verification

Data Verification is the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements.

D.1.3 Data Review

Data Review is the process that evaluates the overall data package to ensure procedures were followed and that reported data is reasonable and consistent with associated QA/QC results.

D.2 VALIDATION AND VERIFICATION METHODS

D.2.1 Validation Methods

All data generated shall be validated in accordance with the QA/QC requirements specified in the methods and the technical specification outlined in this QAPP. Raw field data will be maintained by the Program staff who collect it. Raw laboratory data shall be maintained by the laboratory. The laboratory may archive the analytical data into their laboratory data management system. All data will be kept a minimum of 3 years.

Field data is first reviewed by field personnel performing the field measurement procedures. As with laboratory data, the field personnel have primary responsibility for the technical quality of field data, and for ensuring that field methods are properly performed and instrumentation is in good working order.

Analytical data generated by the laboratory is first reviewed by the testing laboratory and then reported to the Sampling and Analysis Manager. The laboratory has primary responsibility for correctly identifying and quantifying analytes and compounds of interest, for identifying matrix interferences, and for identifying and correcting instrument anomalies when possible. The laboratory is also responsible for the technical quality of the data, for meeting all quality control parameters by correctly following the analytical methods, and for using instrumentation that is in proper working order for the given method.

All laboratory data will be validated according to the laboratory's QAP. The rationale for any anomalies in the QA/QC of the laboratory data will be provided to the Project Manager with the data results. Completed Chain-of-Custody forms will be sent back from the laboratory to the Sampling and Analysis Manager. Data will be qualified as necessary.

The Project QA Officer or his/her designee is responsible for reviewing field log notebooks for accuracy and completeness. Synectics is contracted to HGCMC to conduct the lab data review for the FWMP. The Project QA Officer or his/her designee (Synectics) will fill out a Laboratory Data Review and Validation Checklist (example in Appendix 1.A.E) to be included with the permanent files and the monitoring report. The Laboratory Data Review and Validation Checklist will verify and validate the following items:

- Compare sample information from the field data sheets with the laboratory analytical results to ensure no transcription errors have occurred;
- Verify and validate sample results from the laboratory;
- Verify project QC criteria have been met (i.e., Blind Duplicates, Blanks, Matrix Spikes, Standards, and Completeness).

Unacceptable data (i.e., data that do not meet the QA measurement criteria of precision, accuracy, representativeness, comparability and completeness) will not be used or if used, the problems with the data will be clearly defined, flagged appropriately and data use clearly delimited and justified. Sampling may need to be repeated. Any actions taken to correct QA/QC problems in sampling, sample handling, and analysis must be noted. Under the direction of the Project QA Officer, project staff will document any QA/QC problems and QA/QC corrective actions taken.

D.2.2 Verification Methods

The primary goal of verification is to document that applicable method, procedural and contractual requirements were met in field sampling and laboratory analysis. Verification checks to see if the data were complete, if sampling and analysis matched QAPP requirements, and if Standard Operating Procedures (SOPs) were followed.

The Project QA Officer is responsible for the verification of the data and should verify at least 10% of the generated project data. The field data sheets are compared with the SOPs, sampling requirements and sample sites identified in the Greens Creek Integrated Monitoring Plan.

D.3 RECONCILIATION WITH USER REQUIREMENTS

The Project QA Officer and Sampling & Analysis Manager will review and validate data against the Project's defined MQOs prior to final reporting stages. If there are any problems with quality sampling and analysis, these issues will be addressed immediately and methods will be modified to ensure that data quality objectives are being met. Modifications to monitoring will require notification to ADEC and subsequent edits to the approved QAPP.

Only data that have been validated and qualified, as necessary, shall be provided to ADEC Division of Water.

APPENDICES

Appendix 1.A.A

Geotechnical Visual Inspection Checklist

POND 7/10 DAM SYSTEM SITEWIDE MONTHLY INSPECTION

Name of Dam: POND 7/10 (AK00307; AK00316)		***Date:***	
Weather:			
Pool Elevation Pond 7:	*ft-msl*	***Inflow to Pond 7 WTP:***	*gpm*
Pool Elevation Pond 10:	*ft-msl*	***14-inch Outfall Discharge:***	*gpm*
Inspector:		***Signature:***	
Supervisor:		***Signature:***	

1. Pond 7 Main Embankment	**OK**	**NOT OK**
Is the crest level and free of cracks? Is there settlement occurring? Is settlement or erosion occurring on the slopes of the embankment? Is the pond liner in tact and free of holes? Is the staff gauge in place and secure? Can an excavator and other equpmewnt use the access ramp?	☐	☐
2. Wet Well 7		
Is water from Pond 7 flowing freely into WW 7? Is the cover of WW7 intact and sturdy? Is the fall protection structure intact and sturdy? Is there signs of caisson deformation? Are the pumps functioning appropriately? Are the pump rails free and clear of obstructions?	☐	☐
3. Pond 7 Inlet Spillway		
Is the concrete structure free of cracks? Is the weir inlet clean and clear of obstructions including sediment? Are staff gauges intact and readable? Are the gabion baskets intact? Is the energy dissipater intact?	☐	☐
4. Pond 7 Emergency Spillway		
Is the weir inlet and stilling basin clear and free of obstructions? Is the concrete structure free of cracks? Are the staff gauges intact? Is the rip rap in place?	☐	☐
5. Pond 7 Underdrain System		
Is water running into the caisson from the inlet pipe? Is the caisson deformed? Is the submersible pump functioning? Is the caisson level within specified limits? Is the pump functioning normally?	☐	☐

POND 7/10 DAM SYSTEM SITEWIDE WEEKLY INSPECTION

6. Pond 7/10 Flow Control Structure	**OK**	**NOT OK**
Discharge basin clear and free of obstructions? Are the slide gates free moving and intact? Is the pipe insulation intact around the pipes and flowmeters in the vault? Are the flowmeters functioning? Is the wiring intact? Is the liner below the inlet culvert from the FCS free of wear? Is the concrete free of cracks?	☐	☐
7. Pond 7/10 Connector Culverts		
Are culverts free of obstructions? Are the pipe boots intact?	☐	☐
8. Pond 10 Main Embankment		
Is the crest free of cracks? Is there settlement occurring? Is settlement or erosion occurring on the slopes of the embankment? Is the pond liner in tack and free of holes? Is the staff gauge in place and secure?	☐	☐
9. Pond 10 Inlet Structure and Access Ramp		
Are there obstructions present? Are the gabions intact? Is the access ramp free of vegetation and other obstructions?	☐	☐
10. Wet Wells 10 & 11		
Is water from Pond 10 flowing freely into WW 10? Is water flowing into WW11 from the underdrains? Is there signs of caisson deformation WW 10 or 11? Are the pumps functioning appropriately? Are the pump rails free and clear of obstructions? Are the trends as would be expected?	☐	☐
11. WW 3		
Is the caisson showing signs of deformation? Is the pump functioning? Is there water entering the caisson through the inlet pipe?	☐	☐
12. Wet Well 14		
Is the caisson showing signs of deformation? Are the pumps functioning? Is there water entering the caisson through the inlet pipe? Do the trends look as you would expect?	☐	☐
13. Wet Wells 12, 13, 15 and 16 at South Embankment		
Are the caissons showing signs of deformation? Are the pumps functioning? Is there water entering the caisson through the inlet pipes for WW 12and 13? Is the culvert entering WW16 intact and clear? Do the trends look as you would expect?	☐	☐
14. Pond 9		
Is the pond outlet clear of obstructions?	☐	☐
15. DB-04 and Sump		
Water flowing from DB-04 to sump in building? Pumps functioning?	☐	☐
16. Pond A and Caisson B		
Is the crest free of cracks? Is there settlement occurring? Is settlement or erosion occurring on the slopes of the embankment? Is the pond liner in tack and free of holes? Is the underdrain pump functioning properly? Is the pump in Caisson B functioning?	☐	☐

17. Pond C	**OK**	**NOT OK**
Are embankments free of settlement or erosion? Are caissons showing signs of deformation? Are the pumps function normally? Are the caisson heaters on? Is access free and clear? Is the Upper Pond C liner free of holes or tares? Is Upper Pond C discharge pipeline draining?	☐	☐
18. Pond D		
Is the embankment free of settlement or erosion? Is the caisson showing signs of deformation? Are the pumps functioning properly? Is the fuel tank intact? Are the inlet and discharge lines intact?	☐	☐
19. Pond 23		
Is the embankment free of settlement or erosion? Are the pumps functioning? Is the underdrain functioning? Is the liner intact and free of holes? Do the trends look as they should?	☐	☐
20. 8", 10" and 18" wastewater pipelines from Pond 23 to Pond 7/10		
Are the pipelines intact? Are the AVRs functioning as they should?	☐	☐

Describe noted deficiencies (items checked "not OK"):

Describe immediate actions taken to remedy deficiencies:

Describe marginal items that are still functioning but need attention:

Other remarks:

Appendix 1.A.B

Example Analytical Lab Chain of Custody Forms

Chain of Custody Record / Analysis Request

Lab Sent To:

Company Address:
Hecla Greens Creek Mining Company
P.O. Box 32199
Juneau, AK 99803

Project Name:

Report To: gcenvdata@hecla-mining.com

Sampler:

P.O.Number:

Container

Telephone: (907) 790-XXXX
8482 D. Maller 8420 D. Landes
8457 C. Sell 8461 G. Fredheim

Sample I.D.	Date Collected	Time Collected	Matrix Water / Soil																		pH < 2	RUSH (see below)

Comments:

Deliverable Instructions:
Use naming system below for report. XXXX will be replaced by the lab Project ID

e-mail to: gcenvdata@hecla-mining.com

Shipment Checklist

☐ _____
☐ _____
☐ _____

RELINQUISHED BY SAMPLER:	RECEIVED BY:	RELINQUISHED BY:	RECEIVED BY:	Condition of Sample Containers:
Signature:	Signature:	Signature:	Signature:	Temp Received: _____ °C
Printed Name:	Printed Name:	Printed Name:	Printed Name:	# of Coolers: _____
Firm: *HGCMC*	Firm:	Firm:	Firm:	Seals Intact: _____
Date / Time:	Date / Time:	Date / Time:	Date / Time:	Page _____ of _____

Appendix 1.A.C

PNNL Marine Science Laboratory Quality Assurance Management Plan (QAMP)

PNNL Marine Sciences Laboratory Quality Assurance Management Plan (QAMP)

PNNL Marine Sciences Laboratory
1529 West Sequim Bay Road
Sequim, Washington 98382
(360) 681-4550

April 2016

INTRODUCTION

The purpose of the PNNL Marine Sciences Laboratory (MSL) Quality Assurance Management Plan (QAMP) is to describe the Quality Program implemented at the facility. This plan summarizes the elements of the quality assurance program and discusses the quality control activities routinely used. The objective of the Quality Program is to obtain accurate and precise data consistent with project objectives. The Quality Assurance (QA) Program has evolved over time to meet client needs, but its roots are from the U.S. Environmental Protection Agency's (EPA's) document EPA QA/R-2, "EPA Requirements for Quality Management Plans". This QAMP also addresses the required elements of The NELAC Institute (TNI). While this plan sets forth Quality Program requirements, work plans and QA project plans are used to define project-specific client requirements.

Implementation of the policies and requirements specified in the QAMP and the associated procedures will provide defensible and credible data enhancing the quality of products and services.

CONCURRENCES AND APPROVALS

04/11/2016
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Table of Contents

1.0 INTRODUCTION

The Pacific Northwest National Laboratory (PNNL) is operated by Battelle Memorial Institute, Pacific Northwest Division for the Department of Energy (DOE). For the purpose of this Quality Assurance Management Plan (QAMP), Battelle and PNNL will hereafter be referred to in general as PNNL, except in instances when specifically referring to Battelle as the Battelle Memorial Institute or "Battelle" is in a cited reference title.

Quality requirements for PNNL research and analytical work are determined through contracted customer requirements and on a risk-based graded approach. The requirements of this QAMP are only applicable to MSL's accredited quality program and projects that reference to it.

1.1 QUALITY ASSURANCE MANAGEMENT PLAN

This QAMP describes the Quality Assurance (QA) Program policies, procedures and accountabilities established and implemented at the PNNL Marine Sciences Laboratory (MSL). This QAMP summarizes elements of QA and the quality control (QC) activities used to perform work by collecting accurate, precise and reliable data consistent with project objectives. Detailed methodologies and practices are written in MSL Standard Operating Procedures (SOPs) or project planning or management documents.

This QAMP is designed to meet the requirements of many clients. It is intended to address elements of the Environmental Protection Agency's (EPA) document EPA QA/R-2, "EPA Requirements for Quality Management Plans", the Navy QA Program and the requirements for The NELAC Institute (TNI). While this plan establishes the overall QA program requirements, QA Project Plans (QAPP), sample analysis plans, and/or "kits" assembled at the time of sample receipt are used to define any project specific quality requirements not contained in or that supersede this plan.

A copy of the QAMP is available on the intranet or upon request. Where applicable, personnel are expected to be aware of and perform their assignments in accordance with the QA requirements described in this QAMP. The signature page at the front of this QAMP indicates management's review, consensus, commitment and approval.

To ensure that this QAMP remains current, it is reviewed annually and updated as needed. If only minor changes are needed, red-line changes are applied to the current version. If major changes are needed, the entire document is revised and the effective date is updated.

1.2 POLICY STATEMENT

The MSL is committed to maintaining the highest ethical and professional standards. The MSL's management team is committed to comply with the requirements of PNNL, the client, and any applicable regulations/standards (e.g. TNI standard, when applicable) and continually improve the effectiveness of the management system. Personnel shall conduct themselves in accordance with these standards and in their relationships with each other, with clients, with the public, and with PNNL.

- Personnel shall document calculations, analyses, tests and software required to substantiate results and processes used to develop products/solutions.

- Personnel shall ensure that the scientific and technical information that results from PNNL research is available for maximum possible future use by the scientific community and the public unless contrary to PNNL's interests or the client's requirements.
- Personnel shall identify and appropriately control items and materials affecting scientific results.
- Personnel shall use equipment of known accuracy for process monitoring and data collection.
- Personnel shall maintain records necessary to substantiate results and processes of research or administrative activities, protect records from loss or damage, refer requests from non PNNL personnel for access to records to the Records Manager, and retire records to approved record storage areas.
- Personnel shall be fair and ethical in business operations and not request or make unauthorized business disclosures.
- Research involving human subjects shall be conducted in a manner that will fully protect the subjects.

Personnel must be free of any influence, interest, or relationship that actually or potentially conflicts with the best interests of PNNL or its clients.

- Personnel shall be free of any influence, interest, or relationship that:
  - conflicts, potentially conflicts, or appears to conflict with the best interests of PNNL or its clients
  - could cause embarrassment or public criticism of PNNL
  - could interfere with personnel's ability to perform job duties

Personnel shall comply with all laws, regulations, and contractual obligations and with the conditions imposed by the will of PNNL and PNNL policy.

- Personnel shall comply with applicable PNNL policies, standards, work flows, procedures, permits, and other work instructions. Any deviation from compliance with Laboratory work flows requires a documented variance.
- Personnel shall conduct work within the facility-specific operational boundaries specified in Facility Use Agreements.
- Management system owners shall develop their management systems, standards, and work flows with appropriate input from personnel enabling them to effectively conduct work activities in compliance with applicable requirements.
- Management system owners shall base their work flows on an evaluation of external requirements documents and applicable non-government standards, e.g., orders; directives; federal, state, and local laws; and PNNL policy.

In accordance with these principles, a QA Program was developed to assure that all activities affecting the quality of data or products produced for clients are thoroughly planned and coordinated by project teams. The MSL will ensure that all data generated, processed, or used in completing each task are scientifically valid, legally defensible, and of known and acceptable quality. As part of PNNL, the MSL is committed to the corporate policy of providing quality products and services and committed to their clients to ensure that sampling and analytical procedures are properly executed, sample integrity is not compromised, all QC procedures are implemented and recorded, and only valid data are reported. To attain this goal, the MSL has implemented the QA Program summarized in this QAMP.

1.3 OVERVIEW OF PROGRAM

The MSL works to business, management and quality practices specified by PNNL under the "How Do I" (HDI) system (a web-based system of policies, forms and procedures encompassing safety and QA). This system provides an infrastructure for performing day-to-day work, which includes QA activities. The PNNL system provides documentation of training, reminders for updating training, issuing of formal laboratory record books, a records archive, the chemical ordering and tracking system, and a system for tracking quality problems. The MSL has developed its own QA program as discussed in this QAMP to direct MSL-specific work and address client requirements. The goal is for the MSL QA Program to complement and agree with the HDI system, while meeting MSL needs.

The objective of the MSL's QA Program is to provide clients with quality products and services. A critical element in providing quality products is the maintenance of a QA Program that provides for conducting activities in a planned and controlled manner, thereby permitting the verification of quality performance. The consistent delivery of products of acceptable and documented quality requires commitment and adherence to QA and QC principles and procedures throughout the performance of each task. A commitment to quality is an integral part of every person's job. In addition, the MSL recognizes that formal functions are necessary to assure PNNL Management and its clients that the work performed and the technical products produced meet client needs and conform to their specific data quality objectives and requirements. These formal functions are QA and QC.

- QA includes all systems designed to assure management and the client that data were collected, processed, and interpreted in accordance with the requirements of the planning documents; that all aspects of work performance, including data generation and analysis are adequately documented; and that all data are accurate and fully traceable. For this system to be effective, each individual must understand his or her role in implementing the program. The responsibilities, authorities, and accountabilities with the MSL QA Program are defined in this QAMP.
- QC functions include all activities that are designed to assess or control precision and accuracy of measurements and data. QC functions involve performance of procedures necessary to attain and document the prescribed standards of performance in all measurement and data collection processes.

Project planning is performed in accordance with HDI work flow "Project Review and Approval". Project planning documents may be supported by SOPs, which are detailed documents that describe the tasks and/or approved methods for instrument calibration, data collection, data quality objectives (DQOs) and data processing, reduction and reporting. Planning also involves ensuring that personnel are fully qualified and trained to perform their responsibilities and that facilities and equipment are adequate and appropriate for their use. Procurement of qualified subcontractors is also a key consideration during the project planning stage and is performed in accordance with HDI work flow "Procurement".

A component of the work performed by the MSL involves the collection and analysis of environmental samples for chemical, biological, and physical parameters. A sample control system is essential to ensure that the history of each sample is documented and verifiable. QC activities are implemented during the performance of the work to measure and control the quality of the product. Additional methods of quality assessment are data validation, document reviews and QA verification activities. Deficiencies noted during the assessment process are

reported to management who take the necessary remedial action to bring the system into compliance. Quality improvement processes are implemented to ensure that problems identified are solved, and do not recur.

1.4 SCOPE

The MSL comprises various technical disciplines conducting research in support of environmental programs, primarily those related to the marine environment. The QA program defined in this document generally may be applied to any project performed by the MSL, as required by accreditations/certifications, projects, external clients and other components of PNNL.

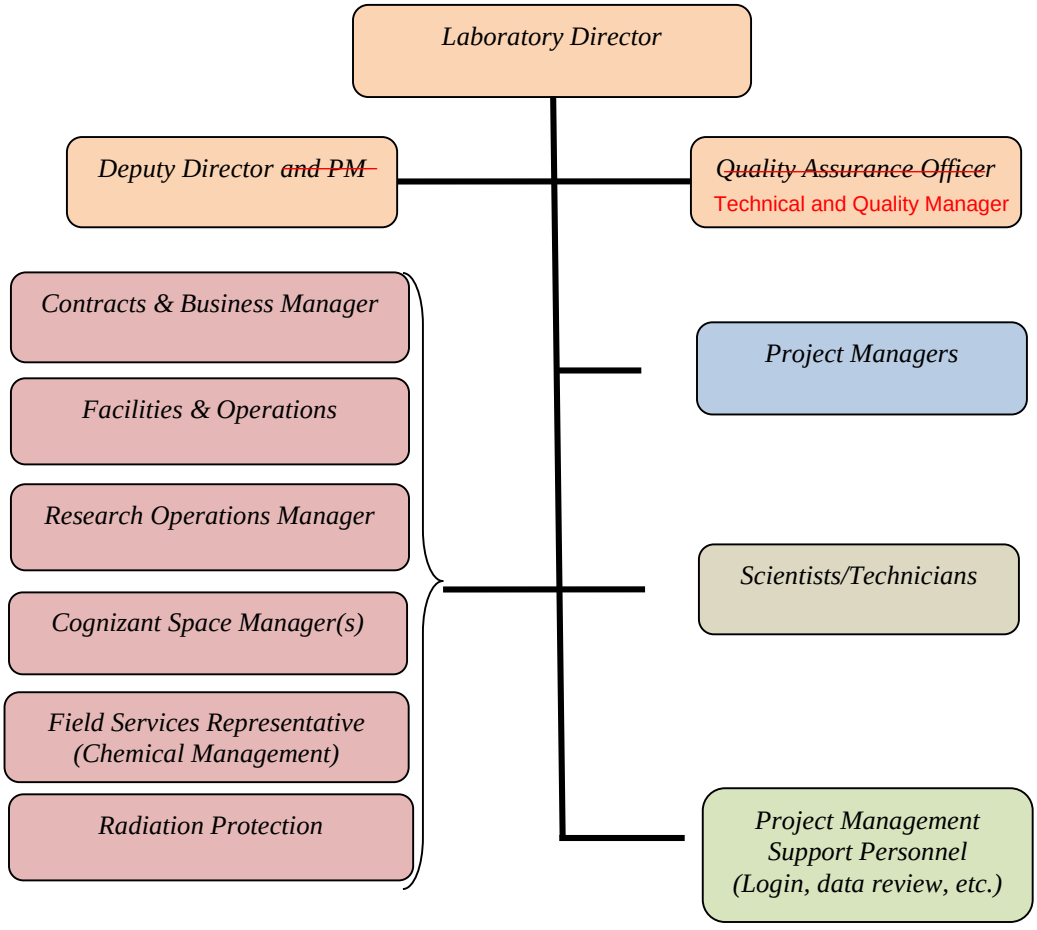
2.0 ORGANIZATION AND PERSONNEL

This section describes the organization of the MSL and defines the associated responsibilities, authorities, and accountabilities.

2.1 ORGANIZATION

QA at the MSL is an interdisciplinary line management function. The MSL's responsibility assignments are that 1) quality is achieved and maintained by those who have been assigned responsibility for performing work, and 2) quality achievement is independently verified by those not directly responsible for performing the work. The organization and key personnel of the MSL is illustrated in Figure 2.1.

FIGURE 2.1: Organization and Key Personnel



Manager (QM)

The Quality Assurance Officer (QAO) has the authority and organizational freedom to identify quality problems, to initiate, recommend or provide solutions, and to verify implementation. All verification activity reports are made available to line and project management. Line and project management are responsible for identifying and assuring implementation of corrective action to all deficiencies.

Any personnel can initiate a stop work on the basis of a safety concern. In the case of a quality concern, the supervisor (which could be the Project Manager (PM), ~~QAO~~ QM, project supervisor, etc.) shall be immediately notified of the concern and then shall initiate investigative activities or initiate implementation of corrective actions. If the nature of the concern is such that the immediate manager cannot be approached, other avenues are also available for raising concerns. It is recommended that personnel seek resolution through the successive levels of management for their organization or through their Human Resource Manager. If personnel do not believe this will lead to resolution of the concern, they may go to a member of management with whom they are comfortable and trust, or any functional director.

2.2 RESPONSIBILIES

➢ Laboratory Director

The Laboratory Director provides overall management and has responsibility for all research operations.

The Laboratory Director is ultimately responsible for ensuring that appropriately qualified personnel are hired, resources for training are allocated, and that appropriate training and professional growth are provided, and records of training are maintained.

➢ Project Manager

The PM has overall responsibility for the management of project activities. Specific responsibilities include:

- Defining DQOs and QA/QC requirements for a project (e.g. TNI standard requirements, when applicable).
- Ensuring a project work plan and QAPP or both is prepared prior to work initiation and that it meets the requirements of the client, and any applicable regulations/standards (e.g. TNI standard, when applicable).
- Ensuring, when applicable, that PNNL, local, state and federal notifications are given, permits obtained and standards/regulations followed.
- Administering and supervising all project tasks to ensure that all project objectives are met, on time, within budget, and are of appropriate quality (e.g. complies to the TNI standard, when applicable).
- Preparing project planning documents, ensuring that the plans are reviewed and approved according to policies and ensuring that the planning documents are made available to participating project personnel.
- Assigning personnel to project tasks in accordance with their experience and skill.
- Identifying project specific personnel training needs, ensuring personnel receive necessary training to perform his/her assigned tasks and ensuring the training is documented.
- Ensuring that the project objectives are communicated to project personnel and that project personnel are trained to perform any procedures unique to the project.
- Reviewing all project reports and deliverables for scientific validity (completeness, accuracy, and appropriate qualifiers) and compliance to the TNI Standard, when applicable.
- Addressing project-specific deficiencies that are identified during verification activities.

➢ Quality ~~Assurance Officer~~ Manager

The ~~QAO~~ QM provides overall direction to, and oversight of, all QA activities. The ~~QAO~~ QM is part of the Quality & Assurance Services Department and reports to the manager of that Department, located in Richland, WA. The ~~QAO~~ QM does not report to anyone at the Sequim facility and thereby maintains independence. Specific responsibilities include:

- Developing the QAMP and updating it, as needed, to reflect policies, procedures and any applicable regulations/standards (e.g. TNI standard, when applicable).
- Ensuring the quality program is compliant to the requirements of the client and any applicable regulations/standards (e.g. TNI standard, when applicable).
- Assisting Project Managers (PMs), when applicable, reviewing project planning documents for conformance to relevant policies, procedures, regulations and requirements and defining QA and QC requirements and budgets at the proposal stage.
- Assisting PMs in defining the QA and QC (e.g. TNI standard, when applicable) procedures to be used during a project.
- Administering a training program related to QA policies and procedures.
- Scheduling, planning, and conducting verification activities (assessments, data audits) of projects and facilities.
- Data package QA reviews.
- Preparing written reports summarizing the results of verification activities for distribution to PMs and management.
- Participating in, or coordinating, inspections and audits conducted by clients and regulatory agencies.
- Preparing periodic status reports of QA activities and verification results for management.
- Reviewing and providing comments on the QA aspects of technical procedures, project planning documents, and reports.
- Preparing SOPs of exclusive QA activities, also adding input for the quality sections of all SOPs.
- Scheduling triennial SOP reviews, distributing SOPs, maintaining an SOP log, and archiving historical SOPs.
- Notifying applicable management of any concerns or conditions that could impact activities or operations and stop-work when applicable.

➢ **Cognizant Space Manager (CSM)**

The CSM is responsible for providing day-to-day oversight activities of the laboratory spaces. Specific responsibilities include:

- Identifying and mitigating hazards from activities and operations in their assigned workspaces
- Conducting periodic assessments of their assigned workspaces and acting to correct any deficiencies observed
- Restricting access to their assigned workspaces when appropriate
- Notifying applicable management of any concerns or conditions that could impact activities or operations within their assigned workspaces and stop-work when applicable..

➢ **Personnel**

- Performing work in conformance with specified procedures, project planning documents and policies and procedures, including ethical and legal responsibilities.
- Notifying applicable management of any deviations to the procedures/methods specified in the planning documents or of any circumstances that could affect the quality or integrity of the data.
- Notifying applicable management of any concerns or conditions that could impact personnel safety and stop-work when applicable.
- Communicating to the appropriate manager any deviation from established procedures or issues requiring corrective action
- Defining appropriate QA requirements for purchased items and services

➢ **Contracts and Business Manager**

- Providing acquisition, contracts, and related business support that assists in meeting the strategic goals and objectives of the MSL and its clients
- Assisting personnel in ensuring that the proposal preparation process meets MSL goals
- Ensuring that QA requirements are specified in procurement documentation
- Ensuring that the proper review of requests for contracts/projects has been completed. HDI work flow "Project Review and Approval" describes the process in detail.

➢ **Operations Manager**

- Overseeing and implementing core Environmental Safety and Health (ES&H) support services to ensure laboratory and personnel compliance with regulations
- Ensuring and assessing that proper waste handling, safety measures, and training are being performed by and for personnel in conjunction with work performed

2.3 PERSONNEL QUALIFICATIONS AND EXPERIENCE

The quality of products depends, in part, on the competence and expertise of the personnel involved. The MSL will ensure that all individuals involved in the conduct or supervision of projects (including laboratory technicians, field personnel, toxicologists, analysts, data-processing personnel, supervisors, PMs and QA personnel) have the necessary education, training, and experience to perform their assigned tasks. This objective is achieved by hiring personnel with the appropriate qualifications and providing continual training and opportunities for professional growth.

Education, work experience and other applicable qualifications are documented and maintained in personnel files. The MSL home page (http://marine.pnl.gov/) provides a list of some key personnel, including a biography and education when applicable.

2.3.1 Responsibilities

The Laboratory Director is ultimately responsible for ensuring that appropriately qualified personnel are hired, resources for training are allocated, and that appropriate training and professional growth are provided, and records of training are maintained.
Each individual's supervisor is responsible for identifying specific training needs, ensuring that the personnel receives the necessary training to perform his/her assigned tasks, and assigning personnel to project tasks in accordance with their experience and skill.

Each individual is responsible for completing required training and submitting training records and certificates to their supervisor, for updating their training file as needed, and for identifying and completing additional training that may be required, but was not assigned.

2.3.2 Training

Training begins the first day of service and continues throughout a personnel's term of employment. Training is specified by personnel's line management and may include policies and organization, QA, ethical and legal responsibilities, and ES&H. Technical training begins prior to work being performed, through reviews of procedural documents and demonstrations by experienced personnel. Introductory courses are augmented by general and project-specific training that is conducted periodically. Personnel assigned to projects receive training to acquire the necessary skills to perform their responsibilities. Technical training is accomplished through a variety of approaches, including

- Direct hands-on training. Training is accomplished by reviewing procedural documents (e.g., SOPs, project work plans), proficiency testing, and supervision by experienced personnel. Each SOP includes the training requirements associated with that procedure, including any proficiency tests.
- Project kickoff meetings. Kickoff meetings ensure that all project personnel are aware of the project objectives and the methods to be used to accomplish the objectives. This also includes field safety training at the beginning of each sampling period.
- Technical seminars. These seminars, which are available to all personnel, are conducted by PNNL personnel or guest speakers and generally cover current projects or related research programs.
- Continuous education through a tuition reimbursement program.
- Attendance at professional meetings and outside workshops.

ES&H training is monitored and provided using Integrated Operations System (IOPS) and Enterprise Learning, both available on-line.

MSL specific QA training is administered by the MSL ~~QAO~~ QM in accordance with procedure MSL-A-006, Marine Sciences Laboratory Training. Personnel complete assigned training activities and acknowledge training on a training form.

PNNL's on-line training modules are administered by the PNNL training program.

2.3.3 Documentation

Records of training and qualifications include the following:

- PNNL Integrated Operations System (IOPS) training
- PNNL Enterprise Learning training
- MSL specific training assignments, including field safety meetings or pre-job/dive briefings
- Certificates attesting to the attendance or completion of external courses
- Resumes and biographies

Records of training and qualifications are maintained in personnel or project files at the MSL by the project manager or the quality ~~assurance officer,~~ Manager at PNNL human resources (HR), on the PNNL on-line computer training system, on the MSL home page (http://marine.pnl.gov/), or on

the intranet. Qualification and training records maintained by HR or in the PNNL on-line computer training system are secure with limited approved access.

2.3.4 Improper, Unethical or Illegal Actions

Training courses in ethical and legal responsibilities including the potential punishments and penalties for violations are provided initially and annually thereafter via on-line computer training. The applicable annual refresher course number and title is 002351, “PNNL Refresher Training”. Topic areas include Business Ethics, Electronic Time Reporting, Human Resources, Property Management, Sustainability and Operational Excellence, Safety and Health, Emergency Preparedness, Safeguards and Security, and Unclassified Cyber Security. Upon completion of the course, a form is signed (manually or electronically) to obtain credit. The signed form is acknowledgement that the personnel have read and understand their personal and legal responsibilities including potential punishments and penalties for violations.

3.0 FACILITIES AND EQUIPMENT

The MSL, located in Sequim, Washington, is part of the Pacific Northwest National Laboratory (PNNL). The PNNL is operated by Battelle Memorial Institute, Pacific Northwest Division for the U.S. Department of Energy (DOE). Battelle Memorial Institute is a non-profit research and development organization.

The MSL campus is on 140 acres fronting Sequim Bay in the Salish Sea, near Puget Sound, making an excellent location for marine based research. The MSL campus consists of two separate areas; the beach area and the uplands area. In addition to general office space, the MSL consists of:

- Over 8,000 square feet of general purpose laboratory space
- Over 6,000 square feet of wet laboratory space
- A research dock and outdoor experimental tanks
- State-of-the-art water supply and treatment system
- Research boats and scientific divers

The MSL supports various researchers, scientists and support personnel, including university students, graduates and post docs.

3.1 WET LABORATORIES

Two wet laboratories provide over 6000 square feet of space for studies requiring flowing freshwater, filtered seawater, and raw seawater through several separate distribution systems. High quality, Class AA seawater is obtained from Sequim Bay through an all- Polyvinyl Chloride (PVC) system with two independent intakes. A redundant system of various pumps provides a continuous supply of filtered and unfiltered seawater to experimental tanks. An emergency diesel generator ensures continuous seawater supply and other essential services in the event of electrical failure. A 14,000-gal reserve tank provides filtered seawater to the laboratories for up to 18 hours (dependent on flow rates required) in the event of failure of all three pumps. Raw seawater at ambient temperature (9-11°C) can be provided at a rate of 250 GPM, and up to 20 GPM of filtered seawater or freshwater can be supplied at various temperatures.

Holding and breeding facilities for a variety of fish, shellfish, and freshwater, estuarine, and marine plants are provided in these laboratories and in outdoor tanks. All water used in testing is passed through a regulated treatment system to ensure no impact is made on the receiving environment.

Two isolation rooms within one of the wet labs provide the capability to isolate pathogens. The isolation rooms share a common waste sump and pumping system and disinfection system on the discharge to the main water treatment system.

3.2 GENERAL PURPOSE LABORATORIES

➢ Beach Facility

General purpose laboratories in the Beach facility consist of chemistry electronics, optics, hyperbaric, biotechnology, and BSL1-2 laboratories and support rooms (e.g., wash rooms, preparation labs, and microscopy labs). A Class-100 Clean Laboratory Facility is also present.

- **Upland Facility**

General laboratories in the Upland facility consist of ten fully-equipped chemistry laboratories, including a Class-100 Clean Laboratory Facility and a radiological laboratory occupying 600 ft^2. The chemistry laboratories are equipped with an array of instrumentation, support equipment and supplies.

Specific styles of clean rooms include: Ultra Trace Hg and Methyl Hg clean rooms for preparing sampling equipment; trace metals grade supplies are stored in clean rooms. The MSL contains a general organic chemistry laboratory for preparation of sample extracts for gas chromatography and mass spectroscopy, and analysis for physical properties of sediment. A high performance liquid chromatography (HPLC) system, with variable wavelength Ultraviolet (UV) light detector, fluorescence detector, auto injector, fraction collector, integrator, and data reduction system is available for specialized sample preparation.

The Upland facility is also equipped with secure sample login, sample holding/acclimation, sample staging/preparation/digestion/extraction. It has the capacity and ergonomic set up to address the specific style of testing to be accomplished. Equipment cleaning stations necessary to provide the level of cleanliness required to support the data generated are also housed in the Uplands facility.

3.3 COMPUTER FACILITIES

Personnel use password protected computer systems connected via a local area network and routinely backed up. The systems are linked to other on- and offsite hardware composed of workstations and servers, minicomputers, database and file repositories, Web servers, and supercomputer facilities.

The MSL has access to the numerous electronic resources available through PNNL Technical Library Services. Commercial databases such as BIOSIS, Chemical Abstracts, Oceanic Abstracts, Enviroline, Aquatic Sciences and Fisheries Abstracts, Pollution and Toxicology Abstracts, and many others can all be accessed quickly by computer. The PNNL Technical Library also provides links to other Department of Energy Laboratory libraries and electronic resources. Through such access to information, literature searches can be conducted efficiently.

3.4 SAFETY AND SECURITY

The safety of personnel is of paramount importance. Therefore, the buildings are equipped with surveillance cameras and structural safety features (e.g., fire doors and extinguishers, emergency lighting systems), alarm systems which serve to alert the personnel in the event of emergencies (e.g., fire/smoke alarm), and engineering controls designed to minimize exposure to potential hazards (e.g., fume hoods).

The security of the facility is an important consideration because of the type of work performed by the MSL. Access to the MSL grounds and buildings is controlled through a card-access and lock and key system. During business hours, all visitors must enter through the main lobby and sign in with the receptionist. Selected areas within the facility are secured at all times and their access limited to authorized personnel. Such areas include the walk-in cold room used for sample storage, the records storage area, the solvent shed, and the data archives. HDI work flow “Access and Protection Requirements at Battelle Facilities” describes the process in detail.

Computer security is a function of the PNNL network and is administered from facilities located in Richland, WA. Personnel have individual responsibility to back up files, instruments and data bases at regularly scheduled intervals which are prescribed in procedure MSL-D-004, Data Reporting, Reduction, Back Up, and Archiving.

4.0 PROCUREMENT AND CONTROL

4.1 MATERIAL PROCUREMENT AND CONTROL

Examples of items that generally have a significant influence on the quality of work, and therefore generally need defined quality requirements are the following:

- Standards and reference materials
- Reagents, chemicals and solutions
- Animals and feed
- Computer software and hardware, and
- Some miscellaneous items such as designed equipment

Procurement activities are prescribed in HDI work flow "Procurement" which should be consulted to determine appropriate QA requirements before initiating procurement actions.

4.1.1 Miscellaneous Procurements

Miscellaneous procurements of items that have a significant influence on the quality of work generally need defined quality requirements. When the purchaser does not know if quality requirements should be specified, the practice is to request the ~~QAO~~ QM or representative to make this determination and document it as a note, letter or email.

4.1.2 Material Receiving Inspection

When materials are ordered that require certification (i.e., standard or certified reference materials (SRMs, CRMs), standards, pre-cleaned sample containers, etc.), a request for certifications shall be made on the purchase order. Standards and reference materials must be traceable to the National Institute of Standards and Technology (NIST) or other nationally-recognized standard (e.g., American Society for Testing Materials [ASTM]). The traceability must be documented by a certificate or label that verifies this link. The traceability documentation must be received and found to be acceptable before material use. Acceptance of these items and certifications shall consist of verifying that the lot numbers on the certifications and the jar and/or boxes are the same. Approval shall be indicated by a signature and date of signature on the certificate. Pending receipt of this documentation and its acceptance, affected material must be segregated to prevent inadvertent use. Certifications received will be maintained by the ~~QAO~~ QM or in the Project files.

4.1.3 Reagent and Standard Inventory Procedures

The procurement of reagents, chemicals and solutions should include requirements for shipping stocked inventory materials with the longest period to the expiration date (i.e., the freshest material) possible, with lot numbers specified. In some cases where extremely high purity material is requested, a request for purity documentation may be necessary.

Procurement procedures should require that a manufacturer's recommended expiration date is provided with every standard material. If manufacturer's expiration dates are not provided, the laboratory must assign an appropriate expiration date in accordance with procedure MSL-A-008, Control of Standards, Reagents, Solutions, Test/Control Articles and Specimens.

The MSL follows the PNNL HDI system requirements for logging in reagents, chemicals and solutions into the associated Chemical Management System (CMS). This system provides policies and procedures regarding tracking and inventory and storage of samples as well as chemical use and disposal. The CMS is used to provide an up-to-date inventory to facilitate emergency response, monitor the location of various classes of materials and identify situations where acceptable limits for the building/facility determined by the assigned chemical hazard group and fire zone might be exceeded before a violation occurs. An assigned Sample Inventory Coordinator provides Radio Frequency Identification (RFID) tags for each tracked chemical item when it is received and assigns it to a location. The item then is tracked in the CMS until disposal. The system is also used to ensure that facility limits based on the chemical hazard group and the assigned fire zone determination are not exceeded.

Personnel are required to document when chemicals are received and expiration dates as prescribed in procedure MSL-A-008, Control of Standards, Reagents, Solutions, Test/Control Articles, and Specimens.

4.1.4 Computer Software and Hardware

Software and hardware is procured in accordance with the PNNL HDI system procurement requirements are maintained under the PNNL Managed Hardware Program. In general, QA requirements for the procurement of software should consider the following guidelines:

- Commercial software that has been developed under the manufacturer's QA Program and fully tested before release is preferable to other types of software developed under lesser or no QA Program
- Documents necessary to demonstrate that software was developed using a Life Cycle approach such as User's Manuals shall be requested when software is ordered.
- Licenses that come with the software and original documentation should be requested, obtained and protected.
- Software that requires a signed site license agreement can only be purchased by individuals with appropriate authority.
- Hardware/Software that exceeds the most recent established PNNL monetary limit can only be purchased with appropriate management approvals.
- Software procured as a product under a subcontract must specify detailed QA requirements for software development and use, and provide plans for testing, verification and validation tests and include acceptance criteria.

4.1.5 Solvent Storage Policies

Solvents used in the laboratory are in containers of 20 liters or less. On receipt they are logged in, RFID-tagged, and tracked, as are all chemicals. No more than a working day's supply of flammable or combustible solvents is permitted out of flammable storage in a laboratory; at the end of the day, these materials must be returned to flammable storage. Large flammable storage cabinets, located in an area separate from the building, are used for storage of solvents that exceed the lab's storage capacity.

4.1.6 Waste Disposal

Hazardous wastes are managed in accordance with Washington State Department of Ecology's (WA-DOE's) Chapter 173-303 WAC, "Dangerous Waste Regulations." The MSL is a "less than

90-day storage" facility and a large-quantity generator and, as such, fulfills all the requirements outlined in the regulation regarding proper labeling, designating, inspections, and timely disposal of hazardous waste. Personnel that generate/handle waste are initially trained in waste management procedures and updated annually of new regulations and requirements. Procedure MSL-A-015, Waste Management and Pollution Prevention, describes the waste streams and their disposal.

4.2 SUBCONTRACTORS

The MSL does not routinely subcontract analyses that can be performed in-house, but in some situations this could occur. The MSL could also subcontract project analyses when there is a project-specific requirement. The MSL is ultimately responsible for the quality of work performed by its subcontractors. Therefore, procedures have been established to ensure that subcontractors determined to have applicable associated risks are qualified to perform their responsibilities, know the project objectives, methods, and responsibilities, and the work performed is monitored to assess conformance to the project specifications.

Whenever work is to be subcontracted to others, the MSL should advise clients of this intent and obtain their permission for this approach. For projects requiring TNI certification, documented permission from the client is required and work may be subcontracted only to TNI-certified laboratories for the specific analysis and matrix of interest or it will be pre-approved and identified in a report or in the project contract that a non-TNI laboratory was used.

In addition to the requirements prescribed in HDI work flow “Acquire Product or Service via Purchase Order-Subcontract”, it is expected that all policies, procedures, and responsibilities required by the project are flowed down to the subcontractor and verified accordingly.

PNNL provides Evaluated Supplier Options which can be used as a starting point to define subcontractors. If the subcontractor does not meet any of the evaluated supplier options, then whenever it is deemed appropriate on a risk based graded approach, an audit of subcontractor may be performed. The audit may include review of the subcontractors QA program, data audits, inspection of facilities, or inspection of project activities. The contract should include a SOW in sufficient detail so that the scope of work, methods, QA requirements, responsibilities, deliverables, and due date are clearly understood between the MSL and the subcontractor.

5.0 PROJECT PLANNING DOCUMENTS

5.1 CONTENT AND FORMAT

5.1.1 General

Project planning documents (e.g., work plans, QAPP, toxicity testing plans, field sampling plans and SOWs) are documents that describe the objectives of a project and the methods, organization, and QA and QC activities necessary to meet the goals of the project. Each project conducted by the MSL must have a planning document that adequately describes the work to be performed, has been approved by the PM, and is in place prior to the start of work.

When applicable, in the absence of client-driven requirements, the following information should be identified in project planning documents:

- A descriptive title, client name, PNNL project number, and effective date;
- The identities of the PM, task leaders, and other key project personnel, including subcontractors;
- A statement of the general goals and the specific DQOs of the project;
- A description of the experimental design and procedures;
- A description of the QA and QC procedures (including DQO's) that will be applied to the project tasks;
- The project schedule, including milestones and deliverables;
- A description of the types of data to be recorded; and
- A statement of deliverable requirements.

5.1.2 Environmental Protection Agency

When work is conducted for the U.S. EPA, it is required that all environmental data-collection activities be covered by a QAPP. Therefore, all project planning documents prepared for the EPA must adhere to specific content and format requirements, as dictated by the EPA office involved. Protocols written for studies conducted under Food and Drug Administration (FDA) or EPA Good Laboratory Practices (GLP) standards must adhere to the specifications of 21 Code of Federal Regulations (CFR) Part 58 (FDA), 40 CFR Part 160 (EPA/ Federal Insecticide, Fungicide, and Rodenticide Act [FIFRA]), or 40 CFR Part 792 (EPA/Toxic Substances Control Act (TSCA), as applicable.

5.2 APPROVAL AND DISTRIBUTION

All planning documents shall be approved by the PM, at a minimum, before work is started on the project.

The project planning document is distributed, or made available to, personnel involved in the project and to the ~~QAO~~QM. It is expected that all work will be conducted according to the planning documents. Modifications to approved planning document procedures should be made only with the concurrence of the PM and client, when applicable.

5.3 DATA QUALITY OBJECTIVES

DQOs are defined as the criteria needed to design an environmental data collection program. DQOs are developed from a multi-step, reiterative process that involves, project management, technical personnel, and the individuals who will be using the data to make decisions. The DQO process may entail the following:

- Stating the problem to be resolved, including limitations of time and resources;
- Identifying the decision that will be made using the data;
- Identifying inputs to the decision, including the environmental measurements needed and the criteria for taking action;
- Specifying how the results will be summarized and used; and
- Specifying acceptable error rates (i.e., limits on uncertainty).

The objective of the DQO development process is to design a cost-effective program that will provide the necessary amount and type of sufficient-quality data.

Once the acceptable error rate has been defined, the program's QA requirements are developed. The specific types of QC samples used to measure data quality are discussed in later in this QAMP.

The QC measurements and acceptance criteria are outlined in SOPs or project planning documents. The precision and accuracy objectives specified are based on standard method performance information (when available) and historical laboratory performance but may change based on project specific criteria. When required by the client or PM, other QC checks for accuracy, precision, comparability and completeness shall be applied to each batch of samples.

During the development of DQOs, the *PARCCS* parameters of precision, accuracy, representativeness, comparability, completeness and sensitivity are commonly considered when measuring data quality. These qualitative and quantitative parameters are described below.

5.3.1 Precision

Precision measures the similarity of individual measurements of the same property, usually under prescribed similar conditions.

Measures of analytical precision may be determined by the analysis of laboratory replicates or matrix spike/matrix spike duplicate recoveries. Laboratory replicates will be prepared by homogenizing and splitting a sample in the laboratory, and carrying the sub-samples through the entire analytical process. Precision can be expressed in terms of relative percent difference (RPD) or relative standard deviation (RSD).

For replicates where duplicates are performed, RPD will be used:

$$RPD = \left(\frac{ABS(C_1 - C_2)}{Average(C_1 : C_2)} \right) * 100$$

ABS = Absolute Value

For replicates where triplicates or more are performed, RSD or CV (coefficient of variation) will be used:

$RSD = \sqrt{\left(\frac{\sum(x-\bar{x})^2}{(n-1)}\right)} * 100$	$CV = \sqrt{\left(\frac{\sum(x-\bar{x})^2}{(n-1)}\right)} * 100$

$\bar{x}$ Sample mean AVERAGE(number1,number2,…)
n = Sample size

5.3.2 Accuracy

Accuracy is a measure of the bias of a system or measurement. It is the closeness of agreement between an observed value and an accepted value.

Accuracy of chemical analysis may be determined [for each matrix of interest (sediment, tissue and seawater)] through the analysis of laboratory control samples, matrix spikes, method blanks, SRMs (when applicable) and surrogate internal standards (organic analyses only).

- **Blank Spike (BS)/Laboratory Control Sample (LCS)** - an aliquot of clean matrix (e.g. reagent water) to which known concentrations are added and prepared, treated and analyzed in the same manner as the associated samples. Its purpose is to determine whether the method is within accepted control limits.
- **Matrix Spike (MS)/Matrix Spike Duplicate (MSD)** - an aliquot of a sample to which known concentrations are added and treated and analyzed in the same manner as the associated samples. Its purpose is to determine whether the sample matrix contributes bias to the results**.**
- **Method Blank (MB)** - an aliquot of clean matrix (e.g. reagent water) prepared, treated and analyzed in the same manner as the associated samples. Its purpose is to determine if method concentrations or interferences are present in the laboratory environment, the reagents, or the apparatus' used that could contribute bias to the results**.**
- **StandardReference Material (SRM)** - a material obtained from an independent source, is certified to a known concentration by a recognized authority (e.g., NIST) and is treated and analyzed in the same manner as the associated samples. Its purpose is to determine whether the method is within accepted control limits.
- **Surrogate Standard** - an analyte which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in the samples. The surrogate is spiked in the sample prior to extraction. The recovery of surrogate is used to quantify extraction efficiency and monitor method performance.

For measurements where matrix spikes or laboratory control samples are used, percent recovery will be used to assess accuracy:

$\%R = \left(\frac{S-U}{C_{sa}}\right) * 100$	%R = percent recovery S = measured concentration in spiked aliquot U = measured concentration in un-spiked aliquot C_{sa} = actual concentration of spike added

For situations where a SRM is used, percent difference (%D or PD) or percent recovery (%R) will be used:

$PD = \left(\frac{C_1 - C_2}{C_2} \right) * 100$	PD = percent difference C_1 = measured value C_2 = certified or consensus value	$\%R = \left(\frac{C_1}{C_2} \right) * 100$	%R = percent recovery C_1 = measured value C_2 = certified or consensus value

5.3.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Representativeness will be addressed primarily by the proper handling and storage of samples and analysis within the specified holding times so that the material analyzed reflects the material collected as accurately as possible. Representativeness of data will be discussed, when appropriate, in deliverable reports.

5.3.4 Comparability

Comparability expresses the confidence with which one data set can be compared to another. Comparability will not be quantified, but will be addressed through the use of laboratory methods that are based on EPA or other recognized methods. The use of standard reporting units also will facilitate comparability with other data sets. Comparability of other data will be discussed, when appropriate, in deliverable reports.

5.3.5 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Target completeness values are 100% for chemical sample analysis.

5.3.6 Sensitivity

Sensitivity is the capability of methodology or instrumentation to discriminate among measurement responses for quantitative difference of a parameter of interest.

6.0 STANDARD OPERATING PROCEDURES

Many routine analytical laboratory activities are directed and controlled by internal procedures or by published procedures. Where possible, U.S. EPA and consensus methods (e.g., National Oceanic and Atmospheric Administration (NOAA) Status and Trends) are used where the technique is applicable to the sample matrix and the overall objective of the analysis.

A list of SOPs is available on the intranet or upon request. The ~~QAO~~ QM maintains and updates the list of controlled documents.

6.1 SCOPE AND PURPOSE

The MSL encourages the preparation of SOPs for routine environmental measurement and analyses and related QA and QC activities. Research and development activities that are not routine, or are unique to a project, can be described in project planning documents such as work plans or in written protocols included in the project files. Subjects that are covered in SOPs include, but are not limited to:

- Sample collection
- Sample handling, preservation, and storage
- Chain of Custody (CoC) procedures
- Digestion and sample preparation
- Sample analysis
- Equipment use, maintenance, and calibration
- Record management
- Data reduction, processing, and validation
- QA verification activities

SOPs are documents that describe procedures that must be followed to ensure the integrity and quality of data. SOPs serve a multi-purpose function, including to

- Reduce the introduction of errors and variables by ensuring the consistent use of appropriate procedures
- Communicate to the necessary people (e.g., client, project personnel) how the work will be conducted, and provide a basis for training
- Increase the effectiveness of training by clearly and consistently communicating the approved method of performing a procedure
- Provide a historical record of the work performed
- Provide a basis for data comparability
- Provide a basis for maintaining reproducible results and producing defensible data

6.2 CONTENT AND FORMAT

Each SOP must be clearly written and include sufficient detail to clearly describe the operation to be carried out so that a qualified individual can perform the procedure. However, it should be flexible enough to accommodate expected variations while maintaining the integrity of the procedure and the quality of the data being generated. SOPs covering equipment must include descriptions of calibration, operation, and maintenance requirements. Procedural SOPs must contain sections on preparation, procedures, calculations, and quality control. Equipment and

procedural SOPs must also include a discussion of the safety concerns associated with the equipment or procedure. All SOPs must state the objective or application of the SOP topic and must stipulate the requirements for the successful completion of training. Specific requirements for content and format are prescribed in procedure MSL-A-003, Guideline's for SOP Format and Control.

6.3 RESPONSIBILITIES

The individual preparing the SOP is responsible for ensuring that the SOP completely and accurately describes the procedures, is based on sound scientific principles or recognized procedures, and conforms to the standards for procedure documentation prescribed in procedure MSL-A-003, Guidelines for SOP Format and Control.

The ~~QAO~~ QM is responsible for

- Assigning each SOP a unique number and entering it into the SOP controlled document log
- Reviewing all SOPs
- Distributing approved SOPs, including posting to the intranet
- Maintaining historical files of SOPs

6.4 REVIEWS AND APPROVALS

Draft SOPs go through a formal review and approval process in accordance with SOP MSL-A-003, Guidelines for SOP Format and Control.

6.5 DISTRIBUTION AND CONTROL

The official controlled copies of SOPs are the versions maintained in the SOP file on the intranet and are readily available to personnel. All other copies (printed or saved in personal electronic files) are considered uncontrolled. All PNNL personnel have signed non-disclosure documents and are trained in the sensitive nature of these documents.

6.6 MODIFICATION AND REVISION

Changes to SOPs must be controlled to ensure documentation and traceability to the modification. SOP modifications will be performed in accordance with SOP MSL-A-003, Guidelines for SOP Format and Control.

7.0 LABORATORY DOCUMENTATION AND RECORDS

A critical component in the generation quality products is proper record keeping and the maintenance of the records after project completion. Documentation must be sufficiently detailed so that the data are traceable and program data can be reconstructed based on the project records. These records must be maintained in a secure location and must be identifiable and retrievable. Should the MSL be unable to retain or maintain documents for any reason (e.g., if the laboratory transfers ownership or goes out of business) all records will be transferred to the PNNL Richland, WA archive system.

7.1 DOCUMENTATION

Data generated during the course of a project must be capable of withstanding challenges to its validity, accuracy, legibility and traceability. To meet this objective, data are recorded in standardized formats and in accordance with prescribed SOPs. Personnel whose responsibilities include recording data must be aware of, and adhere to, the SOPs during the performance of their work. Briefly, data must be entered onto data sheets or in project notebooks directly, promptly, and legibly. All entries must be made in indelible ink, and must be accompanied with the date and initials or signature of the individual making the entry. In some instances (e.g. divers writing underwater or fieldworkers writing in the rain on Rite-in-the-Rain paper), pencil may be used. Changes or corrections to data must not obliterate the original entry, but must be indicated with a single line through the original entry. All changes or corrections must be accompanied by the date and initials or signature of the individual making the change and, when not obvious, an explanation of the change. Specific requirements for documentation are prescribed in procedures MSL-D-001, Recording Data on Data Sheets and Laboratory Notebooks and MSL-D-004, Data Reporting, Reduction, Backup, and Archiving.

7.2 RECORDS

The data archive system is designed to ensure that materials are stored in an orderly manner under secure conditions, and may be easily and promptly retrieved should the need arise. Data archiving requirements and prescribed in procedures MSL-D-003, Archiving of Records, Data, and Retired SOPs and MSL-D-004, Data Reporting, Reduction, Backup, and Archiving.

All material generated during a project should be archived upon completion of the project. All records necessary for the interpretation and evaluation of project data, including planning documents, raw data and other documentation, correspondence, and reports, should be retained. The PM is responsible for ensuring the project materials are collected, organized, and forwarded to the archives at the end of the project. PNNL policy is to retain electronic data files for five years, unless otherwise specified by client request. Hard copy data are stored as prescribed in procedure MSL-D-003, Archiving of Records, Data, and Retired SOPs. Archives are controlled access (locked) storage rooms at the MSL or in Richland, WA. Data are stored and retrieved by project number or central file number.

8.0 SAMPLE HANDLING, TRACKING AND DISPOSITION

Sample handling and tracking requirements are prescribed in procedures MSL-A-001, Sample Log-In Procedure and MSL-A-002, Sample Chain-Of-Custody, and MSL-E-001, Marine Resources Field Operations and Fish Research. The processing of data collected from these activities is prescribed in procedure MSL-D-004, Data Reporting, Reduction, Backup, and Archiving (Archiving may be superseded by client requirements).

8.1 RECORDS

Sample custody responsibilities must be clearly defined and understood by personnel involved for the system to be effective. Samples are considered to be in a person's custody if:

- The samples are in a person's actual possession
- The samples are in a person's view after being in that person's possession
- The samples were in a person's possession and then were locked or sealed to prevent tampering
- The samples are in a secure area

The sample collector is responsible for the proper collection, preservation, and labeling of samples, and for documentation of sample history and custody in the field. The sample collector also is responsible for packaging the samples for shipment, maintaining sample integrity, and for arranging for transportation to the laboratory.

The sample custodian is responsible for receiving and inventorying the samples, placing them in storage, and completing the documentation associated with these procedures. The laboratory sample custodian also is responsible for informing the PM of the samples' arrival and for promptly notifying him/her of any broken, missing, or compromised samples.

8.1.1 General (Non-TNI) Samples

8.1.1.1 Chain of Custody Not required

Samples may not always require a formal log-in and/or Chain of Custody. This may occur when the samples will be returned immediately to the place it was collected, retained by the researcher, or disposed of without having previously left the custody of the researcher. Examples include:

1. While working in the field, fish are collected in beach seines, identified to species, measured, and returned to the water. Data are recorded on data forms and include the date of collection and initials of the recorder. A formal CoC is not completed for the fish sampled.
2. Eelgrass plants are collected offsite during a field project and returned to the MSL. The number of coolers containing eelgrass and being transported to the laboratory is recorded on a field data form or notebook, but a formal CoC is not completed. The exact number of plants harvested is not recorded until the plants are transplanted at the laboratory, to minimize handling of the plants. The number of plants that are transplanted is recorded in the field notebook and subsequent counts of eelgrass shoots in the tank are also recorded to document population changes over time. Each data entry includes the date and initials of the recorder.

3. Eelgrass plants are marked and later harvested from the MSL beach. A record of the location and number of plants sampled is recorded along with the date, recorder's initials and other pertinent information; however a formal CoC is not prepared. The plants are processed and dry weights are obtained to determine the plant's productivity. After the measurements are taken, the plant material is disposed of.

In these circumstances, samples are received and custody is maintained/documented according to the project planning documents.

8.1.1.2 Chain of Custody Required

When chain of custody is required, the MSL documents all sample fates for the client based on objective evidence maintained during the sample processing. Objective evidence will be defined as all information necessary to produce unequivocal, accurate records that document the applicable laboratory activities (including signatures of individuals who physically handle individual samples), accounting for all time periods associated with sample receipt, processing, analysis and storage and disposal.

In accordance with procedure MSL-A-001, Sample Chain of Custody, the PM is responsible to determine which items are required and to ensure that all relevant items are addressed because different programs have different requirements and to assist in project planning.

For test organisims, in place of a CoC a shipping form can be signed and dated and the condition of organism noted. Sample control is the formal system designed to provide sufficient information to reconstruct the history of each sample. This system involves procedural, record keeping and organizational components and is critical for any environmental program that is generating data that may be used for regulatory decisions or in support of litigation.

8.1.2 TNI Samples

Samples to be analyzed under TNI requirements require a formal log-in and Chain of Custody. Login is performed and documented in accordance with procedure MSL-A-001, Sample Log-in Procedure.

8.2 LOGIN

When samples are received from an outside source, they are logged in when received in the shipping area. If a CoC form accompanies the samples, it may used to document the date and time of sample receipt and condition; if not an internal CoC may be initiated. The sample labels are compared to the CoC and, when applicable, assigned an identification code plus sequential numbering of samples upon arrival. Sample containers are inspected for sample integrity (e.g., broken seals, broken or cracked containers, spilled samples and sample temperature). Any discrepancies identified during the process are brought to the attention of the PM who is responsible for contacting the client, when applicable.

8.2.1 Preservation

When sample preservation (e.g. temperature or pH) is indicated by the type of analysis or client specification, preservation is checked and adjusted, when applicable, in accordance with procedure MSL-A-001, Sample Log-in Procedure and/or project documents.

If the samples are not immediately required for use, they are stored under the appropriate conditions in a controlled or secure area.

8.3 SAMPLE TRACKING

Sample tracking while samples are in the laboratory is the responsibility of the individual Laboratory Supervisors and the PM. It is the responsibility of the PM to ensure that the levels of sample custody and tracking needed are specified, samples are given the appropriate priority in the laboratory, and the proper storage, analyses/tests and methods are being performed.

When living organisms are collected, the number of specimens collected is kept to the minimum the investigator determines is necessary to accomplish project goals. If vertebrate species will be collected, handled, or housed during a study, an Animal Care Committee Protocol is submitted to the Institutional Animal Care and Use Committee for review and approval prior to receiving the animals or conducting the research. It is the responsibility of the PM to ensure that sample collection, handling, storage, and/or testing are performed properly.

8.4 SAMPLE ARCHIVING AND DISPOSITION

The PM is responsible for proper disposal of residual sample material (not all samples will have residual left over for disposal). Sample disposition takes three forms: 1) dispose by appropriate means depending on sample content; 2) return to client; or 3) archive for a pre-determined amount of time. Unless arrangements have been made previously, the samples are generally disposed of by the laboratory.

8.4.1 Samples disposed of by a subcontractor laboratory

If the subcontractor laboratory or testing facility is responsible for disposing of the samples, the subcontractor is asked to notify the PM before final disposition. The PM will notify the originator that the samples are scheduled to be destroyed, or will define client requirements for an extended period of storage.

After destruction of samples, the subcontractor laboratory or testing facility is asked to return a copy of the CoC to the PM for placement in project files. The originator may be forwarded a copy of the final Chain-of Custody documentation if requested.

The PM or representative records the date of receipt on the CoC in the "Received by" section of the form space and indicates the samples were destroyed ending the chain of possession.

8.4.2 Samples disposed of by the MSL

For returned samples (should be received with CoCs) or samples that have never left MSL custody, the PM or representative will notify the originator that the samples are scheduled to be destroyed, or will define client requirements for an extended period of storage. If extended storage is not requested, the PM is responsible to ensure samples are disposed in accordance with procedure MSL-A-015, Waste Management and Pollution Prevention.

8.4.3 Samples returned to the client for disposal

Samples may be returned to the client (or the sampling site) by client request. Samples are shipped to meet Department of Transportation regulations. Generally, the samples are shipped in the same way that they were initially shipped to the MSL. Sample disposition should be documented in the central file of each project. The PM shall ensure that completed CoC are filed in the appropriate project files. The originator may be forwarded a copy of the final CoC documentation if requested.

9.0 QUALITY CONTROL

Technical personnel perform QC activities during the conduct of the project. The purpose of these functions is to measure the quality of the data and if necessary, adjust the measurement system so that the specified level of quality is attained.

9.1 GENERAL

9.1.1 Toxicity Testing and Biological Studies

For toxicity testing, each test has its own quality control criteria that are included as part of the test design established in project planning documents. Reference toxicant tests (positive controls), are performed to demonstrate that test organisms used are appropriately sensitive and that the laboratory procedures and techniques are appropriate and repeatable. A reference toxicant test is normally performed with each test, or at a minimum, once with each batch of test organisms as prescribed a procedure (e.g. MSL-T-034, Reference Toxicant Stock Solution Preparation) or project planning documents. It is the responsibility of the PM to ensure the reference toxicant database and control chart(s) are up to date with each set of test results. Each test method contains specific test acceptability criteria for controls, reference toxicant results, test conditions, etc. An individual test may be conditionally acceptable if temperature, Dissolved Oxygen (DO), or other specified conditions fall outside specifications, depending on the degree of the departure from the specified conditions and the overall impact on the test. The acceptability of the test will depend on the professional judgment of the PM or designee. Any deviation from test specifications must be noted when reporting data.

Quality control in biological studies encompasses a wide range of activities such as species identification, organism counts or density estimates, and data entry. QC activities measure the quality of the data and if necessary, adjust the measurement system so that the specified level of quality is attained. For example:

1. Fish species are often identified by two researchers and through consultation of a region-appropriate taxonomic key or guide for reference. This provides a more objective approach to species identification, especially the first time a new species is encountered or the first time a researcher performs species identification.
2. Plants and other resources are often described by the percent of open space they cover within a standardized area (e.g., 1-25%, 26-50%, 51-75%, or 76-100% cover). At the beginning of a field sampling period, researchers may standardize their estimates of percent cover by individually examining several examples of percent cover and comparing their estimates. If the estimates vary, the researchers work together until they can agree on their cover estimates before collecting actual project data individually. Periodic reassessments of standardization between researchers increases the quality of the data.

9.1.2 TNI Analyses

For analyses performed under the TNI standard, work shall be performed in accordance with approved SOPs.

9.2 LIMITS OF DETECTION

Method detection limits (MDLs) are determined for all parameters for a number of different matrices (fresh water collected from the in-house de-ionized water system, filtered seawater from Sequim Bay, Sequim Bay or other clean sediment, chicken tissue, etc.). The method used to determine MDLs is prescribed in procedure MSL-Q-007, Procedure for Determining Method Detection Limits. Limits of quantization may also be reported on request as more conservative estimates of detection limits. MDLs and their determination documentation are available on the intranet or upon request.

9.3 HOLDING TIMES AND PRESERVATION

Holding times typically begin with the day of sample collection. However, holding times can be assessed from both the date of sample collection and the date of sample receipt, depending on project planning documents. In the absence of client-specified holding times, the holding times and requirements provided in Tables 9.3.1 and 9.3.2 are used.

When samples require preservation at the MSL, a holding period before analysis may apply. Holding periods are prescribed in the applicable analytical procedures.

TABLE 9.3.1: Chemistry Sample Holding Times and Preservation

	Analysis	Preservation	Holding Time (Days)
Sediment*	Metals (including Hg)	freeze dried; 4±2°C, or -20±10°C	180[(b)]
	Methylmercury	±2°C, or -20±10°C	180[(d)]
	Organic Compounds	4±2°C, or -20±10°C	30[(b)] extraction; 40 analysis [c]
Tissue*	Metals (including Hg)	freeze dried; 4±2°C, or -20±10°C	180[(b)]
	Methylmercury	4±2°C, or -20±10°C, the freeze dry	180 [(d)]
	Organic Compounds	4±2°C, or -20±10°C	30[(b)] extraction; 40 analysis [c]
Water	Metals (except Hg)	4±2°C in transit, then <2 pH/HNO_3 and ambient	180
	Mercury	4±2°C in transit, then <2 pH/HCl and ambient	90
	Methylmercury	4±2°C in transit, then <2 pH/HCl and ambient	180
	Organic Compounds	4±2°C	7 extraction; 40 to analysis [c]

[(a)] Holding time = 6 months for freeze dried samples.
[(b)] Holding time = 6 months for frozen (-20 °C) sediments and tissues (EPA 1986 and EPA 1989).
[(c)] The 40 day holding time starts the day of extraction for organic analysis.
[(d)] No EPA holding time established; total Hg hold time used as a default.
[(*)] Metals sediment and tissue samples will be refrigerated (4±2°C) or frozen (-20±2°C) by the laboratory until freeze dried

TABLE 9.3.2: Toxicity Sample Holding Times and Preservation

Matrix	Preservation	Holding Time
Sediment	4±2°C dark/airtight	2 weeks is recommended; up to 6 weeks is acceptable; and in some cases up to 8 weeks
Effluent	4±2°C dark/airtight	36 hours from sample collection [a]
SPP/Elutriate	4±2°C dark/airtight	24 hours from preparation

[a] Every effort must be made to initiate the test with an effluent sample on the day of arrival in the laboratory. The holding time should not exceed 36 hours unless a variance is approved by the client.

9.4 CONTROL CHARTS AND PERFORMANCE BASED QUALITY

The TNI and the Navy have withdrawn requirements for control charts for inorganic analytes in favor of performance-based QC data assessment.

9.4.1 Control Charts

Control charts of reference toxicant results obtained from bioassays are used to demonstrate the sensitivity of the stock organism population. Reference toxicant tests are typically conducted concurrently with an aquatic or benthic toxicity test, using organisms from the same batch source. Details of the control charting process and criteria for assessing out of control events are described a procedure MSL-Q-010, Procedures for Control Charting Reference Toxicant Test Results or project planning documents.

When control charts are produced, they are based on normally distributed measurements and short-term variation. Precision is charted over time by calculating a mean recovery for the control sample parameters and then establishing upper and lower warning and control limits. The warning limit is defined as ±2 and the control li samples used for organic parameters are Blank Spikes (BS) and for inorganic parameters results from the analyses of a standard reference material are plotted. A minimum of 20 points are used to set the initial control limits for each parameter.

9.4.2 Performance-Based Quality Control

Performance-based quality control is based on a comparison between *a priori* project or method-specific data quality objectives and the results obtained for each batch of samples. In most cases, both method and project-specific DQOs are evaluated for each batch of samples analyzed. Corrective actions are specified in each analysis method and are followed to ensure that sample data obtained is of high quality and defensible. All issues regarding data quality are discussed in a narrative accompanying sample results. Documentation of the assessment of performance-based DQOs and QC sample results is provided by the use of an analyst checklist on each data package prepared by the analysts. The checklist documents issues that are addressed by completion of the appropriate corrective action during analysis and issues that could not be corrected are documented.

9.5 EQUIPMENT MAINTENANCE AND CALIBRATION

The quality of MSL products is directly related to the validity of the data produced. To produce valid data, equipment must be properly operated, maintained, and calibrated.

Preventive maintenance and primary maintenance of facilities equipment are provided through the PNNL Facilities and Operations Personnel located in Sequim, but located organizationally in Richland, WA.

The MSL maintains a wide variety of research equipment related to the collection and analysis of a variety of parameters (chemical, biological, and physical oceanographic, etc.). This research equipment is maintained to manufacture's specifications through manufacturer service contracts, service calls, factory rehab purchase requisitions, or by qualified personnel. To support the generation of data of known and acceptable quality, the following general guidelines are implemented when applicable:

1. The appropriate and necessary equipment, instruments, and supplies must be available in adequate quantities to perform the proposed work. Spare parts for critical components are maintained to minimize downtime.
2. Measuring and testing equipment is properly handled and stored to maintain accuracy.
3. All equipment involved in the collection and analysis of environmental data is operated, maintained, and calibrated according to approved procedures and specified schedules.
4. Equipment is serviced regularly by qualified individuals, either trained in-house personnel or through service contracts with the manufacturer or an authorized representative. For example, balances are cleaned and calibrated by a PNNL Evaluated Supplier, and analytical instruments have service contracts with manufacturers such as Perkin-Elmer. Most support equipment (e.g., ovens, refrigerators, freezers, hoods) servicing is done internally by PNNL's Facilities and Operations Personnel. When problems arise that cannot be corrected internally, external contractors or manufacturer's representatives are contacted.
5. Equipment that is not operational for any reason must be clearly tagged out to indicate that it is out-of-service
6. Written records of all instrument maintenance, calibration, testing, and inspection are maintained. Maintenance records contain a description of the operation or problem, the remedial action taken (if necessary), date, and the individual responsible.
7. When equipment or instrument maintenance is required, equipment is monitored to ensure correct operation. The responsible analyst monitors analytical instrument operation after maintenance by running a calibration curve and assessing results of standard reference materials (SRM), when applicable.
8. Calibrated equipment is suitably marked to indicate calibration status.
9. Written directions on equipment operation (e.g., operating manual, manufacturer's instruction, and procedures) are maintained with the equipment and are available to personnel using the equipment.
10. Balances are calibrated annually by an approved metrology laboratory and checked daily prior to use by laboratory personnel as prescribed in procedure MSL-C-009, Use and Performance Checks of Balances.
11. Applicable cold-storage facilities are monitored daily as prescribed in procedure MSL-I-026, Use of Laboratory Refrigerators and Freezers.
12. Pipettes are checked quarterly as prescribed in procedure MSL-C-010, Calibration, Verification and Use of Pipettes.

A list of equipment is maintained by the PM, when applicable. The ~~QAO~~ QM maintains and updates a list of equipment used in support of TNI work.

9.5.1 Equipment Calibrations

When applicable, calibrations or performance checks are performed on instruments and support equipment (balances, pipettes, thermometers, etc.) prior to use or at established intervals. Requirements for specific levels and frequency of calibration are described in SOPs or project planning documents. In circumstances, especially during field surveys, where calibration occurs less frequently than described in SOPs or project planning documents, the PM shall notify the client.

Calibration records are kept in the data files and are traceable to date and other applicable parameters (sample runs, standards, etc.). Corrective actions when calibration criteria are not met are described in SOPs or project planning documents.

Whenever data are recorded, the instrument model, serial number (if available), and information on whether a calibration was performed prior to sampling is recorded. If no calibration information is provided with the data, the assumption must be that the instrument was not calibrated immediately prior to use. However, calibration records that indicate the date and results of the previous calibration are acceptable (assuming it is prior to the next recommended calibration date for that instrument) may be referenced.

9.5.2 Preventive Maintenance

Instruments and support equipment are serviced regularly by qualified individuals, either trained in-house personnel or through service contracts with the manufacturer, an authorized representative or other qualified service organization. Written records of all instrument maintenance, calibration, testing, and inspection are maintained. Maintenance records should contain a description of the operation or problem, the remedial action taken (if necessary), date, the individual responsible, and where applicable, documentation of the instrument's return to acceptable use

9.6 INTERNAL QUALITY CONTROL CHECKS

The following are common types of QC analyses implemented by the MSL. It is important to note that measures made for work performed that is not under the TNI standard may be for system monitoring purposes only and are not considered as quantitative measures subject to QC requirements beyond daily calibration verification.

- **Blank Spike (BS)/Laboratory Control Sample (LCS)** - an aliquot of clean matrix (e.g. reagent water) to which known concentrations are added and prepared, treated and analyzed in the same manner as the associated samples. Its purpose is to determine whether the method is within accepted control limits. Blank spikes may be analyzed in duplicate (BSD).
- **Continuing calibration verification sample (CCV)** - A sample of known concentration that is run at the frequency described in the project planning document and/or SOP (typically after every 10 or 20 samples) to ensure that the initial calibration is still valid. The specific project planning document and/or SOP CCV % recovery range for the analysis should be followed. Analysts will attempt to run CCVs such that they bracket the analytical range of the samples run in the analytical batch.
- **Initial calibration verification sample (ICV)** – A sample of known concentration, and of a separate source from the curve is run after the calibration curve to verify instrument control. The specific project planning document and/or SOP ICV % recovery range for the analysis should be followed. For samples that are to be analyzed for the TNI, or when requested by a client, a secondary source ICV shall be run prior to running any samples.
- **Laboratory replicates** - Laboratory replicates consist of splitting a single sample or compositing and splitting two or more samples in the laboratory, and subsequently processed and analyzed as separate samples. Laboratory replicates serve as a measure of the error associated with the analytical process.
- **Matrix Spike (MS)** - an aliquot of a sample to which known concentrations are added and treated and analyzed in the same manner as the associated samples. Its purpose is to determine whether the sample matrix contributes bias to the results.

- **Method Blank (MB)** - an aliquot of clean matrix (e.g. reagent water) prepared, treated and analyzed in the same manner as the associated samples. Its purpose is to determine if method concentrations or interferences are present in the laboratory environment, the reagents, or the apparatus' used that could contribute bias to the results.
- **Standard Reference Material (SRM)** - a material obtained from an independent source, is certified to a known concentration by a recognized authority (e.g., NIST) and is treated and analyzed in the same manner as the associated samples. Its purpose is to determine whether the method is within accepted control limits.

QC samples may also be collected in the field to monitor contamination and to assess sampling error. Common field-related QC samples include

- **Equipment Blanks (EB)** - Equipment blanks are prepared in the field using the freshly decontaminated sampling equipment. De-ionized water is poured over and through the equipment, collected in an identical sampling container, and shipped to the laboratory for processing and analysis. Equipment blanks measure the contamination associated with the entire sampling and analytical process.

- **Field Replicates** - Field replicates are two or more separate samples that have been collected from the same sampling point. Field replicates also serve to measure the error associated with the entire sampling and analytical process, including variation inherent in the sampled media.

- **Reference Samples** - Reference samples are samples for which selected properties are known, generally through historical analysis. Reference samples are used as a benchmark for similar analyses.

- **Split samples** - Split samples are obtained by compositing sample material in the field and dividing the material into separate containers for processing and analysis. Split samples are used to assess the total error associated with sampling and analysis. If split samples are sent to separate laboratories for analysis, inter-laboratory variation may also be obtained.

QC checks are associated with biological toxicity testing (independent recounting of sample, reference toxicity tests, establishment of acceptable water quality measurement ranges) and data processing (proofing or double entry/comparison programs). The specific QC procedures, frequency of performance, and criteria for acceptance for all environmental data collection procedures are defined in SOPs or in the project planning documents.

The immediate monitoring of QC results by analysts allows the data collection process to be continually compared to pre-established acceptance criteria and corrected as necessary. In addition, assessment of QC results is a critical component of the data validation process and is used to interpret the accompanying sample data and to judge its acceptability and usefulness with regard to the project DQOs. QC results are reported with the project data.

10.0 APPROVALS BY EXTERNAL AUTHORITIES

10.1 ACCREDITATION/CERTIFICATIONS

A list of the most current accreditations and accredited methods is maintained by the ~~QAO~~ QM. The MSL's primary TNI accreditation is under the New Jersey Department of Environmental Protection (NJDEP).

Certification is described in procedure MSL-A-013, Laboratory Accreditation and PT Sample Analysis. Certification programs are based on the demonstration of a functional quality program, the existence of planning documents and procedures, the successful analysis of external performance samples at least twice per year for each method, parameter and matric of interest, and in some cases, periodic on-site assessments. The MSL maintains the following documentation to meet these requirements:

Quality Assurance Management Plan (QAMP)
SOPs in the following general areas

- Administration
- Conventional/General Chemistry
- Documentation, Records, and Reports
- Ecological Processes
- Inorganic Chemistry
- Organic Chemistry
- Quality Assurance
- Safety
- Toxicological/Biological Testing
- Water Quality Instrumentation

Training Files
Approved Management Signatures
Signature Log

10.2 OTHER AUTHORITIES

The MSL is inspected semi-annually by the PNNL Institutional Animal Care and Use Committee (IACUC) for compliance with Federal animal welfare regulations that require protocols for all uses of vertebrate animals to be reviewed and approved by the Committee. Approved protocols are also required for animals used in training, animals held as donors for blood and other tissues, breeding stock, and other animals held on site which are not yet assigned to a specific study protocol. Animal use requirements are prescribed in procedure MSL-A-017, Care of Animals.

11.0 PERFORMANCE EVALUATIONS

Analysts performing TNI work are degreed personnel operating analytical instruments on a daily basis. It is the PM's responsibility to ensure analysts supporting non-TNI work have experience and training required by the specific project.

The dedication of analytical personnel to the specific procedures for which they are responsible, their level of training and, daily QC assessments of proficiency through the analysis of blank samples, sample replicates, SRMs, and MSs combine to make the results produced by highly defensible, accurate, precise, and repeatable. The MSL is a specialty laboratory, providing its clients with relatively low detection limits for environmental samples. Daily proficiency is monitored at the bench level, at the level of data assessments performed on sample sets by the analyst and the Data Coordinator (data validation), and at the level of the ~~QAO~~ QM who provides data quality verification.

As part of the TNI accreditation programs, the MSL participates in performance studies at the required frequency for the accredited methods, parameters and matrices as prescribed in procedure MSL-A-013, Accreditation and Performance Testing. Performance Testing samples are purchased from a National Voluntary Laboratory Accreditation Program (NVLAP)-approved vendor. Clients are provided with the results of recent performance studies upon request.

The MSL also participates in inter-laboratory toxicology comparisons whenever offered.

12.0 DATA MANAGEMENT

12.1 DATA REDUCTION

Reduction of raw data shall be accomplished using established techniques. The calculations required for the reduction of data may be performed manually or with the aid of automated data processing systems. In either case, the applicable SOPs for the testing and analysis of samples or the project planning documents will specify the calculations and the mode for raw data processing. If manual processing is to be used for data validation, then the applicable SOP or project planning document will provide the calculation method and the units for reporting derived values. In order to reduce the potential of errors in data transcription the manual transfer of data will be minimized. All calculations performed manually will be checked for accuracy by someone other than the individual who performed the original calculation. Data validation checks shall be documented by the signature and date of the reviewer. Separate documentation is acceptable, provided traceable records are maintained. For automated data reduction methods, the accuracy of calculations will be verified through the use of standards or test case inputs with known resultant values. For TNI projects, all data is reviewed in accordance with procedure MSL-Q-003, Quality Assurance Deliverable Audits.

12.2 DATA REPORTING

Two types of technical reports are produced: Research and development (R&D) reports and data reports. R&D reports are produced from research of a non-standard or non-repetitive nature, data reports are produced from results of standard, repetitive types of analyses. All technical reports go through a formal review process consisting of an author review, technical peer review, editorial or QA review, and a management review. R&D reports must have an editorial review and data reports must have a QA review.

The purpose of the technical peer review is to evaluate the document for technical quality, including scientific validity and logic. This review is performed by senior technical personnel selected for familiarity with the technical discipline of the work being reported. The QA review is conducted by the ~~QAO~~ QM and encompasses accuracy, completeness, adequacy, and conformance to applicable standards and project planning documentation. Editorial review addresses grammatical correctness and consistency of style and format. The management review focuses on scientific validity, logic, conformance to client expectations, and for agreement with policies and procedures. The management reviews are performed by the Laboratory Director or delegate.

The following is a list of data that is typically reported for toxicant results:

- description of test sediment or water; collection, handling, manipulation, storage, and disposal
- description of test organisms; scientific name, age, size (when applicable), life stage, source, and their handling, culturing, and acclimation
- toxicity test method used
- date and time test started and terminated
- percent survival for each test treatment
- percent survival for each test treatment
- control treatment survival
- results of water quality measurements (may be reported as mean, range of measurements, number of times criteria limits were exceeded)

- number of organisms used per test chamber
- number of replicate test chambers per treatment
- summary of statistical endpoints (mortality, growth, LC50, no observed effect concentration [NOEC],)
- gender determinations (when appropriate)
- growth (when appropriate)
- reproduction (when appropriate)
- summaries of biological observations
- summaries of reference toxicant test evaluations
- summary of any problems encountered and corrective actions
- description of any deviations from prescribed laboratory protocols

The following is a list of data that is typically reported for field research results:

- description of study organisms; scientific name, age, size, collection method or other source, and their handling and disposition
- date and times for data collection
- weather and water conditions
- water visibility
- descriptions of sampling equipment (e.g., manufacturer and model number)
- summary of observations
- summary of any problems encountered and corrective actions

The following is a list of data that is typically reported for analytical chemistry results:

- sample receipt date and condition
- date and times for data collection
- the applicable method, matrix, instrument and SOPs
- summary of the results
- summary for DQO results
- summary of any problems encountered and corrective actions

12.3 DATA EVALUATION

Prior to their use, data shall be validated in accordance with project requirements. Validation is defined as the process through which data are accepted or rejected and consists of proofing, verifying, editing, and technical reviewing activities. Data validation requirements are prescribed in procedure MSL-D-004, Data Reporting, Reduction, Backup, and Archiving. Data validation is considered a technical function and should occur prior to the data being audited by the ~~QAO~~ QM.

Data validation occurs at multiple levels as data are collected and processed:

- Individuals recording data during field or laboratory operations are responsible for reviewing their work at the end of the day to ensure that the data are complete and accurate.
- Analysts and instrument users are responsible for monitoring the instrument operation to ensure that instrument has been properly calibrated.
- PMs are responsible for reviewing analytical results and supporting documentation to assess sample holding times and conditions, equipment calibration, and sample integrity. As an additional measure of acceptability, the results of QC samples are compared to the project DQOs.
- Technical personnel are responsible for reviewing the data for scientific reasonableness.

- All manual entries into databases and spreadsheets are verified, either through proofing or by double entry/comparison programs.
- All calculations performed by hand are checked for accuracy.

Data that do not meet the pre-established criteria for acceptance may be flagged (see procedure MSL-D-004*,* Data Reporting, Reduction, Backup, and Archiving), not reported, or reported with an explanation of the limitations, at the discretion of the PM.

12.4 DATA AUDIT PROCESS

Data produced by the MSL for work performed under the TNI standard shall be audited prior to their final release. The reported data are audited, using a process that ensures that the data are complete, accurate, traceable, and defensible. Details of the data auditing process are described in procedure MSL-Q-003, Quality Assurance Deliverable Audits.

Non TNI projects may be audited in accordance with procedure MSL-Q-003, Quality Assurance Deliverable Audits or in accordance with project planning documents.

Data shall be reviewed to ensure that the data are accurate, traceable, defensible, and complete, as compared to the project requirements. The audit procedure is a check that involves comparing selected reported values to the original data. Selection of the reported values to check can either be performed randomly or on a statistical basis. Results of the data audit are documented either on a checklist or in a summary statement. Concerns that can be corrected shall be corrected before the data are released. Deviations are required to be summarized and provided to the client.

12.5 DATA CONFIDENTIALITY

PNNL policy does not allow the release of client data or project-related information to anyone except the client unless expressly directed by the client or an authorized representative. Client confidentiality and proprietary rights are protected whenever requested by marking documents, protecting business sensitive information, sealing records, and/or protecting access on a “need-to-know” basis.

12.6 DATA RELEASE AND EXPORT

Data used for regulatory purposes or for data collection activities that require TNI accreditation will be clearly identified. Non-TNI accredited analytes will be clearly specified and identified as not meeting the TNI standard.

Data are released as electronic files (e.g. Excel, Word, pdf) or in hard copy. Hard copy and electronic files are checked before data are released for consistency and accuracy. This is part of the data audit process. Most hardcopy data is sent to the client via Federal Express, which allows for package tracking and affords a high level of confidence that tampering, does not occur. When data are electronically provided to the client, it is the client’s responsibility to verify that the hard copy matches the electronic file upon receipt. File copies of both formats are signed and dated and kept in the project file. The MSL will assist in resolving any issues that arise during data transmission. Data files will be encrypted upon request, assuming that the encryption programs are either those currently available to PNNL personnel or provided by the client. For confidential data transmissions, the client will be asked to define an acceptable mode

of data transmission that maintains confidentiality. In the past, data has been transmitted as FTP to a secured third party site arranged by the client. Hardcopy data can be footnoted on every page as to their confidentially and evidentiary status. In addition, when required by the client, “need-to-know” cover sheets can be used. A formal procedure has not been developed for these processes because confidential and secure transmission requests to date have been infrequent and very client-specific.

13.0 VERIFICATION ACTIVITIES

To insure the products generated and the services performed meet established standards and client requirements, a systematic approach has been implemented. This approach is graded and intended to provide oversight, assessment, and corrective/verification action for a variety of projects. The goal of the process is to:

- Provide personnel and management accurate technical, business and operational performance information that promotes early identification and resolution of problems that may impact achievement of critical outcomes and objectives.
- Verifies conformance to established requirements.
- Verifies effective conduct of activities to protect the environment and the health and safety of workers and the public.
- Contributes to ongoing improvement in performance

13.1 ASSESSMENTS

Assessments are performed in accordance with the HDI work flow "Integrated Assessments" by personnel and line management to evaluate performance. Assessment methods include, but are not limited to walk through, procedure and program reviews, personnel feedback, and safety, health, and environmental evaluations.

In addition, the ~~QAO~~ QM conducts QA assessments to determine if facilities, equipment, personnel, methods, practices, records and quality control are in conformance to approved planning documents, procedures, regulations, client requirements and PNNL policy. QA assessments are scheduled based on a request from the Director, the definition of critical phase inspections by PMs or clients, and by scheduling by the ~~QAO~~ QM when a new procedure is implemented or significantly revised, when a new study type is initiated, or when data quality reviews indicate technical systems problems. External assessments of suppliers are conducted through the PNNL Environment, Safety, Health and Quality Directorate in Richland, WA and are related to qualifying preferred suppliers.

QA assessments are formal or informal verification activities that are performed in accordance with procedure MSL-Q-002, Quality Assurance Inspections of MSL System and Study Activities and HDI work flow " Integrated Assessments". The purpose of a formal QA assessment is to determine verification with a requirement and includes formal corrective action and follow-up. If the assessment is determined to be informal, the purpose is to determine the status and to report the factual evidence and is not intended to be a verification activity with formal corrective action response, follow-up, etc. Informal assessments are generally requested by management to assess the status of a particular activity.

A schedule of all QA assessments is maintained by the ~~QAO~~ QM. This schedule will include verifications based on client needs, management requests and routine internal verifications (i.e., checking standards logs, sample preparation forms, QC checklists, equipment calibration and maintenance, etc.).

13.2 QA REPORTS TO MANAGEMENT

Biannually, the ~~QAO~~ QM will submit to the Laboratory Director a summary of the past two quarter's QA activities. Subjects to be covered in the biannual QA report are prescribed in procedure

MSL-Q-008, QA Reports to MSL Management, and shall include, but not be limited to, results of assessment activities, results of performance evaluation samples, trends of deficiencies, and other important QA-related issues.

13.3 DEVIATIONS

Each individual engaged in project activities should be alert to problems, deviations from approved SOPs, out-of-control events, or other issues that may require corrective action. The appropriate response is determined by the event. Procedure MSL-A-005, Deviations from Established Requirements provides methods for describes deviations from procedures, planning documents, and client requirements.

All deviations from approved procedures, project planning documents or this QAMP will be documented. Depending on the severity of the deviation, the ~~QAO~~ QM and the PM will determine how the deviation will be addressed and documented (i.e., through use of a Deviation Documentation Form or Quality Problem Report form as prescribed in procedure MSL-A-005, Deviations from Established Requirements). In some cases, the client may be involved in these discussions.

Deviations from project control limits will be documented. In some cases, deviations will be identified in the narrative accompanying the data set or package or in a letter to the client, and the impact of the deviation addressed. The documentation must clearly state the event and the corrective action taken in response, and must be approved by the appropriate management representative. Acceptance of data that exceeds pre-established criteria also must be documented and justified.

Below is a listing of deviation types.

- **Simple Deviation** – A simple deviation is a deviation from project control limits. The situation is documented either in log books, or on project paperwork including the case narrative. It is important to document if the sample integrity or data quality has been adversely affected.
  - **Corrective Action**- Document the situation to client. Look for opportunity to correct the situation.
- **Minor Deviation**- A minor deviation is defined as method or protocol deviation that does not appear to adversely impact the quality of the data. A minor deviation may evolve into a major deviation if an impact on data quality evolves or results.
  - **Corrective Action**- Document either with narration to client or deviation documentation. Determination of a minor deviation will be initiated by either the PM, or ~~QAO~~ QM. The corrective action will be established to assure that the highest quality of data is produced and that all contractual limits are met. It is possible for a minor deviation to result in a major deviation depending upon all circumstances.
- **Major Deviation** - A major deviation is defined as an occurrence or method or protocol deviation with an impact on project data quality or a negative effect on the outcome of a test or analysis.
  - **Corrective Action**- Formal documentation. Major deviation corrective action is tracked to completion, including signatories. The objective is to be able to institute “lessons learned” to improve systems and personnel awareness.

The following are guidelines to resolving deviations:

- All deviations from approved procedures, project planning documents or this QAMP will be documented.
- Issues that affect cost, schedule, or performance of the project will be reported to the PM. The PM will then be responsible for evaluating the overall impact to the project and implementing the necessary corrective actions.
- Deficiencies identified through QA assessment activities will be brought to the attention of the PM. Implementation of corrective action will be the responsibility of the PM.
- When sample integrity is compromised or questionable (e.g., mislabeling, broken or leaking sample containers, improperly preserved samples, expiration of sample holding times), it is the responsibility of the personnel who identify the problem to bring it immediately to the attention of the PM for resolution.
- In the event of an instrument problem, it is the responsibility of the operator to attempt to correct the problem (e.g., recalibrate the instrument). If the problem persists or cannot be identified, the issue should be brought to the attention of the Director for resolution.
- Corrective actions for results outside established DQOs are addressed in applicable SOPs.

13.4 CORRECTIVE ACTION

The need for corrective action may be identified by the technical personnel during the course of their work and through assessments or data audits. It is the responsibility of the analyst to monitor QC sample results, and ensure established criteria in method procedures or project specific criteria are met.

Each individual performing laboratory or data processing activities will be responsible for notifying the PM of any circumstance that could affect the quality or integrity of the data. It is the PM's responsibility to ensure completion of the resulting corrective action by the expected completion date, and to request independent verification (when required).

Corrective actions may include, but are not limited to, review of data and calculations, flagging and/or qualification of suspect data (see procedure MSL-D-004*,* Data Reporting, Reduction, Backup, and Archiving) or re-extraction and/or re-analysis of individual or entire batches of samples. In addition, individual analytical SOPs may contain appropriate corrective actions for various routine problems. The form of documentation is project specific, but at a minimum, the QC data that are outside the established criteria shall be flagged.

When there has been an impact on data, the PM shall ensure that there is a cross reference in the raw data that indicates there is a documented deviation and corrective action.

13.5 QUALITY IMPROVEMENT

Quality improvement is a critical aspect of the Self-Assessment Program and involves both corrective action to identified deviations and continuous improvement processes. The corrective action process involves determining, implementing, approving, and verifying the appropriate remedial action. The continuous improvement process involves determining and prioritizing improvement areas, implementing improvement action and documenting the disposition of each action.

13.6 ASSESSMENT ACTIVITIES

For all assessment activities, a system of notification and verification of corrective action is in place. An assessment report is prepared and submitted to the appropriate PM. The PM reviews the assessment results to determine overall impact and risk and then determines corrective action and prioritizes the actions. The PM assigns the corrective actions to individuals. The PM ensures that the corrective action is tracked to completion and as part of completion, documentation is included that describes the justification for completion of the corrective action. Issues that in the PM's judgment require significant corrective action should be scheduled for verification of that corrective action at a subsequent assessment.

Issues that in the PM's judgment require process improvement instead of, or in addition to, corrective action, are identified as such and any improvement actions are implemented and documented.

13.7 CLIENT COMPLAINTS

The process for tracking and addressing client complaints is the following:

- The PM is the point of contact for any client complaints.
- The client contacts the PM to discuss the concern. The contact is generally made by e-mail or telephone, although a formal written follow up letter may be sent as well.
- The PM will inform the Director of the issue(s). Concerns will be responded to in writing. A determination will be made of an appropriate response (e.g., data review and re-calculation, sample re-analysis, re-sampling and analysis, revision of deliverables), which will be discussed with the client prior to finalizing in a response letter.

A tracking system for client complaints has not been developed because client complaints are rare. If the frequency of client complaints increases (>2/year), a formal tracking system may be developed. The ~~QAO~~ QM will monitor the number of annual client complaints.

APPENDIX A: List of Acronyms

ASTM	American Society for Testing Materials
BS/BSD	Blanks Spike / Blank Spike Duplicate (aka LCS)
BIOSIS	A bibliographic database service, with abstracts and citation indexing
CCV	Continuing Calibration Verification
CDRR	Chemical Disposal Recycle Request
CFR	Code of Federal Regulations
CMS	Chemical Management System
CRM	Certified Reference Material
CSM	Cognizant Space Manager
CoC	Chain of Custody
CV	Coefficient of Variation
DO	Dissolved Oxygen
DOE	Department of Energy
DQO	Data Quality Objective
EED	Energy and Environment Directorate
EPA	Environmental Protection Agency
ES&H	Environment, Safety, and Health
ESE	Environmental and Safety Engineer
FDA	Food and Drug Administration
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
FSR	Field Services Representative
GLP	Good Laboratory Practices
HDI	”How Do I”
HPLC	High Performance Liquid Chromatography
Hg	Mercury
IACUC	Institutional Animal Care and Use Committee
ICV	Initial Calibration Verification
ID	Identification
IOPS	Integrated Operations System
LCS	Laboratory Control Sample
MB	Method Blank
MDL	Method Detection Limit
MSL	Marine Science Laboratory
MS/MSD	Matrix Spike / Matrix Spike Duplicate
MRO	Marine Research Operations
NIST	National Institute of Standards and Technology
NJDEP	New Jersey Department of Environmental Protection
NOAA	National Oceanic and Atmospheric Administration
NOEC	No Observed Effect Concentration
NRCC	National Research Council of Canada
NVLAP	National Voluntary Laboratory Accreditation Program
PARCCS	Precision, Accuracy, Representativeness, Comparability, completeness and Sensitivity
PM	Project Manager
PNNL	Pacific Northwest National Laboratory
PT	Performance Test
PVC	Polyvinyl Chloride
QA	Quality Assurance

QAMP	Quality Assurance Management Plan
~~QAO~~ QM	Quality ~~Assurance Officer~~ Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
QPR	Quality Problem Report
R&D	Research and Development
RFID	Radio Frequency Identification
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
RSO	Radiation Safety Officer
SOP	Standard Operating Procedure
SOW	Statement of Work
SRM	Standard Reference Material
TNI	The NELAC Institute
TSCA	Toxic Substances Control Act
U.S.	United States
UV	Ultraviolet
WA-DOE	State of Washington, Department of Ecology

Appendix 1.A.D

ACZ Laboratories QAP

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Quality Assurance Plan v.24

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1 INTRODUCTION

ACZ Laboratories, Inc. is an environmental testing laboratory that provides data to clients primarily for regulatory purposes. Samples are analyzed for compliance with federal programs including the Resource Conservation Recovery Act (RCRA), Safe Drinking Water Act (SDWA), and Clean Water Act (CWA). Environmental compliance and management decisions are based on the analytical data provided, which are critical to the expenditure of large amounts of money; are important to public health safety; are important in evaluating, monitoring, and protecting the environment; and may be essential in litigation. ACZ's data must be of known and documented quality to support sound decisions and withstand adversarial inquiry.

An effective Quality Management System is the cornerstone of the generation of reliable analytical data. ACZ's Quality Assurance Plan (QAP) outlines the quality assurance and quality control objectives, policies, and procedures determined to be necessary to meet the requirements of the EPA, federal government entities, state agencies, other regulatory authorities, and our clients. This document provides the framework to ensure all ACZ employees have sufficient knowledge and training to perform their job responsibilities in a manner that assures all data reported to ACZ's clients is accurate, reliable, technically sound, legally defensible, and impartial.

For data to be useful, it must be of known and documented quality. The word "quality" has many different meanings, but for the purposes of environmental testing activities can be stated simply as "conformance to requirements." Conforming to requirements allows objective measurements to be applied, rather than subjective opinions, to determine when work is of good quality. *Quality control* refers to all activities that measure conformance (i.e. good quality) of the data. It requires action(s) to be taken and is typically included as part of the procedure. *Quality assurance* provides the records of the results obtained from the required action(s) and refers to the ability of the laboratory to demonstrate or prove to an outside party that the quality of the data is what the laboratory states it is. Quality assurance relies heavily on documentation, and to be effective, the documentation must: (1) assure the quality control procedures are being implemented as required; (2) assure the reported data reflect the sample as it was received, meaning sample mix-up was avoided, the sample was properly preserved prior to analysis, etc.; (3) facilitate traceability of an analytical result; and (4) be subjected to reasonable precautions to protect data from loss, damage, theft, and internal or external tampering.

Quality Policy Statement: To maintain an effective QA program, continually improve the quality of our environmental testing services, and consistently provide clients with technically sound and legally defensible data in a timely manner, the management of ACZ recognizes the importance of its commitment to:

- Ensuring good professional practice by well-trained and qualified employees with the necessary experience and skills to carry out their organizational functions and to meet or exceed ACZ's standards for the quality and reliability of its testing services.
- Ensuring the data provided to our clients is of known and documented quality, and is accurate and impartial.
- Ensuring that all quality assurance and quality control policies and procedures are communicated to and understood by all employees, and that they are implemented by all employees in their work.
- Ensuring that all aspects of the business operations are conducted in a manner that adheres to the TNI Standards and all of ACZ's policies and procedures documented in the QAP, SOPs, emails, memos, etc.
- Upholding the spirit and intent of ACZ's Data Integrity Program and implementing the requirements of the program.

2 QUALITY SYSTEM OBJECTIVES & COMPONENTS

ACZ's QAP provides a framework that guides all technical staff and administrative personnel. The information presented is necessary to ensure all employees perform their duties in a manner that allows the company to achieve its objectives, thereby ensuring the precision, accuracy, completeness, and consistency of the analytical data reported to our clients. This framework is referred to as the Quality System. The Quality System encompasses every documented quality assurance (QA) and quality control (QC) policy and procedure and guides all business functions and laboratory operations by specifying standardized protocols to control both the short-term and long-term activities that influence the quality and defensibility of our testing services.

The Quality System is designed to be appropriate to the type, range, and volume of the environmental testing undertaken. The Quality System is not a static entity and must function in a manner that allows for continuous evolution of all aspects of ACZ's business when improvements have been identified and have been determined to be necessary or beneficial. ACZ management recognizes that the staff is comprised of people who possess varied experience and knowledge and can contribute valuable insight and suggestions regarding these improvements. All employees are encouraged to be involved in this process. The following six (6) key elements form the foundation of ACZ's Quality System:

- Documents & Records
- SOPs
- Training
- Audits
- Corrective Actions
- Management Review of the Quality System

2.1 Documents & Records

The entire history of any sample must be readily understood through the associated documentation. To this extent, a formal and systematic control of documents and records is necessary for accurately reconstructing all events pertaining to any sample and for guaranteeing the quality and defensibility of the data. All information relating to the laboratory facility's equipment, analytical test methods, and related laboratory activities (such as sample receipt, sample preparation, data verification and data reporting) must be documented, and all records, including those pertaining to calibration and test equipment, certificates and reports, must be maintained. Documents and records must be safely stored (protected against fire, theft, loss, deterioration, and vermin), and must be held secure and in confidence to the client for a minimum of 10 years. Refer to §10 for details regarding the storage and control of ACZ's documents and records.

2.1.1 Documents

All official documents are reviewed for accuracy, approved for release by authorized personnel, and distributed through ACZ's LabWeb intranet. LabWeb is a computerized document control system based in HTML that can be accessed from any network computer within the facility. For printed documents to be considered controlled, the header must be in sync with the header on LabWeb. Obsolete or invalid SOPs retained for knowledge preservation or other reasons must be clearly marked to identify their purpose.

All documents are categorized by department and are assigned a unique document ID that is displayed in either the header or footer section. The ID nomenclature starts with either SOP (procedure) or FRM (form), followed by the 2-letter department code, the unique document number, the month and year of issue, and the revision. The effective date for any SOP or other document is included on the title page and header section of each subsequent page and indicates the implementation date.

The QA Officer has full responsibility of the Document Control System. Only employees with the appropriate computer access (IT and QA staff) can upload documents to LabWeb. A new or revised document is reviewed, and following approval, the document control number is updated and the SOP or form is uploaded to Labweb. When a new version of an SOP is added to Labweb, the previous version is removed from the active list, date-stamped and electronically archived in a designated location on the

network. This automatic process guarantees that ACZ can retrieve the version that was in effect at any given time.

2.1.2 Records

A record is any information or data on a particular subject that is collected and preserved. Records are produced on a daily basis and contain original, factual information from an activity or study. For ACZ's purpose, this information may be recorded by the following means: LIMS database, logbooks, raw instrument data, worksheets, and notes (or exact copies thereof) that are necessary for the reconstruction and evaluation of the report of the activity or study. The record management system provides control of records for data reduction, validation, reporting and storage, and also provides control of all laboratory notebooks and logbooks. The system must allow for historical reconstruction of all laboratory activities that produced analytical data, must document the identity of personnel involved in sample receipt, preparation, calibration, and testing; and must facilitate the retrieval of all working files and archived records for inspection and verification purposes. At a minimum, the following criteria for records must be met:

1) Instrument logbooks must be kept up-to-date on a daily basis. Document all relevant activities when the event occurs or as soon as practical thereafter.

2) Dilution factors and observations must be recorded at the time they are made, and notes regarding samples or analyses must be identifiable to the specific task.

3) A detailed description of any departure from a documented procedure, and the reason for the departure, must be provided at the time it is performed.

4) All generated data must be recorded either by an automated data collection system or must be recorded directly, promptly and legibly in permanent ink (blue or black is preferred).

5) Erroneous entries (hard copy or electronic) cannot be destroyed by methods such as erasures, overwritten files or markings. Refer to §16 for ACZ's error correction protocol.

6) Any changes to hard copy records must be clearly initialed and dated by the responsible staff. Changes to electronic records must also be traceable to the individual who made the change, and the reason for the change must be provided.

7) Records generated by computers must have hard copy or write-protected backup copies.

2.2 Standard Operating Procedures

A documented procedure is required for all phases of ACZ's business operations, from sample log-in through sample disposal. A Standard Operating Procedure (SOP) is a written document that details the manner in which an operation, analysis, or action is performed and thoroughly prescribes the techniques and procedures, which are the accepted process for performing certain routine or repetitive tasks. Analytical SOPs must be written with adequate detail to allow someone similarly qualified, other than the analysts who routinely performs the procedure, to reproduce the procedure used to generate the test result. To the extent possible, administrative SOPs [non-technical] must include specific requirements pertaining to the process; however, the procedure itself may be a more general description so as to lend a degree of necessary flexibility to account for client requests and other circumstances which may be outside of ACZ's control.

Proposed revisions to any test SOP shall be submitted by the pertinent department supervisor (exceptions may be granted on a case by case basis) and be reviewed and approved by QA prior to implementation. Changes to provide additional clarification, correct typographical errors, etc. do not require formal approval and/or training. Analytical SOPs must be reviewed annually using the SOP Review Form (FRMQA035), and Administrative SOPs must be reviewed regularly and revised if necessary to ensure the information is accurate and reflects current practice. Documenting changes in the controlled copy of any SOP is prohibited. Refer to §10.5.1 for additional information on SOPs.

SOPs are proprietary documents and ACZ does not distribute them freely. Any copy sent electronically or otherwise to an outside party is considered uncontrolled, and the recipient understands that additional changes can be made without prior notification. Excluding method development, the use of uncontrolled copies of SOPs is not permitted.

Unless the reference method is followed exactly and contains sufficient detail to ensure consistent application, an SOP must be developed before a new procedure, application, or instrument can be implemented. The introduction of a new method must be a planned activity directed by the Production Manager, assigned to the appropriate technical director(s), and overseen by QA staff. Exceptions may be made when the client provides specific procedural instructions. In this event, the client's instructions must be followed exactly and appended to ACZ's test report package. Exceptions are primarily related to the preparation of solid materials for analytical testing (refer to SOPAD043 for additional details). An SOP template (SOPAD025) may be obtained from QA. If a client requests a procedure for which there is no published method or existing SOP, ACZ will utilize the process described in the SOP *Client Service Policies and Procedures* (SOPAD043). Analytical SOPs are written in accordance with the TNI Standards and must include or reference the following items, where applicable:

1) identification of the test method
2) summary, scope & application of the test method, including matrices & parameters to be analyzed
3) references, including documents provided by instrument / equipment manufacturer
4) sample collection, preservation, & storage
5) equipment & supplies
6) reagents & standards, including storage conditions & shelf-life for each
7) safety
8) interferences
9) complete procedure, including details and acceptance criteria for initial & continuing calibration
10) data review & assessment, including protocols for handling out-of-control or unacceptable data
11) quality control, including acceptance criteria & corrective action for handling failed quality control
12) calculation equations (dilution factors, RPD, % recovery, etc.) & calibration formulas
13) method detection limit & quantitation limit
14) method performance, including Demonstration of Capability and Method Detection Limit procedures
15) pollution prevention & waste management
16) definitions
17) tables, diagrams, flowcharts

2.3 Training

It is the responsibility of ACZ's management to ensure the competence of all employees who perform environmental tests and other specific duties, operate equipment or instrumentation, give opinions and interpretations, evaluate results, and sign test reports. Additionally, ACZ management is responsible for formulating the goals and policies with respect to the necessary education, training, and skills of all personnel and for providing training that is relevant to the company's present and anticipated tasks.

Employees must possess the appropriate combination of education, experience, and skills to adequately demonstrate a specific knowledge of their particular functions and to carry out those functions in a manner that meets ACZ's standards and expectations. Additionally, each staff member must demonstrate an understanding of laboratory operations, test methods, related quality assurance and quality control procedures, and management of records and documents to the extent necessary to successfully perform their job duties.

All full-time and part-time personnel must complete a formal training process for Safety, Ethics, Quality Assurance / Quality Control, Quarantined Materials, and Sexual Harassment on the first day of hire and are subsequently responsible for complying with all requirements that pertain to their job duties. For all technical staff, training for analytical procedures must be completed prior to independent generation of client data. In general, any staff member who is undergoing training must be provided with appropriate supervision. It is the responsibility of each supervisor or manager to ensure personnel within his or her department are supervised, competent, and working in accordance with ACZ's Quality System.

2.3.1 Safety Training

Safety training is scheduled with ACZ's Chemical Hygiene Officer (CHO) and includes viewing a video of general laboratory safety, a complete review of ACZ's Chemical Hygiene Plan, and a building tour to identify the location of Safety Data Sheets, emergency showers, eye wash stations, and emergency exits. Following completion of the training, the employee takes an exam, which allows the CHO to evaluate their understanding of the material covered.

2.3.2 Data Integrity Training

ACZ is committed to fostering and enforcing an ethically sound work environment that encourages the conscientious production of accurate, technically sound, and legally defensible data. Data integrity training is required for all full-time and part-time employees (permanent or temporary) as described in ACZ's SOP *Data Integrity Principles & Policies* (SOPAD039). Initial training provides an orientation of ACZ's Ethics program, ACZ's Code of Conduct, Code of Ethics, and zero-tolerance policy. Each new employee is introduced to the company's Ombudsman. Data integrity training is provided for all current employees on an annual basis. At a minimum, employees must review ACZ's Data Integrity Principles & Policies (SOPAD039) and provide documented testimony indicating they have read, understood, & agree to adhere to them.

2.3.3 QA Training

All full-time and part-time employees attend an initial orientation session which is based on the most current version of ACZ's Quality Assurance Plan [QAP] and focuses on the relationship between quality control, quality assurance, environmental testing, and environmental monitoring.

2.3.4 Sexual Harassment Training

Sexual Harassment training is required for each new employee and includes viewing a video that demonstrates the identification, reporting, and remediation of harassment issues in the work place. Any complaints of sexual harassment must be brought to the attention of ACZ's Presidents as soon as possible.

2.3.5 Technical personnel must be thoroughly trained in the analytical techniques and operating principles for all pertinent method procedures. Under no circumstances may any analyst independently generate or review client data for a test procedure before completing the required training and receiving the explicit approval of the technical director overseeing the analysis. §5 provides details of ACZ's technical training program.

2.3.6 Employees may be authorized to perform AREV or SREV for a procedure by the pertinent department supervisor or the QAO. Authorization indicates the employee has been trained on applicable QC requirements, corrective actions, and data qualification. AREV & SREV shall be performed in accordance with effective version of the associated test SOP. Authorization is tracked in an excel spreadsheet located on a network drive. Computer permissions are configured so that all employees may view the spreadsheet but only QA staff may edit it.

2.3.7 Training is required for all employees whose activities are affected by any procedural change(s) to an SOP and is considered to be complete once each employee has submitted documentation attesting they have read, understand, and agree to follow the revised policy.

2.3.8 ACZ recognizes the benefit of continuing education and encourages employee participation in advanced training courses, seminars, and professional organizations and meetings.

2.4 Audits

The purpose of an audit is to verify conformance to documented Quality Assurance and Quality Control policies and procedures, and to identify discrepancies when they exist. In the latter case, any problems shall be addressed and resolved in an appropriate manner to assure the Quality System is continuously improved on all levels.

2.4.1 External Audits

External audits are conducted to ascertain compliance with rules, regulations, and additional criteria for certification, and will have a higher degree of formality than internal audits. Where records are required, compliance will be critically evaluated. Issues of non-compliance identified in previous audits are usually reviewed to verify the laboratory has remediated them effectively. The ease with which important records and information can be retrieved is a criterion for judgment of the management practices of a laboratory and may dictate the depth of the audit. Individual state agencies, laboratory Accrediting Bodies, and current and potential clients routinely audit ACZ.

The on-site assessment is generally a two to four day process during which the regulating agency conducts an entrance interview and tours the facility before performing an in-depth review of documents, workgroups, reports, electronic data files, etc. A critical aspect of the on-site assessment is review and verification of bench-level documentation and analyst interviews to determine actual laboratory practices. ACZ's policy is to always have QA personnel present during an interview. If necessary, the President, may attend the interview. An exit interview is conducted upon completion of all on-site assessments, during which observations and findings are reviewed. The agency will submit a final report to ACZ, generally within 30 days, detailing all pertinent findings and recommendations.

Upon receipt and review of the agency's report, the QA department will meet with department managers to develop a corrective action plan, which must be submitted to the agency by the date indicated in their report. Each finding or group of similar findings is addressed as a major corrective action as described in §2.5.2. Employees shall not make changes to any laboratory or other practice based on comments or opinions expressed by the regulating agency during an interview or any other stage of the on-site assessment without first obtaining approval from QA. ACZ will revise policies and procedures as necessary as part of the major corrective action process.

2.4.2 Internal Audits

ACZ is responsible for the quality of its data and must take reasonable efforts to assure itself and all interested parties confidence can be placed in it. ACZ shall conduct internal audits of its activities to verify compliance with the Quality System. It is the responsibility of the QA Officer (QAO) to plan, direct, and organize internal audits; however, a trained and qualified individual, independent from the area or system being audited, may be designated by the QAO to conduct an internal audit. The area of activity audited, the audit findings, and subsequent corrective actions shall be documented.

The internal audit program shall address all elements of the management system. At least one test method shall be audited annually for each analytical laboratory division. Method audits encompass both qualitative evaluation of the operational details of the QA program and quantitative evaluation of the accuracy of data generated by the laboratory staff. Test method audits include step by step witnessing of the procedure. Laboratory Divisions:

- Wet Chemistry (Prep and Analytical)
- Metals (Instrument & Prep)
- Soils
- Radiochemistry (Prep and Analytical)
- Organics (Prep and Analytical)

When audit findings cast doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test or calibration results, the laboratory shall take timely corrective action, and shall notify
customers in writing if investigations show that the laboratory results may have been meaningfully affected. Client contact should be initiated within two months of discovering the error(s). If data impact

assessment cannot be completed in this timeframe management shall set a deadline commensurate to the demands of the assessment.

More frequent internal audits may be scheduled depending on the following criteria:

- Number and type of corrective actions filed for a method or activity
- Client complaints
- Continued failure to achieve acceptable results for a Proficiency Testing sample
- Findings from an external audit
- Request from management

All findings from internal audits are directed through ACZ's corrective action system. Each finding is assigned a corrective action number (similar findings may be combined). A general description of the process is as follows:

1) Findings and observations are summarized in a report.

2) The report is distributed to the department supervisor, Production Manager, and Presidents.

3) The supervisor reviews the report and composes a plan of corrective action (POC) and projected completion dates for each finding. The POC should be proportional to the finding and the projected completion date commensurate with the demands of the tasks required for the corrective action.

4) The supervisor submits the plan of corrective action to the QAO or designee for review and approval.

5) The QAO or designee reviews the plan of corrective action for each internal audit finding. Once the plan of corrective action is accepted, a major corrective action number is assigned to each planned corrective action or group of similar corrective actions.

6) The supervisor negotiates the corrective action and submits a Corrective Action Report (FRMQA001) for each major corrective action number to the QA department for final review.

7) Once all corrective actions associated with the internal audit have been completed and approved, the internal audit process is complete.

An in-depth review will be conducted if there is any evidence of inappropriate actions or vulnerabilities related to data integrity. This review shall be handled in a confidential manner until a follow up evaluation, full investigation, or other appropriate actions have been completed and the issue(s) clarified. Refer to ACZ's SOP *Data Integrity Policies & Procedures* (SOPAD039).

2.4.3 Proficiency Testing (PT) Program

ACZ is required to participate in a formal Proficiency Testing Program at the frequency stipulated by regulating agencies. These "performance evaluations" are facilitated through the introduction of blind samples, purchased from approved vendors. ACZ analyzes PT samples for most accredited parameters twice in a calendar year, with each study being approximately six (6) months apart. These tests are matrix, technology, and analyte specific, and provide useful information regarding the accuracy of the analytical data being produced. At a minimum, ACZ participates in the Water Supply (WS) study for SDWA, the Water Pollution (WP) study for CWA, a Soil study for RCRA, and a Radiochemistry PT study for Drinking Water. Refer to SOPAD011 for additional details.

2.5 Corrective Action

Corrective action shall be performed when any aspect of ACZ's testing and/or calibration work, or the results of this work, do not conform to established procedures or the agreed requirements of the customer. Corrective actions are a fundamental element of ACZ's QA Program, as a successful Quality System requires the identification of

deficiencies and depends on the development, implementation, and documentation of effective contingency plans and resolutions to effectively remediate the deficiencies. Corrective actions are classified as minor, major, or technical.

2.5.1 Minor Corrective Action

Minor corrective actions address problems or issues isolated to a specific data set or group of data sets that do not meaningfully impact reports *already* issued to clients. The minor corrective action report (FRMQA001) allows for complete documentation of any temporary deviation from the SOP or other protocol. The employee who initiates the corrective action will complete Section 1 of the report. Documentation must be accurate and must provide a complete detailed explanation of the situation for future reference. The need to qualify data shall be critically assessed and appropriately addressed. The department supervisor should always be informed of the need for a minor corrective action and may provide additional information in the appropriate section. The project manager may also provide additional information in the appropriate section if necessary. QA does not need to close a minor corrective action; however, the employee may review the report with QA personnel and request their signature in the appropriate section. Minor corrective actions do not require follow-up.

Complete documentation may be provided either on the workgroup bench sheet or on the data review checklist in lieu of using FRMQA001. Use FRMQA001 if the deviation applies to many workgroups and attach a copy of the completed form to each workgroup before the workgroup is scanned. If the report is generated after the workgroups have been scanned, then the workgroup must be retrieved and rescanned with the report included as part of the data package. In this case, a note is made on the front page of the workgroup package indicating the reason the workgroup was rescanned (i.e. “CAR attached, WG rescanned”).

2.5.2 Major Corrective Action

Major corrective actions address problems which are systematic or meaningfully impact reports which have been issued to clients. It is the responsibility of the QAO to notify laboratory management in writing of departures from the Quality System, and it is the responsibility of the laboratory management to ensure remediation is completed by the assigned due date or to negotiate an extended deadline.

A major corrective action is initiated whenever a system failure has been identified or whenever an audit finding or other circumstance casts doubt on the correctness or validity of the analytical results. The client must be notified in writing if their work is significantly affected. The QA department will work with the Project Manager to determine if a revised report must be issued to the client. See ACZ’s SOP *Client Service Policies and Procedure* (SOPAD043) for details. A major corrective action may also be initiated when the need for preventive action has been identified (refer to §2.5.4).

Only QA department personnel may open and close a major corrective action. When opened, the corrective action shall be assigned a unique tracking number (referred to as the CAR number) to ensure that ACZ maintains a complete and accessible record of all Quality System deviations or failures, root cause determinations and subsequent resolutions, and preventive actions. A remediation deadline shall be assigned for all major corrective actions.

Examples of circumstances requiring a major corrective action include, but are not limited to:

- Contamination trends as indicated by blanks routinely above acceptable levels
- Spikes, surrogates and lab control samples continually outside acceptance limits
-
- “Not Acceptable” Proficiency Testing results
- Findings from internal or external audits
- Discrepancies between what was reported to clients and what should have been reported to clients due to equation errors or incorrect LIMS configuration.

- Hold times or deadlines routinely missed
- Evidence of insufficient or inadequate training

Following initiation, the procedure for a major corrective action proceeds to an investigation by the assigned individual to determine the root cause of the problem and identify possible resolutions to rectify the problem. The action(s) most likely to eliminate the problem and prevent recurrence of the problem must be selected, documented and implemented, and pertinent staff members must be trained, if necessary. Changes resulting from the corrective action will be monitored, if necessary, to ensure the resolution(s) are effective. A general outline of the procedure is as follows:

1) Initiation: Any employee may initiate a corrective action by notifying QA. The department manager should be notified first so that they can assess the need for a major corrective action. If determined to be necessary, QA personnel will open a corrective action, assign a unique tracking number, and a deadline for remediation. Deadlines shall be assigned based on the anticipated demands of remediation and potential threat posed to data integrity.

2) Assignment: QA assigns the corrective action to the person(s) responsible for problem characterization, Root Cause Analysis (RCA), Data Impact Assessment (DIA), corrective (including preventive) actions. Sections 1 and 2 of FRMQA001 shall be completed by the assignee(s).

3) Immediate action shall be taken to eliminate propagation of errors. Stopgap measures may be employed, including but not limited to: subcontracting analyses, imposing a moratorium on data reporting, manual data transformation. Immediate action shall be to a degree commensurate with the magnitude and risk of the problem.

4) Investigation and Action: Must be completed by the assigned deadline. Deadline extensions shall be negotiated with the QA department.

    a. The assigned individual(s) launch an investigation of the problem. There are three major components of the investigation
        1) Characterization of the problem: A thorough, but succinct, description of the problem must be composed. Whenever possible, this includes determination of the exact timeframe during which the error was present and what workgroups and samples were affected.
        2) Root Cause Analysis (RCA): Focuses on establishing the sequence of events or causal chain leading up to the problem, identifying contributing factors and elucidating relationships between them, and determining where intervention could be reasonably implemented to change performance and prevent an undesirable outcome. The depth of the RCA shall be commensurate with the risk and magnitude of the problem.
        3) Data Impact Assessment (DIA): Once the problem has been fully characterized, it shall be evaluated to determine whether client data may have been significantly impacted by the error. The DIA shall be commensurate with the risk and magnitude of the problem.

    b. A resolution to correct the problem and prevent its recurrence must be determined & implemented. Resolution may be done solely by the person(s) who investigated the root cause or it may require input from one or more additional departments.

        1) Conduct additional training if necessary. Training must be documented using the appropriate form and must include a description provided by the person who conducts the training. All trainees are required to sign and date the form to acknowledge he/she has received training, understands the change(s) and agrees to adhere to any change(s) in a policy or procedure.
        2) Revise SOP(s) as necessary. Proposed revisions must be approved by QA prior to implementation.
        3) Configure or enhance automated systems (e.g. LIMS) to correct problems or support preventive measures.

4) Correct data in ACZ's LIMS as deemed necessary by Technical Directors, QA Staff, & Project Managers.
5) Perform additional measures (e.g. instrument or support equipment purchase, etc.) as necessary.
6) Document implementation dates for each corrective action.
7) Attach or reference all supporting documentation in the corrective action report.

5) Project Manager Review: If necessary, the PM will determine whether affected data will be accepted or rejected, contact the client, and issue revised reports accordingly. Project Manager review may not be required for every major corrective action.

6) QA reviews the corrective action. If satisfactory, the corrective action is approved and closed.

7) If deemed necessary, QA conducts follow-up. Follow-up is scheduled after sufficient time has elapsed to observe the efficacy of the corrective action and may need to be done multiple times. If the corrective action is determined to be ineffective, a new major corrective action will be initiated and the process repeated. QA follow-up may be documented on the corrective action report or the CAR spreadsheet located on ACZ's network.

2.5.3 Technical Corrective Actions

Technical corrective actions apply to departures or deviations from the quality control parameters stated in individual test SOPs. Each test SOP must include all required quality control that applies to the procedure (as stipulated by the method and other regulatory agencies) as well as the performance frequency, acceptance criteria and corrective action for handling failed quality control measurements. Each SOP must describe the procedures to be followed for reviewing and assessing data, including corrective action for handling out-of-control or unacceptable data. The required protocol for technical corrective actions is summarized below.

1) Identify the individual responsible for assessing each nonconformance and initiating or recommending corrective action [analyst who performs AREV]

2) Define how the analyst must treat data if associated quality control measurements are unacceptable [section 12 of SOP]

3) Specify how non-conformance and subsequent corrective actions are to be documented [data review checklist]

4) Specify how management reviews the corrective actions [reviewed during SREV]

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control then the corrective action described in the SOP must be performed. Alternatively, report data with the appropriate qualifier if reprocessing and reanalysis is not possible. The qualifier must be assigned to any sample associated with the failed quality control measure. A current list of all extended qualifiers is available in the LIMS database and may be accessed by all employees.

2.5.4 Preventive Action

Preventive action is a pro-active process to identify opportunities for improvement rather than reacting to the identification of problems or complaints. Needed improvements and *potential* source(s) of any nonconformance, either technical or concerning the Quality System, must be identified and addressed. Examples of preventive action include but are not limited to: maintaining a cross-trained staff; maintaining a supply of spare consumable parts; monitoring the performance of support equipment; performing routine maintenance on instruments; maintaining an adequate supply of standards/reagents; ordering supplies before running out; completing log-in review in a timely manner; ensuring ACZ can perform work before

samples are accepted; correcting quotes before samples are logged in; and analyzing samples by the appropriate method.

2.6 Management Review of the Quality System

At least once per calendar year, ACZ's management conducts a review of its Quality System and all activities related to its environmental testing services to ensure their continuing suitability and effectiveness, and to introduce necessary changes or improvements. At a minimum, the review must take the following into account:

- Suitability of policies and procedures.
- Manager and supervisor reports
- Review of internal audits
- Status, review, and discussion of major corrective actions
- Review of recent external audits
- Results of recent PT studies and corrective actions initiated / completed
- Changes in the volume and type of work undertaken
- Customer feedback
- Complaints
- Recommendations for improvement
- Status of state certifications
- Ethics and Data Integrity
- Other pertinent issues
- Resources and training.

2.6.1 Department Reports

Each department manager completes a Department Report (FRMQA041 or FRMQA042) prior to the Management Review meeting. Each item on the report is to be evaluated as it pertains to the individual department.

2.6.2 Management Review Report

The completed department reports are submitted to ACZ's Presidents. At a time specified by the Presidents management meets as a group to discuss the reports. All reviews shall be appropriately documented.

2.6.3 Customer Feedback

ACZ solicits customer feedback on an annual basis through the use of a client survey distributed and received via email. The survey asks for feedback regarding customer service, data quality, staff, value, timeliness, and laboratory standing compared to other labs. Feedback is compiled by the CEO and discussed during management review of the quality system.

2.6.4 When a finding is identified through the management review process ACZ's corrective action protocol shall be initiated.

3 ETHICAL AND LEGAL RESPONSIBILITY

All ACZ employees have an ethical and legal responsibility to produce data that is accurate, reliable, and legally defensible. ACZ's proactive program for the prevention and detection of improper, unethical, or illegal actions includes an Ombudsman who acts as a neutral party and serves as a confidential liaison between ACZ employees and management regarding questions, problems, complaints, suggestions, or ethical dilemmas.

Initial employee orientation includes ACZ's Code of Conduct, Code of Ethics, and zero-tolerance policy. Employees are informed of the processes in place to ensure employees are free from undue internal or external commercial, financial, or other pressures that may adversely affect the quality of an employee's work, endanger the trust in the independence of ACZ's judgment, or compromise the integrity of ACZ's environmental testing activities. A more detailed description of all aspects of the ethics program is provided in ACZ's SOP *Data Integrity Principles & Policies* (SOPAD039).

ACZ will not tolerate unethical or improper activities or behavior. Violation of company policies may lead to repercussions ranging from a warning to termination and possible criminal prosecution if warranted by the situation. ACZ has access to many resources that may be utilized at any time to help clarify any situation determined to be a "gray area." Employees are strongly encouraged to seek further guidance from a supervisor, ACZ's Ombudsman, Presidents, or QA staff whenever doubt is raised. Activities that will not be tolerated include, but are not limited to:

- **Misrepresentation of a procedure or documentation** – Intentionally performing a job duty in a manner that does not comply with a documented procedure, including but not limited to a test SOP or method used for sample analysis; providing inaccurate and misleading documentation associated with a data package or failing to provide the necessary documentation as part of a data package.

- **Falsifying Records** – Providing false information on personal credentials, resumes or educational transcripts, logbooks, raw data and client reports, or creating data without performing the procedure (also known as dry labbing).

- **Improper peak integration** – Intentionally performing improper integration of data chromatograms so quality control samples meet acceptance criteria. This is also known as peak shaving or peak enhancing.

- **Improper clock setting** – Readjusting the computer clock so that it appears samples were analyzed within hold times. Also referred to as time traveling.

- **Improper representation of Quality Control samples** – Failing to treat batch quality control samples in the same manner as client samples (including Proficiency Testing samples) or misrepresenting any type of quality control sample associated with the preparation batch and/or analytical batch.

- **Improper calibration** – Intentionally performing improper manipulation of calibration data or forging tune data so that it meets acceptance criteria.

- **File Substitution** – Replacing invalid data with valid data from a different time so the analysis appears to be successful.

4 PERSONNEL AND RESPONSIBILITIES

ACZ Laboratories, Inc. is an S corporation with two owners.

Refer to FRMAD072 for ACZ's current organizational chart.

It is the responsibility of management to document company policies, objectives, systems, programs, procedures, and instructions to the extent necessary to assure the quality and defensibility of all data.

ACZ's Co-Presidents are responsible for the overall management of the laboratory. On an operational level, one President is the appointed Lab Director with a focus on Production, Sales, & Marketing. The other President is the appointed Quality Assurance Officer with a focus on Quality, Compliance, Information Technology, and Project Management. On a tactical level, the focus on these divisions is less acute, and on a strategic level the Presidents focus on all divisions. Finance shares equal focus across all levels. The Chairman/Owner is the ultimate authority at ACZ but has no formal responsibilities beyond those required by law.

It is the responsibility of all managers to ensure that all documented ACZ policies and procedures, including those in the QAP and associated SOPs, are communicated to, understood by, made available to, and implemented by ACZ personnel.

ACZ only uses personnel who are employed by or under contract to the laboratory.

4.1 Co-President

The President is ultimately responsible for all analytical and operational activities of the laboratory and must ensure that 1) the laboratory carries out all environmental activities in such a way as to meet the requirements of the TNI Standards and 2) the laboratory satisfies the needs of the client and the regulatory authorities. General duties involve budgeting for all departments, making decisions on capital equipment and automation; developing company policies and benefits; addressing personnel issues such as hiring, firing, and promotions; and working with clients on various matters. Day-to-day responsibilities include providing direction to all laboratory departments including laboratory operations, accounting, marketing, QA, and client services. Additional responsibilities are as follows:

- Work directly with ACZ's Ombudsman to provide and maintain a mechanism for confidential reporting of ethical/data integrity issues as well as issues that may directly affect current ACZ policies.
- Define the minimal level of qualification, experience, and skills necessary for all laboratory positions.
- Provide the QA department with defined responsibility and authority for ensuring the successful development, implementation, and management of ACZ's Quality System.
- Provide the Production with defined responsibility and authority for ensuring the technical operations and provision of resources needed to maintain the required quality of laboratory operations.
- Provide adequate supervision of environmental staff by persons familiar with methods and procedures, purpose of each test, and assessment of the test results.
- Ensure all technical staff has demonstrated capability in the activities for which they are responsible and ensure that the training of each member of the technical staff is kept up-to-date.
- Ensure the QA department has access to the highest level of management at which decisions are made on laboratory policy or resources.
- Provide managerial staff the authority and resources needed to discharge their duties.
- Provide technical personnel the resources needed to discharge their duties.
- Specify and document the responsibility, authority, and interrelationship of all personnel who manage, perform or verify work affecting the quality of calibrations and tests.
- Implement appropriate and current guidelines for all lab methods and procedures to ensure data quality and efficiency of analyses. Ensure all method protocols utilized by ACZ meet the QC requirements as established by EPA or other governing agency.
- Document all policies and procedures related to the analytical and operational activities of the laboratory.
- Provide support to technical staff to ensure timely completion of all laboratory work, and develop contingency plans to ensure workflow progresses as planned.
- Meet quarterly (or more often) with department leaders reporting directly to the Presidents.

4.2 QA Officer (QAO)

The QA Officer reports directly to the President; however, the QA department is considered a separate entity from operations in order to ensure data is evaluated objectively. The QAO has direct access to the President, and is therefore able to discuss and/or resolve all concerns, policies, etc. related to quality assurance or quality control. The primary responsibility of the QAO is to develop, implement, and manage all aspects of ACZ's Quality System, and he/she may take any action necessary to ensure all ACZ employees adhere to all policies, procedures, and objectives documented in ACZ's QAP, SOPs, memorandums, emails, etc. If warranted, the QAO has the authority to halt the performance of a single method or the production of a department, and if necessary, the operations of the entire laboratory, and will grant permission to resume when satisfied that the issue(s) have been resolved. Additional responsibilities include but are not limited to those stated in FRMAD060 and the following:

- Review and revise ACZ's QAP and provide training for all employees following approval of a new version.
- Provide QA orientation to new employees.
- Meet quarterly (or more often) with President/Lab Director and Laboratory Department Supervisors.
- Work with department managers to develop and improve training protocols.
- Conduct department training sessions as needed to address specific problems and questions.
- Arrange for or conduct internal audits; notify management of deficiencies; and track corrective actions.
- Organize all external audits; notify management of deficiencies; and assign and track corrective actions.
- Review and approve SOPs.
- Meet at least quarterly with Laboratory department supervisors to provide information, respond to questions, etc.
- Manage Proficiency Testing (PT) program.
- Coordinate and maintain all regulatory and client certification programs.
- Review and validate a determined percentage of all data packages from Log-in to Reporting.
- Work with marketing/client service representatives on QA aspects of proposals.
- Work with Project Managers and the Production Manager to resolve client feedback regarding data quality.
- Review and maintain records and documentation for audits, certifications and all other QA issues.

  <u>Qualifications:</u>

  - General knowledge of the analytical test methods
  - Documented training and/or experience in QA procedures
  - Knowledge of the Quality System as defined under TNI

4.3 QA Coordinator

The QA Coordinator reports directly to the QAO and assists with the development, implementation, and management of the Quality System. Primary job responsibilities are as follows:

- Review and maintain records/documentation for employee training including DOCs, MDLs, etc.
- Provide initial QA orientation to new employees.
- Coordinate annual data integrity training.
- Schedule analyses and compile and report data for Proficiency Testing (PT) program, including DMRQA.
- Initiate and track corrective actions related to PT samples and manage all documentation associated with analyses.
- Review and approve SOPs.
- Conduct internal audits, notify management of deficiencies; and track corrective actions.
- Conduct department training sessions as needed to address specific problems and questions.
- Update control chart-generated QC limits in the LIMS database as needed.
- Monitor control & calibration of support equipment
- Assist QAO with management of certifications.
- Manage ACZ's resume compilation.
- Update ACZ organizational chart as necessary.

Qualifications:

- General knowledge of the analytical test methods
- Documented training and/or experience in QA procedures
- Knowledge of the Quality System as defined under TNI

4.5 Laboratory Department Supervisor

Each Laboratory Department Supervisor is a full-time employee who reports to the President/Lab Director and exercises day-to-day oversight of laboratory operations for their specific area(s) of expertise. Each supervisor must be familiar with the test methods and related theory and instrumentation, as well as the assessment of results. In addition to monitoring the standards of performance, validity of all analyses, conformance to documented requirements, and quality of all data generated in their respective department(s), each supervisor is also responsible for ensuring that a new analyst has successfully completed all training requirements and is adequately prepared to commence work on client samples. Additional responsibilities are described in FRMAD060. If any supervisor is absent for more than 15 consecutive calendar days then another full-time staff member meeting the required qualifications will be assigned to perform the supervisor's duties.

Required Qualifications for a Laboratory Department Supervisor:

1) Chemical analyses (Organics & Metals): BS or BA in chemical, environmental, biological sciences, physical sciences or engineering, with a minimum of 24 college semester credit hours in chemistry and at least two (2) years of experience in the environmental analysis of representative inorganic and organic analytes for which the laboratory seeks or maintains accreditation. A masters or doctoral degree in one of the above disciplines may be substituted for one (1) year of experience.

2) Inorganic Chemical analyses (other than Metals): At least an earned associate's degree in the chemical, physical, or environmental sciences, or two (2) years of equivalent and successful college education, with a minimum of 16 college semester credit hours in chemistry and at least two (2) years of experience performing such analyses.

3) Radiological analyses: BS or BA in chemistry, environmental, biological sciences, physical sciences, or engineering, with at least 24 college semester credit hours in chemistry and at least two (2) years of experience in the radiological analyses of environmental samples. A masters or doctoral degree may be substituted for one (1) year of experience.

   The minimum requirements may be relaxed if the Laboratory Department Supervisor is not the appointed technical director of the laboratory division.

4.6 Business Development Manager

ACZ's Business Development Manager reports directly to the President/Lab Director and supervises all Client Service Representatives, each of who conducts marketing and sales efforts on behalf of ACZ with potential, new and existing clientele, and develops and maintains long-term relationships with customers by working with Project Managers when necessary. Additional responsibilities of the Business Development Manager are described in FRMAD060. ACZ's Client Service staff is authorized to review all contractual agreements with clients, review all proposals and develop price quotations for routine and non-routine analytical projects.

4.7 Project Manager Supervisor

The Project Manager Supervisor reports directly to the President/QAO and is responsible for overseeing the PM department. Additional responsibilities of the Project Manager Supervisor are described in FRMAD060. Each Project Manager serves as the primary laboratory contact for each ACZ client, handles all client service requests, and investigates and resolves any problem brought to ACZ's attention by the customer. In order to provide consistency, each PM is assigned a list of clients, and it is the primary responsibility of each PM to ensure all of their client project needs are managed on a day-to-day basis and met in a timely manner and that all data submitted to the client is of high quality. All PMs work directly with the Laboratory Department Supervisors regarding client data issues (due dates, hold times, retests, data quality, etc.), with Document Control regarding client reports, and with the QA department regarding data quality questions or concerns. The Project Manager Supervisor directly oversees Reporting and the Front Office.

4.8 Instrument Operator

Instrument operators report directly to the respective Laboratory Department Supervisor. The position involves the analysis of various matrices for trace level contaminants using specialized and technical instrumentation. Each operator must be capable of performing all job duties in an accurate and proficient manner. Education will be verified by providing a copy of a college transcript or diploma, which is maintained in the employee's personnel file. Experience is verified by ACZ's CFO prior to completing the hiring process (verbal or documented verification provided by each reference listed on a resume or application is acceptable). The operator must demonstrate understanding of related theory, mathematics, analytical instrumentation, and data interpretation. This work is predominantly intellectual and involves the continuous use of professional and sound judgment. The employee must meet or exceed all requirements for generation of litigation-quality data and must also continue to demonstrate increased proficiency regarding the interpretation of the data as well as the operation and troubleshooting of the assigned instrument(s). These improvements should be attainable through ongoing efforts in-house as well as through specialized instruction at off-site locations. Exceptions pertaining to experience or education will be made on a case-by-case basis.

Qualifications:

- BA or BS in Chemistry or related science or a minimum of 3 years of relevant experience in lieu of degree
- Prior laboratory experience is preferred but is not required.
- Successful completion of training by supervisor or proficient instrument operator

4.9 Laboratory Analyst [Technician]

The laboratory technician reports directly to the respective Laboratory Department Supervisor. The position involves analysis of various matrices using appropriate analytical techniques and support equipment as well as preparation of samples for instrument analyses. Each technician must be capable of performing all job duties in an accurate and proficient manner. Education will be verified by providing a copy of a college transcript or diploma, which is maintained in the employee's personnel file. Experience is verified by ACZ's CFO prior to completing the hiring process (verbal or documented verification provided by each reference listed on a resume or application is acceptable). The technician must demonstrate understanding of related principles and mathematics, must possess common sense and mechanical skills, and must seek professional judgment from the supervisor as necessary. The employee must meet or exceed all requirements for generation of litigation-quality data as well as sample preparation tasks and routine analyses, and must also continue to demonstrate continuous improvements. These improvements should be attainable through ongoing training efforts in-house as well as through training opportunities at off-site locations. Exceptions pertaining to experience or education will be made on a case-by-case basis.

Qualifications:

- BA or BS in Chemistry or related science is preferred but is not required
- Prior laboratory experience is preferred but is not required
- Successful completion of training period by supervisor or proficient technician

4.10 Information Technology (IT) Manager

The Information Technology Manager reports directly to the President/QAO and is responsible for the oversight of the IT department regarding the installation and maintenance of ACZ's computer network and all hardware and software and related equipment deployed on the premise. Additional responsibilities are described in FRMAD060. The department is also responsible for developing, maintaining, and improving custom written applications for laboratory automation and efficiency as well as for ACZ's LIMS, Intranet (Labweb), Internet and electronic data deliverables (EDDs).

4.11 Chemical Hygiene Officer (CHO)

The Chemical Hygiene Officer is responsible for oversight of ACZ's documented Chemical Hygiene Plan, conducting initial and refresher safety training for all employees, monitoring exposures, and maintaining records for Safety Data Sheets, injury reports, chemical exposure reports, etc. Additional responsibilities include working with management to develop and implement policies to improve the program. The person designated as CHO must have completed at least one basic laboratory safety course and have one year of experience performing laboratory work, preferably with responsibility for at least one area of laboratory safety.

4.12 Hazardous Waste Officer (HWO)

The HWO is responsible for managing and, with the collaboration of management, enforcing all aspects of ACZ's Hazardous Waste Management Plan (SOPAD007). The HWO must insure the HWP is compliant with relevant requirements in the US Code of Federal Regulations as well as any additional state regulations. Additional duties include bulking and labeling hazardous materials, filling out required documentation, and arranging for disposal; these activities may be delegated to qualified individuals under the supervision of the HWO. The HWO must know and understand the specific waste streams that ACZ uses and be able to determine how to dispose of unknown chemicals. This is best done by attending a training course on "Laboratory Waste Management." The individual responsible for hazardous waste disposal and signing the waste manifest must maintain HAZWOPER and DOT hazmat certification.

4.13 Radiation Safety Officer (RSO)

ACZ's Radioactive Materials License (RML) requires the laboratory have an RSO. The President/QAO appoints a Radiation Safety Officer to act as his/her representative in implementing the Radiation Safety Program. The RSO's responsibilities include developing radiation safety guidelines in accordance with Nuclear Regulatory Commission (NRC) and Colorado state rules and regulations, and for assuring compliance with those guidelines by ACZ personnel. The RSO will work with ACZ's administration to implement policies and seek ways to improve the safety program. The person designated as RSO must have completed a Radiation Safety Course or have at least 3 years of experience prior to being officially designated as the RSO. The RSO reports directly to the President/QAO of ACZ.

4.14 Chief Financial Officer (CFO)

ACZ's Chief Financial Officer is primarily responsible for all financial matters including payroll, accounts receivable, accounts payable and financial statements; monthly and annual balance and profit and loss statements; and assisting with annual budget preparation. In addition, the CFO maintains and monitors the security system and electronic time clock; invoices client projects from the database; updates customer account information; acts as the administrator for 401k/Profit Sharing Plan; maintains and executes the Employee Benefits Manual; and assists in hiring process by posting job openings, scheduling qualified candidates for interviews, checking references, and ensuring a new employee provides proof of education.

5 TECHNICAL TRAINING

Prior to the independent generation or review of data for client samples (including PT samples), all analysts must undergo a formal, documented training process. Technical personnel must be thoroughly trained in the analytical techniques and operating principles and procedures for the methods utilized by ACZ. This process includes but is not limited to: reading the associated published method, reading all related SOPs, improving laboratory skills, learning troubleshooting, maintenance, calibration and operating procedures for pertinent equipment and instruments, and creating workgroups and reviewing data through the LIMS database.

It is the responsibility of the department supervisor to determine that a new analyst is properly trained, has successfully completed all initial training requirements and is prepared to commence work on client samples. Under no circumstances may an analyst independently generate client data before receiving the explicit approval of the technical director overseeing the analysis.

5.1 The effective version of the test SOP provides the training framework for all sample preparation and analysis. The SOP is typically based on published approved methodologies (EPA or other) and incorporates any necessary activities and protocols not included in the published method(s) as well as requirements stipulated by other regulatory agencies.

5.2 Training for data AREV or SREV only must be documented as specified in §2.3.6. For analysts, approval to perform a procedure includes approval to perform AREV for the procedure. For supervisors and technical directors, approval to perform a procedure includes both AREV and SREV approval. SREV-specific authorization is required for analysts.

5.3 Each employee must be trained either by the department supervisor or by an analyst within the department who is proficient in the area of testing and has been designated by the supervisor. Whenever possible, anyone performing training must meet the following requirements:

1) Documentation of training on the effective version of the test SOP.
2) Documented approval for the analysis.
3) A current IDOC or CDOC.

Exceptions may be granted on a case-by-case basis as approved by the QAO.

5.4 Initial training is documented using the Initial Method Training form (FRMQA004). The General Lab Practice Training Form (FRMQA047) is also required for an analyst's first procedure. Once training has been completed, the trainee and the instructor fill out the form together to ensure all pertinent information has been addressed and to ensure the trainee comprehends the material and is provided an opportunity to ask questions or request additional training. The trainee's signature is an attestation that he/she has read, understands, and agrees to follow the effective version of the SOP.

5.5 To demonstrate an aptitude for the procedure, the analyst must perform a successful Initial Demonstration of Capability (IDOC) prior to independent preparation and/or analysis of client samples. Performance is documented using FRMAD023. The data is reviewed initially by the trainee (AREV). SREV is performed by the pertinent technical director or designee. A new IDOC is required if an analyst does not perform the method within 12 months.

5.6 Prior to performing an IDOC, a new analyst should be provided sufficient opportunity to practice the procedure. This confirms the analyst understands the procedure and feels comfortable performing the procedure independently. Data associated with any practice is not submitted to QA.

5.7 It is not necessary for the first IDOC attempt to pass; however, the supervisor needs to review the analyst's techniques if multiple attempts do not pass.

5.8 A thorough review of the raw data is performed as part of initial method training and should include particular attention to details not presented in LIMS or on the final report, such as generating final sample concentration from the instrument response provided in the raw data (if applicable), verifying correct standard and reagent traceability.

5.9 Where specified by the method or a regulating entity, and as stated in the test SOP, successful demonstration of performance such as Linear Calibration Range determination (LCR) or Method Detection Limit (MDL) study must be completed prior to independent analysis of client samples.

5.10 All initial training documentation must be submitted to the QA department as a complete package. At a minimum, the package must include:

1) Initial Method Training form (FRMQA004), signed by the trainee and instructor (or department supervisor).

2) IDOC documentation:
   - ✓ Completed and signed certification statement (FRMAD023)
   - ✓ Workgroup bench sheet, raw data, and all supporting documentation

3) If applicable, an MDL study. Complete FRMAD031 and attach all related raw data and supporting documentation.

4) If applicable, calibration range study. Complete FRMQA029 and attach all related raw data and supporting documentation.

5) For all determinative methods utilizing a calibration curve or average response factor, the Method Calibration Form (FRMQA050).

   NOTE: For those test methods for which no spiking solution is available only an Initial Method Training form (FRMQA004) is required.

5.11 Following review of all pertinent training documentation, the pertinent technical director will issue procedure-specific clearance for the trainee to independently generate and review data for client samples. This permission is tracked and may be viewed on a designated location on the public network drive.

1) Approval for preparation procedures is granted after the instrument data has been reviewed and approved.

2) An unapproved analyst who is “shadowing” the trainer (observing, learning the organization of the lab, reagent room, etc.) may not assist with the procedure, and the workgroup documentation must bear only the initials of the trainer, who is fully responsible for the data.

3) If the analyst has successfully completed training for a procedure and generates client data or reviews client data prior to the technical director’s approval, then any workgroups or data review checklist must also bear the initials of a proficient analyst, with current approval for the method, who oversees the analyst’s work for the procedure and assumes full responsibility for the data. The primary analyst must always be aware that he/she is responsible for the workgroup. The use of another employee’s initials without their explicit approval is expressly prohibited.

5.12 The supervisor is responsible for ensuring the training of each analyst is kept up-to-date. Each analyst must read, understand, and agree to follow the effective version of the SOP and continued proficiency must be demonstrated and documented annually for each analyst. A one month grace period is allowed for submitting CDOC documentation. Thereafter, the analyst is prohibited from performing the procedure until a successful CDOC or IDOC is submitted to QA.

5.13 Each Laboratory Department Supervisor routinely conducts department meetings to discuss procedures, work schedules, resources, questions and concerns, problems, QA, etc.

6 SAMPLE COLLECTION AND HOLDING TIMES

Sample collection procedures are well documented by the EPA and other agencies. ACZ's clients are instructed to provide representative samples whenever possible. ACZ supplies its clients with the containers and other materials necessary to maintain sample integrity (to the extent possible) from the time of collection through analysis. Although ACZ does not perform sample collection activities, each project manager or client service representative will assist a client with specific sampling requirements as needed. When necessary, they will direct a client to other resources. The following sections include general information on sample containers, preservatives. and holding times. These are essential components in preserving the chemical and physical properties possessed by the sample at the time of collection.

6.1 Sampling Containers and Preservatives

The EPA outlines the requirements for sample container types, sample volume, and preservation. ACZ's inventory includes various sizes of plastic and glass containers that range from pre-sterilized to certified-clean by the supplier. Amber bottles are used when specified by the method. Glass containers are obtained from vendors that specialize in the sales of environmental sample containers, and all non-certified bottles are purchased from reputable lab/industry vendors. Refer to FRMAD045 and FRMAD046 for bottles types and preservation techniques for specific analyses. Refer to the pertinent test method SOP for specific information regarding EPA requirements for container types, chemical, and thermal preservation.

All sample containers shipped to our clients are new, contain the appropriate preservative(s), and are color-coded to identify preservation and storage. Out-going containers are packed in clean coolers with a copy of ACZ's Sample Acceptance Policy, general directions for sample collection, bottle labels, ice packs, sampling information, blank chain of custody, return shipping labels, and custody seals. Trip blanks and rinsette water are included when requested by the client or when mandated by a specific analytical method. After samples have been collected they are cooled to a temperature ≥ 0 °C and ≤ 6.0 °C. Samples that require thermal preservation must be maintained within this temperature range until all analyses have been completed.

6.2 Holding Times

The EPA has conducted lengthy studies of sample degradation versus time to establish a maximum holding time for each parameter, and the results of these studies are compiled into holding-time tables to provide guidelines for litigation purposes. Data for a sample prepared / analyzed outside of the established holding time may be rejected by regulators as unusable. Holding times will vary slightly from regulation to regulation, thus further emphasizing the need for a client to consult with their Project Manager prior to sample collection. The holding time typically begins at the time or date of collection in the field. Holding times observed by ACZ are specified in the laboratory's test method SOPs.

If ACZ Laboratories, Inc. receives samples past holding times or near the expiration of the holding time, sample analysis will proceed unless the client has indicated on the CCOC that an attempt to contact the client must first be made. Analyses performed outside of holding time will be appropriately qualified on the final report. Holding times ≤ 72 hours are calculated based on the hour of the sample date/time. Holding times > 72 hours are calculated based on the day of the sample date/time.

In general, and unless otherwise noted in the test SOP, sample preparation and analysis must be completed within the stated holding time. For analyses that extend beyond the intended scope of the method for an analyte or matrix, the hold time stated in the SOP must be met or the samples must be appropriately qualified.

7 SAMPLE CUSTODY & SAMPLE HANDLING

Sample custody begins with receipt of sample containers from the client and continues beyond preparation and analysis to the proper disposal of primary and secondary sub-samples. Complete and accurate documentation must be provided at all stages of custody. There are many key elements to sample custody including laboratory security, chain of custody records, sample storage, internal custody logs, sample tracking within the laboratory, control of subcontracted work, and sample disposal. Unless otherwise specified, ACZ is contractually committed to retain samples for a minimum of 30 days after the invoice of a project[2].

7.1 Sample Receipt and Log-in

Refer to SOPAD045, *Sample Receipt & Log-In Procedure / Maintenance of Sample Integrity,* for the details of ACZ's sample receipt and log-in procedures. Upon delivery of samples to ACZ, Log-In personnel evaluate the condition of the cooler and custody seals. The custody seals are then broken to retrieve the Chain of Custody (COC), which must be signed by the sample custodian to document transfer of sample possession to ACZ.

Sample conditions are evaluated and any problems, such as expired hold times, lack of preservative or improper cooler temperature, are noted. Clients are notified of problems as soon as possible so that a contingency plan can be initiated if necessary. Samples are logged-in and are delivered to the assigned storage areas. Samples (including subsamples, extracts, etc.) must be stored away from standards, reagents, food, and other potential contaminants. Following log-in, every project is reviewed by the assigned PM. Upon completion of the review, the client receives an electronic summary that details the project information. This summary allows the client an opportunity to make changes to the project before samples are analyzed. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043) for additional information.

7.2 Internal Custody Logs

Some clients may specify additional custody tracking of the samples once they have been logged in. Internal custody may require that samples are stored in a manner that ensures limited access. The internal custody log (FRMQA015) shall accompany the samples from log-in through completed analysis. The person responsible for the work signs and dates each entry and/or page in the logbook. When all data from a sample set is compiled, copies of all logbook entries shall be included in the final report package. For projects requiring internal custody, ACZ will adhere to the procedure described in the SOP *Client Service Policies and Procedures* (SOPAD043).

7.3 Sample Tracking

Sample flow through the laboratory is facilitated by the use of an Oracle-based LIMS database (Laboratory Information Management System). Every product (requested analysis) logged into the LIMS for a sample has a specific, pre-determined department path. All products have default paths of at least Login Review and Reporting. Between these two departments, a product may go through, for example, Soil Prep and Metal Analysis or Soil Prep, Organic Prep and GC Analysis. At each department step in a product's path, the status can be updated and viewed at any time. Analytical product statuses are defined below. Additional information regarding sample tracking is available in the SOP *Client Service Policies and Procedures* (SOPAD043).

NEED	Prep or Analysis has not been started
WIP	Prep or Analysis has been started (Work In Progress)
PREP	Sample preparation is complete and sample is ready for analysis
UPLD	Analytical data has been uploaded into LIMS
AREV	Analyst has reviewed and accepted analytical data
SREV	Supervisor has reviewed and accepted analytical data
DONE	Analysis or task has been completed
REDO	Sample requires reanalysis
REDX	Sample requires re-digestion/extraction

[2] Currently, samples scheduled for any radiochemistry parameters are held for a minimum of 90 days to facilitate radioactive material inventory monitoring. This policy is subject to change at any time in harmony with ACZ's Radiation Safety Plan.

CANT	Sample preparation or analysis cannot be performed
NREV	Project requires PM review before work can begin
HOLD	Prep or analysis postponed
SENT	A final report has been issued to the client

8 PROCUREMENT, INVENTORY AND TRACEABILITY OF SUPPLIES

8.1 Procurement / Inventory

All consumable supplies are purchased from reputable vendors that have been evaluated for service, quality, and price. To the extent possible, materials traceable to national or international standards of measurement are purchased for use in technical operations. Supplies are purchased using ACZ's purchase order (PO), remote inventory management system (RIMS), and the Aestiva ordering system. The Purchasing Agent is not permitted to make a substitution for any material specifically requested unless the Laboratory Department Supervisor approves the substitution. Upon receipt, reagents, chemicals, standards, and other laboratory consumables are stored in the Chemical & Supply Room, which has limited access, or are delivered to the laboratory. Refer to ACZ's SOP *Purchase, Receipt, and Storage of Consumable Materials for Technical Operations* (SOPAD037) for additional information.

8.2 Glassware

ACZ uses only laboratory grade glassware. Prior to use, glassware is cleaned to meet the sensitivity of the method. Refer to individual test SOPs for detailed cleaning procedures.

8.3 Other Supplies

Routine consumables (centrifuge tubes, autosampler tubes, pipette tips, etc.) are purchased through an automatic system managed by Fisher (RIMS). All other supplies are purchased on an as-needed basis through ACZ's Purchase Order and the Aestiva ordering system. Refer to SOPAD037 for additional information.

8.4 Traceability of Standards and Reagents

To provide complete traceability, each data package must reference every standard and reagent used for sample preparation or analysis, including but not limited to acids, bases, preservatives, color reagents, pH indicators, buffers, and instrument reagents. Each PCN and/or SCN must be documented either on the workgroup bench sheet, data review checklist, or a current standard/reagent form. The open date for all original containers is not tracked in LIMS; however, good laboratory practice dictates the open date be noted on the sample container.

8.4.1 Primary Control Number (PCN)

Upon receipt, all stock chemicals, standards, and reagents are assigned a unique PCN in LIMS for tracking and traceability purposes. A label with the PCN and the expiration date is affixed to the container and the Certificate of Analysis is scanned or downloaded and saved in the public drive (if applicable). The data for each PCN is entered using the certified value(s) supplied by the vendor, as indicated on the Certificate of Analysis. Because the certified value is entered, the final concentrations for prepared standards may vary slightly from the theoretical value indicated in the test SOP. Certified values shall be used for standards when available. If certified values are not available, informational values may be used. If the certified reference values for any PCN are changed after the PCN has been used in the laboratory, then complete documentation must be provided as a major corrective action (FRMQA001).

8.4.2 Secondary Control Number (SCN)

To ensure complete traceability, a unique SCN must be created when any intermediate or working standard is prepared from one or more stock solutions, stock chemicals, or intermediate solutions. A standardized format is used for creating the SCN: a two-letter code indicates the lab section and is followed by the prep date and then by a daily sequential number. For example, the SCN **II051128-2** denotes the second standard prepared on November 28, 2005 in the Inorganic Instrument lab. An acceptable alternative is to let LIMS assign a unique number when prompted.

An SCN for any working standard subjected to a LIMS calculation must be created electronically in LIMS. The initial volume and concentration of each constituent and the final volume of the prepared

solution are entered in the SCN Wizard program to calculate the final concentration of each analyte using the formula $C_1V_1 = C_2V_2$. The preparation date, expiration date, and preparer's initials are included as part of this electronic record. A hard copy of the SCN report may be affixed to the standard/reagent logbook, depending on individual department practice; however, it is not required.

Prepared reagents do not require an SCN be created electronically in LIMS; however, preparation must be recorded in the department's designated logbook. At a minimum, the logbook entry must clearly identify what reagent was prepared, its subcomponents, the preparer's initials, the preparation date, and the expiration date. This information is sufficient for color reagents, buffer solutions, instrument reagents, etc. because details of the preparation are stated in the test SOP.

8.5 Preparation and Expiration of Standards and Reagents

8.5.1 Preparation of Standards and Reagents

Refer to individual test SOPs for detailed information regarding standard and reagent preparation. In general, either Class A pipettes or mechanical pipettes are used to measure and dispense aliquots of any solution used to prepare a standard or reagent. Accurate delivery of mechanical pipettes must first be verified as described in ACZ's SOP *Control, Calibration, and Maintenance of Measuring and Test Equipment* (SOPAD013).

All containers of prepared reagents and standards stored for more than one day must be properly labeled with the SCN (or other unique identifier), name/description, preparation date, and expiration date. Preparation of reagents and standards must be documented as described in §8.4.2.

8.5.2 Expiration of Purchased Standards and Chemicals (PCNs)

When provided, the manufacturer's expiration date will be assigned. If the manufacturer does not provide an expiration date, an expiration date of 5 years from receipt is assigned unless the laboratory has knowledge indicating a longer or shorter shelf life is appropriate.

An expired stock material may continue to be used if its reliability can be verified. For the purpose of ensuring transparency, the rationale for extending the expiration date must be documented on FRMQA051 and submitted to the QA department or pertinent technical director for approval. If the extension is granted, FRMQA051 is saved on a network drive. Unusable materials should be replaced and the standard or reagent remade as soon as possible. Remove the container from the lab or the supply room and dispose of properly. Contact ACZ's HWO for assistance.

8.5.3 Expiration of Prepared Standards

Storage conditions and shelf life for prepared standards are provided in the individual test SOPs. The following guidelines may be used to determine the shelf life for a prepared standard if the method does not prescribe a shelf life:

1) A standard that has been prepared in-house may continue to be used after its assigned expiration date for as long as its reliability can been verified. Whenever possible, reliability should be verified by comparison to another, unexpired standard containing the same constituents. For applicable procedures, instrument response may be considered when determining whether or not a solution is still reliable.

    - In cases where reliability has been verified, the expiration date of the SCN must be updated in LIMS and/or the standard/reagent logbook. The rationale for extending the expiration must be documented on FRMQA051 and submitted to the QA department for approval.

    - In the event the solution was used prior to updating the SCN then documentation must be provided as part of the workgroup to indicate the solution was used past the shelf life stated in the SOP (a minor corrective action or FRMQA051 may be used if more than one workgroup is affected). The expired standard must be remade as soon as its reliability becomes questionable – it is the responsibility of the analyst to use their best judgment.

2) The shelf life of any prepared standard with any analyte concentration < 10 mg/L is 90 days from the preparation date. This is a general guideline – if any constituent does not remain in solution for 90 days, then the standard must be prepared more often. If the manufacturer's expiration date for any stock standard is sooner, then the expiration date of the SCN is the manufacturer's expiration date for a single analyte solution or the earliest manufacturer's expiration date for a multiple analyte solution.

3) The shelf life of a prepared standard with analyte concentration $\geq$ 10 mg/L is one year from the preparation date. This is a general guideline – if any constituent does not remain in solution for one year, then the standard must be prepared more often. If the manufacturer's expiration date for any stock standard is sooner, then the expiration date of the SCN is the manufacturer's expiration date for a single analyte solution or the earliest manufacturer's expiration date for a multiple analyte solution.

4) In general, prepared Radiochemistry standards expire one year from the preparation date. The solution may be re-evaluated using control charts, efficiency checks, or other criteria and the expiration date extended by year intervals if the solution is still deemed usable. Refer to the specific test SOP for details.

8.5.4 Expiration of Reagents

In general, a reagent is a solution, which does not contain the target analyte(s). Storage conditions and shelf life are stated in the individual test SOPs. The expiration date can be extended for a prepared reagent provided its reliability can be verified. LCS/LFB performance (QC criteria met) may be used to verify reagent stability if the control standard is a valid indication of the reagent's continued functionality/stability. Reagents used to treat samples for interference may not be verified this way. Reagents used to dissociate complexed target analytes may not be verified this way unless the LCS is an appropriate complex. FRMQA051 must be submitted to QA or the pertinent technical director for approval whenever an expiration extension is requested.

9 MAINTENANCE AND CALIBRATION OF INSTRUMENTATION & EQUIPMENT

9.1 Maintenance of Instruments and Support Equipment

The best protocol for producing quality work is to prevent errors and non-conformances rather than react to and correct problems after they occur. An essential part of this protocol is ensuring that all laboratory instrumentation and equipment used for the generation of data have been optimized and are functioning properly before commencing work on client samples. Performing routine maintenance and optimizing instrument-operating conditions prior to sample analysis minimizes instrument downtime, thereby improving productivity and ensuring quality of the data. It is the responsibility of the designated analyst(s) to perform and properly document daily and routine maintenance, instrument optimization, troubleshooting, instrument service or repair, and repair or replacement of parts.

All manufacturer-prescribed inspection and maintenance shall be performed according to the schedule indicated in the operator's manual (or similar) provided by the manufacturer and must be documented in the instrument logbook, a separate maintenance logbook, or on the instrument maintenance checklist (available in LabWeb). ACZ management recognizes that performing all maintenance procedures at the frequency indicated by the manufacturer may not be necessary to sustain instrument optimization. Therefore, at a minimum, instrument part(s) and optimization shall be inspected according to the schedule. The analyst must use their professional judgment to determine if maintenance or replacement is necessary at that time. Decisions to deviate from the manufacturer's schedule shall be documented.

All support equipment (any device that may not be the actual test instrument, but is necessary to support laboratory operations) must be monitored regularly to confirm proper functioning. The temperature of all drying ovens, refrigerators, freezers, and incubators must be checked each day the equipment is in use and each check recorded on the associated Temperature Logsheet. Refer to SOPAD013 for more detail.

Equipment that does not meet performance specifications must be taken out of service and FRMAD029 attached to indicate the instrument or equipment is waiting for repair and cannot be used. During this downtime the department supervisor, Production Manager, and Project Manager may collectively determine it is necessary to sub-contract samples until correct performance of the repaired instrument or equipment has been demonstrated by a successful calibration or other suitable test. Document all contact with the manufacturer, as well as all repairs and other services, in the instrument or maintenance logbook to be used as a reference for solving future instrument problems. Transport and storage of measuring equipment shall be done in accordance with manufacturer recommendations. Additionally, when instrumentation or equipment goes outside of the direct control of the laboratory, the functioning and calibration status must be checked and shown to be satisfactory before it is returned to service. Refer to SOPAD013 for additional information.

To minimize downtime and prevent analytical delays, each laboratory should maintain an adequate inventory of reagents, stock standards, glassware, etc. and should keep a sufficient supply of extra "critical" parts in-house. Instrument redundancy should be established for all analyses and Instrument Qualification (IQ) should be maintained on backup instruments.

9.2 Instrument Calibration

The accuracy of all instrument-generated data relies on proper calibration. In general, calibration or standardization involves defining the relationship between instrument response and the amount or concentration of analyte introduced into the instrument. The graphical depiction of this relationship is referred to as the calibration curve.

Calibration frequency must be performed in accordance with the manufacturer's guidelines, test method or other regulatory requirements, or client contract stipulations, whichever is most stringent. Every calibration or standardization must meet the acceptance criteria stated in the SOP and shall be subsequently verified by analyzing an initial calibration verification standard (ICV) or other control standard (if specified in the SOP) that contains all target analytes and has been prepared or obtained from a different source than the one used to prepare

the calibration standards.[3] Calibration standards and the second-source verification standard should be prepared on different days. If they are prepared concurrently, then another qualified analyst should prepare the second-source verification standard. This minimizes the risk of both solutions being prepared consistently incorrectly.

A continuing calibration verification standard (CCV) containing all analytes of interest must be analyzed at the frequency stated in the test SOP to ensure the stability of the initial calibration curve has not varied over time due to any change in the analytical instrument and its detection system, such as instability of standards, instrument cleanliness, column performance, matrix effects, flow changes, and changes within the laboratory environment.

For applicable methods, all initial and continuing calibration steps must be clearly detailed in the test SOP. Additionally, each test SOP must specify the frequency and acceptance limits for the calibration and subsequent verification (ICV and CCV). In general, acceptance criteria are method-specific; however, the SOP may also include requirements of other regulatory agencies. Prior to resuming sample analysis, immediate corrective action must be taken if the calibration, ICV, or CCV is outside of the acceptance criteria. Technical corrective actions are described in the individual test SOPs. Refer also to §11.2 for additional information.

General calibration guidelines are listed below. Additional information is provided in the individual test SOP's and ACZ's SOP *Control of Measuring & Test Equipment* (SOPAD013).

- Understand the method requirements for calibration (minimum number of standards, etc.)
- Use the correct calibration model (linear, second-order, etc.)
- Include all target analytes in the calibration standards and second-source standard
- Analyze a calibration standard with a concentration less than or equal to the quantitation limit.
- Do not remove points from the middle of the calibration (only high or low standards may be dropped).
- Calibration is a single-event process. A retest of a calibration standard must be performed immediately.
- Documentation and resolution of calibration abnormalities is critical

[3] If a second source standard is not available calibration shall be verified using a standard from a different lot. If a different lot is not available, an analyst who did not prepare the calibration standards may prepare the calibration verification standard. For some standards, it is important to consider whether manufacturers have obtained their material from the same lot.

10 CONTROL AND STORAGE OF RECORDS AND DOCUMENTS

A formal and systematic control of records and documents is necessary to accurately reconstruct the entire history of any sample and guarantee the quality and defensibility of the data. All information pertaining to instrumentation and equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, data verification, audits, corrective actions, method validation, and data reporting must be documented, must identify all personnel involved, and must be readily understood. All records, including those pertaining to calibration and test equipment, certificates and reports, must be maintained, and the management system must facilitate the retrieval of all working files and archived records for inspection and validation purposes. Documents and records must be safely stored (protected against fire, theft, loss, environmental deterioration, and vermin) and must be held secure and in confidence to the client for a minimum of 10 years. The hard copy of all records and documents must be maintained in a designated storage area with limited access. To the extent possible, hard copies for the most recent two (2) years are stored on-site, and if necessary, may be moved to off-site storage after two years. Off-site storage conditions must meet the same criteria that apply to on-site storage.

10.1 Workgroups

10.1.1 Changes made to any workgroup record (hardcopy or electronic) must be documented.

1) If a workgroup is “dissolved” or its data deleted from LIMS, the analyst is prompted in LIMS to provide an explanation of why he/she is performing the task.

2) Changes to upload files must be documented on the hard copy of the workgroup.

10.1.2 Workgroup data that is re-uploaded *for any reason* must first be deleted. If any of the data changes, the Run Approval report shall be corrected. The workgroup shall be rescanned if necessary.

10.1.3 Document Control or other administrative personnel use a multi-page scanner with its own PDF scanning software to scan all hardcopy portions of workgroups.

1) Before the workgroup is scanned, the top page is reviewed to make sure it has both the AREV and SREV initials and dates.

2) The person scanning the workgroup must initial in the lower right hand corner of the front page of the workgroup. This provides an indication the document has been scanned.

3) The workgroup is scanned to the designated network directory and is then moved through an automated process to the appropriate read-only LabWeb directory. This directory is accessible to all employees. When a workgroup is rescanned, the previous file is maintained. A copy will be automatically created so as not to overwrite any files and will have a letter appended; starting with “A” the first time the workgroup is rescanned. <u>The most current file will not have a letter appended</u>.

10.1.4 The hard copy is filed by workgroup number in a file cabinet in the supply room by the front office. When capacity is reached, the workgroups are boxed and prepared for long-term storage. The front of the full storage box is labeled with the year and the workgroups contained in the box. The first box of each new calendar year is “1.” Full boxes are consecutively numbered, transferred to a designated location and stored in numerical order. The storage room is locked at all times and access is limited to authorized staff.

10.1.5

10.2 Electronic File Retention & Storage

All electronic records, stored either on instrument computers or on the network, are systematically

backed up to both fixed and removable media. These records include Oracle data, instrument raw data, workgroups, client reports, instrument upload files, SOPs and other controlled documents, and department data.

10.2.1 Critical system data is protected by Microsoft's Data Protection Manager. The Data Protection Manager is configured to maintain data for a period of 10 years.

10.2.2 All archived data is moved to a secondary machine on a weekly basis. From there, it is backed up to removable media to provide additional data redundancy.

10.2.3 The removable media from the first week of the month is pulled from service and moved to ACZ's safe deposit box at a local bank. The most recent 6 months of tapes are kept in the bank safe deposit box. Months 7 through 12 are placed in a secure, data rated, 4-hour fireproof safe. Note that this removable media only contains data from December 1st of the previous year to the present date.

10.2.4 At the conclusion of the calendar year, a master copy is made that comprises all of the data from December 1st of the previous year through January 31st of the following (current) year. This 14-month span of data is then moved to ACZ's safe deposit box at a local bank. At that time, the removable media that has aged 10 years is removed from the safe deposit box and its contents are destroyed. All data on the secondary machine from prior to December 1st of the previous year is removed from the system so that it is no longer included on the weekly backup.

10.2.5 Data that has aged 5 years is deleted from the Oracle Database on a monthly basis.

10.3 Instrument Data Files

Instrument raw data files are backed up by ACZ's Instrument Data Backup Application (IDBA). IDBA is a program that accesses local directories from instrument computers. Each morning the program retrieves and backs up individual data files from the specified directory on each instrument computer. Refer to ACZ's SOP *Backup and Archive of Instrument Data Files* (SOPAD044) for details.

10.4 Client Reports

10.4.1 Client reports are generated and signed electronically and are automatically stored as a PDF at a designated location on the network that has limited access. If a copy of any report exists on the network, and a new report is generated, then the existing copy will be renamed so that it is not overwritten. This way ACZ maintains a copy of all reports generated for a client.

10.4.2 Hardcopy documentation associated with a client project (CCOC, invoice, Login Review Form, etc) is filed by project number and stored in the document storage location.

10.4.3 Electronic Data Deliverables (EDD) are stored on the network at a designated location.

10.4.4 Once a project has been invoiced, the working directory is moved to the designated storage network location as a read-only PDF. If a project is un-invoiced, the project folder is copied back to the working directory where changes may take place. If an invoice is altered, a revised invoice is included with the project hardcopy.

10.4.5 In general, changes are not allowed to projects (including compilation) if the project has been invoiced. If a change needs to be made, the project must first be un-invoiced. At the time of un-invoicing, the user must provide a reason in LIMS to explain why the project was un-invoiced. This information is then stored in the Oracle database.

10.4.6 If a test report requires amendment after it has been issued to the client, the entire report shall be re-issued with the amendment. The amended report shall include a case narrative describing change(s) from the original report. Amended reports shall be uniquely identified and contain a

reference to the original report. Typically, the laboratory number assigned to the project serves as the link to the original report. Amendments shall meet all the requirements of ACZ's quality system.

10.5 Documents

10.5.1 Standard Operating Procedures

10.5.1.1 Refer to §2.2 or SOPAD049 for additional information pertaining to SOPs.

10.5.1.2 The original master copy of each SOP is stored as a PDF in a secured public directory. The cover page indicates approval authorities. Approval is documented through emails. Whenever an SOP or SOP revision is approved, QA emails all staff whose job activities intersect the SOP or SOP revision with training instructions and a request for read receipt. The pertinent technical director or supervisor shall be included in the email distribution list. This email constitutes QA's signature of approval. Read receipts constitute training signatures and the technical director's or supervisor's signature of approval. Emails requesting read receipts for training documentation purposes shall contain a statement that by sending read receipt the sender attests they have read, understand, and agree to follow the identified policy. MS Outlook's read receipt is mimicked for this process. To send read receipt, trainees and approval authorities reply to the sender and replace "Re" with "Read" at the beginning of the subject field. Emails documenting training and/or approval shall be saved in a public outlook email folder or converted to PDF's and stored in the same directory as the SOP master copy or a subdirectory therein.

SOP master copies pre-dating the above paperless policy are located in the document control office.

10.5.1.3 A printed controlled copy of any SOP may be obtained from ACZ's LabWeb.

1) To ensure outdated information is not inadvertently used as a reference, Invalid or obsolete SOPs must be promptly removed from all points of use or clearly marked to indicate the purpose of retention

10.5.1.4 SOP Revisions: Any revision to a procedure must be approved by QA before changes may be implemented.

10.5.2 When documents are found to contain conflicting policies or procedures, the most recent document will be followed unless the conflict is prescribed as an exception to general protocol by a document more specific to the application.

10.5.3 Forms containing procedures or equations shall be controlled. Equations shall be validated and protected from inadvertent alteration.

10.5.4 All controlled forms must be printed from LabWeb and may not be stored on a separate network drive. If photocopies are used then any unused copies of the expired version must be disposed of as soon as a new version is uploaded to LabWeb. This ensures that the effective version of any controlled form is in use at all times. Exceptions may be granted by the QAO on a case by case basis.

10.5.5 The original certificate of analysis for any stock material, if provided, is stored in electronic format on ACZ's network.

10.5.6 Accreditation certificates are stored as PDF files to a designated network location. Original copies are maintained by QA. Certificates are also posted to ACZ's website.

10.5.7 Original calibration certificates and related documentation for support equipment (including but not limited to pipettes, thermometers, and glass micro liter syringes) are maintained by the QA Department.

10.5.8 LIMS and other problems pertaining to IT are documented and managed by the electronic system called Help Desk. If an employee encounters a problem that requires attention, that employee will submit a request through Help Desk. The request requires a priority be assigned. This system allows ACZ to track all changes made to computer systems.

10.6 Records

10.6.1 Records include, but are not limited to: all logbooks; phone logs; raw data, derived data, and calibration data; training documentation (training forms, MDL studies, DOCs, etc.); proficiency testing results; calibration and certification records; internal audit reports; external audit reports; corrective action reports; management reports; and regulatory correspondence.

10.6.2 Records related to sample log-in are maintained as described in SOPAD045.

10.6.3 Records related to support equipment calibration and calibration verification are maintained as described in SOPAD013.

10.6.4 Certificates of cleanliness and volumetric accuracy received with consumable supplies (e.g. sample containers, centrifuge tubes) shall be submitted to and maintained by QA. Any other type of certificate that does not have a defined storage location shall be submitted to QA.

10.6.5 Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, dictated observations, and recorded data from automated instruments.

10.6.6 Original copies of records, except those pertaining to analytical data, are maintained by the QA department or Document Control, and access is limited.

10.6.7 Relevant qualifications, training skills, and experience of technical personnel are maintained in the employee's training file.

10.6.8 Records such as transcripts, applications for employment, performance evaluations, etc. are maintained in the personnel files, which are stored in the secured office of the CFO.

10.6.9 The DOC certification statement (FRMAD023), initial method training form (FRMQA004), General Lab Practice Training Form (FRMQA047), and Method Calibration Training Form (FRMQA050) are filed with the workgroup if the DOC was logged-in; otherwise, the DOC package is filed in the method files. An analyst training spreadsheet referencing training dates and documentation locations is maintained on a public drive.

10.6.10 Each employee's legal name, legal signature, and initials are documented on the New Employee Checklist (FRMAD043). The form is maintained in the employee's personnel file, which is stored in the CFO's office. Additionally, employee names, signatures, and initials are documented in a logbook maintained by ACZ's CFO. In the event an employee legally changes their name, the CFO is responsible for garnering new signatures and initials in the logbook; FRMAD043 is not updated in this event.

10.6.11 Each Organic Instrument ICAL data package is scanned to the designated network directory as a PDF and the hard copy stored in labeled boxes. Alternatively, a PDF may be generated directly from the instrument files. ICAL information that needs to be attached to any subsequent workgroup(s) must be printed from the PDF.

10.6.12 Logbooks shall be maintained and controlled as described in SOPAD013.

10.6.13 Project Managers are responsible for maintaining all emails pertaining to a client and/or project. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043).

10.6.14 Changes to electronic records must be traceable to the individual who made the correction, and the reason for the change must be provided. Erroneous entries cannot be destroyed by methods such as overwritten files.

10.6.15 Record Storage and Retention

10.6.15.1 The minimum record retention period of 10 years may be increased dependent upon client request, regulatory requirement, or civil action order.

10.6.15.2 Records stored by a computer must have hard copy or software backup copies.

10.6.15.3 Records stored only on electronic media must be supported by the hardware and software necessary for their retrieval and utilization in the proper format.

10.6.15.4 Records stored on electronic media must be stored in a way to provide protection from electronic or magnetic sources.

10.6.15.5 If there is a change in ownership and/or a change in location, all records and documents will be made available to clients for 10 years. Under no circumstances shall any records or documents be destroyed – all records and analyses performed that pertain to TNI accreditation are subject to inspection by the TNI accrediting authorities for a 5 year period. (The 10 year record retention policy is client driven, TNI standards require records be retained for a minimum of 5 years.) A new owner of ACZ will assume possession of all records and documents.

10.6.15.6 If ACZ goes out of business, all records and documents will be stored and maintained according to protocol in a location to be determined at the time of closure.

10.6.16 Access to Archived Records

10.6.16.1 Access to archived information must be documented with an access log. A log is kept in each storage location, and any person entering a storage location must provide the required information in the log.

10.6.16.2 Hard copy records are stored in a locked environment with limited access. When a record is removed from its location, a "checkout card" must be filled out to indicate who removed the record, the date the record was taken, and a description of the record. The card marks the place in the storage box, and when the record is returned the card is pulled from the box.

10.6.16.3 Any changes to be made to archived electronic data will require assistance from IT to do so.

10.6.16.4 Electronic data that has been archived to removable media is stored in a bank safety deposit box. Access is limited to ACZ's Presidents, IT staff, and CFO and recorded in a logbook maintained by the IT Manager.

10.6.17 Record Disposal

10.6.17.1 Records are disposed of in a manner that ensures client confidentiality.

10.6.17.2 Stored records will be reviewed to determine which ones can be destroyed in compliance with ACZ's record retention policies.

10.7 Computer Data and Records

10.7.1 Network File Server

Computer files pertaining to all aspects of ACZ's business are stored on a series of file servers. To gain access, an employee logs on to the "LAB" domain. Each employee has a unique network user name so that security rules may be enforced. No "guest" logon is permitted. Every employee belongs to a specific "group" and directory security is enforced through privileges granted to these groups. An employee is granted access to files that pertain to their job functions. Other files will be granted read-only or no access as appropriate to the employee's position.

Data generated and reported by ACZ is extremely confidential and the company may be liable for the consequences of the release of this data to any unauthorized person. The implementation of password security is not arbitrary and ensures data is protected and cannot be disclosed to outside parties. Weak passwords that are not changed frequently make this scenario more likely.

In general, the network will prompt employees to change their password every 30 days. The password must be at least five (5) characters. Numeric characters are optional. Passwords may not be shared with other employees, unless necessary for work purposes. The use of another employee's password without permission from ACZ's Presidents, or IT Manager (with the exception of common passwords for shared computers) is grounds for disciplinary action.

10.7.2 LIMS Server

a. Information stored in LIMS consists of all sample and client information needed for day-to-day production activities. The information is stored in an Oracle database. Access is controlled through membership in "groups." Employees may update and change database records according to their job responsibilities. Otherwise, information is restricted to read-only access or no access.

b. No modifications to data can be made through applications not authorized by ACZ's IT department unless a CAR or Help Desk ticket is submitted or documentation is provided on the hardcopy of the workgroup. Unauthorized applications include any form of direct database manipulation.

c. Tracked changes will be audited on a regular basis by the QA department or its designee to ensure sufficient information is being supplied as to why changes occur. The explanations must be both professional and specific.

10.7.3 Docs Server

For general users, access to the docs server is read-only and is permitted through Internet Information Services (IIS) authentication and is logged in IIS log files. Direct access is limited to authorized users or groups who need to bypass the IIS to perform their job duties. The server is updated on a regular basis by automated scripts.

11 ELEMENTS OF QUALITY CONTROL

A critical focus of ACZ's quality control policies and protocols involves monitoring sample preparation and measurement processes to determine matrix effects and to evaluate laboratory performance. Quality control samples are typically analyzed with every batch of environmental samples. Each test SOP provides detailed information regarding quality control sample types, acceptance criteria, and corrective actions, if applicable to the procedure, and reflects the requirements of the method and/or other regulatory authorities.

Performance control samples demonstrate precision or accuracy and expose out-of-control events. Matrix-specific control samples indicate possible effects of the matrix on method performance and may also identify data as in-control or out-of-control. Data that is out-of-control dictates corrective action ranging from re-preparation and re-analysis to reporting data with qualifiers. The corrective action specified in the SOP shall be performed if any quality control sample does not meet the acceptance criteria.

To the extent possible, client samples are reported only if all quality control measures are acceptable. If any measure is outside of the acceptance criteria, and the data will be accepted and reported to the client, then the appropriate data qualifier(s) must be assigned to all associated samples. The list of current extended qualifiers is maintained in the LIMS database.

11.1 Method Performance

11.1.1 Negative Control – Prep Blank (Method Blank)

A prep blank or method blank shall be analyzed at a minimum of one per batch. The blank shall be processed along with and under the same conditions as the associated samples. Method blanks are not applicable for certain analyses, such as pH, Conductivity, Flash Point, and Temperature.

The prep blank is used to assess possible contamination introduced during sample processing steps. A prep blank is prepared using Type I water or other similar matrix that is free of the target analyte(s) and contains all reagents in the same volumes used to prepare the client samples. Unless specified in the test SOP, sample concentration may not be corrected for the prep blank value.

While the goal is to have no detectable contaminants, each prep blank must be carefully evaluated as to the nature of the interference and the effect on the analysis of each sample in the batch. Contamination in the prep blank results from four principle sources: the environment the analysis is performed in; the reagents used; the supplies and apparatus used; and the analyst performing the analysis. Contamination sources vary and the test SOP must be referenced to determine appropriate corrective action.

When method blanks fail acceptance criteria, potential sources shall be investigated and measures taken to correct, minimize or eliminate the problem, and associated client samples must be reprocessed and reanalyzed. Alternatively, data may be reported with the appropriate qualifier if reprocessing and reanalysis is not possible or if one of the following criteria is met:

1) The concentration of a target analyte in the blank is at or above the acceptance limit and the measured concentration of the analyte in an associated sample is greater than 10 times the measured concentration of analyte in the blank.
2) The concentration of the target analyte in the associated sample is less than the MDL.
3) Corrective actions could not be performed or are ineffective. Thoroughly document any corrective action taken and the outcome.

11.1.2 Positive Control (however named)

Laboratory Fortified Blank (LFB), Laboratory Fortified Blank Duplicate (LFBD), Laboratory Control Sample Water (LCSW), Laboratory Control Sample Water Duplicate (LCSWD), Laboratory Control Sample Solid (LCSS), Laboratory Control Sample Solid Duplicate (LCSSD).

1) The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps.

2) The LCS is a quality system matrix, known to be free of the analytes of interest, spiked with known concentrations of analytes. Alternatively, an appropriate Certified Reference Material (CRM) containing the analytes of interest may be used.

3) If no separate preparation method is used (e.g. dissolved metals), an ICV or CCV may double as the LCS. If different acceptance criteria are specified, the most stringent criteria shall be observed.

4) Each test SOP must define the positive control to be used for the procedure, the required frequency, acceptance criteria, and contingencies for corrective action.

5) Unless the reference method specifies a different frequency, the LCS shall be analyzed at a minimum of one per batch, not to exceed 20 environmental samples.

6) Any affected samples associated with a failing LCS shall be re-processed for analysis or the results reported with appropriate data qualification. A failing LCS may be re-tested once to confirm the failure. Additional re-tests must be accompanied by documented corrective action taken between tests. For example, the instrument did not sample from the correct tray position in the first two tests; alignment was corrected for the third test.

   Note: In general, qualification of data for LCS failures is only permitted if there is insufficient sample for re-analysis, the data is extremely time sensitive, or the LCS failed high but the analyte was not detected above the reporting limit in the sample.

7) The components to be included in the LCS shall be as specified by the method, regulation, or as requested by the client. **In the absence of such specifications, the following rules shall be observed (Radiochemistry excluded)** :

   a) For those components that interfere with an accurate assessment, such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike shall be chosen that represents the chemistries and elution patterns of the components to be reported.

   b) For those methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected shall be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked. **However, the laboratory shall insure that all targeted components are included in the spike mixture over a two (2) year period**:

      i. For methods that include one (1) to ten (10) targets, spike all components.

      ii. For methods that include eleven (11) to twenty (20) targets, spike at least ten (10) or 80%, whichever is greater.

      iii. For methods with more than twenty (20) targets, spike at least sixteen (16) components.

8) An LCSW duplicate may be prepared and analyzed with the batch, typically in lieu of a matrix duplicate or spike duplicate. Data is acceptable if the LCSW and/or LCSWD is within the acceptance limits and the RPD passes. Associated samples must be re-prepped and reanalyzed if either of the following occurs:

   a) LCSW/D RPD fails the acceptance criteria specified in the SOP.

   b) % R of both the LCSW and LCSWD is outside the acceptance limits.

9) For a solid or semi-solid matrix, an LCSS and LCSSD are often prepared and analyzed.[4] The data is acceptable if the LCSS and/or LCSSD are within the acceptance limits and the RPD passes. Associated samples must be re-prepped and reanalyzed if any of the following occurs:

   a) LCSS/D RPD fails the acceptance criteria specified in the SOP.

   b) % R of both the LCSS and LCSSD is outside the acceptance limits.

[4] Corrective action for Recommendation #5 cited in the 2002 ADHS audit report.

10) When the acceptance criteria for the LCS are exceeded [i.e. high bias] then any associated client sample with a measured concentration less than the reporting limit (MDL or PQL) may be accepted and reported with the appropriate qualifier.

11) Refer to §11.1.3.3 for additional information regarding data assessment for solid-matrix workgroups prepared with both LCSS/LCSSD and MS/MSD.

12) An LCS is not required for those analytes for which no spiking solution is available.

13) The following apply to radiochemistry only:

    a) The activity of the LCS shall be at least 10 times the Lower Limit of Detection (LLD) or Minimum Detectable Activity (MDA). Note: this requirement does not apply to DOCs.

    b) Whenever possible, the standards used to prepare the laboratory control sample shall be from a source independent of the standards used for instrument calibration.

    c) Where a radiochemical method, other than gamma-ray spectroscopy, has more than one reportable analyte isotope (e.g. plutonium, 238Pu and 239Pu, using alpha-particle spectrometry), only one of the analyte isotopes needs to be included in the laboratory control sample at the indicated activity level. However, where more than one analyte is detectable, each shall be assessed against the specified acceptance criteria.

    d) Where gamma-ray spectrometry is used to identify and quantify more than one analyte, the laboratory control sample shall contain gamma-emitting radionuclides that represent the low (e.g., Am^{241}), medium (e.g., Cs^{137}) and high (e.g., Co^{60}) energy range of the analyzed gamma-ray spectra. As indicated by these examples, the nuclides need not exactly bracket the calibrated energy range or the range over which nuclides are identified and quantified.

    e) The laboratory control sample shall be prepared with similar aliquot size to that of the routine samples for analyses.

11.1.3 Sample Specific Controls

The effect of different sample matrices on the performance of any method can be profound; therefore, matrix spikes, duplicates, and surrogate compounds are analyzed to evaluate matrix effects on data quality. Each SOP includes specific information regarding the usage and evaluation of matrix-specific QC samples and also states the required corrective action to take if any matrix QC fails.

ACZ provides analytical services to numerous and varied clients. Therefore, the possibility of routinely favoring one client or sample is highly unlikely, and over time the samples from all routine sources should be fortified. ACZ recommends that analysts, to the extent possible, select samples to spike or duplicate that are representative of the workgroup. Analysts are not to associate QC with a client sample known to be or believed to be any type of blank or Proficiency Testing sample. Several exceptions exist for selecting samples for spiking or duplicating:

1 A sample is not spiked or duplicated if the volume is inadequate and the client sample and QC sample(s) would require dilution; however, if no other option is available then the client sample and Duplicates should be prepared and analyzed on the same dilution whenever possible. Matrix spikes will not be accepted on different dilutions (minor d.f. variations in soils samples are acceptable) unless no other alternative exists. The data must be qualified in this event.

2 Use the same weights (as close as possible) to prepare duplicates of solid matrix samples.

3 A client may require that one or more of their samples be spiked or duplicated. A “RUN QC” comment is added when the sample is logged in to notify the analyst that QC must be performed for a specific sample or project. Alternatively, the Special Instructions function in ACZ’s LIMS may be used to communicate the request. If a client requests that their sample(s) be spiked or duplicated, ACZ will accommodate the client for a fee.

4 A reactive sample is unpredictable and is a poor choice for spiking or duplicating.

5 A PT sample is not a real-world sample and is a poor choice for spiking or duplicating, because the data does not provide any useful information about possible matrix effects. When selecting samples for batch QC such as spikes or duplicates, PT samples should be avoided. If insufficient volume exists to spike or duplicate any other samples in the batch, it is appropriate to select a PT sample. It is better to use the PT sample for a duplicate than a spike if this choice is presented. If a batch consists solely of a PT sample, QC designed to assess matrix effects is not required (e.g. spike, SDL); an assessment of precision is still required and may be accomplished by duplicating the PT sample, or preferably, running a duplicate of the positive control.

11.1.3.1 Surrogates

Surrogates are organic compounds that are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but are not normally found in environmental samples. Surrogates are included in the scope of Organic methods and are used to evaluate accuracy, method performance and extraction efficiency and are added to environmental samples, controls, and blanks, in accordance with the method requirements.

When surrogate recoveries fail acceptance limits, corrective action stated in the test SOP shall be performed. If corrective action cannot be performed or is ineffective, reported data must be appropriately qualified.

11.1.3.2 Matrix Spike Samples

A matrix spike sample (however named) is used to determine the level of bias (accuracy) associated with a particular matrix. For the purposes of this document, “MS” designates a matrix spike, and “MSD” designates a matrix spike duplicate. Spikes are prepared by adding a known and appropriate quantity of each target analyte to a replicate aliquot of client sample.

The required analytical frequency is specified by the method or other regulating entity and is indicated in the test SOP. Each result is evaluated against the acceptance criteria, and matrix effects are determined and reported to the client. The following evaluation criteria apply to spikes that are subjected to processing steps and post-digestion spikes (analytical spikes).

- Percent Recovery (%R) is considered for all spikes.
- %R is evaluated only if the theoretical concentration in the spiked aliquot is greater than or equal to the PQL; otherwise, each associated client sample must be reported with the appropriate qualifier, regardless of %R, unless a representative number of analytes as described in §11.1.3.2.1 are evaluated for %R.
- If %R for the MS and/or the MSD is outside of the acceptance limits, the RPD passes, and all other pertinent prep and instrument QC passes, each associated client sample may be accepted and reported with appropriate qualification.

  11.1.3.2.1 The components to be included in the MS & MSD shall be as specified by the method, regulation, or as requested by the client. **In the absence of such specifications, the following rules shall be observed (Radiochemistry excluded)**:

  1) For those components that interfere with an accurate assessment, such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike shall be chosen that represents the chemistries and elution patterns of the components to be reported.
  2) For those methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected shall be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked. **However, the laboratory shall insure that all targeted components are included in the spike mixture over a two (2) year period**:

a) For methods that include one (1) to ten (10) targets, spike all components.

b) For methods that include eleven (11) to twenty (20) targets, spike at least ten (10) or 80%, whichever is greater.

c) For methods with more than twenty (20) targets, spike at least sixteen (16) components.

11.1.3.3 Matrix Duplicates and Matrix Spike Duplicates

The matrix-specific precision associated with an analysis is determined through the use of a matrix duplicate (DUP) or spike duplicate (MSD), which are performed at a frequency specified by the method or other regulating entity (refer to the specific test SOP). If the method does not prescribe a frequency, a duplicate shall be included in each workgroup, not to exceed 20 samples. The results are evaluated, and the matrix affect on precision are determined and reported to the client.

- Relative Percent Difference (RPD) is used to evaluate precision, unless the test SOP specifies a different technique (§12.4.6).

- RPD for a spike duplicate is evaluated only if the observed concentration is greater than or equal to the PQL; otherwise each associated client sample must be reported with the appropriate qualifier.

- RPD for a matrix duplicate is evaluated only if the observed concentration is greater than 10 times the MDL or 2 times the PQL if an MDL has not been established; otherwise each associated client sample must be reported with the appropriate qualifier, regardless of RPD.

- In the absence of other contributing factors, a DUP failure for a solid or semi-solid matrix is attributed to non-homogeneity of the sample, and each associated client sample may be reported with the appropriate qualifier.

- For an aqueous matrix, if the DUP fails then all associated samples must be retested. If permitted by the instrument software the sample and DUP can be reanalyzed at the end of the analysis in lieu of retesting all associated samples.

- For an aqueous matrix, if the MS/MSD RPD fails then the associated samples must be reanalyzed. If permitted by the instrument software the sample and MS/MSD can be reanalyzed at the end of the analysis in lieu of retesting all associated samples.

- If applicable, evaluate the LCS/LCSD if the RPD fails for a matrix duplicate or spike duplicate. Each associated client sample may be reported with the appropriate qualifier if the LCS/LCSD meets the criteria stated in §11.1.3.2.

- For a solid or semi-solid matrix, if both the LCSS and LCSSD recoveries pass but the RPD fails, then acceptable precision may be demonstrated by a passing RPD for the MS/MSD, and each associated client sample may be reported with the appropriate qualifier.

- A sample and duplicate may only be re-analyzed once before additional corrective action is required. If more than one re-analysis is performed, the workgroup documentation must include justification.

11.2 Instrument Specific Controls

All data must be associated with a passing instrument calibration and initial calibration verification. To the extent possible, all data must be associated with passing continuing calibration verification. If the initial calibration verification results (ICV/ICB) are outside of the acceptance criteria, then the source of the failure must be

identified, necessary corrective action performed, and the instrument recalibrated if necessary before proceeding with sample analysis.

If the continuing calibration verification results (CCV/CCB) do not meet the acceptance criteria, then the source of the failure must be identified and corrective action performed, including recalibration if necessary, before continuing with sample analysis. If reanalysis of any sample associated with failing calibration verification is not possible and results will be reported, the data shall be appropriately qualified.

For instruments that permit the analysis of subsequent workgroups using the most recent calibration, two (2) consecutive attempts of the opening CCV/CCB are allowed. If both attempts fail to produce acceptable results then the sources of failure must be identified and corrective action performed, including recalibration if necessary, before commencing sample analysis.

If a CCV or ICV *retest* fails and the instrument is not recalibrated, 2 consecutive passing CCVs or ICVs are required before continuing with analysis.

Unless stated otherwise by the test SOP, passing calibration verification must bracket all batch quality control samples, and results for additional instrument check standards, if applicable, must be within the acceptance criteria stated in the SOP. However, when the acceptance criteria for a CCV or CCB are exceeded (i.e. high bias) any associated client sample with a measured concentration less than the MDL may be accepted and reported with the appropriate qualification. This exception is not allowed if the workgroup contains a batch LCS (however named) which fails low.

11.3 Other Control Indicators

11.3.3 Internal Standards

Internal Standards (IS) are measured amounts of certain compounds added after preparation or extraction of a sample to be analyzed. The IS is an analyte not likely to be found in the environment and is used in a calibration method to correct sample results affected by column injection losses, purging losses or viscosity effects. The IS is added to client samples, controls and blanks in accordance with the method requirements. When the results are outside of the acceptance limits for applicable quality control samples, corrective actions shall be performed. Once system control has been reestablished, all samples analyzed while the system was malfunctioning shall be reanalyzed. If corrective actions could not be performed or are ineffective and associated sample results will be reported, the data must be appropriately qualified.

11.3.2 Trip Blank

The trip blank is a sample container filled in the laboratory with Type I water that is shipped to the collection site in the sample cooler, returned to the laboratory, logged-in, and analyzed in the same manner as the client samples. With the exception of Hg-1631, trip blanks are not opened in the field.

11.3.3 Instrument Blank

The instrument blank is an aliquot of Type I water processed only through the instrument steps of sample analysis and is used to determine presence of instrument contamination. For Organic instrument methods, neither surrogate nor IS standards are added.

11.3.4 Equipment Blank

An equipment blank is provided by the client and is used to assess the effectiveness of equipment decontamination procedures. Type I water is poured into (or over) or pumped through the sampling device, collected in a sample container and transported to the lab to be analyzed for all parameters requested for the environmental samples collected at the site.

11.3.5 Ambient Blank

The ambient blank consists of Type I water poured into a VOA vial at the sampling site (in the same vicinity as the associated samples). It is handled like an environmental sample and transported to the laboratory for analysis. Ambient blanks are prepared when samples are to be analyzed for VOA analytes and are used to assess the potential introduction of contaminants from ambient sources (e.g. active runways, engine test cells, gasoline motors in operation) to the samples during sample collection. The frequency of collection for ambient blanks is specified in the client's field-sampling plan. Ambient blanks are not required for all projects.

11.3.6 Radiological Tracers & Carriers

Radiological tracers and carriers are used for radiological analyses. The control reacts in the same manner as the target isotope and is used to assess analyte recovery. The tracer is added to client samples and QC in accordance with the requirements stipulated in the test SOP. Because the tracer recovery has a direct impact on the LLD, the recovery must be high enough to yield LLDs that are within the scope of the project or meet ACZ's acceptance criteria. Refer to the test SOP for evaluation criteria and corrective action(s) for out-of-control tracer recovery.

11.4 Titrants – Where applicable, test SOPs shall include procedures for verifying the concentration of titrants prepared by the laboratory. Verification is not required for purchased titrants with certified values. If a purchased titrant is diluted, verification is required.

12 EVALUATING QUALITY CONTROL SAMPLES

In general, acceptance criteria for quality control samples are method-specific; however, compliance with the requirements of clients and regulatory or other accrediting agencies must also be demonstrated. Immediate corrective action must be taken if any quality control is outside of the acceptance criteria. Appropriate corrective actions are described in the test SOP. To the extent possible, client samples are reported only if all quality control measurements are acceptable. If a quality control measure is outside of acceptance criteria, and the data will be reported, then all samples associated with the failed QC must be appropriately qualified. Clients will occasionally request limits different from those in a published method. Deviations from ACZ's policies pursuant to client request must be explicitly noted on client reports. ACZ will not be held liable in the event such deviations do not meet client regulatory needs.

Unless otherwise stated, for the purpose of determining conformance to specifications, ACZ employs the rounding method described in ASTM E29. When using this method, observed values are rounded to the same decimal place limits are expressed before assessing conformance. For example, if the calculated percent recovery for an LCS is 89.5% and the QC limits are 90 to 110%, the percent recovery would be rounded to 90% and evaluated as passing. (Note: double rounding is NOT permitted, e.g. 89.48 rounds to 89.5 rounds to 90.) Conversely, if limits were expressed as 90.0 to 110.0%, the same LCS would be evaluated as failing acceptance criteria. Analysts must consider whether the QC limits expressed in the test method cohere in a technically sound manner with the rounding method. If they do not, the SOP must express the limits to a technically sound numerical place value, or the absolute method must be employed. The absolute method takes a limit of 6˚C, 6.0˚C, and 6.000˚C all to mean the same thing, exactly 6 degrees Celsius.

For methods that permit the use of control charts or do not specify acceptance criteria for quality control measurement, limits may be generated by plotting historical data obtained from analytical processes considered in control. Whenever practical, a minimum of 20 data points is used. The process of rejecting data points relies heavily on the statistician's judgment and control chart activities are therefore restricted to supervisors and experienced analysts. All points must be associated with passing calibrations and calibration verification(s). Data points with known anomalies must be rejected. Data points should not be rejected solely because they fail acceptance criteria. Control chart documentation must clearly indicate rejected data points. ACZ's LIMS has a utility for querying and retrieving historical data for control chart applications. Control chart limits are typically set at ± 3σ. All control chart limits are reviewed and approved by the QA department prior to implementation. When possible, a comparison to previous limits is included in the review and may form the basis for rejecting new limits and requiring an investigation of the analytical system's condition. Previous limits are archived in a network folder. Default acceptance criteria established by the Arizona Department of Health Services (ADHS) may be used in lieu of generating a control chart to establish limits; however the SOP must specify which limits are in use. [5] **NOTE:** For all data evaluation, final results ending with 1 – 4 are rounded down and results ending with 5 – 9 are rounded up.

12.1 Accuracy

Accuracy is defined as "The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations". Control samples (LCS or LFB) and spiked samples are analyzed with every batch of samples or as stipulated by the specific test SOP to assess accuracy and matrix effects.

- Percent Recovery (%R) for a control sample is calculated as follows:

$$\%R = \frac{M}{S_p} \times 100$$

Where: M = Measured concentration of the control sample

S_p = True value of the control sample

[5] Arizona Administrative Code (A.A.C.), Title 9, Ch. 14, Table 6.4 (September, 2016)

- Percent Recovery (%R) for a spike is calculated as follows:

$$\%R = \frac{M - S}{S_p} \times 100$$

Where: M = Measured concentration of the spiked sample
S = Measured concentration of the sample aliquot
S_p = True value of the spike concentration

12.2 Precision

Precision is defined as "The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms." Matrix duplicates and spike duplicates are analyzed with every batch of samples or as stipulated by the test SOP to determine the precision associated with the analysis. If any method does not specify acceptance criteria for the RPD, then default criteria of RPD $\leq$ 20 is used (a value that rounds to 20 is acceptable). [6] The Relative Percent Difference (RPD) as an absolute value is calculated as follows:

$$|RPD| = \frac{(S - D)}{[(S + D) / 2]} \times 100$$

Where: S = Sample Value
D = Duplicate Value

12.3 Other Calculations

- Solids Dilution Factor:

$$\text{Dilution Factor} = \frac{V}{(W)(\%\ \text{solid})}$$

Where: V = Final digestate volume, in mL
W = Sample weight used, in g
%solid = %solid of the sample as a fraction

- Sample Concentration for Solids:
  - wet weight [biota tissue, fruit or vegetable matter, etc.]: $mg/Kg = \frac{DF * C * V}{W}$
  - dry weight [plant matter, grasses, soil, sludge, etc.]: $mg/Kg = SF * C * DF$

Where: DF = instrument dilution factor
C = raw data value, in mg/L
V = Final volume of digestate, in L
W = sample (as received) weight used, in Kg
SF = soil dilution factor

- Percent Difference for Serial Dilution (SDL):

$$|\%D| = \frac{[I - (s * 5)]}{I} \times 100$$

Where: I = initial sample result
s = serial dilution result (raw data value)

For SDL calculations in LIMS, "s" is multiplied by 5 and the resulting "reg value" is compared to the "found value" to calculate %D.

[6] ADHS Information Update #87 (July 7, 2005)

12.4 Radiochemistry Calculations: (**NOTE**: Specifications in the individual test SOPs supercede the information detailed below.)

12.4.3 Activity

The results of radioactivity are typically reported in terms of activity per unit volume or mass. Units are normally expressed in picocuries (pCi), which equal 2.22 disintegrations per minute (dpm). Specific formulas to determine activity are in the SOP for each method. The general formula is as follows:

$$C = \frac{R_{net}}{(e)(y)(i)(v)(u)}$$

Where: C = activity per unit volume (pCi/L)
R_{net} = net counts per minute
e = counting efficiency, cpm/dpm
y = chemical yield
i = ingrowth correction factor
v = volume or mass being counted (L)
u = units correction factor, 2.22 for cpm to pCi

12.4.4 Counting Error

Radiochemical data are considered incomplete without reporting associated random and systematic errors. For this reason all radiochemical results should be accompanied by a counting error at the 95% confidence level (1.96*standard deviation). The general counting error formula is as follows:

$$E = \frac{1.96\,(R_o / t_1 + B / t_2)^{1/2}}{(e)(y)(i)(v)(u)}$$

Where: E = counting error
R_o = gross sample, cpm
t_1 = sample count duration, min
B = background, cpm
t_2 = background count duration, min
e, y, i, v, and u are as previously defined.

12.4.5 Lower Limit of Detection (LLD)

LLD (also referred to as Minimum Detectable Activity or MDA) is considered the smallest quantity of sample radioactivity that will yield a net count for which there is a pre-determined level of confidence that radioactivity is present. At the 95% confidence level, the following equation calculates the LLD for any single nuclide. The calculation uses the standard deviation for the background counting rate, assuming the sample and background counting rates should be very similar at the LLD. A formula for determining LLD is as follows:

$$\mathrm{LLD}_{95} = \frac{4.66\,S_b}{(e)(y)(i)(v)(u)}$$

Where : LLD_{95} = Lower limit of detection at the 95% confidence interval
S_b = Standard deviation of the instrument background counting rate, cpm
e, y, i, v, and u are as previously defined

12.4.6 Precision

The normalized absolute difference, or Replicate Error Ratio (RER), between the sample and the laboratory duplicate, given by the following equation shall be used to determine that results do not differ significantly when compared to their respective 2* sigma uncertainty.

$$RER = \frac{\left| Sx - Dup \right|}{\sqrt{(Sx_{error})^2 + (Dup_{error})^2}} \leq 2.0$$

Where: Sx = sample concentration in pCi/L
Sx_{error} = sample counting error (in pCi/L) at the 95% confidence level.
Dup = duplicate concentration in pCi/L
Dup_{error} = duplicate counting error (in pCi/L) at the 95% confidence level.

NOTE: For Radchem samples, both RPD and RER may be used to evaluate precision. RPD is the default assessment for Drinking Water samples; RER is the default assessment for non-Drinking Water samples. Data for both RER and RPD are uploaded to LIMS for all analyses. Use the following guidelines to correctly assess precision. Further details are provided in ACZ's Wiki and should be consulted to ensure data for each workgroup is correctly evaluated. Go to LabWeb \ Wiki \ Analytical Departments \ Radio Chemistry.

Drinking Water:

RPD $\leq$ 20, RER < 2.0 – Precision is judged to be in control
RPD $\leq$ 20, RER > 2.0 – Precision is judged to be in control;
RPD > 20, [sx] < 5x [LLD], RER < 2.0 – Precision is judged to be in control; qualify data.
RPD > 20, [sx] > 5x [LLD], RER> 2.0 – Precision of the prep batch is questionable.
RPD > 20, [sx] > 5x [LLD], RER < 2.0 – Precision of the prep batch is questionable.

Non-Drinking Water:

RER < 2.0, RPD $\leq$ 20 – Precision is judged to be in control.
RER < 2.0, RPD > 20 – Precision is judged to be in control;
RER > 2.0, RPD $\leq$ 20 – Precision of the sample prep batch is questionable.
RER > 2.0, RPD > 20 – Precision of the sample prep batch is questionable.
RER > 2.0, [sx] > 5x [LLD], RPD $\leq$ 20 – Precision is judged to be in control; qualify data.

13 VALIDATION AND REVIEW OF ANALYTICAL DATA

ACZ has a responsibility to provide the best data possible to ensure our clients can make sound and cost-effective decisions regarding public health and the environment. In order to generate and report reliable data, the analytical systems used need to be properly functioning, and the review process must be conducted in a manner that is logical and reasonable and would be defensible if subjected to legal scrutiny. Decisions regarding data quality must be backed by good science and sound professional judgments.

The entire validation and review process encompasses more than solely evaluating the final results for client and quality control samples. To this extent, the necessary steps must also be performed *prior* to sample preparation or analysis to ensure the quality of the data. Following sample analysis, data is uploaded to the LIMS database and then submitted to a variety of process chains such as calculations, rounding, application of qualifiers, etc. A multi-level data review process is utilized to verify the uploaded analytical data meets all documented ACZ requirements as well as any client-specific quality objectives. At a minimum, the validation process must include the following steps, as applicable:

- Monitor the expiration dates for all stock, intermediate, and working standards, reagents, and chemicals.

- Prior to analysis, determine that holding times have not been exceeded. Unless otherwise specified by the test SOP, sample preparation and analysis must be completed within the holding time.

- Prior to analyzing samples, verify the correct set-up and operation of the instrument or equipment. Perform calibration, maintenance, and optimization as necessary to ensure proper functioning.

- QC Association
  1) In general, for QC frequency of 1 per10 or less client samples, the first set of QC is associated with samples 1 – 10. If there are fewer than 20 samples in the workgroup, then the remaining client samples are associated with the second set of QC.
  2) If sample characteristics or amount dictate that 2 of the first 10 samples be spiked or dup'd, then the first spike or DUP is associated with samples 1 through 11 excluding the 2nd sample spiked or dup'd, and the 2nd spike or dup is associated with itself and samples 12-20. For example, if samples 3 & 5 are spiked in a 20 sample batch, sample 3 is associated with 1-4 & 6-11, and sample 5 is associated with 5 and 12-20. The same principle applies if both spiked or dup'd samples reside in the 2nd set of ten within the workgroup sequence.
  3) Variations to the QC association rules noted above are permitted but must be documented with the WG. The documentation must define the altered QC association and provide a compelling, technically sound reason for the deviation. QC association may **not** be changed after data has been acquired.
  4) QC association must be properly defined in LIMS.

- Before completing workgroup creation, verify the correct PCNs and/or SCNs have been entered. Percent recovery for control samples and spikes is calculated using the information in LIMS for each.

- Verify the proper sub-sample (green dot, yellow dot, etc.) is being used for preparation or analysis.

  - Notify the supervisor or Production Manager as soon as possible if a sample cannot be located.
  - Document on the bench sheet if a sub-sample other than the type indicated in the SOP is used.

- Clearly label tubes, beakers, autosampler cups, etc. to identify the sample (and dilution factor, if applicable).

- Manage sample volume to ensure all analyses from a bottle type can be completed.

- Document all dilution factors at the time the dilution is performed.

- Record complete and accurate observations when an analysis, sample preparation, or sample matrix is unusual or problematic.

- Ensure transcription errors do not occur. Verify all data manually entered into LIMS is correct before completing the upload process.

- The calibration workgroup must be associated with all subsequent workgroups. Record the calibration workgroup number (or calibration file name) on the data review checklist.

- Provide complete traceability for all standards and reagents used for sample preparation and analysis.

- Batch quality control samples must be treated in the same manner as client sample, including preparation.

- If it is necessary to perform a calculation manually, use the values in the raw data [do not truncate] and then round the final result to the appropriate numerical place value. If the final result passes the acceptance criteria then pass the QC in LIMS and note on the data review checklist that it passes.

- LIMS performs several additional QC calculations on the approved data including cation/anion balance (CAB) checks, calculated TDS versus actual TDS ratios, and Total versus Dissolved ratios. The Project Manager may update the status of pertinent samples to REDO if one of these calculations indicates a discrepancy with the associated data.

- If two attempts fail to produce acceptable data then notify the supervisor or Production Manager before taking further action. It may be necessary to first determine if a larger problem is interfering with the analysis. Investigate the problem before qualifying the associated data.

- If there is an indication the analytical system is out of control, the issue must be investigated. Notify the supervisor immediately. Conduct troubleshooting in a systematic, organized manner.

- All data must be reviewed initially in LIMS [AREV] by the analyst who performed the analysis or another individual authorized to perform or AREV the procedure. The department supervisor or another individual authorized for SREV performs the secondary review [SREV]. The following are data review guidelines:

  1 A data review checklist must be completed during the review process. Verify all items listed and note any errors, problems or non-compliances and the corrective action(s) taken.

  2 If applicable, review the raw data to verify the analytical system was in control and to ensure no anomalies exist. Check for notes on the bench sheet regarding the preparation or analysis.

  3 For client samples and quality control samples, ensure all results are within the measurement range and are bracketed by a passing calibration and passing calibration verification [ICV/ICB or CCV/CCB]. Sample values outside of the measurement range must be appropriately qualified if reanalysis is not possible.

  4 The corrective action specified in the SOP must be performed if any quality control sample does not meet the acceptance criteria.

  5 Data generated after the hold time has elapsed may not be usable for the client. If reprep or reanalysis will be conducted outside of the holding time, check first with the supervisor.

  6 Confirm all dilutions are appropriate. A reasonable explanation must be provided on the bench sheet if a sample was diluted and the value is less than the quantitation limit (refer also to §15).

  7 If a spike fails, determine if the sample concentration is disproportionate to the spike added. If the analyte concentration in the sample is more than **4 times** the spike concentration, note the failure on the checklist and appropriately qualify the associated samples.

8 If a spike recovery suggests the sample was not spiked, matrix interference must be confirmed prior to qualifying samples. If matrix interference cannot be confirmed, then re-prep and/or re-test all associated samples.

9 Each associated client sample must be appropriately qualified if the matrix spike, matrix duplicate, or spike duplicate data cannot be used for validation purposes.

10 Confirm failed QC by verifying the correct PCN or SCN was entered. Make corrections if necessary before proceeding with data review.

11 Verify all assigned qualifiers are appropriate. Does use of a particular qualifier make sense? Could data be defended using the qualifier assigned to the scenario or problem?

12 If a case narrative is necessary, the reason for accepting and reporting the data must be sound and logical. Provide sufficient and accurate verbiage to ensure the data is legally defensible.

13 If a sample was retested in the same workgroup, verify the correct data will be reported. All other data for the sample must be failed; LIMS cannot report multiple datum for the same sample-product-analyte combination.

14 Confirm all samples have the correct status (PASS, FAIL, REDO, REDX) before completing the review process. For multi-parameter workgroups, all analytes must have the correct status.

15 Refer also to §11 for data evaluation criteria.

14 DETECTION LEVELS

Current practice identifies several detection levels, each of which has a defined purpose: Instrument Detection Limit (IDL), Method Detection Limit (MDL), Reporting Limit (RL), and Practical Quantitation Limit (PQL). The MDL and PQL are stated in each test SOP and are adjusted accordingly in LIMS when data is uploaded to reflect the use of smaller sample volume (dilution) or larger sample volume (concentration).

14.1 Instrument Detection Limit (IDL)

The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 10
replicate measurements of the calibration blank signal at the selected analytical mass. (EPA Method 200.8 definition.)

14.2 Method Detection Limit (MDL)

The EPA defines the MDL as the "minimum concentration of substance that can be measured by a specific testing protocol and reported with 99% confidence that the analyte concentration is greater than zero…" This confidence interval means that any substance detected at a concentration equal to the MDL is 99% likely to be present, but it also means there is a 1% chance that the substance will be considered falsely present (false positive). The MDL procedure is designed so that the probabilities of both false positive and false negative errors are acceptably small; however, the procedure has limitations. Data users must understand the limitations when evaluating low level data and must proceed with caution when interpreting data reported between the MDL and PQL in order to minimize the risk of making poor environmental decisions.

MDLs are dependent on variables (temperature, instrument conditions, analysts, matrix, etc.) and are typically determined by processing, preferably over the course of several days, at least seven individual replicates of a fortified blank sample through the method's preparation and analytical schemes. MDLs determined for the same method / matrix / technology must be compared to ensure they are in agreement.

ACZ maintains a current MDL for each applicable method. A qualitative verification of the MDL must be performed annually for each applicable method, analyte, instrument, and matrix and before a new instrument or method is utilized for client samples. Refer to ACZ's SOP *Demonstration of Capability & Method Detection Limit Studies* (SOPAD001) for additional information.

14.3 Practical Quantitation Limit (PQL)

The PQL represents the lowest quantitative level that can be reported with a specified degree of confidence. Data reported at or above the PQL is considered reproducible, allowing for comparison of analytical results over a relatively long period of time, which is important to the monitoring of environmental data. ACZ *typically* defines the PQL as a value 2 – 10 times the MDL with an accuracy of 70 to 130% in a matrix free of interferents. The low calibration standard shall be at or below the PQL. Reported values less than the PQL are qualified as estimated. The region between the MDL and PQL is a continuum of uncertainty, lacking distinct cutoff points, and the error below the PQL is increased to the extent that the statistical validity of the result is questionable.

15 SAMPLE DILUTIONS

Sample dilution may be necessary for one or more of the following reasons: (1) sample concentration exceeds the established measurement range of the procedure/method; (2) sample volume or material is limited; (3) matrix interference is indicated or suspected; (4) sample matrix is reactive; (5) aqueous sample contains high sediment; (6) color, odor or other physical characteristics are present; (7) For ICP and ICPMS, TDS is greater than 2000 mg/L. In all cases, the analyst must use good professional judgment when determining the most appropriate dilution. Whenever possible, prepare and analyze client samples and any complimentary duplicates or spikes on the same dilution.

For samples that contain high concentration of analyte(s), the analyst will use their knowledge of the measurement range of the procedure to determine an optimal dilution that yields quantifiable data with minimal error propagation. In general, prepare the dilution so the final concentration is near the mid-point of the measurement range. A sample must be retested on a smaller dilution if analyte concentration is less than the reporting limit; exceptions must be explained on the bench sheet. For multi-parameter analyses, it may not be practical to report all analytes within the desired range, and the analyst must use their best judgment when determining a reasonable dilution factor.

The following requirements pertain to all dilutions:

- Document all dilution factors when the dilution is performed.
- Assign the appropriate “D” qualifier if data for the diluted sample is less than the quantitation limit
- Retest sample on smaller dilution if the result is less than the quantitation limit (or document justification for accepting the data on the bench sheet or data review checklist)
- Document the reason for any dilution on the bench sheet [not required for sample values that exceed the measurement range of the procedure]

16 ERROR CORRECTION PROTOCOL

When an error occurs in any type of record it must be crossed out with a **single line**. The error must not be erased, deleted, overwritten, obliterated, or made illegible. Alterations to make data legible are considered error corrections. The correct value must be entered alongside. All changes to hard copy records must be initialed and dated by the person making the correction. [7]Under no circumstances may White-Out® or any other substance be used to conceal data. Concealing or improperly altering data is fraudulent and may be grounds for termination from ACZ. Equivalent measures must be taken to avoid loss or change of original data in the case of records stored electronically. Refer to §10 for details of corrections made to electronic records. The following is an example of proper error correction:

fleece BWC 10-20-06
Mary had a little lamb, it's ~~feet~~ as white as snow. And everywhere that ~~Lary~~ went, the lamb was sure to go.
Mary BWC 10-20-06

[7] There is one exception to this rule. Client identification may be obliterated from a record if it’s presence compromises client confidentiality (e.g. client ID is mistakenly entered in a logbook). In this event, the rationale for obliteration must be clearly stated and initialed and dated by the person making the correction.

17 COMPUTER / AUTOMATED PROCESSES

ACZ employs its proprietary LIMS (Laboratory Information Management System) to acquire, record, process, store, and archive data. LIMS is the primary application for all employees and encompasses the combination of hardware and software throughout the entire facility. Tasks performed with LIMS include but are not limited to creating workgroups, reviewing data, and generating client reports. ACZ implements the defined standards of Good Automated Laboratory Practices (GALP) to establish a uniform set of procedures to assure that all LIMS data used by our clients is reliable, credible, and legally defensible.

17.1 Software

The software used to achieve GALP goals is a combination of industry standard commercial off the shelf (COTS) software and internally developed applications. COTS software is purchased through professional and well-developed companies such as Oracle, Microsoft, and Lab Vantage Systems that complete sufficient testing and quality control to assure their products function properly. Internal applications undergo testing before being implemented and distributed throughout the laboratory.

Electronic records are protected, backed up, and archived to prevent unauthorized access or amendment. Refer to §10 of this document and ACZ's SOP *Backup and Archive of Instrument Data Files* (SOPAD044) for details.

17.2 Hardware

ACZ deploys many servers using industry standard architecture. All servers run standard enterprise operating systems such as Microsoft Windows Server and SuSE Linux. All data residing on network servers is routinely backed up.

To the extent possible, instrument PCs comply with at least the minimum recommendations of the instrument manufacturer and they are connected to ACZ's network. This allows transparent backup and access to computers by system administrators.

17.3 Security

GALP security is controlled through a set of passwords. A log-in name and password are required to access ACZ's network. User passwords must be at least five characters and must be changed when the user is prompted. Each user has a given set of network rights and is restricted to software necessary to complete their job functions as well as his/her own documents. Refer also to §10.7.1 for additional information.

A firewall protects the network from internet traffic. The only traffic permitted access to the internal network is protocols approved by ACZ such as IMAP, SMTP and HTTP. Incoming and outgoing E-mails are scanned for viruses and content. Email that fails this automated scan is quarantined for further review. Web traffic that is potentially harmful or inappropriate is automatically blocked by ACZ's proxy server.

17.4 Electronic Signatures

ACZ permits the use of electronic signatures to approve documentation produced by the laboratory and to enter contractual agreements. Electronic signatures meeting the following criteria are considered equivalent to a handwritten signature:

(1) Each electronic signature shall be unique to one individual and shall not be reused by, or reassigned to, anyone else.
(2) Signing shall be password protected.
(3) Signatures shall be embedded with a timestamp.
(4) Faxed handwritten signatures are considered equivalent to a wet signature unless proven unreliable.
(5) E-documents that contain contractual commitments must be signed by an officer of the company.

18 CLIENT SERVICES

18.1 Contracting Services

ACZ's sales representatives and project managers are responsible for reviewing requests, preparing quotes, and entering contractual commitments with clients. Prior to accepting new work, it must be verified that the laboratory has appropriate facilities and resources to meet client needs. To the extent possible and pragmatic, ACZ shall use the latest valid edition of a standard. This is dictated largely by what ACZ's accrediting authorities will issue certification for. Where an older standard is universally recognized by ACZ's accrediting authorities but the latest is not, ACZ will typically use the older standard. As necessary, sales representatives and project managers must collaborate with ACZ's Production Manager, QAO, and/or technical directors to evaluate laboratory capacity, capability, and resources. Refer to SOPAD043 for additional details.

18.2 Subcontracting

ACZ utilizes subcontract labs to perform analyses for various reasons. A subcontracted lab must meet the clients DQOs and laboratory certification requirements for the subcontracted analysis. When applicable, ACZ advises its clients in writing of its intentions to subcontract any portion of the testing to another party. Any non-accredited tests shall be clearly identified as such to the client. ACZ scans this report as an attachment to be included as part of ACZ's final report. A comment is added to ACZ's final report indicating which subcontracted laboratory performed the analyses, if the name is not indicated on the attachment. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043) for additional information.

18.3 Data Reporting

Once all analyses and the entire review process have been completed, a client report is generated and submitted for final validation by the Project Manager. If necessary, a case narrative is written describing the details of the project and any non-conformances or other relevant issues. The PM electronically signs the report, and the Document Control department sends the report to the client in an electronic format. At a minimum, the following information appears on an ACZ analytical report:

Client Name	Sample Matrix
Client Address	Parameter/Analyte
Client Contact	Method Reference
Lab Sample ID	Result
Client Sample ID	Units
Client Project ID	LIMS Qualifier (U, B, J, H)
ACZ Report ID	MDL or LLD
Date/Time Sampled	PQL
Date/Time Received	Analyst's Initials
Date/Time Analyzed	Extended Qualifiers (as separate page)

A complete electronic data package contains the analytical reports, the external chain of custody records, sample shipping documentation, and any other relevant project information. Department Reference Sheets explaining acronyms, qualifiers, and method references are also included. All of these documents are an integral part of the final data package and must always be viewed as a whole. To prevent the separation of reports, each page identifies the project number, the sequential page number, and the total number of pages in the data package. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043) for more detail.

If requested by a client, custom and standard Electronic data deliverables (EDDs) are generated by the Document Control department. These deliverables, containing data in client specified format, are sent by e-mail with the client report. EDDs and analytical reports access data from the same Oracle tables, thus eliminating the possibility of inconsistent results. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043) for more detail.

Results may be reported in a simplified manner for internal customers or in accordance to a written agreement with a customer.

18.3.1 ACZ Report Packages

ACZ provides different levels of data packages based on client request. ACZ defines the different levels as follows:

Level 2: Standard analytical reports

Level 3: Standard analytical reports; Electronic Data Deliverable (EDD); Standard QC summary

Level 4A: Standard analytical reports, Extended QC Summary (standard QC plus calibration verification checks, interference checks and serial dilutions) EDD, raw data and run logs. This package can be provided either on a disk or in a full paginated data package with the raw data

Level 4B: "CLP like" data package: CLP like forms 1-12; Run Logs and raw data incorporated into the full paginated data package.

NOTE: Surcharges apply for non-standard reports.

18.4 Data Confidentiality

ACZ has an obligation to each client to maintain custody of samples, data, and reports and to keep all data or other information confidential. To uphold this responsibility, ACZ retains custody of the information at all times – data or other client information obtained by ACZ is not allowed to leave the premises. This includes but is not limited to Chains of Custody, raw data, workgroups, run logs, logbooks, reports, QC summaries, data packages and other media containing data. Client data cannot be released to anyone except the client (as directed on the Chain of Custody) or the client's designated representative, and project data, including any client information, is not to be discussed with anyone other than ACZ employees and/or the client without first receiving written permission from the client. Additionally, client-specific information is not to be documented on raw data, workgroups, logbooks, or other records that may be provided to any client as part of an extended data package. All information must be referenced using only the ACZ log-In number. Refer to ACZ's SOP *Data Integrity Principles and Policies* (SOPAD039) for additional details of policies pertaining to confidentiality.

External access to the ACZ network is limited to employees that may need to access information remotely. Employees requiring such access use ACZ's Virtual Private Network (VPN). The VPN client is setup on the employee's computer so that it adheres to ACZ security standards. These standards include (1) a unique user name (2) a password with at least 12 characters, and (3) 128 bit encryption of data to and from the client from the ACZ servers. After the VPN server has authenticated the employee, the employee must logon to the ACZ domain through normal domain security in order to access any ACZ network resources. Most employees initiate a "Remote Desktop" connection to their office PCs, thus ensuring that ACZ data is never accessible from the client PC hard drive.

18.5 Client Feedback

Handling client feedback is a joint effort between QA, Project Managers, Laboratory Department Supervisors, and Client Service representatives. If a client has a concern or complaint, either a Project Manager or Client Service Representative takes the call and initiates the feedback procedure by documenting the complaint or problem and requesting the assistance of the Laboratory Department Supervisor and/or QA Officer. If the issue cannot be easily resolved, then it must be documented using FRMAD024, which is routed from the initiator to other appropriate parties, including the QAO if necessary. All client feedback is submitted to management as part of the Management Review of the Quality System. Refer to ACZ's SOP *Client Service Policies and Procedures* (SOPAD043) for additional information.

19 FACILITIES

ACZ Laboratories, Inc. inhabits a modern 31,000 square foot laboratory facility architecturally designed and specifically organized to ensure efficient operation and meet the needs of a large capacity analytical laboratory. Complete lists of instrumentation, balances, thermometers, & weight sets are maintained on a network drive. Incompatible activities are effectively separated. Refer to FRMQA066 for ACZ's floor plan.

19.1 Accommodation of Environmental Test Conditions

19.1.1 Temperature and room pressure are controlled by an HVAC system which maintains 19 independent zones. The clean room, metals lab, and organic instrument lab are kept under positive pressure to prevent contaminant infiltration. The radiochemistry and organic prep labs are kept under negative pressure to prevent the migration of fire, smoke, and chemical releases from the laboratory space. All other zones are maintained at a neutral pressure.

19.1.2 In humid environments, a sudden rise in temperature can result in condensation on microcircuitry leading to problems such as reduced life cycle, inaccurate readings, corrosion, etc. Due to the laboratory's location at 6730 feet above sea level, these concerns are irrelevant and humidity monitoring is only required for desiccators and the clean room.

19.1.3 Servers have a 20 minute backup power supply. If there is an interruption in power, the IT Manager receives a text. This provides sufficient time to ramp down the servers.

19.2 Security

A secure facility is essential to maintaining sample and data integrity and to providing safety to employees and visitors. ACZ has an electronic security system, which controls and limits access to only authorized personnel. The following steps have been taken to ensure this security:

- All entryways are secured. ACZ has three entries equipped with proximity readers which allow access to an employee only after he/she presents their access card. Access to the front visitor entry is controlled by an interior push button monitored by ACZ staff.
- All employees are required to use their access cards to enter and exit the building.
- If any employee does not have their access card, they must sign in at the front desk. This ensures a record is maintained of which personnel were in the building at any time. A temporary access card will then be activated and issued to the employee for the day. These access cards are identified by the word "Temporary" written on a scenic background.
- During normal business hours, public access into the building can be made at the front entrance and the west shipping entrance. Both doors are equipped with a buzzer.
- Visitors must enter and exit through the main entrance and must sign the register at the front desk upon arrival and before departure. A visitor pass is issued at sign in and collected at sign out. There are two types of visitor passes. A red pass identified by the word "Visitor", will not function as an access card and symbolizes the visitor requires an escort. The other visitor pass is identified by the word "Visitor Pass" written on a scenic background and will not function as an access card. This visitor does not require an escort. The determination of which pass the visitor gets is made first, by the visitor's trust level and, second, by the visitors access needs. Visitor passes must be collected when the visitor leaves for the day.
- Companies or individuals under contract to perform recurring or extensive work for ACZ are assigned an access card similar to employees. Contractor passes function as an access card for a defined period of time commensurate with the contract work.
- Emergency Exit doors are to be used only for emergency purposes. If a door is opened, an alarm will sound.
- Loaning or transferring access cards to anyone, including other ACZ employees, is prohibited.

20 RADIOCHEMISTRY

20.1 DATA TRANSFORMATION

ACZ's radiochemistry department utilizes excel spreadsheets to transform instrument response into final results. Spreadsheet equations are locked and password protected in order to reduce the likelihood of inadvertent modifications. Additionally, spreadsheet equations are validated by the radiochemistry supervisor or a sufficiently experienced analyst on an annual basis. Initial validation must be performed by hand calculating results. Annual validation may be performed by populating the current template with data that has been hand calculated in a previous validation and comparing the calculated results from the current template to the hand calculated results from the previous validation. Documented secondary review is required for all updates to spreadsheet templates (e.g. incorporating new mass attenuation coefficients).

20.2 INSTRUMENTATION

Radioanalytical instrumentation is located adjacent to the radiochemistry prep lab. In order to maintain appropriate temperature control in the instrument lab, separation must be maintained. The door between the two lab areas must be kept closed when not in use. Except as noted, instrument checks and other determinations must be performed and documented annually, or more often if necessary.

NOTE: To eliminate potential contamination, planchets must be stored in a covered container or in a drawer.

20.2.1 Gas-Flow Proportional Counter

20.2.1.1 Instrument Reliability Test (Voltage Plateau Determination) – The proper voltage plateau for alpha and beta is where the counting rate is consistent (should not exceed > 5% over a 150 volt change in anode voltage).

20.2.1.2 Cross Talk (Carryover) Check - Cross talk is defined as the percentage of alpha counts represented on the beta plateau. Once the amount of cross talk is determined, the cross talk settings are adjusted on the instrument to eliminate cross talk.

20.2.1.3 Detector Efficiency Curve (Self Absorption) - Efficiency curves are graphs plotting counts versus sample residue density and determine the efficiency of the alpha and beta counter as a function of sample residue density. This factor is part of the overall determination of sample activity.

20.2.1.4 Background Determination - Characteristic of most detectors is a background or instrument count rate attributed to cosmic radiation, radioactive contaminants in instrument parts, counting room construction material and/or the proximity of radioactive sources. The background is determined weekly by counting an empty planchet for 12 hours. On each day of use the instrument is checked for background drift by counting an empty planchet for 90 minutes. Background counts must fall within established control chart limits or corrective action must be taken before analyzing samples. Although most radiation measurement systems are noteworthy for their stability, sudden changes can occur due to instrument component failure, loss of gas pressure, vacuum, or contamination of a detector or sample chamber from a high activity sample. Subsequently, instrument drift in detector efficiency and background must be checked both before and after measuring samples used for drinking water compliance monitoring. Refer to individual test SOPs for additional details.

20.2.1.5 Instrument-Response Check (Performance Check) – This continuing calibration check verifies the instrument response and stability and is performed daily for each detector. For a performance check measurement, the same calibration sources must be used as for the calibration measurement in order to verify the current measuring results still match the results of the calibration measurement stored last. At the end of the check the count rates and the relative deviations from older calibration measurements are displayed. The system signals

"OK" if the deviations do not exceed the maximum deviation defined by the user. Samples used for drinking water compliance monitoring must be bracketed by passing performance checks. Refer to individual test SOPs for additional detail.

20.2.2 Liquid Scintillation Counter

20.2.2.1 *Optimal Window* - When determining radionuclides by liquid scintillation, it is necessary to select the optimal window by counting a standard for five minutes and generating a sample spectrum. For better clarity, a log scale for the channel number axis should be used. On the graph, the region of interest is determined by the energy of the peak one is trying to quantitate. The optimal window is formed by extending this region by 10% on each side of the alpha peaks.

20.2.2.2 *Efficiency Quench Curve* – The liquid scintillation instrument, a Beckman LS 6000TA, automatically corrects for quenching by the H - Method. Refer to SOPRC010 for details.

20.2.2.3 *Background Check* - Three background blanks are run with every batch. The first two are run immediately after calibration. The third, the CCB, is employed as a measurement of instrument drift and is run immediately before the final LCS. For both checks, the counting duration must be equivalent to the longest sample counting duration.

20.2.2.4 *Instrument-Response Check Source* - This continuing calibration check verifies instrument response and stability and must be performed daily. If the source count is within two standard deviations (sigma) of the previously determined average count rate, instrument reliability and stability is established. If the source rate is outside the ±2 sigma-warning limit then the variability should be further investigated. If the source check is outside the ±3 sigma out of control limits, then no further samples should be analyzed until the problem is resolved. Resolution might include a new efficiency curve, background checks, and/or instrument maintenance. If insufficient data exists for control charts, ±10% of the initial source value is considered acceptable. The source for this check is a Tritium standard.

20.2.3 Alpha Spectrometer

20.2.3.1 *Energy vs. Channel Calibration* - Each alpha spectrometer has a set number of channels associated with it. To associate these channels to a specific alpha particle, the channels must be calibrated. One known calibrated solid source is placed into the detector and analyzed for five minutes to determine its associated channel to its calibrated energy peak. Since the energy is linear across the channels, all of the channels now have an associated energy. This determination is performed on an annual basis, or whenever maintenance is performed that could potentially affect the calibration.

20.2.3.2 *Background Checks* - Characteristic of most detectors is a background or instrument count rate attributed to cosmic radiation, radioactive contaminants in instrument parts, counting room construction material and/or the proximity of radioactive sources. Placing an empty sample tray in the counting chamber and counting it for as long as the longest sample-counting duration can determine the background rate (or a background check can be completed overnight). An overnight background determination must be completed at least quarterly.

20.2.3.3 *Instrument-Response Check Source* - This continuing calibration check verifies the instrument response and stability and is performed daily. If the source count is within two standard deviations (sigma) of the previously determined average count rate, instrument reliability and stability is established. If the source rate is outside the ±2 sigma-warning limit, then the variability should be further investigated. If the source check is outside the ±3 sigma out of control limits, then no further samples should be analyzed until the problem is resolved. Resolution might include a background check, and/or instrument maintenance. If insufficient

data exists for control charts then ±10% of the true value is considered acceptable.

20.2.4 Gamma Spectrometer

20.2.4.1 *Background Checks* –Characteristic of most detectors is a background or instrument count rate attributed to cosmic radiation, radioactive contaminants in instrument parts, counting room construction material and/or the proximity of radioactive sources. A cave background must be measured monthly and the background gross activity recorded. The cave background is determined by counting the empty cave for a period of time at least as long as the longest sample-counting duration. When drinking water samples are present in the batch, and additional background check is measured at the end of the batch to monitor instrument drift.

20.2.4.2 *Instrument-Response Check (Performance Check)*- The total activity of a calibration or check source will check the efficiency calibration currently in use and the general operating parameters of the system, including source positioning, contamination, library values, and energy calibration. This activity calculation uses the general analysis program to ensure that the total system is checked. This check is performed for every workgroup. If the performance check is within the defined acceptance limits, instrument reliability and stability is established. If the performance check does not meet acceptance criteria, then no further samples should be analyzed until the problem is resolved. Samples used for drinking water compliance monitoring must be bracketed by acceptable performance checks. Resolution might include a background check, and/or instrument maintenance. Refer to SOPRC016 for additional information.

21 CERTIFICATIONS

ACZ has primary or secondary (reciprocal) certification with numerous states and EPA regions. Current certificates can be viewed at http://acz.com/certifications/. Each certificate contains a scope of accreditation listing each method the laboratory is accredited for by the issuing authority.

APPENDIX A REFERENCES UTILIZED BY ACZ

- “TNI Standards,” National Environmental Laboratory Accreditation Conference, (current version).
- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act," USEPA, Federal Register Vol. 67, No. 205, October 23, 2002.
- "Manual for the Certification of Laboratories Analyzing Drinking Water," USEPA, (current version).
- "Methods for the Chemical Analysis of Water and Wastes," USEPA, EPA-600/4-79-020, March 1983.
- "Test Methods for Evaluating Solid Waste," USEPA, SW-846 Third Edition, Update IV, January 2008.
- “Guidelines in Establishing Test Procedures for the Analysis of Wastewater Pollutants,” Code of Federal Regulations 40, Parts 136, 141, 143.
- "Quality Assurance of Chemical Measurements,” Taylor, J., Lewis Publishers, Michigan, 1987
- "Annual Book of Standards, Water Analysis," ASTM, 1989.
- "Quality Control in Analytical Chemistry," Kateman, G., Vol. 60, 1985.
- "Principles of Environmental Analysis, Analytical Chemistry," Keith, L.H., et al., Vol. 55, 1983.
- "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," USEPA, 1979.
- “Guidance for the Data Quality Assessment: Practical Methods for Data Analysis,” USEPA, EPA 600/R-96-084, July 2000.
- “Methods for the Determination of Metals in Environmental Samples,” USEPA, EPA 600/4-91-010, June 1991.
- “Methods for the Determination of Metals in Environmental Samples,” Supplement I [to EPA 600/4-91-010], USEPA, EPA 600/R-94-111, May 1994.
- “Methods for the Determination of Inorganic Substances in Environmental Samples,” USEPA, EPA 600/R-93-100, August 1993.
- "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater," USEPA, EPA 821/B-96-005, December 1996.
- "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," USEPA, EPA 600/4-80-032. August 1980.
- “Determination of Lead-210, Thorium, Plutonium and Polonium-210 in Drinking Water: Methods 909, 910, 911, 912,” 01A0004860 (Region 1 Library), March 1982.
- “Good Automated Laboratory Practices - Principles and Guidance to Regulations for Ensuring Data Integrity in Automated Laboratory Operations” USEPA, 2185, 1995.
- “Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications”, ASTM E29-08

APPENDIX B DEFINITIONS OF TERMS

Acceptance Criteria: specified limits places on characteristics of an item, process, or service defined in requirement documents.

Accreditation: verification by a competent, disinterested, third party that a laboratory possesses the capability to produce accurate test data, and that it can be relied upon in its day-to-day operations to maintain high standards of performance.

Accrediting Body: The Territorial, State, or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation.

Accreditation body: Authoritative body that performs accreditation.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Aliquot: A discrete, measured, representative portion of a sample taken for analysis.

Analyte: The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)

Analytical Spike (AS): an aliquot of client sample to which a known amount of target analyte is added and that demonstrates the absence or presence of interference in the matrix. The AS is prepared exactly the same way as the LFB, only spiking into sample instead of reagent blank, and is not prepped (digested) prior to analysis. The AS may also be referred to as a post-digestion spike.

Analytical Spike Duplicate (ASD): a second replicate analytical spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Analytical System: the combination of events, techniques, and procedures used to generate analytical results.

Analyst Review (AREV): See Primary Review.

Atomization: A process in which a sample is converted to free atoms.

Audit: a systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.

Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same matrix, meeting the above criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of 20 or less prepared environmental samples (extracts, digestates or concentrates) that are analyzed together as a group. QC samples (e.g. LCS, MS, MSD) do not count towards the maximum of 20.

All required QC samples must be prepared and/or analyzed with each batch at the frequency required by the method, even if there are less than 20 client samples in the batch. If the workgroup has more than 20 samples, then sufficient batch QC must be analyzed for additional samples. Every batch of environmental samples is assigned a unique (i.e. traceable) six-digit numerical identifier called the LIMS Workgroup number.

Blank: a sample that has not been exposed to the analyzed sample stream utilized to monitor contamination during sampling, transport, storage, or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical

results. See also Equipment Blank, Field Blank, Instrument Blank, Method Blank, Reagent Blank. Refer to §11.3 for types of blanks.

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst or laboratory may know the identity of the sample but not its composition. It is used to test the analyst or laboratory's proficiency in the execution of the measurement process.

Calibration: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration Curve: the graphical relationship between the known values, such as concentrations, or a series of calibration standards and their instrument responses.

Calibration Range: The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.

Case Narrative: Additional documentation provided in the client report that describes any abnormalities and deviations that may affect the analytical results and summarizes any issues in the data package that need to be highlighted for the data user to help them assess the usability of the data.

Certified Reference Material (CRM): A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.

Chain of Custody Form: a legal record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses.

Client: Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations. (ANSI/ASQ E4-2004)

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:
- Second column confirmation;
- Alternate wavelength;
- Derivatization;
- Mass spectral interpretation;
- Alternative detectors; or
- Additional cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ ASQC E4-1994)

Congener: A member of a class of related chemical compounds (e.g., PCBs, PCDDs)

Continuing Calibration Blank (CCB): the same solution as the calibration blank, it detects baseline drift in the calibration of the instrument. When specified by the method, analyze a CCB immediately after each CCV, including the final CCV.

Continuing Calibration Verification (CCV): a solution of method analytes of known concentrations used to confirm the continued calibration of the instrument. The CCV is analyzed at the frequency indicated in the test SOP.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e. the data meet specified acceptance criteria)

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

Definitive Data: Analytical data of known quality, concentration, and level of uncertainty. The levels of quality and uncertainty of the analytical data are consistent with the requirements for the decision to be made. Suitable for final decision-making. (UFP-QAPP)

Demonstration of Capability (DOC): a procedure to establish the ability of the analyst to generate acceptable accuracy [and precision, if applicable].

Detection Limit: the lowest concentration or amount of target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value (see Method Detection Limit).

Digestion: A process in which a sample is treated (usually in conjunction with heat) to convert the sample to a more easily measured form.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Eluent: A solvent used to carry the components of a mixture though a stationary phase. (Skoog, West, and Holler. *Fundamentals of Analytical Chemistry*. 1992)

Elute: To extract; specifically, to remove (adsorbed material) from an adsorbent by means of a solvent. (Merriam-Webster's Collegiate Dictionary, 2000)

Elution: A process in which solutes are washed though a stationary phase by the movement of a mobile phase. (Skoog, West, and Holler. *Fundamentals of Analytical Chemistry*. 1992)

Equipment Blank: a sample of analyte-free media that has been used to rinse common sampling equipment to check the effectiveness of decontamination procedures.

False Positive (Type I or alpha error): concluding that a substance is present when it truly is not.

False Negative (Type II or beta error): concluding that a substance is not present when it truly is.

Field Blank: a blank prepared in the field by filling a clean container with Type I water and appropriate preservative, if any, for the specific sampling activity being undertaken.

Holding Time (Maximum Allowable Holding Time): the maximum time that samples may be held prior to analysis and still be considered valid or not compromised.

Homologue: One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, CH3OH (methanol), C2H5OH (ethanol), C3H7OH (propanol), C4H9OH (butanol), etc., form a homologous series. (*The Condensed Chemical Dictionary* G.G. Hawley, ed. 1981)

Initial Calibration Blank (ICB): a solution identical to the calibration blank and confirms the absence of background contamination in the calibration blank. When specified by the method, an ICB is analyzed immediately after the ICV.

Initial Calibration Verification (ICV): a solution of method analytes of known concentrations intended to determine the validity of the instrument calibration. The ICV must be analyzed immediately after each

calibration and must be prepared from a source independent of the calibration standards, preferably purchased from a different manufacturer.

Instrument Blank: an aliquot of Type I water or solvent processed through the instrument steps of the measurement process; used to determine presence of instrument contamination.

Interference, spectral: Occurs when particulate matter from the atomization scatters the incident radiation from the source or when the absorption or emission of an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible. (Skoog, West, and Holler. *Fundamentals of Analytical Chemistry*. 1992)

Interference, chemical: Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte. (Skoog, West, and Holler. *Fundamentals of Analytical Chemistry*. 1992)

Internal Standard (IS): a known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Isomer: One of two or more compounds, radicals, or ions that contain the same number of atoms of the same elements but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane. (Websters)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Laboratory Fortified Blank (LFB): a reagent blank spiked with a known concentration of analyte. The LFB is analyzed exactly like a sample and determines whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

Legal Chain of Custody Protocols: procedures employed to record the possession of samples from the time of sampling until analysis and are performed at the special request of the client. These protocols include the use of a Chain of Custody form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Linear Dynamic Range (LDR): concentration range over which the instrument response to analyte is linear.

Matrix Duplicate (DUP): a second aliquot of a client sample that is prepared and analyzed in the same manner as all other samples in the same workgroup. The DUP demonstrates the precision of the method.

Matrix Spike (spiked sample or fortified sample): a sample prepared by adding a known amount of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes (MS or LFM) are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate: a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Maximum Contamination Limit (MCL): the numerical value expressing the maximum permissible level of contaminant in water that is delivered to any user of a public water system.

May: denotes permitted action, but not required action.

Measurement Quality Objectives (MQOs): The desired sensitivity, range, precision, and bias of a measurement.

Measurement System: A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as client samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for the sample analyses.

Method Detection Limit: the minimum concentration of an analyte, in a given fortified matrix, that can be measured and reported with 99% confidence that the concentration is greater than zero.

Method of Standard Additions: A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.) (Modified Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Must: denotes a requirement.

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Nonconformance: An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also the state of failing to meet the requirements.

Outlier (Statistical): an observation or data point that deviates markedly from other members of the population.

Performance Audit: the routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

Primary Review (AREV): The first level of data review conducted after data has been generated and uploaded to LIMS. Primary review is typically conducted by the analyst who generated the data but may be performed by another authorized individual. Quality control and corrective actions are evaluated as part of this review. Where acceptance criteria fails, samples are scheduled for re-preparation and/or re-analysis or data is appropriately qualified.

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Testing Study Provider: Any person, private party, or government entity that meets stringent criteria to produce and distribute TNI PT samples, evaluate study results against published performance criteria and report the results to the laboratories, primary accrediting authorities, PTOB/PTPA, and TNI.

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Protocol: a detailed written procedure [SOP] for laboratory operation that must be strictly followed.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality.

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Quality Manual [QAP]: a document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products, and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control.

Quantitation Limit [Limit of Quantitation, Practical Quantitation Limit]: level, concentration, or quantity of a target variable (i.e. target analyte) below which data is reported as estimated. The quantitation limit may or may not be statistically determined, or may be an estimate that is based upon analyst experience or judgment.

Quantity Sufficient (QS): Refers to the addition of appropriate diluent to the solution to achieve the final volume.

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for reconstructing and evaluating the report of the activity or study.

Reagent Blank (method reagent blank): a sample consisting only of Type I water and reagent(s) without the target analyte(s) or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so (EPA, etc.). The reference method is included on the client report.

Reporting Limit (RL): The lowest analyte level (concentration or mass) the laboratory will report as a detected result. ACZ's default reporting limit is the MDL; however the RL may be defined as the PQL or another level dependent on project needs.

Retention Time: The time between sample injection and the appearance of a solute peak at the detector. (Skoog, West, and Holler. *Fundamentals of Analytical Chemistry*. 1992)

Sample: Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis

Sample Tracking: procedures employed to record the possession of the samples from the time of sampling until analysis, reporting, and archiving. These procedures include the use of a Chain of Custody form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.

Secondary Review (SREV): The second level of data review conducted after primary review (AREV) has been completed. Secondary review is typically conducted by the pertinent department supervisor but may be performed by another authorized individual. Quality control and corrective actions are evaluated as part of this review. Data qualifiers and sample statuses assigned at AREV are evaluated and corrected if necessary.

Selectivity: (Analytical chemistry) The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (i.e. concentrations) of a variable of interest.

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there is no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled.

Should: denotes a guideline of recommendation whenever noncompliance with the specification is permissible.

Signal to Noise Ratio (S/N): a dimensionless measure of the relative strength of an analytical signal (S) to the average strength of the background instrumental noise (N) for a particular sample.

Spike: a known amount of target analyte added to a blank sample or client sub-sample; used to determine the recovery efficiency or for other quality control purposes.

Standard Deviation: the measure of the degree of agreement (precision) among replicate analyses of a sample. The population standard deviation (n degrees of freedom) should only be used for more than 25 data points; otherwise, when referenced, standard deviation implies sample standard deviation (n-1 degrees of freedom).

Standard Operating Procedure (SOP): a written document which details the manner in which an operation, analysis, or action is performed. The techniques and procedures are thoroughly prescribed in the SOP and are the accepted process for performing certain routine or repetitive tasks.

Supervisor [however named]: the individual designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training, and experience to perform the required analyses.

Surrogate (SURR): a substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.

Test Method: adoptions of a scientific technique for a specific measurement problem, as documented in a laboratory SOP or published by a recognized authority.

The NELAC Institute (TNI): a voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories.

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

Tuning: A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

Validation: The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification: Confirmation by examination and provision of evidence that specified requirements have been met.

APPENDIX C TECHNICAL DIRECTORS

Name	Department	Degree
Steve Pulford	Metals	BS, Chemical Engineering, Minor in Biochemistry
Gus Torde	Organics	BS, Chemistry
Alyssa Dybala	Wet Chemistry	BS, Pharmaceutical Marketing
Matt Sowards	Radiochemistry (reserve) Wet Chemistry	BA, Neuroscience
Keith Hensley	Wet Chemistry	BS, Chemistry & Biology
Brett Dalke	Geochemistry	BA, Geology & English
Mark McNeal	Radiochemistry	BS, Biology
If a technical director is absent for a period exceeding fifteen calendar days, another qualified full time employee shall be assigned to temporarily fulfill the duties of technical director. Defined reserve technical directors shall assume these duties by default. Where reserves are not yet defined, management shall appoint a qualified individual as necessary. If a technical director is absent for more than 34 days, it is the QAO's (or delegee's) responsibility to notify accrediting bodies in writing.		

Appendix 1.A.E

Laboratory Data Review and Validation Checklist

LABORATORY DATA REVIEW AND VALIDATION CHECKLIST – GREENS CREEK PROJECT

Sample Point(s):	Laboratory #(s):
Parameter list requested:	Date Samples Collected:
	Date Samples Received by Lab:

Category		Yes	No	N/A	Comments
Reported Data					
1. COC & other field documents included?					
2. All reporting requirements satisfied?					
3. Parameters reported match parameters requested?					
4. Methods reported match methods requested?					
5. Reporting limits and units as requested?					
6. Electronic file matches hard copy?					
Sample Analysis					
1. Analysis holding times met?					
Laboratory QA/QC Requirements					
1. Blanks	proper frequency?				
	acceptance criteria met?				
2. LCSs	proper frequency?				
	acceptance criteria met?				
3. Spikes	proper frequency?				
	acceptance criteria met?				
4. Duplicates	proper frequency?				
	acceptance criteria met?				
General *Note any additional comments/observations on back of sheet.*					
1. Are sample results consistent with historical data for specific sample point(s)?					

Reviewed by: **Date:**

FIELD DATA REVIEW AND VALIDATION CHECKLIST – GREENS CREEK PROJECT

Sample Point(s):	Date Collected:			
	Date Shipped to Lab:			
	Collected By:			
Category	**Yes**	**No**	**N/A**	**Comments**
Reported Data				
1. Are all appropriate data fields filled out?				
2. Are water level data measurements calculated and recorded correctly?				
3. Are flow measurements calculated and recorded correctly?				
General				
1. Are sample results for field measurements consistent with historical data for specific sample point(s)?				

2. Note additional comments/observations (use back of sheet if necessary):

Reviewed by: **Date:**

CORRECTIVE ACTION FORM

Sample I.D.(s)_____ **Date Sampled** _____

Laboratory Job Number(s)_____ **Date Analyzed** _____

Reviewed By_____

Describe the deficiency:
Document all correspondence involved:
(Include date and time of the communication(s), as well as the name and position of all individuals contacted. Also include a synopsis of each communication, attach extra pages as necessary)

Define a corrective action:

Explain the resolution:

Appendix 1.A.**F**

Chester LabNet
Quality Assurance Management Plan (QAMP)

Quality Assurance Management Plan

CHESTER LabNet

12242 S.W. Garden Place, Bldg. 1
Tigard, Oregon 97223
P: 503.624.2183 F: 503.624.2653
www.chesterlab.net

Paul Duda – President, Laboratory Director, LIMS Administrator, Client Services Technical Director

Sheri Heldstab – QA Officer, Conventional Chemistry Laboratory Technical Director, Analyst

Richard Sarver - XRF Laboratory Technical Director, XRF Analyst

Lisa Ball – Project Manager, Sample Custodian

Jennifer (Jen) Schleis – Gravimetry Laboratory Technical Director & Technician, XRF Analyst, Analyst

T. Mike May - Analyst, Gravimetry Laboratory Technician

Julie Delarue – Analyst, Gravimetry Laboratory Technician, XRF Analyst

Theodore (Ted) Perry – Analyst, Gravimetry Laboratory Technician, Chemical Hygiene Officer, Health and Safety Officer

Kevin Healey – Gravimetry Laboratory Technician

This document supplants any previous versions.

Revision Number:	**February, 2019**	**Effective Date:**	**5 March 2019**
Document Number:	QAMP March 2019		

Review History

Review Date	Changes	Author
2/13/19	Reinserted references to 2009 TNI standard. Updated changes in staffing, equipment and SOP lists. Added rounding rules, significant figures protocol, and protocol for determination of LL-LCS and LL-CCV acceptance limits. Added information on thermometer calibrations, volumetric ware verification and tracking, and sampling media being exempted from DL study. Changed initial calibration requirements.	S. Heldstab
2/27/18	Updated changes in staffing, equipment, and SOP lists. Updated to 2016 TNI Standard requirements (primarily Appendix H). Added LL-CCV and LL-LCS requirements. Changed "Limit of Detection" to "Detection Limit" per 2016 TNI Standard. Added definitions in glossary to reflect those in 2016 TNI Standard. Defined "absence" for Technical Directors and QA Officer.	S. Heldstab
1/31/17	Updated changes in staffing, equipment, and SOP lists. Typographical and grammatical errors corrected.	S. Heldstab
1/25/16	Updated changes in staffing, equipment, and SOP lists. Typographical and grammatical errors corrected.	S. Heldstab
1/28/15	Updated changes in staffing, equipment, floorplan and SOP lists. Typographical and grammatical errors corrected.	S. Heldstab
1/15/14	Updated changes in accredited methods, staffing, equipment, SOP lists. Typographical and grammatical errors corrected.	S. Heldstab
2/21/13	Updated changes in accredited methods, staffing, equipment, SOP lists. Typographical and grammatical errors corrected.	S. Heldstab
1/8/12	Updated to NELAC template. Updated Personnel and Organizational Structure to reflect current staffing, capital equipment and manuals lists. Updated floor plan to reflect current floor plan; build-out is on-going.	S. Heldstab
6/14/11	Updated to NELAC template. Updated Personnel and Organizational Structure to reflect current staffing, capital equipment and manuals lists. (Revision incomplete)	S. Heldstab
2/17/11	Updated equipment and manuals lists. Changed client surveys section.	S. Heldstab
2/26/10	Changed Personnel and Organizational Structure to reflect current staffing. Minor clarifications to text.	S. Heldstab
2/12/09	Minor changes to text.	S. Heldstab
12/16/08	Changed Personnel and Organizational Structure to reflect current staffing. Minor clarifications to text.	S. Heldstab
2/19/08	Changed Personnel Organizational Structure to reflect staffing, incorporated new equipment to Capital Equipment Inventory, added section to 'Backup of LIMS System' to include other computers containing essential information, updated final data report page, minor changes to text for clarification.	J. Schleis

Review Date	Changes	Author
2/15/07	Changed Corrective Action Report documentation system, minor change to quality policy statement to include metric reviewable goals, moved 'Traceability of Measurements' section to Section 7, added section on client surveys, merged client complaints & corrective action documentation into a single system, added appendices "Listing of Document Locations" and "Glossary & Definitions of Terminology"	S. Heldstab
2/5/07	Changed references to staffing where staffing has changed, added text on handling of PE samples to Section 10.6, added section on detection limits (7.8), added details for clarification throughout.	S. Heldstab
2/7/06	Definition of "basic laboratory skills" added to Personnel qualifications, SOP formatting elements changed to reflect new NELAC-compliant format, acceptance/rejection criteria for non-standard methods added, section added on documentation of customer inquiries/complaints, updated staff resumes, updated SOP listing, added 'failure to report' to the list of breaches of ethical behavior in Appendix D.	S. Heldstab
7/8/05	Expanded Section 7.7, revised Section 10.4 and 10.5 to reflect new internal audit process, updated staff references.	S. Heldstab
1/5/05	Added Appendix D (Ethics training).	S. Heldstab
10/21/04	Major updating to incorporate elements of NELAC/ORLAP requirements. Sections 3.3.2.1, 7.3–7.7, 8.2.4-8.2.7, 8.3, 8.3.1–8.3.5, 8.4, 9.4, 10.6–10.8, 11.8–11.10 added.	S. Heldstab
1/23/04	Updated floor plan and Capital inventory table. Minor grammatical changes.	S. Heldstab
3/10/03	Changes in organization of Section 3. No changes to content, only to formatting.	S. Heldstab
12/10/02	Minor changes to text, some text rearranged for clarity and conciseness.	S. Heldstab
11/8/01	This document has been in existence since at least 1988. The number and types of revisions are unknown. As of this date, the document is being revised to standardize the formatting of ***CHESTER LabNet*** documents.	S. Heldstab
Unknown	No Changes - date of origination.	C.R. Lytle

Controlled Copy #1: This document
Controlled Copy #2: Sent to ORELAP

Concurrences

The following approved signatories, by their signature, attest to having read and understood the most current version of the Quality Assurance Management Plan for CHESTER LabNet:

Name	Title(s)/Responsibility(ies)	Initials	Signature	Date
Paul Duda	President Laboratory Director LIMS Administrator Customer Service Technical Director	PDD	Paul Duda	2/27/19
Sheri Heldstab	QA Officer Conventional Chemistry Laboratory Technical Director Lead Analyst	SH	Sheri Heldstab	2-21-19
Lisa Ball	Project Manager Sample Custodian	LMB	Lisa Ball	3-4-19
Rick Sarver	XRF Technical Director Lead XRF Analyst	RS	Rick Sarver	3-1-19
Jennifer (Jen) Schleis	Gravimetry Laboratory Technical Director Lead Gravimetry Laboratory Technician XRF Technician Analyst	JMS	Jen Schleis	3.03.19
T. Mike May	Analyst Gravimetry Laboratory Technician	TMM	T. Mike May	3.03.19
Julie Delarue	Analyst Gravimetry Laboratory Technician XRF Technician	JD	Julie Delarue	3-5-19
Theodore (Ted) Perry	Analyst Gravimetry Laboratory Technician Chemical Hygiene Officer Health & Safety Officer	TP	Ted Perry	2-27-19
Kevin Healey	Gravimetry Laboratory Technician	KH	Kevin Healey	3-4-19

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Disclaimer

In 2011, ORELAP, the accrediting body for the state of Oregon, requested that the laboratory use the 2009 TNI Standard QAMP Template in creating its QAMP, to make it easier for auditors to find information. This document is organized following the 2009 TNI Standard QAMP Template, however it has been updated to include changes from the 2016 TNI Standard. Historically, all versions of the TNI Standard and its associated QAMP templates have been designed for water/soil analysis.

CHESTER LabNet specializes in inorganic air quality analyses, both ambient and source emissions. Volume 1, Module 1, Section 1.3.2 of the 2016 TNI Standard states *"This Standard applies only to fields of accreditation (FOA) that are also designated as fields of Proficiency Testing (FoPT) by the TNI Proficiency Testing Program Executive Committee (PTPEC)."* As of this writing, no ambient air or source emissions methods have FoPTs; thus, according to the 2016 TNI Standard, ***CHESTER LabNet*** is not eligible for accreditation for any ambient air or source emissions methods. However, ORELAP has accredited the laboratory for some methods since 2005, despite their ineligibility for accreditation.

Due to the significant differences between sampling gaseous samples and sampling water/soil samples, there are some requirements of the 2009 and 2016 TNI Standard which are unachievable based on the nature of the methods utilized. Throughout this document, the requirements as set forth in the 2009 and 2016 TNI Standard have been included. Also included are the reasons those requirements are not applicable to the laboratory's field of work as well as explanations of how the laboratory attempts to meet the intent, if not the letter, of the requirement.

Below is a short list of issues which are described in greater detail throughout various sections of this document:

1. "Media": This term is used throughout the 2009 and 2016 TNI Standard in reference to microbiological testing. For the purposes of air quality testing, media is generally comprised of either filters (various sizes/compositions), sorbent tubes, or solutions used in impinger trains. Most media used by the laboratory has some form of contamination present from the manufacturing process and contamination is considered a routine part of analyses. The manufacturer, not the laboratory, is responsible for the quality of the media. The laboratory does attempt to purchase the cleanest media commercially available.

2. Sampling: The laboratory performs no sampling. Most air quality methods, source or ambient, involve quite complex sampling equipment and procedures. The laboratory's clients perform the sampling, from collection to shipment. The laboratory has no control over the actions or inactions of the client in the field. Many requirements of the 2016 TNI standard make reference to sampling, sample containers and sample rejection. These requirements are not suited to the nature of air quality sampling or analysis.

3. Sample containers: To capture a gaseous sample, either a filter, an absorbing solution or a sorbent material of some sort must be used. Thus, the primary container for the sample is either "filter," "solution," or "sorbent." Secondary containers are then used for containing the sample containers. Filters may or may not be provided by the laboratory in a variety of secondary containers. Containers

for liquids are nearly always provided by the client as the laboratory has no idea how much liquid will be collected. The absorbing solution acts as a preservative by its nature, thus, no preservatives are added to any secondary container.

4. Method selection: Air Quality methods, particularly the Code of Federal Regulations (CFR) methods, tend to not be regularly updated to incorporate technological improvements.

   CHESTER LabNet has no control over the selection of methods used during analysis. Frequently, our clients have no control over method selection either. The client is responsible to their client (the "ultimate" client), who, in turn, is typically responsible to a regulator at some governmental level (e.g., federal, state, county, city). The regulator for the particular facility or project being monitored is the body responsible for method selection. The laboratory and the laboratory's clients have little sway over the decisions of these regulators.

   In addition, some methods are in conflict with the laws of physics and are, therefore, not possible to perform as written.

   The 2016 TNI Standard has many requirements pertaining to the selection of methods to be used by the laboratory. These sections cannot be applied to the type of work performed at ***CHESTER LabNet***.

5. Proficiency Testing: Proficiency testing is required only when a field of proficiency testing table (FoPT) exists. No FoPT exists for either source or ambient air analyses, consequently, this requirement does not apply to ***CHESTER LabNet.*** Source Emissions sampling requires that an audit sample, procured from an accredited provider, be analyzed with any sample from which data will be utilized for regulatory or compliance purposes. The facility being regulated is responsible for obtaining the audit, and the regulator is responsible for choosing an appropriate concentration range for the audit. The laboratory is entirely removed from the acquisition of the audit. The client sends the audit to the laboratory along with the associated samples. This typically results in far more than two audits per year being analyzed for a given Source Emission method.

CHESTER LabNet will make every effort to meet the intent of the requirements as given in the 2009 and 2016 TNI Standard, just as it makes every effort to meet the intent and chemistry of archaic or contradictory methods.

Section 3

INTRODUCTION AND SCOPE
(TNI V1:M2 – Sections 1, 2, 3)

The purpose of this Quality Assurance Management Plan (QAMP) is to outline the management system for ***CHESTER LabNet***. The Quality Assurance Management Plan defines the policies, procedures and documentation that assure analytical services continuously meet a defined standard of quality. That standard is designed to provide clients with data of known and documented quality and, where applicable, demonstrate regulatory compliance.

CHESTER LabNet has specialized in the inorganic analyses of ambient particulates and source emission samples since its inception as NEA, Inc. in the late 1970's. The laboratory as an organization, its management, and its personnel are committed to the production of the highest quality data achievable with current methodologies and instrumentation; and to compliance with contractual, regulatory and accreditation standards and requirements.

This QAMP is heavily based upon the 2009 TNI QAMP Template, with updates to reflect the 2009 and 2016 TNI Standard. The template is designed for water, wastewater and soil/sludge samples, and does not work well for air quality samples. As a result, many sections have been significantly modified in an effort to meet accrediting requirements. In addition, many of the requirements of the 2016 TNI Standard also do not apply to air quality sampling (ambient or source) and have been significantly modified.

The Quality Assurance Management Plan sets the standard under which all laboratory operations are performed, including the laboratory's organization, objectives and operating philosophy. The Quality Assurance Management Plan has been prepared to assure compliance with the 2009 and 2016 TNI Environmental Laboratory Sector Standard, Volume 1. This Standard is consistent with ISO/IEC 17025:2005 requirements that are relevant to the scope of environmental testing services and thus, the laboratory operates a quality system in conformance with ISO/IEC 17025:2005(E). In addition, the policies and procedures outlined are compliant with the various accreditation and certification programs listed in Appendix E of this document.

For any activity involving a service or the creation of an analytical result, quality may be defined as conformity to a given set of requirements. To ensure acceptable quality, three conditions must be met: (1) requirements and objectives must be clearly delineated before work begins; (2) the major steps in the production of the service or analytical result must have a component that allows for the control of quality, based on the end-result objectives; (3) the components of quality control must include control limits and corrective actions designed to both effectively monitor quality and modify procedures if quality is compromised.

Quality Assurance (QA) comprises the overall program elements designed to maintain any activity within the stated objectives. Examples of such program elements are: clearly stated precision and accuracy targets; written standard operating procedures for all laboratory and instrumental protocols; the selection of sample preparation and analytical methods that are most appropriate for the matrices and analytes to be encountered; etc.

Quality Control (QC) comprises the individual checks used to monitor laboratory procedures, the precision and accuracy statistical control limits for each individual check, and the specific corrective actions to be followed when QC results are outside control limits. An example of a QC element is the matrix spike. Good quality control would set the frequency of analysis, the particular QC statistic to be used (i.e., percent recovery), the control limit (based on published statistics for the particular analysis or on QC limits developed in house), and the corrective action for QC results that are out of control.

3.1 Scope of Testing

CHESTER LabNet specializes in Inorganic Air Quality Analysis of ambient air and source emissions, including analysis of PM_{10} and $PM_{2.5}$ samples. The laboratory's scope of analytical testing services includes those listed in Appendix D.

At present, the six methods accredited are:

- Hexavalent Chromium in ambient air (modified CARB SOP MLD039 and modified ASTM D7614-12);
- 40 CFR 60 Method 202, "Condensable Particulate Matter, Rev. 12/1/2010";
- NIOSH 5040 "ELEMENTAL CARBON (DIESEL PART.)"; DRI SOP#2-216r2 (Organic & Elemental Carbon by Improve_A parameters);
- PM_{10} 40 CFR 50 Appendix J,
- $PM_{2.5}$ 40 CFR 50 Appendix L, and
- 40 CFR 60 Method 26A, "Hydrogen Halides and Halides in Stationary Sources".

Note that at present, none of these methods are contained in a FoPT table.

3.2 Table of Contents, References and Appendices

The Table of Contents is in Section 2 and Appendices are at the end of this document.

Where applicable to air quality analyses, this Quality Assurance Management Plan uses the references included in the 2009 and 2016 TNI Environmental Laboratory Sector Standard, Volume 1.

Unlike Water and Soils methods, Air Quality reference methods can be difficult to locate and, in some cases, a reference method may not exist for the analysis requested by the client. The majority of reference methods utilized at ***CHESTER LabNet*** can be found in 40 CFR Part 50, 40 CFR Part 60, 40 CFR Part 61, NIOSH Methods Compendium, OSHA Methods Compendium, US EPA IO Methods Compendium, US EPA "Other Test Methods," US EPA "Conditional Test Methods," published peer-reviewed papers, and a variety of methods developed in-house to satisfy the needs of our clients. See Appendix D for a listing of reference methods commonly used at ***CHESTER LabNet*** and Appendix B for a listing of all ***CHESTER LabNet*** SOPs.

3.3 Glossary and Acronyms Used

Quality control terms are generally defined within the Section that describes the activity.

3.3.1 Glossary

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service and defined in requirement documents.

Accreditation: The process by which an agency evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques, and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analyte: A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as a part of the analysis.

Archaic: A method that requires the use of equipment that is no longer available, no longer in use, or that has become obsolete by virtue of technological advancements (e.g., requiring the use of instrumentation that has not been supported since 1972, requiring hand-injection where autosamplers are commonly available).

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation).

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Batch: environmental samples that are received, prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.

Analytical Batch: a group of prepared samples (extracts, digests, etc.) which are analyzed together as a group, although they may have been prepared separately. An analytical batch may exceed 20 samples.

Preparation Batch: a group of ≤20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours and which are prepared together as a group, and which share common QC samples. [Note: Some source emission methods have preparation times exceeding 24 hours].

Sample Delivery Batch (SDG): a group environmental samples that arrive at the laboratory as one shipment or delivery group. There is no limit on the number of samples that may be delivered as a single group.

Blank: (note: "clean" for most Air Quality sampling media is defined as media that has had no sampling performed on it. Many air sampling media, including filters and sorbents materials, are not "analyte free")

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage, or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value.

Calibration Blank: An unspiked clean matrix of similar constitution as the sample extracts or digests (e.g., reagent Water, 5% HNO3, etc.) used to establish the zero intercept of the calibration curve.

Instrument Blank: a clean matrix (e.g., deionized water, 5% HNO3, etc.) processed through the instrumental steps of a method, used to determine instrument contamination.

Field Blank ("FB"): A blank prepared by the client in the field. The laboratory has no control over the actions of the client in the field. This blank is treated as a sample by the laboratory.

Laboratory Blank: (Gravimetric analysis only) A clean non-sampled filter or container that has been subjected to the same physical handling in the laboratory as the samples.

Method Blank ("MB"): A sample of a matrix similar to the batch of associated samples that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. No sampling media is contained in this blank (see "Sample Media Blank"). This blank demonstrates cleanliness of reagents and of the preparation process itself.

Reagent Blank ("RB"): All reagents, mixed in correct proportion, used in the preparation of samples, however, not taken through the preparation process. In the laboratory, this blank is rarely used, and usually only used when some question arises as to the source of contamination (reagents vs. process). Clients submit reagents blanks routinely; client reagent blanks are treated as a sample.

Proof Blank ("FTPB"): A blank prepared by the client in the field. The laboratory has no control over the actions of the client in the field. This blank is treated as a sample by the laboratory.

Field Train Recovery Blank ("FTRB"): A blank prepared by the client in the field. The laboratory has no control over the actions of the client in the field. This blank is treated as a sample by the laboratory.

Sample Media Blank ("SMB"): An unspiked aliquot of unsampled sampling media, taken through the entire preparation and analytical processes associated with a method. This blank determines if the sampling media may be contributing any analyte of interest to the samples, and may be used by the client to adjust or correct analytical results. The laboratory reports the SMB results to the client, however, performs no blank corrections of sample results prior to reporting results. "Blank subtraction" is the responsibility of the client. The laboratory has no control over the actions of the client.

Train Blank ("FTB"): A blank prepared by the client in the field. The laboratory has no control over the actions of the client in the field. This blank is treated as a sample by the laboratory.

Trip Blank ("TB"): A container with unsampled sampling media that is shipped from the lab to the field and back again, or from the field to the lab, without ever having been exposed to the sample gas stream or airshed. This blank is treated as a sample by the laboratory.

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

Support equipment calibration: the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

Analytical equipment calibration: the values realized by the standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Standard: A substance or reference material used for calibration.

Certified Reference Material (CRM): Reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute (e.g., NIST Class 0 and Class 1 weights, certified by an A2LA accredited laboratory).

Chain of Custody Form (**CoC**): A record that documents the possession of the samples from the time of collection to receipt by the laboratory. This record generally includes: the number and types of containers; the method of collection; the date of collection; and requested analyses.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: Second column confirmation, Alternate wavelength, Derivatization, Mass spectral interpretation, Alternative detectors or Additional cleanup procedures. No reference methods utilized by the laboratory require confirmation analysis.

Continuing Calibration Blank (**CCB**): A blank "standard" analyzed at the end of an analytical batch and at least every 10 samples during an analytical batch to verify that the lower end of the calibration curve remains valid during the course of the analytical run. See ICB.

Continuing Calibration Verification Standard (**CCV**): A second source standard, of a different lot or manufacturer from the calibration standards, analyzed at the end of an analytical batch and at least every 10 samples during an analytical batch to verify that the calibration curve remains valid during the course of the analytical run. See ICV.

Control Limit: A mathematical representation of acceptable limits for a given Quality Control Metric such as percent recovery or percent difference. Limits may be in the form of an absolute number or represented as a percentage.

Corrective Action: The action taken to address and/or eliminate, where possible, the causes of a non-conformity (such as exceeding a control limit) or failing to follow any documented non-analytical protocols (e.g., revising report numbers when resubmitting reports).

Corrective Action Report (**CAR**): A document, filled in by the person or persons finding a non-conformity with documented protocols, which documents the non-conformity and actions taken to correct it.

Correlation Coefficient: The statistical representation of how closely a set of x, y coordinates approaches the line of best fit. A correlation coefficient of 1.000 is considered a perfectly straight line of data points. Correlation coefficients above 0.995 are normally attainable by most instruments.

Data Integrity: The condition that exists when data are sound, correct and complete, and accurately reflect activities and requirements.

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more useful form.

Demonstration of Capability (**DoC**): A procedure to establish the ability of an Analyst to perform analyses with acceptable accuracy and precision.

Detection Limit: The lowest concentration of an analyte of interest that can be identified, measured and reported with 99% confidence that the analyte concentration is not a false positive value. The minimum result which can be reliably discriminated from a blank with 99% confidence.

Frequency: The number of occurrences of a specified event within a given interval. The number of samples or analytical runs with which a given QC sample or metric must be analyzed or verified.

Holding Time: The maximum time that can elapse between two specified events.

In-depth Data Monitoring: When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.

Initial Calibration Blank (**ICB**): A blank "standard" analyzed at the beginning of an analytical batch immediately after calibration to verify that the calibration curve is valid at the beginning of the analytical run.

Initial Calibration Verification Standard (**ICV**): A second source standard analyzed at the beginning of an analytical batch immediately after calibration that verifies that the calibration curve is valid at the beginning of the analytical run. This standard must be of a different source than the standards used to calibrate the instrument.

Laboratory Control Standard (**LCS**): A sample matrix, free from analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. Used to establish intra-laboratory or Analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Laboratory Control Standard Duplicate (**LCS**-**D**): A second LCS prepared and analyzed in the same manner, with the same preparation batch as the LCS. Used to assess precision when the sample or reference method precludes the ability to run a sample duplicate or sample spike duplicate.

Laboratory Information Management System (**LIMS**): A comprehensive computerized database system that the laboratory uses for sample tracking and data management, from sample receipt to reporting and disposal.

Limit of Quantitation (LoQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The laboratory sets the LoQ at five times the detection limit. It is used in setting acceptance limits for precision targets between duplicate analyses and determining concentration levels for LL-CCVs and LL-LCSs (see below).

Lot: A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.

Low Level Calibration Verification Standard (LL-CCV): Formerly Contract Required Detection Limit standard (CRDL or CRI or CRA). A standard at the LoQ used to verify the low end of the calibration curve. May be a primary or secondary standard. For instruments with only two points in the calibration curve (e.g., ICP), the LL-CCV will always be made from a second source standard.

Low Level Laboratory Control Sample (LL-LCS): A sample matrix, free from analytes of interest, spiked at a concentration between the DL and the LoQ, and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. Used to verify performance of the method at the low end of the calibration curve and to determine detection limits.

Matrix/**Matrices**: The component or substrate of a test sample. For the purposes of NELAP, this is comprised of "aqueous," "solid," or "air" matrices.

For the purposes of ***CHESTER LabNet***, the matrix is more specifically designated as size/type of filter, chemical composition of impinger solution, type of sorbent tube or other descriptor of the substance used to capture the analyte of interest. In addition, the matrix may also refer to characteristics of the gas stream from which the sample was obtained, such as moisture content, acidity, or interfering compounds present.

Matrix Duplicate ("Dup"): A second aliquot of a sample prepared and analyzed in the same manner, with the same preparation batch as the original sample aliquot. Used to assess precision.

Matrix Replicate ("Rep"): A second aliquot of prepared sample, analyzed when insufficient sample is present to perform a true duplicate analysis.

Matrix Spike ("spike"): A sample prepared by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. The spiked sample is taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. Matrix spikes are used to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (MSD): a second matrix spike of the same sample as used in the initial matrix spike sample, prepared and analyzed in the same manner, with the same preparation batch as the initial matrix spike sample. Used to obtain a measure of the precision of the recovery for each analyte. Only possible when sufficient sample is present to take a third aliquot from the original sample.

Measurement System: A method, as implemented at a particular laboratory, which includes the equipment used to perform the test and the operator(s).

Media: This term is used throughout the 2016 TNI Standard in reference to microbiological testing. For the purposes of air quality testing, media is generally comprised of either filters (various sizes/compositions), sorbent tubes, or solutions used in impinger trains.

Method: A body of procedures and techniques for performing an activity (e.g., chemical analysis, quantification, reporting), systematically presented in the order in which they are to be executed.

Method Detection Limit: See Detection Limit

National Institute of Standards and Technology (NIST): A federal agency of the US Department of Commerce's Technology Administration that is designated as the United States' National Metrology Institute (NMI).

NEAT (adj.): "Nothing Else Added To". The physical state of having not been altered (e.g., diluted, solubilized, etc.) from an unadulterated state.

Physical Parameter: A physical characteristic or property of a sample as distinguished from the concentrations of chemical components.

Post-Digestion Spike or Analytical Spike ("post spike"): an aliquot of prepared sample to which a known amount of target analyte is added for which an independent test result of target analyte concentration is available. Analyzed when a true spike is not possible which typically occurs with small sample sizes, or when the entirety of the sample is reduced to a single digestate.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms (e.g., relative percent difference, RPD).

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis (e.g., kept cold).

Procedure: A specified way to carry out an activity or process. Procedures can be documented or not.

Proficiency Evaluation Samples (Audit sample): A sample, the composition of which is unknown to the laboratory, designed to test whether the laboratory can produce analytical results within the specified acceptance criteria. Not all air methods have audit samples commercially available. It is not possible to create audit samples for some air methods.

Protocol: A detailed, written procedure for laboratory operation which must be strictly followed (see "Standard Operating Procedure").

Quality Assurance (**QA**): An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality Control (QC): The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as spikes, blanks, and duplicates, intended to demonstrate that a measurement system or activity is in control.

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to users. This document.

QC Statistic: Any of a number of statistical permutations performed on raw data to generate a metric capable of being subjected to control limits and corrective actions.

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products, and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities.

Quality System Matrix: This definition is to be used for the purpose of batch and QC requirements. Air and Emissions Matrices consist of whole gas or vapor samples. The matrix also includes the analytes of interest from a gas or vapor stream that are collected and the media with which the analytes are collected (e.g., sorbent tube, impinger solution, filter, or other media).

This laboratory primarily analyzes gas samples captured on filters, on sorbent media (filter or tube), and in impinger solutions.

Reagent: A single chemical, combination of chemicals, or a chemical solution used in the preparation or analyses of samples. Note: media, for the purposes of this document, is <u>not</u> a reagent.

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, electronic data, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.

Reference Material: Material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Method: A reference method is a published method issued by an organization generally recognized as competent to do so. ISO 17025:2015 also refers to this as a "standard method".

When a laboratory is required to analyze an analyte by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method.

Replicate: See "Matrix Duplicate."

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte from another component that may be a potential interferent or that may behave similarly to the target analyte within the measurement system.

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard (analytical): A solution or matrix (solid or liquid, prepared in the laboratory or purchased from a vendor such as a standard weight for balances) of a known amount of analyte(s).

> **Stock standard**: a standard received from a vendor with NIST or equivalent traceability.
>
> **Working standard**: any standard created when mixing, diluting or otherwise manipulating aliquots of primary standards; may be called "working standards" or "intermediate standards."
>
> **Primary Standard**: A standard typically used for calibration and no other purpose, received from a vendor with NIST or equivalent traceability.
>
> **Secondary Standard**: A standard of a different lot, manufacturer, or serial number from the primary standard received from a vendor with NIST or equivalent traceability typically used to verify calibration.

Standard (**NELAP**): The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and that meets the approval requirements of standard adoption organizations procedures and policies.

Standard (adjective): usual or typical; as in "The *standard* way to extract a solid for metals is with a hot acid digestion."

Standard (ambiguous): use of the word "standard" more than once in a sentence without enough context to make the meaning comprehensible, as in "The standard must be in keeping with the standard standard."

Standard Deviation (ambiguous): a quantity calculated to indicate the extent of deviation for a group as part or all of a whole.

> **Sample Standard Deviation:** a quantity calculated to indicate the extent of deviation for a data set, where the deviation only of the data points contained within the set is being described. This calculation is what is most commonly meant when "standard deviation" is discussed. The Excel formula is "=stdev"
>
> **Population Standard Deviation:** a quantity calculated to indicate the extent of deviation for a data set, where the calculated deviation of a data set is considered to be a subset of the entire population of data points being considered. This number is smaller than the results for Sample Standard Deviation. The Excel formula is "=stdev.p"

Standard Operating Procedure (**SOP**): A written document that details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. SOPs are not training manuals and thus may not contain all of the fine details a trained Analyst should know.

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.

Verification: Confirmation by examination and objective evidence that specified requirements have been met. In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation, or specification peculiar to the management of the measuring equipment.

For further definitions, please refer to the 2016 TNI Environmental Laboratory Sector Standard – Volume 1.

3.3.2 Acronyms

A list of acronyms used in this document and their definitions are:

AB	–	Accrediting Body
ASTM	–	American Society for Testing and Materials
Blk	–	Blank
°C	–	degrees Celsius
cal	–	calibration
CAR	–	Corrective Action Report
CARB	–	California Air Resources Board
CAS	–	Chemical Abstract Service
CCV	–	Continuing calibration verification
CFR	–	Code of Federal Regulations
CLP	–	Contract Laboratory Program (US EPA)
CoA	–	Certificate of Analysis
CoC	–	Chain of custody
CVAA	–	Cold Vapor Atomic Absorption Spectrophotometer
DL	-	Detection Limit (formerly “LoD”)
DoC	–	Demonstration of Capability
DQO	–	Data Quality Objective
dscf	–	dry standard cubic feet
dscm	–	dry standard cubic meters
EPA	–	Environmental Protection Agency
FoPT	–	Field of Proficiency Table
IC	–	Ion Chromatograph(y)
IC-PCD	-	Ion Chromatography with Post Column Derivatization
ICP	–	Inductively Coupled Plasma (Atomic Emission Spectrophotometer)
ICV	–	Initial calibration verification
IO	–	Inorganics Air Compendium
ISO	–	International Organization for Standardization
LCS	–	Laboratory Control Sample
LCS-D	-	Laboratory Control Sample Duplicate
LL-CCV	-	Low Level Continuing Calibration Verification
LL-LCS	-	Low Level Laboratory Control Sample
LoQ	–	Limit of Quantitation
MB	-	Method Blank
mg/Kg	–	milligrams per kilogram
mg/L	–	milligrams per liter
mg/Sx	–	milligrams per sample
MS	–	matrix spike
MSD	–	matrix spike duplicate
NELAC	–	National Environmental Laboratory Accreditation Conference
NELAP	–	National Environmental Laboratory Accreditation Program
NIOSH	–	National Institute of Safety and Health
NIST	–	National Institute of Standards and Technology
OC/EC	–	Organic Carbon/Elemental Carbon (analyzer)
ODEQ	-	Oregon Dept. of Environmental Quality
ORELAP	-	Oregon Laboratory Accreditation Program
OSHA	–	Occupational Safety and Health Administration
PE	–	Proficiency/Performance Evaluation sample

PM	–	Particulate Matter
$PM_{2.5}$	–	Particulate Matter 2.5 µm or smaller
PM_{10}	–	Particulate Matter 10 µm or smaller
PTP	–	Proficiency Testing Provider
PTPA	–	Proficiency Testing Provider Accreditor
QA	–	Quality Assurance
QAMP	–	Quality Assurance Management Plan
QAO	–	Quality Assurance Officer
QC	–	Quality Control
RE	-	Relative Error (percent, also %RE)
RO	–	Reverse Osmosis
RPD	–	Relative Percent Difference
RSD	–	Relative Standard Deviation
SMB	-	Sample Media Blank
SOP	–	Standard Operating Procedure
spk	–	Spike
std	–	Standard (analytical)
TNI	–	The NELAC Institute
TSP	–	Total Suspended Particulate
µg/L	–	micrograms per liter (air or liquid volume, dependent on context)
$\mu g/m^3$	–	micrograms per cubic meter
µg/Sx	–	micrograms per sample
UV	–	Ultraviolet
XRF	–	Thin Film X-ray Fluorescence spectrophotometer

3.4 Management of the Quality Assurance Management Plan

The Quality Assurance Officer (QA Officer) or their designated alternate is responsible for maintaining the currency of the Quality Assurance Management Plan (QAMP).

The Quality Assurance Management Plan is reviewed annually by the QA Officer and laboratory personnel to ensure that it reflects current practices and meets the requirements of any applicable regulations or client specifications. The manual is updated and the revision number is changed to the month and calendar year of the revision. If more than one revision in a given month/year is required, a letter is added after the year to indicate the new revision (e.g., “February, 2012B”). The cover sheet of the Quality Assurance Management Plan is then signed and the Table of Contents is updated.

The Quality Assurance Management Plan is considered confidential and proprietary, and may not be altered in any way except by approval of the QA Officer. If it is distributed to external users, it is for the purpose of reviewing ***CHESTER LabNet***'s management system for accreditation or contractual purposes and may not be used for any other purpose without the written consent of the laboratory.

3.5 Data Quality Objectives (DQOs)

For environmental laboratory activities, data quality objectives (DQOs) may be defined as qualitative and quantitative statements that specify the quality of the data required to support defined analytical requirements (U.S. EPA 1987). Data quality objectives provide the driving force for the level of quality control (QC) required for any analytical task. For example, a field laboratory providing only screening data would have DQOs much less stringent than a laboratory providing data to be used in enforcement actions. Thus the Quality Assurance Management Plan (QAMP) must be written to provide the level of quality control demanded by the end use of the data.

The paramount analytical requirement for ***CHESTER LabNet*** is that all measurement data be of the quality required to withstand the scrutiny of litigation. To meet this DQO, the ***CHESTER LabNet*** QAMP is structured to enable the laboratory to provide data of known and acceptable quality. The quality of data is considered known when all components associated with its derivation are thoroughly documented and traceable back to NIST standards. Data are of acceptable quality when a QA/QC program is carried out and the QC indicators fall within predefined limits of acceptability. One of the primary functions of the QAMP is to detail the methods of documentation and to define the mechanisms to be used in generating data traceable to NIST.

QA/QC requirements vary widely depending on the task being performed and the methodology utilized in performing said task. As such, it is the responsibility of the Analysts performing the work to be familiar with the QA/QC requirements of each analytical test performed, the SOPs describing those tests, and to ensure that work they are performing meet these requirements.

3.5.1 QA Mechanisms for Attaining DQOs

The quality assurance mechanisms used to attain predefined data quality objectives fall with five broad categories: precision, accuracy, comparability, representativeness, and completeness. The characteristics of these mechanisms are defined below. Targets for DQO's are summarized in Section 3.5.2.

3.5.1.1 Precision.

Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. For two measurements (duplicates), the relative percent difference (RPD) is used to represent precision. For more than two measurements, the percent relative standard deviation (%RSD, also known as the coefficient of variation or CV) is used to represent precision.

3.5.1.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator. The accuracy is represented by percent recovery (%R).

3.5.1.3 Comparability

Comparability is defined as the confidence with which one data set can be compared to another. Comparability in laboratory operations is important in analyzing samples for large projects where sample analysis may occur continuously over many days or may occur sporadically over a long period of time. Comparability is evaluated primarily on the basis of accuracy and precision statistics. There are no quality control estimators specific to comparability, and comparability must be approached as a data assessment task at a level above that of simply compiling QC statistics. In order to ensure data comparability, ***CHESTER LabNet*** has standard operating procedures and accepted analytical methods, and data is reported in units of measurement usable by the client.

3.5.1.4 Representativeness

Representativeness can be defined both qualitatively and quantitatively, and is dependent upon the selection of sampling site and choice of sampling methods. The degree of representativeness is important in planning for the collection of samples and has significant ramifications in the subsequent uses of the data. Sample collection methodology is the most significant contributor to sample representativeness. Unless the laboratory is directly involved in the sampling process, this element of representativeness is beyond the laboratory's control.

For air sampling, the laboratory can assist in the collection of representative data by minimizing spurious results caused by defective filter and sorbent media. This is accomplished by acceptance testing filter media and by conducting pre-sampling operations (e.g., tare weighing) in a controlled environment designed to prevent media contamination.

3.5.1.5 Completeness

Completeness is the amount of valid data actually obtained compared to the amount of data that was expected to be obtained under anticipated sampling/analytical conditions. As in the case for representativeness, the laboratory can assist in sampling completeness by providing air sampling media that have been acceptance tested, and have been prepared and shipped to ensure that samples are not lost due to physical deficiencies or higher than normal contamination.

The analytical component of completeness is controlled by employing qualified Analysts; by adhering to training protocols; and by using written standard operating procedures.

3.5.2 Targets for the DQO Mechanisms

The basis for the targets for the quantifiable DQO mechanisms is that of the U.S. EPA Contract Laboratory Program (U.S. EPA 1990). The default targets are as follows:

TNI Matrix	Sampling Medium	Precision	Accuracy	Completeness
Air	Impinger solution	20%	75 - 125%	99%
Air	Filter	20%	75 - 125%	99%
Air	Sorbent Tube	20%	75 - 125%	99%

Complete directives for all Precision and Accuracy limits are located in the QA/QC section of the Standard Operating Procedure for each analytical technique. DQOs vary from one analytical methodology to another; the table shown above is to be considered a general guideline.

DQOs may also vary from project to project, and from client to client. ***CHESTER LabNet*** works closely with the client to ensure that the quality of data generated is of a caliber suitable for the client's purposes.

Section 4

ORGANIZATION
(TNI V1:M2 – Section 4.1)

The laboratory is a legally identifiable organization. The laboratory is responsible for carrying out testing activities that meet the requirements of the 2009 and 2016 TNI Standard and ISO 17025:2005, and that meet the needs of the client, their client or their client's regulatory agency. Through application of the policies and procedures outlined in this Section and throughout the Quality Assurance Management Plan, the laboratory assures that:

- It is impartial, and that personnel are free from undue commercial, financial, or other pressures that might influence their technical judgment.
- Management and technical personnel have the authority and resources to carry out their duties, and the procedures to identify and correct departures from the laboratory's management system.
- Personnel understand the relevance and importance of their duties as related to the maintenance of the laboratory's Quality Management system.
- Ethics and data integrity procedures ensure personnel do not engage in activities that diminish confidence in the laboratory's capabilities (see Section 5 and Section 19 of Appendix A).
- Confidentiality is maintained.

4.1 Organization

CHESTER LabNet is an employee-owned, independent, commercial laboratory, incorporated in the state of Oregon as LabCor, Inc. DBA ***CHESTER LabNet.*** The laboratory has no legal ties to any other entity that might have any influence over or conflict of interest with the testing performed on site. The federal tax identification number is available upon request, solely on an as-needed basis.

The laboratory operates in Tigard, Oregon.

Additional information regarding responsibilities, authority and interrelationship of personnel who manage, perform or verify testing is included in Section 5, "Management" and Section 20, "Personnel". These Sections also include information on supervision, training, technical management, job descriptions, quality personnel and appointment of deputies for key managerial personnel.

The laboratory has the resources and authority to operate a management system capable of identifying departures from that system and from procedures during testing; and initiates actions to minimize or prevent departures.

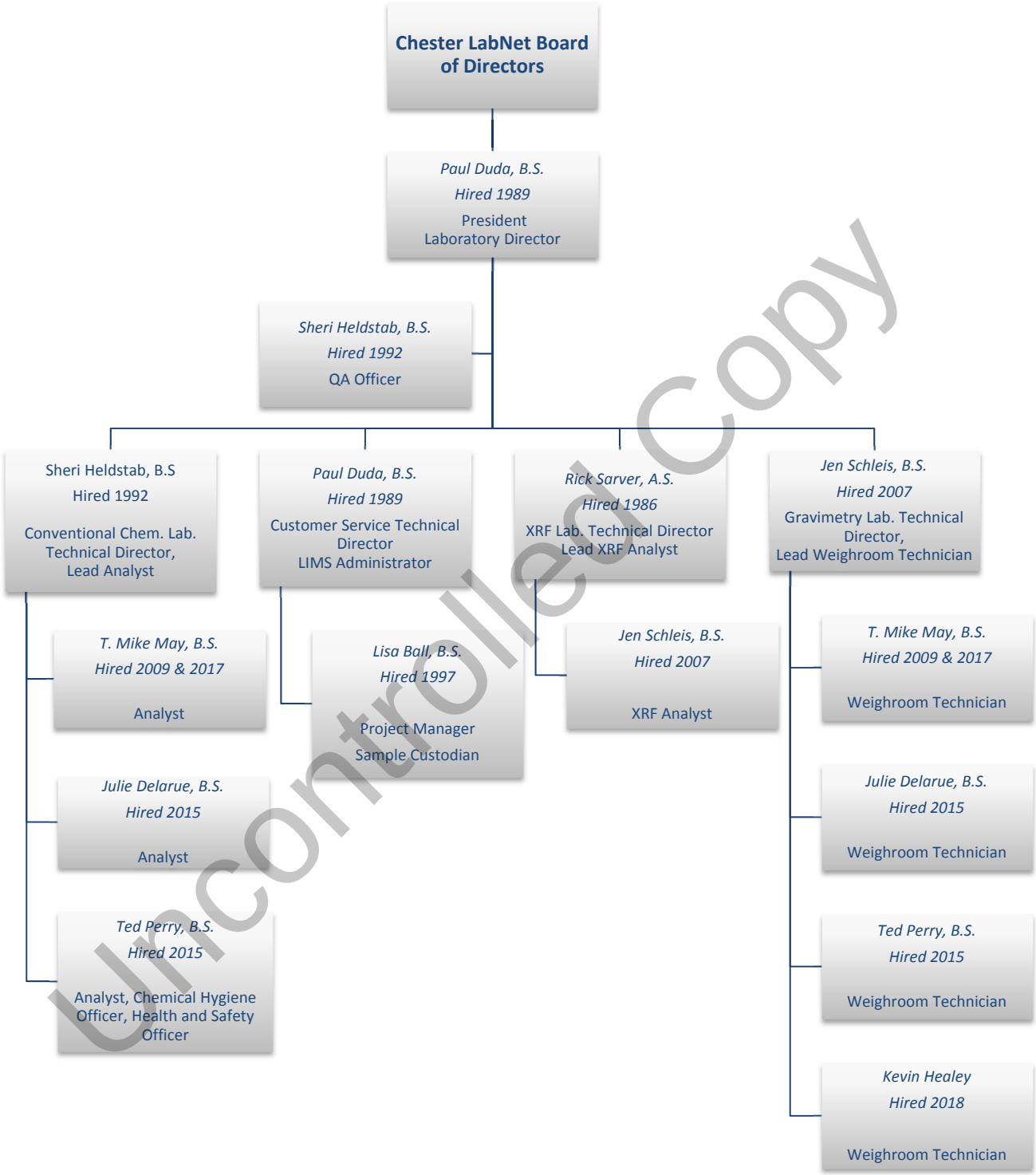


Figure 4-1. ***CHESTER LabNet*** Organizational Chart

4.2 Conflict of Interest and Undue Pressure

The organizational structure minimizes the potential for conflicting or undue interests that might influence the technical judgment of analytical personnel. In addition, procedures are in place to prevent outside pressures or involvement in activities that may affect competence, impartiality, judgment, operational integrity, or the quality of the work performed at the laboratory.

Due to the small size of the laboratory, some conflicts of interest are inevitable. For instance, the QA Officer and Analyst may be the same person. In some situations, it may be necessary for a person to audit an area of the laboratory in which they perform analysis or to verify their own data. The corporate culture of ***CHESTER LabNet*** is such that this is not considered by Management to be a significant conflict of interest. All employees understand the need for ethical integrity and perform their current task without regard for any previous or future tasks they may be performing. In other words, when the QA Officer puts on her QA Officer hat, she is acting as a QA Officer, not as an Analyst. All employees, from the time they begin working at ***CHESTER LabNet*** are imbued with the understanding that a failure to uphold ***CHESTER LabNet***'s ethics will result in an investigation and possible termination of the employee. See Section 5.4, "Ethics and Data Integrity" and Appendix A, "Ethics and Data Integrity Policy."

Policies and procedures to prevent commercial, financial or other influences that may negatively affect the quality of the work or negatively reflect on the competence, impartiality, judgment or operational integrity are described in the Ethics and Data Integrity Policy found in Appendix A.

Section 5

MANAGEMENT
(TNI V1:M2 – Section 4.2)

The laboratory maintains a management system that is appropriate to the scope of its activities.

5.1 Management Requirements

Top management includes the Laboratory Director, Technical Directors, and the QA Officer.

Management's commitment to good professional practice and to the quality of its data is defined in the Quality Policy statement, Section 5.3.

Management has overall responsibility for the technical operations and the authority needed to generate the required quality of laboratory operations. Management ensures communication within the organization to maintain an effective management system and to communicate the importance of meeting customer, statutory and regulatory requirements. Management assures that the system documentation is known and available so that appropriate personnel can implement their part. When changes to the management system occur or are planned, managers ensure that the integrity of the system is maintained.

Management is responsible for carrying out testing activities that meet the requirements of the 2009 and 2016 TNI Standard and ISO 17025:2015 Standard, and that meet the needs of the client.

Managers implement, maintain and improve the management system, and identify non-compliance with the management system of procedures. Managers initiate actions to prevent or minimize non-compliance.

Management ensures technical competence of personnel operating equipment, performing tests, evaluating results or signing reports, and limits authority to perform laboratory functions to those appropriately trained and/or supervised. Competence is ensured via review of previous experience/education, signed training documentation, DoC's, and Managerial oversight. Continuing competence is evaluated and documented during the annual employee review cycle, or more often if necessary. See Section 20, "Personnel."

All personnel performing work at ***CHESTER LabNet*** possess the necessary knowledge, skills and abilities to perform the work required. No duties or activities will be assigned to staff members not having the qualifications, training and experience to conduct such work. Training is provided as needed to each employee by a Lead Analyst or Technical Director (this excludes method development, which is performed by a Technical Director or designated alternate). Refer to SOP QA-001 "Laboratory Training" for further detail.

All personnel performing work at ***CHESTER LabNet*** are degreed professionals. See Appendix G for resumes of all personnel. Each employee's QC file contains a copy of their diploma or university transcripts and résumé as evidence of their educational background and laboratory skills.

All personnel performing analytical duties possess basic laboratory skills such as the ability to use an analytical balance, to properly read a meniscus line, to use autopipets and burettes, to perform basic mathematical calculations including proper dimensional analysis, and to properly identify glassware and its functions. In addition, these personnel possess knowledge of general laboratory vocabulary (e.g., "buffer", "titrant", "reflux", etc.), fundamental computer skills (e.g., saving files, opening software applications, finding files on a computer, etc.), and general laboratory safety.

Management is responsible for defining the minimal level of education, qualifications, experience and skills necessary for all positions in the laboratory, and assuring that technical staff have demonstrated capabilities in their tasks.

Training is kept up to date as described in Section 20, "Personnel" by periodically reviewing training records, examining QC data from each analysis, and reviewing employee performance annually via DoC's, ongoing QC checks, and annual employee competency reviews.

Management bears specific responsibility for maintenance of the management system. This includes defining roles and responsibilities for personnel, approving documents, providing required training, providing a procedure for confidential reporting of data integrity issues, and periodically reviewing data, procedures and documentation. The assignment of responsibilities, authorities and interrelationships of the personnel who manage, perform, or verify work affecting the quality of environmental tests is documented in Section 20, "Personnel."

Management ensures that audit findings and corrective actions are completed within required time frames.

Designated deputies may be appointed by management during the absence of the Laboratory Director, Technical Director(s) or the QA Officer, and are always appointed if the absence is more than 15 days. The accrediting body will be notified if the QA Officer or Technical Director is absent for more than 35 days. A Manager is not considered to be absent if still available electronically or telephonically and if the Manager is capable of making rational decisions and giving pertinent and timely instructions to other staff.

5.2 Management Roles and Responsibilities

5.2.1 Laboratory Director

The Laboratory Director is responsible for the financial, human resource and service performance of the laboratory. The Laboratory Director provides the resources necessary to implement and maintain an effective quality and data integrity program.

5.2.1.1 Responsibilities

The Laboratory Director is responsible for:

- ensuring that personnel are free from any commercial, financial and other undue pressures that might adversely affect the quality of their work;
- overseeing company financials, to include the purchase of new instrumentation and equipment;
- reviewing of all tenders and contracts;
- overseeing accreditation(s);
- ensuring adequate staffing (in tandem with Technical Directors);
- ensuring that the management system related to quality is implemented and followed at all times;
- engaging in management reviews of laboratory systems;
- ensuring clients are notified within 5 business days if events cast doubt on the validity of reported data; and
- overseeing client-specific analytical requirements.

5.2.2 Quality Assurance Officer

The QA Officer (or designee) is responsible for the oversight and review of quality control data. Due to the small size of the laboratory, the QA Officer is not free from other obligations in the laboratory. Refer to Section 4.2 for a description of ***CHESTER LabNet***'s resolution of conflicts of interest. The QA Officer's training and proof of experience in QA/QC procedures, knowledge of analytical methods, and the laboratory's management system can be found in the QA Officer's resume in Appendix G, "Staff Resumes."

5.2.2.1 Responsibilities

The QA Officer is responsible for:

- serving as a focal point for QA/QC;
- arranging or conducting annual internal audits without outside (e.g., managerial) influence;
- overseeing accreditation(s);
- notifying management of deficiencies;
- overseeing and reviewing quality control data;
- monitoring corrective actions;
- ensuring that the management system related to quality is implemented and followed at all times;

- monitoring and maintaining laboratory certifications;
- keeping this Quality Assurance Management Plan current;
- ensuring all SOPs are reviewed annually and keeping them current;
- ensuring that all Analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and that this training has been documented;
- ensuring that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits - procedures that do not meet the standards set forth in the Quality Manual, laboratory SOPs or laboratory policies may be temporarily suspended by the QA Officer;
- reviewing all logbooks for completeness and correct usage (in tandem with Technical Directors);
- ensuring all project specific data quality objectives and QA/QC targets are satisfied;
- ensuring all mandated systems requirements are met;
- issuing and archiving laboratory logbooks;
- reviewing and approving all SOPs and policies prior to their implementation; and
- ensuring availability of and adherence to all approved SOPs and policies.

5.2.3 Technical Director

The Technical Directors are laboratory staff members, and supervise laboratory operations and data reporting. The Technical Directors' proof of experience in the fields of accreditation can be found in their resumes in Appendix G.

If a Technical Director is absent for fifteen (15) calendar days or more, a deputy (see Table 5-1 below) with appropriate qualifications will perform the Technical Director's duties. If no employee has "appropriate qualifications," the most senior Analyst will become the deputy or work will cease in that department until such time as the Technical Director returns. Beyond a thirty-five (35) calendar day absence, management will notify the primary accreditation body in writing of the absence of the Technical Director and the appointment of the deputy. A Technical Director is not considered to be absent if still available electronically or telephonically, is capable of making rational decisions, and is capable giving pertinent and timely instructions to other staff.

The Technical Director is not the Technical Director of more than one accredited environmental laboratory. Due to the wide variation in Air Quality methods and analytical techniques, each department has its own Technical Director.

5.2.3.1 Responsibilities

The Technical Director is responsible for:

- meeting the general and education requirements and qualifications found in Sections 4.1.7.2 and 5.2.6.1 of the 2009 and 2016 TNI (note: no requirements are given for gravimetry or thin-film XRF laboratories);
- monitoring performance data and the validity of the analyses for the laboratory;
- providing technical direction to staff and clients;
- overseeing instrument and equipment certification, maintenance and repairs;
- monitoring data compilation and interpretation;
- training new employees or delegating training to a qualified Analyst;
- assessing qualifications of employees (education, experience, training, and performance);
- ensuring training records are completed for employees;
- where feasible, ensuring completion of an Initial DoC before newly trained Analysts are released from training;
- officially releasing newly trained Analyst(s) from training;
- coordinating operations within the laboratory to ensure smooth flow of samples through the analytical process (may need to be done in tandem with other Technical Directors); and
- supervising all Analysts to ensure compliance with all accreditations, regulations and client specific requirements.

5.2.4 Current Technical Directors and qualifications:

Rick Sarver: XRF Laboratory Technical Director
A.S. Science, 1980 (Chemekta Community College)
College Credit Hours in Chemistry: 33
Year hired at ***CHESTER LabNet***: 1986

Paul Duda: Customer Service Technical Director
B.S. Engineering Management, 1987 (University of Portland)
College Credit Hours in Chemistry: N/A
Year hired at ***CHESTER LabNet***: 1989

Sheri Heldstab: Conventional Chemistry Laboratory Technical Director
B.S. Biology, 1990 (University of Oregon)
College Credit Hours in Chemistry (B.S.): 37
Year hired at ***CHESTER LabNet***: 1992

Jennifer Schleis: Gravimetry Laboratory Technical Director
B.S. Environmental Management, 2003 (The University of Georgia)
College Credit Hours in Chemistry: 4
Continuing Education – Portland Community College, Portland State University
College Credit Hours in Chemistry: 21
Year hired at ***CHESTER LabNet***: 2007

Note that 2009 and 2016 TNI Volume 1, Module 2, Section 5.2.6.1 does not make reference to any requirements for the Technical Director of a laboratory or laboratory department in which only gravimetric analysis is performed. ***CHESTER LabNet***'s Laboratory Director and QA Officer have, together and in unison, agreed that 2 years of work experience in the Gravimetry Laboratory, coupled with a thorough understanding of the QA/QC requirements given in the various appendices of 40 CFR 50, other similar reference methods, and the applicable QA guidance documents, are grounds for promoting a person into the Gravimetry Laboratory Technical Director position.

5.2.5 Laboratory Key Personnel Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

Table 5-1 Key Personnel Deputies

Key Personnel	Deputy	Comment
Laboratory Director	QA Officer/Project Manager	Choice of deputy is dependent on nature of task.
QA Officer	Laboratory Director/most experienced Analyst	Choice of deputy is dependent on nature of task.
Technical Director – Conventional Chemistry	Most Experienced Analyst	
Technical Director – XRF	Most senior XRF Analyst	
Technical Director – Gravimetry	Most senior Gravimetry Laboratory Technician	

5.3 Quality Policy

Management's commitment to quality and to the management system is stated in the Quality Policy below, which is upheld through the application of related policies and procedures described in the laboratory's Quality Assurance Management Plan, SOPs and policies.

Quality Policy Statement: Our goal is to provide the most informed and accurate inorganic analyses of air quality samples available from a commercial laboratory by supplying our clients with data of known and documented quality. ***CHESTER LabNet***'s management is committed to good professional practice and to the quality of its environmental testing in servicing its clients. This policy is implemented and enforced through the unequivocal commitment of management, at all levels, to the Quality Assurance (QA) principles and practices outlined in this manual. All

personnel are familiar with the quality documentation requirements, and implement the policies and procedures in their work as attested to by their signatures on the Concurrences page of this document. The laboratory and its management are committed to complying with all requirements of any accreditations, contracts and governmental mandates.

CHESTER LabNet is proud of having specialized in the inorganic analysis of ambient particulates and source emission samples since its inception (as NEA, Inc.) in the late 1970's. The laboratory as an organization, its management and its personnel are all committed to the production of the highest quality data achievable with current methodologies and instrumentation, as well being committed to complying with contractual, regulatory and accreditation standards and requirements.

5.4 Ethics and Data Integrity System

The laboratory has an Ethics and Data Integrity policy that is included in Appendix A. The laboratory's Ethics and Data Integrity program, training and investigations are discussed in Section 19, "Data Integrity Investigations".

5.5 Documentation of Management / Quality System

The management system is defined through the policies and procedures provided in this Quality Assurance Management Plan and written laboratory Standard Operating Procedures (SOPs) and policies.

5.5.1 Quality Assurance Management Plan

The Quality Assurance Management Plan contains the following required items:

5.5.1.1 document title;

5.5.1.2 laboratory's full name and address;

5.5.1.3 identification of all major organizational units covered by this Quality Assurance Management Plan and the effective date of the version;

5.5.1.5 identification of the laboratory's approved signatories;

5.5.1.6 the signed and dated concurrence (with appropriate names and titles) of all responsible parties including the QA Officer, Technical Director(s) and the Laboratory Director;

5.5.1.7 the objectives of the management system and references to the laboratory's policies and procedures (where not explicitly contained herein);

5.5.1.8 the laboratory's official quality policy statement including the management system objectives and management's commitment to ethical laboratory practices and to upholding the requirements of all contractual, regulatory and accreditation standards and requirements; and

5.5.1.9 a table of contents and applicable lists of references, glossaries and appendices.

This Quality Assurance Management Plan contains or references all required elements as defined by the 2009 and 2016 TNI Standard, Volume 1, Module 2, Section 4.2.8.4.

5.5.2 Standard Operating Procedures (SOPs)

Standard operating procedures (SOPs) represent all phases of current laboratory operations and include an effective date, revision number and signature of the approving authorities as described in SOP QA-003, "Implementation, distribution and control of Standard Operating Procedures." SOPs are available to all personnel, and contain sufficient detail such that someone with similar qualifications could perform the procedures. There are two types of SOPs used in the laboratory: 1) administrative SOPs which document non-analytical procedures, and 2) method SOPs, which have specific requirements as outlined below.

Each accredited analyte or method has an SOP. The laboratory's method SOPs include the following topics:

i. identification of the method;
ii. applicable matrix or matrices;
iii. limits of detection and quantitation;
iv. scope and application, including parameters to be analyzed;
v. summary of the method;
vi. definitions;
vii. interferences;
viii. safety;
ix. equipment and supplies;
x. reagents and standards;
xi. sample collection, preservation, shipment and storage;
xii. quality control;
xiii. calibration and standardization;
xiv. procedure;
xv. data analysis and calculations;
xvi. method performance;
xvii. pollution prevention;
xviii. data assessment and acceptance criteria for quality control measures;
xix. corrective actions for out-of-control data;
xx. contingencies for handling out-of-control or unacceptable data;
xxi. waste management;
xxii. references;
xxiii. any tables, diagrams, flowcharts and validation data; and
xxiv. Deviations from reference methods and technical justifications for those deviations.

Refer to SOP QA-003, "Implementation, distribution and control of Standard Operating Procedures" for more detail on the written structure of each SOP.

5.5.3 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows unless otherwise noted:

- Client Directives ("client trumps all")
- Quality Assurance Management Plan
- SOPs and Policies
- Reference methods and associated QA Guidance documents

Section 6

DOCUMENT CONTROL
(TNI V1:M2 – Section 4.3)

This Section describes how the laboratory establishes and maintains a process for document management. Procedures for document management include controlling, distributing, reviewing, and accepting modifications. The purpose of document management is to preclude the use of invalid and/or obsolete documents.

Documents can be SOPs, "Quick Guides", policy statements, specifications, calibration tables, charts, textbooks, posters, notices, memoranda, software, drawings, plans, etc. These may be on various media, whether hardcopy or electronic, and they may be digital, analog, photographic, or written.

The laboratory manages four major types of documents: 1) controlled and approved, 2) suspended, 3) deactivated and 4) obsolete. In addition, they fall into three broad categories: Administrative documents (e.g., QAMP, Chemical Hygiene Plan), SOPs, and laboratory logbooks.

A controlled document is one that is uniquely identified, issued, tracked and kept current as part of the management system. Controlled documents are always internal and, consequently, must be approved.

An approved document means it has been reviewed, signed and dated by the issuing and approving authorities.

A suspended document is one which has not been used for an extended period of time, typically three to five years. These documents could be unsuspended at any time and brought back into use if needed. For example, ***CHESTER LabNet*** did not analyze pH using an electrode for over a decade, during which time the SOP for pH by electrode was suspended. A massive change to a reference method required the use of a pH electrode, consequently, the method was unsuspended, updated and brought back into use again. The suspension of a document is not necessarily permanent. While suspended, the document does not go through the annual review cycle. Once unsuspended, the document resumes its place in the annual review cycle. The purpose of this designation is to avoid the loss of time in reviewing a method which is not being actively utilized.

A deactivated document is one which the laboratory believes will never be used again. Frequently, these are project or client specific documents or documents for an instrument no longer in service. Deactivation of a document may also be the result of merging that document with another (e.g., combining two SOPs into one). Deactivated documents are not reviewed annually and are retained for evidentiary purposes only. By definition, a deactivated document is also an obsolete document.

Obsolete documents are documents that have been superseded by more recent versions or are no longer needed. These documents have an "effective until:" date noted at the bottom of the Cover Page and are retained in the QA Officer's files. In addition to the "effective until:" date, these documents are stapled together along the right-hand margin to indicate that it is not to be used.

6.1 Controlled Documents

Documents will be reviewed, revised (as appropriate) and approved for use by the QA Officer and the pertinent Technical Director prior to issue. In cases where the QA Officer is the person most familiar with the document requirements, a second person with or without the same degree of knowledge shall read and sign the document.

Documents are reviewed annually by all pertinent staff to ensure the contents are suitable, in compliance with the current management systems and requirements, and accurately describe current operations. A master list of SOPs is maintained electronically by the QA Officer to ensure annual review. Internal documents are uniquely identified with: 1) a unique name and number identification 2) date of issue, 3) revision identification. All documents are fully paginated in the form of "Page X of Y."

Original, signed SOPs are kept in 3-ring binders in the main office area of the laboratory. The obsolete versions are stapled along the right margin and retained in the archived SOPs file drawers. Deactivated or suspended SOPs are kept in the binders, are clearly marked as being deactivated or suspended, and are stapled along their right-hand edge. The production of new SOPs and the revision of existing SOPs are under the supervision and control of the QA Officer. Each SOP must be approved, signed and dated by a minimum of two people. The Annual Review is signed by the pertinent Technical Director.

Within the laboratory, original SOPs are always used as references. Copies are only made for submission to outside authorities and only on specific contractor or accreditor request. Electronic copies are watermarked on each page with "Uncontrolled Copy." Any physical copies of SOPs used for submission materials for new projects or for proposals are scanned and watermarked "Uncontrolled Copy" prior to printing for submission. "Controlled copies" do not exist for SOPs within the confines of the laboratory.

Originals of the general laboratory QAMP, project-specific Quality Assurance Management Plans, and the laboratory Chemical Hygiene Plan are kept with the original SOPs in the main office area of the laboratory. Production of new QA Manuals and revision of existing QA Manuals is under the supervision and control of the QA Officer. Each QA Manual must be signed and dated by the author. In addition, QA Manuals must be read, signed and dated by all affected laboratory personnel. This process is conducted annually for the general laboratory QA Manual and for all project-specific QA Manuals where an annual review is required. The date of issue is clearly marked on the title page.

As copies are only made for submission to outside organizations, ***CHESTER LabNet*** does not allow for a controlled copy within the laboratory. This makes the need to trace dispersed documents moot.

A master list of controlled copies submitted to outside organizations is maintained by the Laboratory Director. The list includes, by reference, the title, author, copyright date, date of publication and location. The controlled copy list is maintained

electronically and is updated each time a new document is sent to a requesting entity.

6.1.1 Document Changes to Controlled Documents

6.1.1.1 Paper Document Changes

Document changes are approved by the QA Officer or pertinent Technical Director during the annual review cycle.

The document management process allows for handwritten modifications to documents. As no controlled copies are distributed throughout the laboratory, and as Analysts must always refer to the original document, there is no need to track the changes (changes are "tracked" by their existence in the one master copy). Changes must be written in the original document in non-black ink, and dated and initialed by the person making the change.

All document modifications are approved by the personnel making the change. Changes that are not process modifications but clarifications may be performed without revision, but must still be dated and initialed. Process amendments/modifications to documents are incorporated into a new revision. The document is reissued when it has been reviewed and updated during its scheduled review cycle. Approval of changes by the QA Officer or pertinent Technical Director is required for the issuance of a new version or clean copy of the document.

A reason for the modification or change is provided as historical information in the revised document. All internal documents have a Cover Page and a Review History Page. The Review History Page tabulates the changes made over time to the document. Any major changes to the document content will be noted in this table (see Review History Page of this document).

6.1.1.2 Electronic Document Changes

CHESTER LabNet does not maintain electronically available documents. All documents, including SOPs and quality manuals, whether laboratory or project specific, are used by personnel in hardcopy form only. The QA Officer is responsible for maintaining the electronic versions of the documents. No other personnel are involved in the electronic maintenance or use of documents. Anytime a new electronic version of a document is created, such as during annual reviews, the QA Officer will rename the electronic version of the document by appending a year, or if necessary, a the year and a letter (i.e., 2016B), to the electronic file name of the document. Changes to the document are tracked by comparing the obsolete document to the current hardcopy document in use. Scanned versions of the current hardcopy are in .pdf format, and, aside from the "uncontrolled version" watermark, are unchanged from the original.

6.2 Obsolete Documents

All suspended, deactivated, invalid or obsolete documents are prevented from unintended use.

Obsolete documents retained for legal use or historical knowledge preservation are appropriately marked and retained. The obsolete versions have an "effective until:" date noted at the bottom of the Cover Page and are retained in the QA Officer's files. In addition to the "effective until:" date, these documents are stapled together along the right-hand margin to indicate that it is not to be used. Deactivated, suspended, invalid or obsolete documents are retained for a minimum of five years.

Section 7

REVIEW OF REQUESTS, TENDERS AND CONTRACTS
(TNI V1:M2 – Section 4.4)

The review of all new work assures that oversight is provided so that requirements are clearly defined, the laboratory has adequate resources and capability, and the method is applicable to the customer's needs or requirements. If the method is mandated by a regulator despite being archaic or contradictory, the laboratory will follow that method as closely as possible and in keeping with the chemistry or intent of the method.

This process assures that all work will be given adequate attention without shortcuts that may compromise data quality. Note: Air Quality methods not applicable to the needs of the client may be stipulated by a regulatory body. In such cases, the regulatory body has the final decision in methodology, even if the methodology is not suitable to the process being regulated.

A contract is defined as an agreement between the laboratory and their client to perform work and report results in a manner agreeable to both parties. This includes analytical methods, reporting formats, and any ancillary work requested such as cleaning of field sampling equipment or loading filters into cassettes.

Contracts for new work may be in the form of formal bids, signed documents, or verbal or electronic agreement. The client's requirements, including the methods to be used, must be clearly defined, documented and understood. Requirements might include target analyte lists, project specific reporting limits (if any), project specific quality control requirements (if any), turnaround time, and requirements for data deliverables. The review must also cover any work that will be subcontracted by the laboratory.

7.1 Procedure for the Review of Work Requests

The Laboratory Director and the appropriate Technical Director(s) for the area(s) being affected determine if the laboratory has the necessary accreditations, resources (including schedule), equipment, deliverables and personnel to meet the work request. The review for most work is documented in email exchanges between the Project Manager and the client. Note that many samples may arrive without forewarning. This process only applies to work requests made in advance of the receipt of samples. A notice of impending receipt of samples is not considered to be a contractual request (e.g., an email received from a client saying "we shipped the samples today, you should receive them tomorrow" does not qualify as a work request made in advance, nor does "we might be sending you samples in six months").

The Laboratory Director, Project Manager or Technical Director informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily.

The client is informed of any deviation from the contract, including the method or sample handling processes. All differences between the request and the final contract are resolved *and documented* before any work begins. It is necessary that the contract be acceptable to both the laboratory and the client. This information may be documented in email exchanges between the client and the personnel listed above. For large or ongoing projects, the Laboratory Director prints email exchanges and retains them in a project/contract specific file. For small or one-time projects, documentation may be recorded by hand on the Chain of Custody or Sample Receipt Checklist.

The review process is repeated when there are amendments to the original contract by the client. The participating personnel are notified, verbally or by email, of the amendments. For small projects or one-time-only projects (e.g., one sampling event) the amendments are maintained in email exchanges between the client and the laboratory or by handwritten changes on the Chain of Custody. For large or ongoing contracts requiring more stringent documentation, a contract specific hardcopy file is maintained by the Laboratory Director.

For routine projects and other simple tasks, a review by the Laboratory Director, Project Manager or Technical Director is considered adequate. The aforementioned person confirms that: the laboratory has all required certifications, can meet the client's data quality and reporting requirements, and has the capacity to meet the client's turn around needs.

For new, complex, or large projects, the proposed work contract is given to the Laboratory Director and the Technical Director(s) whose area(s) will be affected by the contract. The Laboratory Director, in tandem with the QA Officer and relevant Technical Director(s), will evaluate such items as:

- contractual obligations, bonding issues and payment terms;
- method capabilities, analyte lists, reporting limits and quality control limits;
- turnaround time feasibility;
- QA/QC issues, including certification/accreditation;
- formal laboratory quote;
- final report formatting and electronic deliverable documents;
- post-analytical sample storage requirements;
- final sample disposal requirements; and
- review of audit sample results, if any.

The Laboratory Director submits the bid and formal quote to the client, and maintains copies of all signed contracts.

For repetitive routine tasks, the review may be made only at the initial inquiry stage or on granting of a contract for on-going routine work performed under a general agreement with the client, provided the client's requirements don't change.

Any changes to contractually agreed upon processes are documented in a manner that is easily retrievable and in proportion to the change in the contract. If the client changes the scope of the project, but fails to notify the laboratory, the laboratory is not required to perform the review as described above.

7.2 Documentation of Review

Records are maintained for every contract or work request, when appropriate. This includes pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. Refer to Section 7.1 of this document for record keeping procedures.

Records of all project-related communication with the client (including e-mails, telephone conversations, etc.) are generally kept in each project file. For large contracts, the Laboratory Director creates a contract specific files and records are retained in this file, not in each project file/report. Refer to Section 7.1 of this document for record keeping procedures.

Section 8

SUBCONTRACTING OF ENVIRONMENTAL TESTS
(TNI V1:M2 – Section 4.5)

A subcontract laboratory is defined as a laboratory external to this laboratory, or at a different location than the address indicated on the front cover of this manual, that performs analyses for this laboratory.

When subcontracting analytical services, the laboratory assures work is placed with a laboratory that meets applicable statutory and regulatory requirements for performing the tests, unless otherwise requested by client.

8.1 Procedure

CHESTER LabNet very rarely subcontracts work. The Laboratory Director maintains a list of subcontractors.

For the rare occasions that the laboratory does subcontract work, ***CHESTER LabNet*** uses a laboratory with current NELAC accreditation status, unless otherwise requested by the client. The majority of subcontracting requests made by clients involve specialty laboratories that are not NELAC accredited. In these cases, the subcontracted lab is typically specified by the client. If the client requests that a non-NELAC accredited laboratory be used, the status of the subcontracting laboratory's accreditation will be noted in the Case Narrative of the final data report.

The laboratory may send sub-samples, or the samples or sample residues in their entirety, to the subcontracted lab(s), or the client may ship samples directly to the subcontracted lab(s) with a request to report the results to ***CHESTER LabNet*** for final reporting. The subcontracted laboratory's report is included *in toto* in the final report issued by ***CHESTER LabNet***. Billing is then performed by ***CHESTER LabNet.***

CHESTER LabNet reports do not contain data generated by another laboratory, unless that data is clearly indicated to have originated from another laboratory.

Section 9

PURCHASING SERVICES AND SUPPLIES
(TNI V1:M2 – Section 4.6)

The laboratory ensures that purchased supplies and services that affect the quality of environmental tests are of the required or specified quality.

The laboratory has procedures for purchasing, receiving and storage of supplies that affect the quality of environmental tests.

9.1 Procedure

The Laboratory Director reviews and approves the supplier of services and supplies, and verifies or approves technical content of purchasing documents prior to ordering.

Most frequently used services are provided by companies with known reputations and are procured from the manufacturers when possible. For servicing of instruments where the Analyst cannot repair the failure on site, the instrument manufacturer's field service technician is called. For the ICP servicing, this is primarily performed by Perkin-Elmer. IC servicing is performed by a Thermo field technician. OC/EC servicing is performed by Sunset Laboratories. The CVAA is serviced by a Nippon field representative. XRF's are serviced by Thermo-Electron. Balances are serviced annually by Quality Control Services, which is also the company responsible for recertifying all weights used in the calibration and verification of the balances, and all thermistors, thermometers, and thermometer/hygrometers. The HVAC system and fume hoods are serviced by USA Mechanical.

Evaluation of suppliers is accomplished by reviewing the packing slips, Certificates of Analysis, or other supply receipt documents to ensure the supplier has shipped the product or material ordered and that the material is of the appropriate quality and arrives fit for use, then stamping the packing slips and any accompanying Certificates of Analysis "received [date of receipt]." The purchasing documents, including Certificates of Analysis, contain the data that adequately describe the services and supplies ordered. The description may include type, class, grade, identification, specifications or other technical information.

The supplies received are inspected for breakage, leaks or any other damage. The supplies and chemicals are checked for expiration dates, then are marked "r: [date and initials of person receiving the supply]". They are stored according to manufacturer's recommendations, laboratory SOPs or reference method specifications. Consumable non-perishable supplies such as gloves, pipet tips, paper towels, etc., do not have a received date noted on the container.

Any documents received with the supplies and services including specifications, certificates of analyses, warranties, maintenance records, calibration records, etc., are kept on file. For further information, refer to SOP AD-005, "Reagent Procurement and Control."

The Conventional Chemistry Laboratory maintains 3-ring binders for the retention of certificates of analysis for: 1) dry chemicals, 2) inorganic liquids, 3) organic liquids, 4) standards, and 5) conventional chemistry laboratory support equipment calibrations. The person who writes the "r:" date on the container is responsible for obtaining the certificate of analysis, recording the received date (and their initials) on the certificate of analysis, verifying the expiration date (if any) on the certificate of analysis, and placing the certificate in the appropriate 3-ring binder.

The Gravimetry Laboratory maintains a 3-ring binder containing all the certifications of all NIST traceable weights currently in use, as well as balance maintenance and servicing records for all balances and NIST traceable weights. NIST traceability certificates for all NIST traceable electronic thermometers and hygrometers are maintained in the same binder. NIST traceable glass bodied thermometer certificates are retained in the Conventional Chemistry Laboratory's Support Equipment binder.

The purchased supplies and reagents that affect the quality of the tests are not used until they are inspected or otherwise verified as complying with requirements defined in the reference method.

9.2 Approval of Suppliers

As previously detailed (see Section 9.1), evaluation of suppliers is accomplished by reviewing the packing slips or other supply receipt documents to ensure the supplier has shipped the product or material ordered, that the material is of the appropriate quality, and then stamping the packing slips or other supply receipt documents "received [date of receipt]". The purchasing documents contain the data that adequately describes the services and supplies ordered. The description may include type, class, grade, identification, specifications or other technical information.

9.3 Reagent Water

Reagent water is manufactured onsite using a Millipore system. The manufacture of reagent water is discussed thoroughly in SOP AD-006. Briefly, water is generated using the system noted above. At the time of production, the resistivity of the water is measured by the De-ionizing system, and, at the start of each day of use, the resultant measurement is recorded in a reagent Water Control Chart located near the system. Resistivity is verified, but not documented, with each use throughout the day.

Reagent water is produced in compliance with, and meets the resistivity and TOC requirements of, ASTM D1193-06(2018), "Standard Specification for Reagent Water".

Section 10

SERVICE TO THE CLIENT
(TNI V1:M2 – Section 4.7)

The laboratory collaborates with clients and/or their representatives on clarifying their requests and monitoring laboratory performance related to the client's work. Each request is reviewed to determine the nature of the request and the laboratory's ability to comply with the request within the confines of prevailing statutes and/or regulations and without risk to the confidentiality of other clients.

10.1 Client Confidentiality

The laboratory confidentiality policy is to not divulge or release any information to a third party without proper authorization. A "third party" is defined as a person or entity who did not pay for the data generated, and therefore has no legal rights of ownership to the data. Third party requests for data and information are referred to the client. Data and records are not disclosed to third parties without permission from the owner (client), except in the case of subpoena. All subpoenas are sent to the laboratory's attorney prior to being acted upon.

All electronic data (storage or transmissions) are kept confidential, based on technology and laboratory limitations, as required by client or regulation.

All data produced by ***CHESTER LabNet*** is the property of the client. As such, ***CHESTER LabNet*** will not and does not release data to any other person, agency or business without prior verbal or written consent of the client. Verbal consent is documented and maintained in the data report.

In cases where data is subpoenaed, ***CHESTER LabNet*** will contact the laboratory's attorney prior to submitting data. In these rare instances, only the data directly mentioned in the subpoena are released to the subpoenaing authority. Data which may be related to the subpoena but was generated for a different client must be subpoenaed independently. Any situations that arise involving legal action are brought to the attention of the Laboratory Director and shall involve ***CHESTER LabNet***'s representing attorney to ensure the subpoena is correct, pertinent, legally viable and that any actions taken by ***CHESTER LabNet*** in releasing data are legally defensible.

In a situation where the laboratory goes out of business or changes ownership, each client shall be contacted and their wishes regarding the disposition of their data shall be carried out. The laboratory will not release any data to any organization who is not the client without the client's permission. The laboratory will follow the client's wishes in regards to all data, be it hardcopy, electronic, data packages, or electronic raw data.

10.2 Client Support

Communication with the client, or their representative, is maintained to provide proper instruction and modification for testing. Technical staff are available to discuss any technical questions or concerns the client may have.

The client, or their representative, is provided reasonable access to laboratory areas for witnessing testing or for auditing purposes. The laboratory reserves the right to limit access in the case of potential business competitors.

Delays or major deviations to the testing are communicated to the client immediately by phone or email. Communication may be made by the Project Manager, applicable Technical Director, or Analyst, depending on the nature of the communication. Major deviations may also be documented in the Case Narrative of the report.

The laboratory will provide the client with all requested information pertaining to the analysis of their samples. An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or not previously agreed upon.

If events cast doubt on the validity of results already reported to a client, the client will be notified of the concerns not more than five business days from the time of discovery. The laboratory will attempt to resolve or document the cause of concern prior to notifying the client. Documentation may be in the form of the email notifying the client of the concerns, or in the form of a Corrective Action Report.

10.3 Client Feedback

The laboratory seeks both negative and positive feedback following the completion of projects and periodically for ongoing projects. Feedback provides acknowledgement, corrective actions, where necessary, and opportunities for continuous improvement.

Negative customer feedback is documented as a customer complaint (see Section 11, “Complaints”).

For the duration of one month per year, ***CHESTER LabNet*** sends customer surveys out to every client receiving a data report that month. The highest rate of return of these surveys has been 40%, while the typical rate of return is around 20% – 25%. ***CHESTER LabNet***’s clients tend to be highly brand-loyal, and thus it is the laboratory’s experience that customer surveys always yield positive responses for the surveys returned.

Section 11

COMPLAINTS
(TNI V1:M2 – Section 4.8)

The purpose of this Section is to ensure that customer complaints are addressed and corrected. This includes requests to verify results or analytical data. Complaints provide the laboratory an opportunity to improve laboratory operation and client satisfaction.

Complaints by customers or other parties are reviewed by management and an appropriate action is determined. All customer complaints are documented by the person receiving the complaint and addressed to the appropriate member of Management.

If it is determined that a complaint is without merit, that is documented and the client is contacted by the person receiving the complaint, the appropriate Project Manager or the appropriate Technical Director.

While inquiries into data are not uncommon, complaints requiring action by the laboratory are rare. In either case, once contacted by the client, the Project Manager notifies the appropriate Technical Director or the QA Officer of the nature of the issue. That person locates the original data report and investigates the client's concern. If it is determined that the complaint has merit, the procedures outlined in Section 14, "Corrective Action" are utilized.

Errors Made by the Laboratory: While rare, errors do occasionally occur. If the issue at hand is the result of an error made by the laboratory (i.e., miscalculation, transposed numbers, decimal point errors, incorrect sample IDs, etc.), the laboratory will correct the error and issue a revised report to the client. In some cases, the client may request that the samples be re-analyzed. Where possible, this is performed.

Issues resulting from sample characteristics: Air Quality sampling is not a simple matter. Issues may arise over which the laboratory has little or no control (e.g., filter deposits not adhering to the filter, source emission samples having interfering analytes, impinger solutions with large quantities of particulate matter, etc.). In these cases and where possible, the client is notified prior to work being performed, and client agreement as to how to reconcile the matter is noted in the report. Where the client had been previously notified of such issues, the client complaints are referred back to the client's original statements. In cases where the issue could not be detected until after analysis (such as interfering compounds), the client will be notified prior to receiving the data if the problem is severe, otherwise the issue will be documented in the Case Narrative of the data report. The laboratory will explain the cause of the problem to the client, as well as what, if any, other courses of action may be taken to resolve the issue. In some cases, the client may request that the samples be re-analyzed or analyzed following a different method. Where possible, this is performed.

Unethical or Illegal requests by clients: On very rare occasions, client complaints take the form of the laboratory refusing to commit unethical or illegal actions. Requests made by clients to perform such actions are declined and an explanation given as to why the request is declined. Such requests have taken the form of asking the laboratory to analyze Sample A, but report the results as Sample B due to the loss of Sample B during shipping;

requesting that re-analysis be performed until the number desired by the client is obtained; or requesting that the laboratory take legal liability for actions performed in the field.

CHESTER LabNet does not and will not report false data or make false claims to any client. Samples from clients who persist in making such requests are refused in the future. ***CHESTER LabNet*** will not knowingly participate, directly or indirectly, in fraudulent activity. Any indications that the client may be using the laboratory's data in a fraudulent manner are documented in the Case Narrative and/or other areas of the data report. Requests for the laboratory to oversee or otherwise assume legal liability for actions occurring outside the laboratory's control are summarily refused, to include development of sampling plans and monitoring of field activities.

Billing Complaints: Client complaints regarding billing errors are directed to the Laboratory Director, who also executes all accounting functions for the company. The Laboratory Director will investigate the billing in question. Where errors are found, a revised invoice or credit memo to rectify the financial records will be issued.

Media and Supplies Complaints: Complaints regarding sampling media are referred to either the Project Manager or the Technical Director responsible for that particular media. In instances where the incorrect media was shipped to a client, the error will be corrected and appropriate media sent in a timely fashion to the client. It is ***CHESTER LabNet***'s policy that media, once sent to a client, cannot be returned unused as the laboratory cannot vouch for the integrity of the media once outside of its control. Complaints about the inability to return unused media are explained by this policy, with which most clients agree once they understand the logic behind it.

A complaint such as a concern that data is repeatedly late should be reviewed for preventative action (see Section 15, "Preventative Action") to minimize a future occurrence.

Section 12

CONTROL OF NON-CONFORMING ENVIRONMENTAL TESTING WORK
(TNI V1:M2 – Section 4.9)

Non-conforming work is work that does not meet acceptance criteria or requirements. Non-conformances can include departures from standard operating procedures or reference methods, or unacceptable quality control results (see Section 27, "Quality Assurance for Environmental Testing"). Identification of non-conforming work can come through various means including customer complaints, quality control, instrument calibration, consumable materials evaluation, staff observation, final report review, management reviews, and internal and external audits.

12.1 Exceptionally Permitting Departures from Documented Policies and Procedures

Requests for departures from laboratory procedures are approved by the Technical Director for the department in which the departure occurs and documented in the Case Narrative of the report to the client. Planned departures from procedures or policies do not require audits or investigations, and are also documented in the Case Narrative of the report to the client. In the field of Air Quality testing, departures at the request of the client or as a result of sample characteristics are quite common.

12.2 Non-Conforming Work

The laboratory's policy for control of non-conforming work is to identify the non-conformance, determine if it will be permitted, and take appropriate action. All employees have the authority to stop work on samples when any aspect of the process does not conform to laboratory requirements.

The responsibilities and authorities for management of non-conforming work are as follows: The employee who discovers the non-conformance is responsible for notifying their Technical Director. The Technical Director then notifies the appropriate client services personnel, as needed, and in proportion to the magnitude of the non-conformance. If the non-conformance affects data either in process or already reported, the client will be contacted by the QA Officer, Project Manager or Laboratory Director. The client is responsible for making the decision as to what to do with the non-conforming work (proceed, stop, change methodology, etc.).

The procedure for investigating and taking appropriate corrective actions of non-conforming work are described in Section 14, "Corrective Actions". Section 14.3 describes procedures for Technical Corrective Actions. Formal corrective action procedures must be followed for non-conforming work that could recur (beyond expected random QC failures) or where there is doubt about the laboratory's compliance with its own policies and procedures.

The investigation of and associated corrective actions for non-conforming work involving alleged violations of the company's Ethics and Data Integrity policies must follow the procedures outlined in Section 19, "Data Integrity Investigations".

The laboratory evaluates the significance of the non-conforming work and takes corrective action immediately. The customer is notified if their data has been impacted within five business days of discovery. The laboratory allows the release of non-conforming data only with approval by the appropriate Technical Director on a case-by-case basis. Non-conforming data is clearly identified in the Case Narrative of the final report (see Section 28, "Reporting the Results").

The discovery of a non-conformance for results that have already been reported to the customer must be immediately evaluated for significance of the non-conformance, its acceptability to the customer, and determination of the appropriate corrective action.

Corrective action for routine, non-recurring exceedances can be documented on raw data worksheets, logbooks, e-mail, a database or other documents. More serious corrective actions (non-conforming work that could recur or where there is doubt that the laboratory is in compliance with its own policies and procedures) will require a more formal corrective action process that typically includes the use of a corrective action report.

12.3 Stop Work Procedures

Personnel notify the appropriate Technical Director of any non-conformance not addressed in the SOP for that method. The Technical Director reviews the significance of the non-conformance and develops a course of action. If data are questionable, the QA Officer may be involved in the review and clients are notified.

When an investigation of non-conformance indicates that the cause of the non-conformance requires a method be restricted or not used until modifications are implemented, the Laboratory Director and Technical Director will immediately notify all personnel of the suspension/restriction. The lab will hold all relevant reports to clients pending review. The QA Officer must be involved in resolution of the issue and must verify that the issue is resolved before work may resume. Personnel are notified by the Technical Director when resumption of work is authorized. The Technical Director and QA Officer will document the issue, root cause and resolution using the corrective action procedures described in Section 14, "Corrective Action".

The Technical Director for the affected department authorizes resumption of work after it has been stopped.

The reporting of non-conforming work involving alleged violations of the company's Ethics and Data Integrity policies must be reported to the QA Officer and applicable Technical Director. Procedures described in Section 19, "Data Integrity Investigations" are followed.

Section 13

IMPROVEMENT
(TNI V1:M2 – Section 4.10)

Improvement in the overall effectiveness of the laboratory management system is a result of the implementation of the various aspects of the laboratory's management system: quality policy and objectives (Section 5, "Management"); internal auditing practices (Section 17, "Internal Audits"); the review and analysis of data (Section 27, "Quality Assurance for Environmental Testing"); the corrective action (Section 14, "Corrective Action") and preventative action (Section 15, "Preventative Action") processes; and the annual management review of the quality management system (Section 18, "Management Reviews") in which the various aspects of the management/quality system are summarized and evaluated, and plans for improvement are developed. The resulting Annual Managerial Report is the primary means by which Management monitors, and works to improve, laboratory systems (Section 18, "Management Reviews").

The Annual Managerial Review includes a detailed summary of the previous twelve months' records (see Section 18.1, "Management Review Topics").

Based on the Annual Managerial Review, Management may make changes to improve overall systems. When the Annual Managerial Review is completed, a staff meeting is held, the results are discussed, and input from all employees is taken for means of improving the laboratory's performance and client service.

All staff are expected to share ideas for improvement with their Technical Director or QA Officer. Most improvements implemented are the result of employees seeing novel approaches to various systems and methods. If an employee has an idea that is approved by the pertinent Technical Director or QA Officer, that idea is communicated to all affected personnel either verbally or by email. All employee-generated improvement schemes must be approved by either the pertinent Technical Director or QA Officer prior to being implemented on a regular basis.

Section 14

CORRECTIVE ACTION
(TNI V1:M2 – Section 4.11)

Corrective action is the action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence.

Deficiencies cited in external assessments, internal quality audits, data reviews, customer feedback/complaints, control of non-conforming work or managerial reviews are documented and require corrective action. Corrective actions taken are appropriate for the magnitude of the problem and the degree of risk.

14.1 General Procedure

All corrective actions not specified in method SOPs or other in-house documents are documented using a Corrective Action Report (CAR). This includes corrective actions by Analysts, client services personnel, findings from internal audits, customer inquiries and complaints, etc.

The first section of the CAR is completed by the person who discovers the issue. The second section is completed by the person who investigates the issue, determines the root cause and causes the corrective action to be carried out (may be a Technical Director or Analyst). The third and fourth sections are completed by the QA Officer, with the fourth section being completed after a check to ensure that the corrective action has been effective.

Completed Corrective Action Reports are retained in a 3-ring binder with a copy of the CAR placed in the client's job file, if applicable.

The person who discovers the non-conformity is responsible for initiating the corrective action where a non-conformance is found that could recur (beyond expected random QC failures) or where there is doubt about the compliance of the laboratory with its own policies and procedures. The QA Officer is responsible for monitoring and recording the corrective action.

All deficiencies are investigated and a corrective action plan is developed and implemented, when it is determined to be necessary. The implementation is monitored for effectiveness.

14.1.1 Cause Analysis

When failures due to systematic errors have been identified, the first step of the corrective action process starts with the initial investigation and determination of root cause(s) of the problem. Records of non-conformances requiring corrective action are maintained in a 3-ring binder of Corrective Action Reports including the results of the investigation to show that the root cause(s) was investigated.

Where there may be non-systematic errors, the initial cause is readily identifiable, or the failure is an expected random event (e.g., failed quality control), a formal root cause analysis is not performed. In this case, the process begins with selection and implementation of corrective action (also see Section 14.3, "Technical Corrective Actions").

14.1.2 Selection and Implementation of Corrective Actions

Where uncertainty arises regarding the best approach for analysis of the cause of exceedances that require corrective action, involved personnel will recommend corrective actions that are appropriate to the magnitude and risk of the problem and will be most likely to eliminate the problem and prevent recurrence.

The QA Officer, in tandem with affected personnel, ensures that corrective actions are discharged within the agreed upon time frame. It is not uncommon for the corrective action to have been implemented immediately upon discovery of the non-conformance.

14.1.3 Monitoring of Corrective Action

The QA Officer will monitor implementation and documentation of the corrective action to ensure that the corrective actions were effective.

After a Corrective Action Report (CAR) has been initiated and the root cause determined and addressed, the report is given to the QA Officer. The QA Officer allows the CAR to "age" for a period of 30 to 60 days. The QA Officer then interviews the involved personnel to ensure that the corrective action was both taken and effective, and that the problem has not recurred. At this point, the QA Officer signs off on the Corrective Action Report and places it in the 3-ring binder. A copy of the CAR is also placed in the affected job file, where applicable, for traceability.

14.2 Additional Audits

Where the identification of non-conformances or departures from normal lab procedures cast doubt on the laboratory's compliance with its own policies and procedures or on its compliance with the 2009 and 2016 TNI Standard, the laboratory ensures that the appropriate areas of activity are audited in accordance with Section 17, "Internal Audits" as soon as possible.

These additional audits are follow-ups after the corrective action has been implemented to ensure that it was effective. These are rare and done only when a serious issue or risk to the laboratory has been identified. Since 1994, there has not been a single need to implement this policy.

14.3 Technical Corrective Action

Sample data associated with a failed quality control parameter are evaluated for the need to be re-analyzed or qualified. Unacceptable quality control results are documented in the data report and, if the evaluation requires cause analysis, the cause and solution are recorded on a Corrective Action Report (also see Section 12, "Control of Non-conforming Environmental Testing Work"*).*

Analysts routinely implement corrective actions for data with unacceptable QC measures. First level correction may include re-analysis without further assessment. If the method SOP addresses the specific actions to take, they are followed. Otherwise, corrective actions start with assessment of the cause of the problem.

Corrective actions for non-systematic errors or expected random failures are documented in the Case Narrative of the data report. Depending on the severity of the non-conformance, documentation may be noted in the raw data or the non-conformance may be discussed in the Case Narrative of the report. Corrective actions for non-conformances that may recur (beyond expected random QC failures) or where there is concern that the laboratory is not in compliance with its own policies and procedures require that a Corrective Action Report be completed (see Section 14.1).

The QA Officer, in tandem with the Technical Directors and Analysts, reviews the Corrective Action Reports and suggest improvements, alternative approaches and procedures where needed.

If the data reported are affected adversely by the non-conformance, the affected data is clearly identified in the Case Narrative of the report and the customer is notified. It is common to contact the client for direction prior to the issuance of a report.

Section 15

PREVENTATIVE ACTION
(TNI V1:M2 – Section 4.12)

Preventative action is a proactive process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

Preventative actions include, but are not limited to: encouraging staff to discuss any ideas they may have for improving processes with the appropriate Technical Director, review of QC data to identify quality trends, review of client feedback to look for improvement opportunities, review of proficiency testing data to look for analytes that were nearly missed, annual managerial reviews, scheduled instrument maintenance, and other actions taken to prevent problems.

When improvement opportunities are identified or if preventative action is required, action plans are developed by the pertinent Technical Director, implemented, then monitored to reduce the likelihood of the occurrence of non-conformities.

Procedures for preventative actions include initiation of the actions and subsequent monitoring to ensure that they are effective.

All personnel have the authority to offer suggestions for improvements and to recommend preventative actions. Management is responsible for coordinating, implementing, and monitoring preventative action.

Section 16

CONTROL OF RECORDS
(TNI V1:M2 – Section 4.13)

Records are a subset of documents, usually data recordings, which include annotations such as daily refrigerator temperatures posted to a laboratory form, lists, spreadsheets, or Analyst notes on a chromatogram. Records may be on any form of media including electronic and hardcopy. Records allow for the historical reconstruction of laboratory activities related to sample-handling and analysis.

The laboratory maintains a record system appropriate to its needs, documents all laboratory activities, and complies with applicable standards or regulations as required. Records of original observations and derived data are retained to establish an audit trail. Records help establish factors affecting the uncertainty of the test and enable test repeatability under conditions as close as possible to the original.

16.1 Records Maintained

Records (or copies of records) are kept of all procedures to which a sample is subjected while in the possession of the laboratory. The laboratory retains all original observations, calculations and derived data (with sufficient information to produce an audit trail), calibration records, personnel records and a copy of the test report for a minimum of five years from generation of the last entry in the records. At a minimum, the following records are maintained by the laboratory to provide the information needed for historical reconstruction:

i) all raw data, whether hardcopy or electronic, for calibrations, samples, and quality control measures, including Analysts' worksheets and data output records (chromatograms and other instrument response readout records);

ii) a written description of, or reference to, the specific method(s) used, including a description of the specific computational steps used to translate parametric observations into reportable analytical values (a copy of all pertinent Standard Operating Procedures);

iii) laboratory sample ID code;

iv) date of analysis;

v) time of analysis, required when the holding time is seventy-two (72) hours or less, or when time-critical steps are included in the analysis (e.g., extractions);

vi) instrument identification and operating conditions/parameters (or reference to such data);

vii) all manual calculations (including manual integrations);

viii) Analyst's or operator's initials/signature or electronic identification;

ix) sample preparation, ID codes, volumes, weights, filter deposit area, instrument printouts, and reagents;

x) test results (including a copy of the final report);

xi) standard and reagent origin, receipt, preparation and use;

xii) calibration criteria, frequency and acceptance criteria;

xiii) data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

xiv) quality control protocols and assessment;

xv) electronic data security, software documentation and verification, software and hardware audits, backups and records of any changes to automated data entries;

xvi) method performance criteria including expected quality control requirements;

xvii) proficiency evaluation sample results;

xviii) records of demonstration of capability for each Analyst;

xix) a record of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record;

xx) correspondence relating to laboratory activities for a specific project;

xxi) corrective action reports;

xxii) preventative action records;

xxiii) copies of internal and external audits including audit responses;

xxiv) copies of all current and historical laboratory SOPs, policies and Quality Manuals;

xxv) sample receiving records;

xxvi) sample storage records;

xxvii) data review and verification records;

xxviii) personnel qualification, experience and training records;

xxix) archive records; and

xxx) management reviews.

16.2 Records Management and Storage

Program Documents: Process, Approval and Distribution

Currently, ***CHESTER LabNet*** has two program documents: Laboratory Quality Assurance Management Plan (QAMP) and the Chemical Hygiene Plan (CHP). All program documents are written and polished to a draft condition by the QA Officer or designated alternate prior to being submitted to the Laboratory Director as a draft version for review. The Laboratory Director makes comments on the draft version and returns it for revision. After making the requested revisions, program documents are circulated to the rest of the staff for comments. Once all comments have been addressed, the final copy is read and signed by all ***CHESTER LabNet*** personnel. The documents are stored in the main office area of the premises and are reviewed annually by all personnel.

Document Control

All original program documents and SOPs are stored in a bookcase in the main office area. Original finalized copies of these documents or binders containing documents may not be removed from the premises under any circumstances. Photocopies, electronic copies and/or draft copies of documents may only be removed from the premises with the approval of the QA Officer or Laboratory Director.

Due to the small size of ***CHESTER LabNet***, the laboratory has no formal document control system, nor does the laboratory have an SOP describing document control within the company. As no controlled copies of documents are permissible, excepting use for accreditation or project proposals, the need to trace dispersed copies is moot. SOP QA-003 does include a thorough description of the processes the laboratory uses to govern document generation, control, and archiving of obsolete documents. The QA Officer maintains an electronic master list of all in-house written documents on the QA Officer's computer. Technical Directors and Analysts are responsible for maintaining control of equipment/instrument specific manuals and literature.

All laboratory employees have access to the original documents at all times. All laboratory documents are reviewed on an annual basis by the QA Officer, the appropriate Technical Director or an alternate designated by them, and the employee(s) who perform the procedure. The QA Officer is also responsible for the preparation, approval and issuance of new documents.

Copies are not allowed to be made, except for submission to the client for the purposes of meeting contractual or proposal obligations. Any photocopy or electronic copy must be approved by either the Laboratory Director or the QA Officer. No copies of client specific documents (QAMP or any SOP) will be submitted to any other client. All original SOPs or laboratory documents must be signed on their Cover Page by at least two laboratory employees in BLUE ink. The Review History Page is signed by the appropriate Technical Director or QA Officer.

Any physical copy of a document will be clearly stamped with the word "COPY" on the Cover Page and can be distinguished from the original by a lack of signatures in blue ink. Electronic copies are distinguishable by a watermark reading "uncontrolled copy" on each page of the document.

Instrument Manuals are kept in a location near the instrument to which they apply, with the exception of support instrumentation/equipment, which may be kept in a binder in the laboratory where the support instrumentation/equipment is used. Instrument manuals are available at all times to all personnel. A listing of all manuals and their locations is located in Section 23.1, "General Equipment Requirements," of this document.

Standard Operating Procedures (SOPs) and "Quick Guides"

Original signed SOPs are kept in 3-ring binders in the main office area of the laboratory. Obsolete versions are stapled along the right margin and retained in the archived SOPs file drawers for a minimum of 5 years from their last effective date. The production of new SOPs and the revision of existing SOPs are under the supervision and control of the QA Officer. Each SOP must be approved, signed, and dated by a minimum of two people. In cases where the author is the person most familiar with the technique, a second person with or without the same degree of technical knowledge shall read and sign the SOP. Within the laboratory, originals are always used as references. Copies are only made for submission to outside authorities and only by specific contractual request. Electronic copies are notable by a watermark reading "uncontrolled copy" on each page of the document. Any physical copies of SOPs (e.g., to be used for submission materials for new projects or for proposals) must be stamped "COPY" in red ink across the title page. All SOPs are reviewed annually and a master list of SOPs is maintained electronically by the QA Officer to ensure annual review.

"Quick Guides" are appendices to some SOPs which contain frequently referenced information contained in the SOP. The production of new Quick Guides and the revision of existing Quick Guides are under the supervision and control of the QA Officer. Quick Guides are reviewed and approved annually as part of the SOP review.

QA Guidance Manuals

Originals of the general laboratory Quality Assurance Management Plan, project-specific Quality Assurance Management Plans, and the general laboratory Chemical Hygiene Plan are kept along with the original SOPs in the main office area of the laboratory. The production of new QA manuals and the revision of existing QA manuals are under the supervision and control of the QA Officer. Each QA manual must be signed and dated by the author. In addition, Quality Assurance Management Plans must be read, signed and dated by all affected laboratory personnel. This process is conducted annually for the general laboratory Quality Assurance Management Plan and for all project-specific Quality Assurance Management Plans where an annual review is required. The date of issue is clearly marked on the title page and the total number of pages is clearly marked at the top of each page, next to the specific page number.

Laboratory Logbooks

Bound laboratory logbooks are assigned numbers and dispensed by the QA Officer who maintains a bound logbook containing the dispensed logbook number, date of origination, use, and date the logbook is retired. The master logbook-tracking logbook is kept in the main office area of the laboratory. Filled logbooks are decommissioned by the QA Officer. The decommission date and archive location are noted in the same bound book and the decommissioned logbooks are kept in a series of labeled banker's boxes in the laboratory archive area.

Documentation stored in 3-ring binders is decommissioned by the QA Officer. The decommission date and archive location are noted in the same bound logbook as decommissioned logbooks. Decommissioned 3-ring binders are stored on archive shelves near the front office.

Document Production and Maintenance

All internal documents have a Cover Page and a Review History Page. The Review History Page tabulates the changes made over time to the document. Any major changes to the document content will be noted in this table (see Review History Page of this document). Minor changes can be noted in the original document in NON-BLACK ink, as long as those changes are dated and initialed by the person making the change. If a document is in the process of a major revision, handwritten changes do not need to be dated or initialed as those changes will propagate immediately to the new version, and the date will be noted in the revision history at the front of the document. Upon the introduction of a newly revised document, the obsolete version is retained in the QA Officer's files. Upon introduction of a newly revised SOP, the obsolete version is marked with an "effective until" date, stapled along its right-hand margin, and retained in the archived SOPs file drawer.

The production of new documents and the revision of existing documents are under the supervision and control of the QA Officer. Each document is approved, signed, and dated by the QA Officer and at least one other laboratory employee. Any copies of documents (e.g., to be used for submission materials for new projects or for proposals) must be stamped "COPY" in red ink across the title page. Electronic copies are notable by a watermark reading "uncontrolled copy" on each page of the document.

All documents are reviewed annually by the QA Officer and all pertinent employees for currency, accuracy, and clarity. Any revisions are noted in the Review History table at the front of the document. If no significant changes are needed, the review is documented by signing and dating the annual review line of the Review History Page. The Review History Page is signed by the pertinent Technical Director or QA Officer. Program documents are signed by, at a minimum, the QA Officer and Laboratory Director.

All electronic versions of documents are maintained on the QA Officer's computer. Changes to electronic documents are performed by the QA Officer, or, rarely, by the Laboratory Director. Handwritten corrections may be made to the original hardcopy by any employee, as long as that change is dated and initialed. If there

are numerous significant changes to content, the document is re-issued as soon as practicable. If the changes are minor, such as typographical errors, re-issuance may be delayed until such time as a major change requires it. The review history of each document notes changes made to the document and the name of the person making the changes. Electronic copies of all SOPs and Administrative Documents (such as this document) are password protected.

Analytical Record Keeping

Data are recorded immediately and legibly in permanent ink. Data generated by automated data collections systems are recorded electronically. Corrections are initialed and dated with the reason noted for corrections (other than transcription errors). A single line strikeout is used to make corrections such that the original record is not obliterated.

Analytical records associated with analysis are retained in varying formats and locations such as to create a documentation trail sufficient to create a historical account of the analysis of any given sample. These records and their locations are listed in the table below:

Record	**Location**
Client/Laboratory Sample ID	LIMS and final data report
Date/time of analysis	Raw data (final data report)
Instrument ID (may be in the form of analysis type – e.g., only one IC is used for the determination of Anions.)	Header of the instrumental printout or data sheets (final data report), LIMS
Instrument operating conditions	Instrumental method is usually noted in the header of the instrumental printout (final data report)
Analysis type	Final data report (data sheets or Case Narrative)
Manual calculations	Raw data (final data report)
Analyst's initials	Raw data (final data report)
Sample Preparation Logs	Raw data (final data report)
Sample Analysis	Raw data (final data report)
Standard and reagent origin, receipt, preparation and use;	Ordering and Receipt files, Certificate of Analysis, standards/reagents logs, SOPs (for reagents needing preparation immediately prior to use), sample preparation logs.
Calibration Criteria, frequency and acceptance criteria	Applicable SOPs
Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions	Final data report, applicable SOPs
Quality control protocols and assessment	Protocols contained in applicable SOPs. Assessment found in raw data and final

Record	Location
	report.
Method performance criteria	Applicable SOPs

The following records are maintained in the finalized hardcopy or electronic data report that is retained for a period of at least 5 years:

- all original data, in hardcopy format, or a record of where the hardcopy data is located;
- reference to the specific reference method used, where one exists;
- Cover Page, Case Narrative and copies of final data sheets;
- correspondence relating to the specific project;
- the identity of the personnel involved in sample receipt and log in;
- the identity of the personnel involved in sample preparation;
- the identity of the personnel involved in sample analysis;
- the identity of the instrumentation used during analysis;
- original observations;
- derived data;
- calibration records;
- staff records; and
- the identity of the person who checks the data QC indicators prior to reporting.

In addition to the above, the record keeping system allows for the retrieval of all working files and archived records via run logs, dates, data file names, preparation logs, etc. All handwritten changes to any logs are lined out using a single line so as not to obscure the original entry, then dated and initialed, and the reason for the change (other than transcription errors) is noted. All data recorded manually are recorded directly, promptly and in indelible black ink. Erasures or intentional overwriting of files are not permissible. Observations, data and calculations are labeled so as to link them unequivocally to the specific task and are recorded at the time they are made.

Changes to electronic files are rare and are usually performed by a Project Manager during the final report production stage. The person making the change is evident by which Project Manager is working with that data package. Entries to electronic records are made in such a way as to not erase or overwrite files. Reasonable measures are taken to avoid loss or change of original data in electronic records; however, unforeseen catastrophic electronic failures can occur, resulting in loss of electronic records.

Control of Data Reports

SOPs AD-007 and QA-008 document the production of data reports. Each data report will have all associated hardcopy documents necessary for the historical reconstruction of data contained within it, or within appropriate bound logbooks. This includes, but is not limited to: final data report pages, case narratives, chains of custody, raw data, digestion logs, any notes concerning client directives, observations of the samples, QC summary pages and any other documentation required by verbal or written contract with the client. All software-generated data are stored in hardcopy format within the data report.

Hardcopy data reports of all documentation not easily regenerated by the LIMS are retained for a period of no less than 5 years (e.g., raw data, CoC's, correspondence, Case Narratives, etc.). Copies of all reports submitted to clients, including documentation easily regenerated by the LIMS, are retained in .pdf format. Disposal of old records is carried out per client specifications (e.g., shredded, recycled, returned, etc.). Archived hardcopy reports are stored on-site in banker's boxes on ventilated shelves in a location with fire suppression devices. Active hardcopy reports are stored in lateral file cabinets. All documents pertaining to data generation are stored in a safe and secure environment, and held in confidence to the client.

Electronic Data Control

Primary control of electronic data occurs at the physical security level, by preventing any non-authorized persons access to the premises without an escort. Secondary control of electronic data is achieved by employing only personnel with proven ethical understanding of data integrity. Tertiary data control at the instrument level is controlled by the software auditing mechanisms built into the major instrumental software utilized by the laboratory. Quaternary electronic data control is achieved by retaining hardcopy records of all electronic data produced by the laboratory in appropriate project files.

The laboratory backs up electronic data including instrument data files, company data files, client data reports, emails, company financials, accreditation information, Standard Operating Procedures and administrative documents, detection limit studies and spreadsheets used for data reduction (for non-electronically generated data). This backup is performed daily, first to a different dedicated backup hard drive and then to 'cloud' storage.

Contract or Accreditation Specific Records

Contract or accreditation specific records are maintained for a period of time in keeping with the contract- or accreditation-specific requirements. These documents are stored safely and securely, as are all other documents, and are available at all times to the accrediting authority or contract representative.

Records, including electronic records, are easy to retrieve, legible and protected from deterioration or damage; held secure and in confidence; and are available to accrediting bodies for a minimum of five years or as required by regulation or contract. Records that are stored *only* on electronic media are supported by the hardware and software necessary for their retrieval. To prevent unauthorized

access or amendment, access to protected records is limited to employees in the department in which the records were generated.

Additional information regarding control of data is included in Section 22.5, "Control of Data".

The QA Officer or pertinent Technical Director notes when repositories of Quality and Technical records need archiving. The responsible person collects and, if necessary, binds the records. Records are then archived by the QA Officer using the master tracking logbook.

Archived information and access logs are protected against fire, theft, loss, environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources. Archived records have limited access, and are checked out through an access log. Both hardcopy and electronic archived records are stored onsite, with the exception of 'cloud' backup records.

In the event that the laboratory transfers ownership or goes out of business, records will be maintained or transferred according to client instructions. Appropriate regulatory and state legal requirements concerning laboratory records are followed. Note that the laboratory is owned in shares. Sales of shares does not constitute a change of ownership unless the laboratory changes names, Taxpayer Identification Number (TIN), or is otherwise no longer the legal entity responsible for the data produced.

16.3 Legal Chain of Custody Records

Evidentiary sample data are used as legal evidence. ***CHESTER LabNet*** does not *knowingly* analyze data for evidentiary purposes.

To establish the documentation necessary to trace sample possession, a chain of custody record should be filled out at the time of collection and accompany every sample. The record should contain the following minimum information:

- sample identification (***CHESTER LabNet*** laboratory identification number or client sample ID);
- sample tag number (if separate tag present);
- site (client sample ID or site location identifier);
- signature of sampler;
- date and time of sample collection;
- type of sample or referenced method number;
- signatures of all persons involved in the chain of custody;
- inclusive dates of possession; and
- analyses requested.

Each person who has custody must sign the chain of custody form. Samples must not be left unattended unless secured and sealed. Note that ***CHESTER LabNet*** has no control over whether or not a client submits a legally defensible chain of custody.

Due to its small and secured facilities, ***CHESTER LabNet*** does not utilize internal chains of custody. Samples are kept in a secure part of the facilities at all times, and visitors are not allowed within the confines of the facilities without an escort.

Section 17

AUDITS
(TNI V1:M2 – Section 4.14)

Audits measure laboratory performance and verify compliance with accreditation/ certification and project requirements. Audits specifically provide management with an on-going assessment of the management system. They are also instrumental in identifying areas where improvement in the management/quality system will increase the reliability of data. Audits are of four main types: internal, external, performance and system. Section 17.5 discusses the handling of audit findings.

17.1 Internal Audits

Internal Audits are conducted annually (every 12 – 14 months). These audits verify compliance with the requirements of the management/quality system, including analytical methods, SOPs, the Quality Assurance Management Plan, ethics policies, data integrity, other laboratory policies and the 2009 and 2016 TNI Standard.

It is the responsibility of the QA Officer to plan, organize, and ensure the performance of audits as required by the schedule. These audits are carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited.

In addition to the scheduled internal audits, it may sometimes be necessary to conduct special audits as a follow-up to corrective actions, PE results, complaints, regulatory audits or alleged data integrity issues. These audits address specific issues. If deficiencies found affect data, the client shall be notified and the issue shall be addressed.

The area audited, the audit findings, and corrective actions are recorded. Audits are reviewed after completion to assure that corrective actions were implemented and effective.

17.2 External Audits

It is the laboratory's policy to cooperate and assist with all external audits, whether performed by clients or an accrediting body. Management ensures that all areas of the laboratory are accessible to auditors as applicable and that appropriate personnel are available to assist in conducting the audit.

17.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, on-site auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such

information is entitled to such treatment." When information is claimed as business confidential, the laboratory includes the word "proprietary" in the title page of the document. Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory, however, sample identifiers may not be obscured from the information.

17.3 Performance Audits

Performance audits may be SSAS Audit Samples, internal single-blind samples, double-blind samples through a provider or client, or anything that tests the performance of the Analyst and method.

Proficiency Evaluation Samples are discussed in Section 27, "Quality Assurance for Environmental Testing".

Note that no TNI-defined Field of Proficiency Testing table (FoPT) exists for the Quality Matrix of "Air", therefore, the requirement for biennial analysis of PE Samples does not apply to the laboratory.

17.4 System Audits

The Laboratory's management system is audited though annual management reviews. Refer to Section 18, "Management Reviews" for further discussion of management reviews.

17.5 Handling Audit Findings

Internal or external audit findings are responded to within the time frame agreed upon at the time of the audit. The response may include action plans that could not be completed within the response time frame. A completion date is established by management for each action item and included in the response.

Development and implementation of corrective actions to findings is the responsibility of the QA Officer in tandem with affected personnel. Corrective actions are documented through the corrective action process described in Section 14, "Corrective Actions". If the corrective action reports described in Section 14 are unmanageable due to the size of the document required, a separate response to audit findings is created by the QA Officer. The response to audit findings is retained by the Laboratory Director in a file pertinent to the auditing body.

Audit findings that cast doubt on the ability of the laboratory to produce data of known and documented quality or that question the correctness or validity of sample results must be investigated. Corrective action procedures described in Section 14, "Corrective Action" must be followed. Clients must be notified in writing if the investigation shows the laboratory results have been negatively affected and the client's requirements have not been met. The client must be notified within five business days, verbally or in writing, electronically or hardcopy,

after the laboratory discovers the issue. Laboratory management will ensure that this notification is carried out within the specified time frame.

All investigations resulting in findings of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients. See Section 19, "Data Integrity Investigation," for additional procedures for handling inappropriate activity.

Section 18

MANAGEMENT REVIEWS
(TNI V1:M2 – Section 4.15)

Top management reviews the management system on an annual basis and maintains records of review findings and actions.

18.1 Management Review Topics

The following are reviewed to ensure their suitability and effectiveness:

- policies and procedures;
- reports from managerial and supervisory personnel;
- the outcome of recent internal audits;
- corrective and preventative actions;
- assessments by external bodies;
- the results of inter-laboratory comparisons or audit samples;
- changes in the volume and type of the work;
- customer feedback;
- complaints;
- recommendations for improvement;
- completeness record; and
- other relevant factors, such as quality control activities, resources and staff training.

18.2 Procedure

Once annually, the QA Officer, with the help of the Laboratory Director, shall gather records pertinent to the topics listed in Section 18.1. The Annual Managerial Review document compiles a detailed analysis of those items. This review is then presented to the entire company during a company meeting for input and discussion.

Managerial reviews are part of the annual internal audit/review process. If needed, at the end of the annual audit cycle, an annual audit summary report is written. This report summarizes the findings, corrective actions, follow-up procedures and any other items of note found during the annual audit. The report is retained by the QA Officer in that year's internal audit file.

The managerial review shall take account of:

- the suitability of policies and procedures including a review of the QAMP to verify that all elements are being followed;
- reports from managerial and supervisory personnel;
- the outcome of the annual internal audits;
- corrective and preventative actions;
- assessments by external bodies;
- the results of SSAS audit samples (See Section 18.1);
- changes in the volume and type of work;
- summary of client surveys;
- completeness record;
- quality control activities;
- resources: facilities and equipment;
- resources: staff and training; and
- goals, objectives and corrective action plans.

Findings and follow-up actions from management reviews are recorded. Management will determine appropriate completion dates for action items and ensure they are completed within the agreed upon time frame.

Section 19

DATA INTEGRITY INVESTIGATIONS
(TNI V1:M2 – Section 4.16)

In addition to covering data integrity investigations, this Section covers all topics related to ethics and data integrity policies, procedures and training.

CHESTER LabNet is committed to ensuring the integrity of its data and providing valid data of known and documented quality to its clients. The elements in ***CHESTER LabNet***'s Ethics and Data Integrity program include:

- annual data integrity training (held in tandem with the annual safety training);
- an audit program that monitors data integrity (see Section 17, "Audits") and procedures for handling data integrity investigations and client notifications;
- procedures for confidential reporting of alleged data integrity issues; and
- documented data integrity procedures in the form of an Ethics and Data Integrity Policy signed and dated by all management and staff (see Appendix A) as well as the signatures on this QAMP. All staff shall read and sign the Policy annually (see Appendix A). This policy is an integral part of the QAMP, and is signed, dated, and distributed by the QA Officer as part of the annual QAMP review cycle. It is available to all staff at any point in time by referencing the QAMP.

19.1 Ethics and Data Integrity Procedures

The Ethics and Data Integrity Policy provides an overview of the program. Written procedures that are considered part of the Ethics and Data Integrity program include:

- the Ethics and Data Integrity Policy (see Appendix A);
- manual integration procedures (see SOP QA-012);
- corrective action procedures (see Section 14);
- Data Integrity Investigations (see below);
- data recall procedures (see Section 14);
- data integrity training procedures (see Section 19.2);and
- annual management review of data integrity.

19.2 Training

Data integrity training is provided as a formal part of new employee orientation and a refresher is given annually for all employees. Employees are required to read and sign the Personal Ethics and Data Integrity Policy included in this

document (see Appendix A) during new employee orientation and again annually. This policy clearly states that any infractions of the laboratory data integrity procedures shall result in a detailed investigation that could lead to very serious consequences including immediate termination or civil/criminal prosecution. Attendance for required training is documented through the signatures and accompanying dates on the Data Integrity and Ethics Policy.

Data integrity training emphasizes the importance of proper written narration on the part of the Analyst in those cases where analytical data may be useful, but are in one sense or another partially deficient. All topics contained in the Personal Ethics and Data Integrity Policy are covered, and employees are given the opportunity to ask questions at the end of the training session.

The following topics and activities are covered:

- the organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting;
- how and when to report data integrity issues;
- record keeping;
- training, including discussion regarding all data integrity procedures;
- data integrity training documentation;
- in-depth data monitoring and data integrity procedure documentation; and
- specific examples of breaches of ethical behavior such as improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards.

When contracted technical or support personnel are used, the QA Officer is responsible for ensuring that they are trained in the laboratory's management system and data integrity procedures, are competent to perform the assigned tasks, and are appropriately supervised. These personnel are treated in the same manner as a regular employee and often become regular employees.

Topics covered are provided to all trainees in writing in the form of the Personal Ethics and Data Integrity Policy located in Appendix A of this document.

19.3 Confidential Reporting of Ethics and Data Integrity Issues

Confidential reporting of data integrity issues is assured through the following procedure: All staff members have the authority and responsibility to bring any problems, discrepancies, or concerns to the attention of their appropriate Technical Director. In situations where privacy is of concern, all staff have access to all other staff members' phone numbers. It is understood and encouraged that when needed, employees contact each other via their private phone numbers. All employees are required to inform their Technical Director or other member of Management if they have reason to believe that an investigation into data integrity is necessary.

19.4 Investigations

All investigations resulting from data integrity issues are conducted confidentially. They are documented and notifications are made to clients who received any negatively affected data that did not meet the client's data quality requirements.

The first stage of investigation is data inspection including, but not limited to: all associated documentation including reagents logbooks, calibrations, run logbooks, computer clocks and/or date/time stamps on the raw data, computer controlled environmental monitoring, etc. The inspection is conducted by the appropriate Technical Director, in conjunction with the Laboratory Director/President and QA Officer. If the inspection results raise concerns, those results and the concerns they raised, along with copies of the associated documentation are retained in the employee's personnel file.

If, in their opinion, a breach of ethical conduct has occurred (as opposed to an honest mistake or oversight), the same Management staff member shall then interview the employee. If the interview raises concerns, other employees may be interviewed and the results of the interviews are documented.

If the situation is deemed to be a breach of ethics by Management, documentation from both the records inspection and any subsequent interviews, along with a record of any actions taken, is placed in the employee's personnel file maintained by the Laboratory Director/President. The employee will be informed of the outcome of the investigation.

CHESTER LabNet does not tolerate unethical behavior of any sort by its employees, whether said behavior is related or unrelated to data production. If a breach of ethics is found to be supported by evidence, the employee may expect to be terminated. Conversely, Management begins with the assumption that errors are unintentional and oversights are honest mistakes. Management avoids the judgment error of assuming intent by testing against Hanlon's Razor.

Section 20

PERSONNEL
(TNI V1:M2 – Section 5.2)

CHESTER LabNet employs competent personnel based on education, training, experience and demonstrated skills as required. The laboratory's organization chart is located in Section 4, "Organization," of this document.

Competency is defined for three sub-sets of employees: Technical, Client Services, and Management. See Appendix J. Employees must achieve "Fair" or better ratings for all applicable competencies to be deemed competent. Competency is monitored and documented annually during the annual employee performance reviews. Documentation is stored in employee personnel files.

20.1 Overview

All personnel are responsible for complying with all quality and data integrity policies and procedures that are relevant to their area of responsibility.

All personnel who are involved in activities related to sample analysis and/or the evaluation of results, or who sign test reports must demonstrate competence in their area of responsibility. See above for definition of "competence." Appropriate supervision is given to any personnel in training or not fully competent, and the trainer is accountable for the quality of the trainee's work. Personnel are qualified to perform the tasks for which they are responsible based on education, training, experience, and demonstrated skills as required for their area of responsibility.

The laboratory provides goals with respect to education, training, and skills of laboratory staff. These goals are outlined in the specific job descriptions in Section 20.2.

Training needs are identified at the time of employment and when personnel are moved to a new position or new responsibilities are added to their job responsibilities. Ongoing training, as needed, is also provided to personnel in their current jobs. The effectiveness of the training must be evaluated before the training is considered complete.

Contracted personnel, when used, must meet the same competency standards and follow the same policies and procedures that laboratory employees must meet.

20.2 Job Descriptions

Job descriptions are available for all positions that manage, perform, or verify work affecting data quality, and are located below. Job descriptions include the specific tasks, minimum education, qualifications, skills and experience required for each position. An overview of top Managements' responsibilities is included in Section 5, "Management."

President

Goal:

- Ensure the business operates within compliance of all local, state, federal laws, including financial tracking and reporting;
- Communicate with board of directors.

Area of Responsibility: Corporate Affairs

Minimum education required: Higher degree in a hard science

Training required: Minimum 5 years as an employee of ***CHESTER LabNet***. Training, by necessity, may be autodidactic.

Minimum experience required: 5 years Project Management with ***CHESTER LabNet*** or 2 years equivalent Presidential job duties at another laboratory (environmental or other).

Qualifications:

- Knowledge of legal requirements of running a business;
- knowledge of bookkeeping and legal financial reporting; and
- knowledge of submission of Requests for Proposals (RFPs).

Brief description (including Managerial Duties):

- Oversees marketing and sales;
- performs legal financial reporting including calculating and disbursing dividend checks to owners, ensuring all taxes are paid in a timely manner, and managing payroll and pay rates for employees; and
- where possible, performs no functions related to testing, reporting or method development/modification.

Laboratory Director

Goal:

- Ensure human resource and service performance of the laboratory.
- Provide the resources necessary to implement and maintain an effective quality and data integrity program.

Area of Responsibility: Laboratory Management

Minimum education required: Higher degree in a hard science

Training required: Minimum 5 years as an employee of ***CHESTER LabNet***. Training, by necessity, may be autodidactic.

Minimum experience required: 3 years Project Management or Technical Director experience with ***CHESTER LabNet*** or 1 year equivalent Laboratory Director job duties at another laboratory (environmental or other).

Qualifications:

- Knowledge of legal requirements of human resource management; and
- knowledge of submission of Requests for Proposals (RFPs).

Brief description (including Managerial Duties):

- Ensures that personnel are free from any commercial, financial and other undue pressures that might adversely affect the quality of their work;
- oversees company financials, to include the purchase of new instrumentation and equipment;
- reviews all tenders and contracts;
- oversees accreditation(s);
- ensures adequate staffing (in tandem with Technical Directors);
- engages in management reviews of laboratory systems;
- oversees client specific analytical requirements; and
- where possible, performs no functions related to testing, reporting or method development/modification.

Quality Assurance Officer (QA Officer)

Goal:

- Review all data prior to reporting;
- write/maintain all Quality documents including SOPs and this document;
- ensure compliance with this Quality Assurance Management Plan; and
- ensure compliance with Data Integrity and Ethics Policy.

Area of Responsibility: Laboratory Management

Minimum education required: Higher degree in a hard science

Training required: Minimum 5 years as an Analyst with ***CHESTER LabNet***. Training, by necessity, may be autodidactic.

Minimum experience required: 5 years analytical experience with ***CHESTER LabNet*** or 1 year equivalent QA Officer job duties at another laboratory (environmental or other).

Qualifications:

- Knowledge of current TNI Standard requirements;
- knowledge of all QC requirements associated with methods performed; and
- knowledge of the general chemistry and techniques of all methods performed.

Brief description (including Managerial Duties):

- Serves as a focal point for QA/QC;
- arranges or conducts annual internal audits without outside (e.g., managerial) influence;
- notifies management of deficiencies and monitors corrective actions;
- oversees and reviews quality control data;
- monitors corrective actions;
- ensures that the management system related to quality is implemented and followed at all times;
- monitors and maintains laboratory certifications/accreditations;
- maintains currency of this Quality Assurance Management Plan;
- ensures all SOPs and Administrative Documents are reviewed annually and maintains currency of all SOPs and Administrative Documents;
- ensures that all Analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented;
- ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits - procedures that do not meet the standards set forth in the Quality Manual, laboratory SOPs or laboratory policies may be temporarily suspended by the QA Officer;
- reviews all logbooks for completeness and correct usage;
- ensures all project-specific data quality objectives and specific QA/QC targets are satisfied;
- ensures compliance with mandated systems requirements;
- issues and archives laboratory logbooks;
- reviews and approves all SOPs and policies prior to their implementation, and ensures availability of and adherence to all approved SOPs and policies;
- evaluates all results based upon QA elements described above and QC requirements for the pertinent testing;
- ensures new methods brought online meet QC criteria for Precision and Bias studies as required;
- ensures new methods brought online meet QC requirement for detection limit studies as required;
- performs statistical analysis for annual DL verifications or Initial DL studies for new methods; and,
- reports opinions and interpretations of data, where applicable.

Technical Director (all departments)

Goal:
Supervise laboratory department operations, data generation and reporting.

Area of Responsibility: Laboratory Management

Minimum education required: Higher degree in a hard science. In addition, the Technical Director must have a minimum of 24 credit hours of college level chemistry for departments where chemical analysis occurs (does not apply to gravimetry or customer service).

Training required: Minimum 3 years as an employee of ***CHESTER LabNet***. At the discretion of the QA Officer and Laboratory Director, an employee's past work experience may be substituted for 2 of the 3 years. Training, by necessity, may be autodidactic.

Minimum experience required: 2 years Analyst or technician experience with ***CHESTER LabNet*** or 1 year equivalent Technical Director job duties at another laboratory (environmental or other).

Qualifications:

- Knowledge of the general chemistry and techniques of all methods performed in respective department;
- knowledge of all instruments used in respective department;
- knowledge of all QC requirements associated with methods performed in respective department;
- knowledge of reporting requirements associated with methods performed in respective department; and
- meets the general and education requirements and qualifications found in Sections 4.1.7.2 and 5.2.6.1 of the 2009 and 2016 TNI Standard, Volume 1, Module 2, unless not specified.

Brief description (including Managerial Duties):

- Monitors performance data and the validity of the analyses for the laboratory;
- provides technical direction to staff and clients;
- oversees instrument and equipment installation, maintenance and repairs, to include preventative maintenance;
- monitors data compilation and interpretation;
- trains new employees (or delegates training to a qualified Analyst);
- assesses qualifications of employees (education, experience and training);
- ensures training records are completed for employees;

- ensures completion of initial DoC before newly trained Analyst may be released from training, where feasible;
- officially releases newly trained Analyst from training;
- coordinates operations within the laboratory to ensure smooth flow of samples through the analytical process (may need to be done in tandem with other Technical Directors);
- supervises all Analysts to ensure compliance with all accreditations, regulations and client specific requirements;
- plans tests and ensures adequate flow of work through the department;
- evaluates results against QC criteria as required;
- reports opinions and performs data interpretation, where applicable;
- oversees all method development, modifications and validation in the department; and
- coordinates day to day operations of the department including: flow of work, resource allocations, sample disposal, laboratory hygiene, supplies procurement and instrument maintenance and repair.

LIMS Administrator

Goal:
Ensure proper functioning, configuration and use of the LIMS.

Area of Responsibility: Administrative

Minimum education required: Higher degree in a hard science

Training required: Minimum 2 years as an employee of ***CHESTER LabNet***. Training, by necessity, may be autodidactic.

Minimum experience required: 2 years LIMS usage at ***CHESTER LabNet*** or 1 year equivalent job duties at another laboratory using NWA LIMS software (environmental or other).

Qualifications:
- Computer literate, and
- a general understanding of database operations.

Brief description:
- Operates and maintains (hardware/software) Laboratory Information Management System (LIMS);
- creates/edits/validates report scripts and worklists in tandem with the appropriate Technical Director; and
- where possible, performs no functions related to testing, reporting or method development/modification.

Project Manager

Goal:

& Lead Project Manager

Coordinate sample receipt, log-in, analysis and reporting to suit the clients' needs.

Area of Responsibility: Administrative

Minimum education required: Higher degree in a hard science

Training required: Minimum 2 years as an employee of ***CHESTER LabNet*** or Project Management training/experience at another laboratory (environmental or other). Training by LIMS Administrator in all functions of the LIMS.

Minimum experience required:

- Lead Project Manager: 2 years Project Management at ***CHESTER LabNet***
- Project Manager: none

Qualifications:

- Computer literate;
- professional demeanor, both written and spoken; and
- good communication skills.

Brief description (including Managerial Duties):

- Functions as primary contact person for client interactions;
- receives samples;
- performs chain of custody procedures;
- interfaces with client and Laboratory on corrective actions;
- packages and ships sample media to clients;
- performs data entry in LIMS;
- reports data;
- coordinates sample receipt, sample analysis and data reporting activities to ensure project turnaround times; and
- performs no functions related to testing or method development/modification.
- Additionally, the Lead Project Manager acts as a resource to other Project Managers, may act as another layer of Quality Control prior to submission of reports, and is responsible for adequate flow of work through the department.

Analyst / Lead Analyst

Goal:
Produce the most accurate data possible that meets QC and documentation requirements of all applicable methods, accreditations and contracts.

Area of Responsibility: Analytical

Minimum education required: Higher degree in a hard science

Training required: Must be trained in all areas, including the Quality System and Technical/Analytical procedures by a Lead Analyst. For the Lead Analyst, training may, by necessity, be autodidactic.
Minimum experience required:

- Lead Analyst: 2 years as an Analyst at ***CHESTER LabNet***.
- Analyst: none.

Qualifications:

- Computer literate;
- professional demeanor, both written and spoken;
- good communication skills;
- mathematical skills at the algebraic level or higher; and
- 1 year college-level general chemistry OR 1 year experience in performing laboratory analysis at another laboratory.

Brief description (including Managerial Duties):

- Analyzes samples under the direction/coordination of either the Technical Director or Lead Analyst in compliance with requirements of this document, pertinent SOPs, contracts or guideline documents;
- keeps records in compliance with requirements of this document, pertinent SOPs, contracts or guideline documents;
- enters data into LIMS;
- safely handles chemicals and laboratory equipment;
- troubleshoots, maintains and repairs instrumentation, with or without input from the Technical Director or Lead Analyst;
- evaluates data against pertinent QC requirements;
- reports opinions and performs data interpretation, where applicable;
- Lead Analyst is defined as the person with the most experience with a given method or analytical technique; and
- the Lead Analyst acts as a resource to other Analysts and may act as another layer of Quality Control prior to submission of data.

XRF Analyst / Lead XRF Analyst

Goal:
Produce the most accurate data possible that meets QC and documentation requirements of all applicable methods, accreditations and contracts.

Area of Responsibility: Analytical

Minimum education required: Higher degree in a hard science

Training required: Must be trained in all areas, including the Quality System and Technical/Analytical procedures by a senior Analyst.

For the Lead Analyst, training may, by necessity, be autodidactic.

Minimum experience required:

- Lead XRF Analyst: 2 years as an XRF Analyst at ***CHESTER LabNet***.
- XRF Analyst: none.

Qualifications:

- Computer literate;
- professional demeanor, both written and spoken;
- good communication skills;
- mathematical skills at the algebraic level or higher; and
- 1 year college physics or 1 year experience operating a thin-film XRF spectrophotometer (experience may include on-the-job training).

Brief description (including Managerial Duties):

- Analyzes samples under the direction/coordination of the Technical Director in compliance with requirements of this document, pertinent SOPs, contracts or guideline documents;
- keeps records in compliance with requirements of this document, pertinent SOPs, contracts or guideline documents;
- performs data compilation and spectral interpretation;
- enters data into LIMS;
- troubleshoots, maintains and repairs instrumentation, under the supervision of the Technical Director;
- evaluates data against pertinent QC requirements; and
- reports opinions and performs data interpretation, where applicable; and
- the Lead Analyst acts as a resource to other Analysts and may act as another layer of Quality Control prior to submission of data.

Gravimetry Laboratory Technician

Goal:
Produce the most accurate data possible that meets QC and documentation requirements of all applicable methods, accreditations and contracts.

Area of Responsibility: Analytical

Minimum education required: High School Diploma

Training required: Must be trained in all areas, including the Quality System and Technical/Analytical procedures by a Lead

Technician or Technical Director.

Minimum experience required: none

Qualifications:

- Computer literate;
- extremely good attention to detail;
- extremely good organizational skills;
- professional demeanor, both written and spoken; and
- good communication skills.

Brief description (including Managerial Duties):

- Performs all Gravimetry Laboratory operations and QA/QC, including acceptance testing and gravimetry;
- maintains appropriate inventory levels of filters and supplies;
- archives analyzed samples;
- follows all QA/QC protocols; and
- evaluates data against pertinent QC requirements.

20.3 Training

All personnel, including non-analytical personnel, are appropriately trained and competent in their assigned tasks before they contribute to functions that can affect data quality unsupervised. It is Management's responsibility to ensure personnel are trained. Training records are used to document Management's approval of completed training. The date on which authorization is confirmed is included.

Training records are generated by the person who performs the training and are maintained by the QA Officer. Records include the dates that training occurred and a brief description of what the training was, along with a "training completed" date.

An Analyst is considered trained when they can pass an IDoC study with no input from any other employee and the department Technical Director evaluates the Analyst's competency and finds is satisfactory. Training on a given method typically takes less than six months.

For further detail, refer to SOP QA-001, "Laboratory Training."

20.3.1 Training for New Staff

New staff members are trained in the following:

- requirements of the Quality Assurance Management Plan;
- requirements of the Chemical Hygiene Plan;
- relevant reference methods or SOPs which they will be performing;
- LIMS operation to the extent necessary for their job requirements; and

- administrative tasks to the extent necessary for their job requirements.

20.3.2 Ongoing Training

Refer to SOP QA-001, “Laboratory Training.”

Section 21

ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS
(TNI V1:M2 – Section 5.3)

21.1 Environmental

The laboratory facility is designed and organized to facilitate testing of environmental samples. Environmental conditions are monitored to ensure that conditions do not invalidate results or adversely affect the required quality of any measurement, such as the temperature and humidity in the Gravimetry Laboratory.

If the laboratory environment is required to be controlled by a method or regulation, the adherence is recorded. Gravimetry Laboratory temperature and humidity are recorded using a combination temperature and humidity digital data logger, and the balances are placed upon marble slabs to avoid problems with vibration. Environmental tests are stopped when the environmental conditions jeopardize the results. Relevant temperature/humidity readings are recorded in the raw data for methods that have specific temperature/humidity requirements, but are not performed in the Gravimetry Laboratory.

21.2 Work Areas

Work areas include access and entryways to the laboratory, the administrative area (which may serve as a sample receipt area), sample storage areas, sample processing and analysis areas, and chemical and waste storage areas.

Access to and use of areas affecting the quality of the environmental tests is controlled by restriction of areas to authorized persons only. See Section 21.4 below. Due to the small size of the laboratory, all employees are, by default, authorized personnel in all areas of the laboratory. Unauthorized persons could be people such as plumbers, field service technicians, visitors, etc.

The laboratory work spaces are adequate for their use, and appropriately clean to support environmental testing and ensure an unencumbered work area.

Laboratory space is arranged to minimize cross-contamination between incompatible areas of the laboratory. The Gravimetry Laboratory is on a separate HVAC system from the rest of the laboratory areas to ensure proper temperature and humidity control. Areas with high mineral acid usage are located in a different part of the conventional chemistry laboratory from where ion chromatography is performed. Sulfuric acid is not utilized near areas where reference Method 202 is being performed. Resuspension of particulates onto filters is performed in a separate area to prevent particulate contamination of other samples.

Testing occurs only within the laboratory's analytical areas (e.g., Conventional Chemistry laboratory, Source Particulate Laboratory, XRF Laboratory or Gravimetry Laboratory). Adequate laboratory space is maintained for the testing performed in

each area. Electronic balances are located away from drafts and doorways, and are mounted on marble slabs in areas where their use is affected by vibrations. Neighboring test areas of incompatible activities are effectively separated. Specific work areas are defined and access is controlled. (Only authorized laboratory personnel and escorted visitors may enter the work areas.) Good housekeeping measures are employed to avoid the possibility of contamination. Smoking is prohibited.

All equipment and reference materials required for accredited tests are available on-site. Records are maintained for all equipment, reference measurement materials and services used by the laboratory.

Reference materials traceable to national standards of measurement (NIST) or to national standard reference materials (SRM's) are stored away from heavy use areas or major equipment that may affect the proper operation of the materials. Certificates of Traceability are available for NIST traceable thermometers and hygrometers, for Class 0 and Class 1 weights, and for all commercially prepared aqueous standards. The reference materials are used only for calibration or calibration verification in order to maintain the validity of performance. Certificates of Analysis are available for all standards and reagents.

21.3 Floor Plan

See Appendix C.

21.4 Building Security

The laboratory is kept secure during off hours by the use of locks and an alarm system.

Access to the facilities is by cardlock during non-business hours, seven days a week. During business hours, the main door is unlocked and is monitored by personnel. The back door is locked 24/7 and only accessible via cardlock. Visitors are allowed in the laboratory under escort only.

Section 22

ENVIRONMENTAL METHODS AND METHOD VALIDATION
(TNI V1:M2 – Section 5.4 and Sections 1.4, 1.5 and 1.6 of Technical Modules TNI V1:M 3-7)

Methods and/or procedures are available for all activities associated with the analysis of the sample including preparation and testing. For purposes of this Section, the term "method" refers to both the sample preparation and determinative methods, except where the term "reference" precedes the word "method". See Appendix B for a listing of all ***CHESTER LabNet*** SOPs. See Section 25, "Collection of Samples," and Appendix D, "***CHESTER LabNet*** utilized reference methods" for a listing of the most common reference methods in use at ***CHESTER LabNet***.

Before being put into use, a method is confirmed by a demonstration of capability or method validation process, where possible.

All methods are published or documented. Deviations from the methods are allowed only if the deviation is documented, technically justified, authorized by management and accepted by the customer. Note that most source sampling methods (CFR methods) are extremely outdated and archaic, and ***CHESTER LabNet***'s clients are well aware of this. The laboratory does not notify clients of changes to methods necessitated either by the laws of chemistry and physics, or by the EPA's failure to update methods to reflect current technology.

Every SOP has an appendix which lists all of the differences between the laboratory's SOP and the reference method. Examples include using a computer rather than a strip chart recorder, using correct stoichiometry in calculations, and using instrument software to generate calibration curves rather than hand-plotting on graph paper.

22.1 Method Selection

A reference method is a method issued by an organization generally recognized as competent to do so. When ISO refers to a "standard method", that term is equivalent to "reference method". When a laboratory is required to analyze a parameter by a specified reference method due to a regulatory requirement, the parameter/method combination is recognized as a reference method.

The laboratory will use methods that meet the needs of the customer. Such methods will be based on the latest edition of the reference method unless it does not meet the needs of the customer. Generally speaking, the customer has little room for guesswork as there are a very limited number of reference methods for the analysis of either ambient air or source emissions. Both the client and the laboratory may be forced by a regulatory agency to use a reference method that is not appropriate to the ultimate client's needs. When the regulatory authority mandates a specific reference method, the laboratory will follow that method as closely as possible and in keeping with the chemistry and intent of the reference method. This statement is most commonly applicable to archaic methods found in the CFR for source emission testing.

CHESTER LabNet does not inform the client when a reference method is considered to be inappropriate or out-of-date, as our clients are already aware of those issues.

If a reference method is not specified by the client, the client is contacted and clarification is obtained prior to proceeding with any analytical work. Air quality methods (ambient or source) have two major components to them: sampling and analytical. ***CHESTER LabNet*** only performs the analytical portion of the method. The client will be aware of what method/analytes are needed. Typically, the omission of a reference method request is a simple documentation error.

All communications between the laboratory and the client are documented via printed email correspondence or notes on the Chain of Custody that have been dated and initialed by the laboratory agent contacting the client.

22.2 Laboratory-Developed Methods

If the laboratory develops a method, the process of designing and validating the method is carefully planned and documented. One person, usually the Technical Director of the affected department, will be responsible for developing the method.

In some cases, methods may be developed to fill in gaps found in other published or reference methods. On rare occasions, a method will be developed due to a lack of a reference method (e.g., Alkalinity in Teflon Filters). All methods developed in house, for whatever reason, will have an associated SOP. The in-house methods will undergo the same annual review cycle as all other SOPs, and shall contain the following information:

a) appropriate identification;
b) scope;
c) description of the type of sample to be tested;
d) parameters or quantities and ranges to be determined;
e) apparatus and equipment, including technical performance requirements;
f) reference standards and reference materials required;
g) environmental conditions required and any stabilization period needed;
h) description of the procedure, including:
    i. affixing of identification marks, handling, transporting, storing and preparation of samples,
    ii. checks to be made before the work is started,
    iii. checks that the equipment is working properly and, where required, calibration and adjustment of the equipment before each use,
    iv. the method of recording the observations and results,

v. any safety measures to be observed;

i) criteria and/or requirements for approval/rejection;

j) data to be recorded and method of analysis and presentation; and,

k) the uncertainty or the procedure for estimating uncertainty.

Laboratory-developed methods are only used if they are validated by a demonstration of capability study (where possible) and only if they are appropriate for the intended use. As with other new methods, the Technical Director is responsible for ensuring that the laboratory is capable of performing the method in such a manner as to meet all applicable QC requirements. This is achieved via a Demonstration of Capability Study and a Precision and Bias Study, where possible. Where not possible, other method specific QC criteria may be utilized to demonstrate capability. The development of new methods will always be assigned to the staff member with the greatest knowledge relating to that method and the ability to obtain all the resources needed to carry out the method.

When an occasion arises in which the employment of a non-reference method is needed, the method will be developed in conjunction with the client. These methods are almost never used by any other client or for any other project, and tend to fall into one of two categories: contingency and contractual.

Contractual methods are usually developed based on a method or methods supplied by the client. These may or may not be publicly available reference methods. Method development then proceeds until such time as the laboratory can demonstrate method proficiency and Precision and Bias, where possible (see SOP QA-006). Methods developed for contractual reasons must meet the client's approval prior to samples being analyzed. As method development may generate large quantities of documentation, most documentation is kept in a 3-ring binder in chronological order with notes as to what changes were being made during the maturation of the method.

Contingency methods tend to be utilized on a one-time basis, for engineering purposes or for the purposes of testing obscure/difficult/non-regulated matrices. As such, full validation of the method may not be possible and presents an undue burden on the laboratory. Such analyses will be documented fully, all directives issued by the client will be noted in the data file, and attempts are always made to get written confirmation from the client as to the acceptance of the proposed methodology. Documentation by the Analyst will include specifics, where not obvious, pertaining to the analysis.

The acceptance/rejection criteria for non-reference methods default to methods of similar chemistries or to CLP guidelines, if at all possible. When not possible, acceptance/rejection criteria may be based upon Precision and Bias studies and/or DL studies, or other method specific QC where DL and Precision and Bias Studies are not applicable (e.g., Method 202, filter impregnation, etc.).

22.3 Method Validation

Validation is the confirmation, by examination and objective evidence, that the particular requirements for a specific intended use are fulfilled.

At a minimum, reference methods are validated by performing an initial demonstration of capability, where possible. Additional requirements are discussed for each technology.

All non-reference methods are validated before use, where possible. The validation is designed to enable the laboratory to demonstrate that the method is appropriate for its intended use. All records (e.g., planning, method procedure, raw data and data analysis) are retained while the method is in use. Based on the validation process, the method's SOP will contain a statement of the intended use of the method and whether or not the validated method meets the use requirements.

Method validation and Demonstration of Capability procedures for methods in use at ***CHESTER LabNet*** is located in Appendix H, "Chemistry."

22.4 Estimation of Analytical Uncertainty

Analytical Uncertainty is a subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.

When requested and where possible, the laboratory will provide an estimate of the analytical uncertainty (reported as a percent or absolute numbers) determined by calculating the standard deviation of a statistically significant set of recovery values for known standards analyzed by the same method, then multiplying the standard deviation by the appropriate 99% confidence Student *t* value for the degrees of freedom present in the data set.

Due to the unique analytical requirements for air testing, this may not always be possible, as is the case with all gravimetric analyses (e.g., particulate measurements).

Two methods utilized at the laboratory issue uncertainties as part of standard reporting formats: analysis of metals by XRF and analysis of Carbon by OC/EC. A full explanation of determination of uncertainty for measurements by XRF is contained within SOP XR-005. Determination of uncertainties for OC/EC is performed by the instrument software.

22.5 Control of Data

To ensure that data are protected from inadvertent changes or unintentional destruction, the laboratory uses procedures to check calculations and data transfers (both manual and automated).

22.5.1 Computer and Electronic Data Requirements

The laboratory assures that computers, user-developed computer software, automated equipment, or microprocessors used for the acquisition, processing, recording, reporting, storage, or retrieval of environmental test data are:

- documented in sufficient detail and validated as being adequate for use;
- protected for integrity and confidentiality of data entry or collection, data storage, data transmission and data processing;
- maintained to ensure proper functioning and provided with the environmental and operating conditions necessary to maintain the integrity of environmental test data; and
- held secure, including the prevention of unauthorized access to, and the unauthorized amendment of, computer records. Data archive security is addressed in Section 16, "Control of Records" and building security is addressed in Section 21, "Accommodations and Environmental Conditions".

Primary control of electronic data occurs at the physical security level, by preventing any non-authorized persons access to the premises without an escort. Secondary control of electronic data is achieved by employing only personnel with proven ethical understanding of data integrity. Tertiary data control at the instrument level is controlled by the software auditing mechanisms built into the major instrumental software utilized by the laboratory. Quaternary electronic data control is achieved by retaining hardcopy records in appropriate job files of all electronic data produced by the laboratory.

Note that due to the small staff size of the laboratory, all employees are considered "authorized users" on all computers. For analytical computers, the user's initials will appear on the electronic files and in the associated run logs for that instrument.

The laboratory uses spreadsheets to calculate final results from the raw data for some analyses. Before reporting any results derived from these spreadsheets, the laboratory validates the underlying calculations by performing a sample calculation on at least 10% of the total data, selected at random throughout the spreadsheet. All mathematical steps from raw data to final reported data are verified manually. If a new calculation is created within the LIMS, the procedure for verifying spreadsheets is used.

After a spreadsheet has been developed and verified, subsequent use of the spreadsheet is verified by testing each set of cells used for input and output of the calculation. Any changes made to the spreadsheet are revalidated manually as described above. Some spreadsheets may be used only rarely; any spreadsheet that has not been used in more than 3 months will be revalidated as described above.

Electronically stored data from all electronic media, including the LIMS, emails, company financials, client contracts, SOPs and program documents, and all computers governing instruments are backed up to both a separate hard drive and to 'cloud' storage daily. See SOP AD-009 for further details.

22.5.2 Data Reduction

In cases where the Analyst calculates final results manually from raw data, all manual calculations will be verified by a second individual. The second individual shall document their verification by writing out the formula(e) they used to determine the final results on the raw data, then placing a tick or check mark next to each calculation they verified and, finally, writing "QC OK" at the top right corner of the raw data sheet along with their initials and the date they verified the data.

In addition and wherever possible, 100% of all manual data transfers (e.g., hand transcription, data entry into the LIMS) will be checked for accuracy by a different Analyst than the one performing the transfer. The second individual shall document their verification by placing a tick or check mark next to each entry they verified, and writing their initials and the date they verified the data at the top right corner of the page.

Appropriate computer programs may provide the results in a reportable format, although that is not typical for air quality methods. Usually, several different data sets are entered into the LIMS and the LIMS reports the data in the units as requested by the client. Typical units requested by the client include: ng/Sample, mg/Sample or µg/Sample; ng/m^3, mg/m^3 or $\mu g/m^3$; and µg/L (gas volume, not liquid volume).

The methods may provide required concentration units, calculation formulae and any other information required to obtain final analytical results, but do not always do so in a manner that meets the clients' needs. The clients' requests shall always be honored above any calculations given in any method. For example: 40 CFR 60 Method 8 requires reporting units of "meq SO_x/m^3", with an intermediary calculation of "meq SO_x/sample". Most clients request results in µg H_2SO_4/sample or µg SO_2/sample. In these cases, the client's requested reporting units are the units reported, regardless of the calculations in the reference method.

Some reference methods, particularly CFR methods, have errors in the reference calculations. In such cases, the laboratory calculates the data using correct formulae, regardless of that contained in the reference method. Any changes from the reference method calculations are documented in the "Differences from Reference Method" appendix in each SOP.

The laboratory has manual integration procedures that must be followed when integrating peaks during data reduction. These procedures are taught to new Analysts by the Technical Director or Lead Analyst overseeing the instrument that the trainee is learning to operate. Manual integration procedures are described in SOP QA-012.

The laboratory reports data to the number of significant figures specified by the method or client, or, if not specified by method or client, to three significant figures.

In cases where dilutions are performed solely to remove matrix interferences (not for samples over the calibration range), the same rules apply; however, the reported detection limit will be multiplied by the dilution factor.

Data is rounded following the guidelines in Section 7 of ASTM E29. Any "5" followed by a non-zero digit will round up. Any "5" followed by a "0" or lacking a trailing digit will round to the even number. Thus, 52.052 will round to 52.1, whereas 52.050 will round to 52.0 and 52.150 will round to 52.2.

All raw data are retained in hardcopy form in the report folder and in the form in which it was generated (e.g., computer files, logbooks, spreadsheets, etc.). It is maintained as described in Section 16, "Control of Records".

22.5.3 Confidentiality, Storage, Transmission and Processing

Data confidentiality is discussed in Section 10.1, "Client Confidentiality," and applies to all stages of data production.

Data storage is described in Section 16, "Control of Records" and Section 22.5, "Control of Data."

Data transmission is described in Section 28, "Reporting the Results."

Data processing is described in Section 22.5, "Control of Data."

22.5.4 Data Review Procedures

Data review procedures are located in Section 27.4, "Data Review".

Section 23

CALIBRATION REQUIREMENTS
(TNI V1:M2 – Sect 5.5 and Section 1.7 of Technical Modules TNI V1:M 3-7)

23.1 General Equipment Requirements

The laboratory provides all the necessary equipment required for the correct performance of the scope of environmental testing performed by the laboratory.

All equipment and software used for testing and sampling are capable of achieving the accuracy required for complying with the specifications of the environmental methods as specified in the laboratory SOPs.

Equipment is operated only by authorized and trained personnel (see Section 20, "Personnel").

The laboratory has procedures for the use, maintenance, handling and storage of equipment, and they are readily available to laboratory personnel. Manuals received from the manufacturer of the equipment provide information on use, maintenance, handling and storage of the equipment. Below is an equipment manual table that includes additional information on storage location:

Document	Title	Location
OC/EC Manual	Sunset Laboratory OC/EC Instruction Manual	Drawer below instrument
ICP Manual	Perkin Elmer Optima 8300 Hardware Guide	Drawer below instrument
WinLab32 software guide	Perkin Elmer WinLab32 for ICP software CD-ROM	Drawer below instrument
XRF Manuals	XRF Instruction Manual	Cabinet in center island near XRFs & bookshelf by Quant'X
Sartorius Manual (B120S)	Sartorius 120 Basic Series Instrument Manual & Operating Instructions	Drawer in center island in XRF room
CAHN Manual	CAHN C30/31 Instruction Manual	Drawer in center island in XRF room.
Sartorius Manual (ME5)	Sartorius ME & SE Series Operating Instructions	Drawer in center island in XRF room
Sartorius Manual (CPA224S)	Sartorius GemPlus Series	Drawer under balance in conventional chemistry lab
Sartorius Manual (MSA225S)	Sartorius Cubis Series	Drawer under balance in SPM laboratory

Document	Title	Location
Dehumidifier Manual	Kenmore Dehumidifier Manual	Drawer in center island in XRF room.
Humidifier Manual	Kenmore Humidifier Manual	Drawer in center island in XRF room.
Dehumidifier Manual	Whirlpool Dehumidifier Manual	Drawer in center island in XRF room.
Humidifier Manual	LASKO Recirculating Console Humidifier Model 1128	Drawer in center island in XRF room.
Humidifier Manual	Holmes Console Humidifier	Drawer in center island in XRF room.
Humidifier Manual	Duracraft Moisture Humidifier Owner's Manual	Drawer in center island in XRF room.
Reagent Water system manual	User Manual Milli-Q Direct 8/16 System	Countertop next to system cartridges
Air compressor dryer	Hankison Compressed Air Dryer Instruction Manual	Black bookcase in laboratory
CVAA Manual	Nippon 3320A Instruction Manual	Drawer under CVAA computer
UV/Vis Manual	Spectronic UV/Vis Model 20D Owner's Manual and Bench Manual	Black bookcase in laboratory
Hot Plate manuals	Cimarec Hot Plate/Stirrer Operating Instructions	Black bookcase in laboratory
	Thermolyne Hot Plate Operating Instruction	
Vortex Mixer	Vortex Mixer User Manual	Black bookcase in laboratory
Centrifuge	CL2 Centrifuge	Black bookcase in laboratory
Sonicator Manual	Branson Model 8510 and Model 8800 Operator's Manuals	Black bookcase in laboratory
Heated Sonicator Manual	ElmaSonic P Ultrasonic Cleaning Units	Black bookcase in laboratory
Hot bath Manual	Precision Water Bath 280 Series Installation and Service Manual	Black bookcase in laboratory
Oven Manual (VWR)	VWR Utility Oven Operating Instructions	Drawer near muffle furnace
Oven Manual (Binder)	Binder Operating Manual Heating Oven with Forced Convection	Drawer near muffle furnace
Muffle furnace manual	American Scientific Products FP-41 Furnace Owner's Manual	Drawer near muffle furnace
pH/mV meters (2)	VWR SympHony benchtop model	Center drawer, titration desk

The laboratory also has a policy for planned equipment maintenance. A summarized plan for equipment maintenance is located in table 23.5. These procedures ensure proper functioning of the equipment and prevent contamination or deterioration.

Each instrument has a bound maintenance logbook in which all malfunctions, repairs, preventative maintenance, and service visits are documented. Support equipment for each department has one common maintenance logbook. The maintenance logbooks are stored next to the appropriate instrument or in the department in which they are used. Every entry in a maintenance logbook shall have the following elements:

- date and initials of the Analyst making the entry;
- name of the person performing the repair or maintenance, if different than the Analyst;
- a complete description of the nature of the problem, symptoms or preventative maintenance;
- a description of the parts repaired/replaced/realigned; and
- proof that equipment is functioning properly after service.

The description of the maintenance/repair shall be thorough enough that another person reading the entry can identify what the symptoms were (if any), what the suspect parts were (if any), and what steps were taken to repair or maintain the instrument. Any hardware or software upgrades shall be noted in the maintenance logbook.

Preventative maintenance is scheduled based on guidance from the manufacturer and Analyst familiarity with their respective instruments. Preventative maintenance is noted in each instrument's maintenance logbook. All Technical Directors are responsible for scheduling/performing preventative maintenance on their instruments. Corrective maintenance can be performed either by the Technical Director, an Analyst, or by a field service technician, depending on the complexity of the repair needed. Both corrective and preventative maintenance is noted in the maintenance logbook and the name of the field service technician (if any) is included in the description of the repair. These procedures ensure proper functioning of the equipment and help prevent contamination or deterioration.

All equipment is calibrated or verified before being placed in use to ensure that it meets laboratory specifications and relevant standard specifications. Records are maintained by the QA Officer in tandem with the Laboratory Director for each major item of equipment and its software used for testing. The records include checks that equipment complies with the specifications; dates, results and copies of reports; and certificates of all calibrations, adjustments, acceptance criteria and the due date of next calibration where applicable; and the date received and date placed in service (if available). This record is the same record as described below.

Test equipment, including hardware and software, are safeguarded from adjustments that would invalidate the test result measurements by limiting access

to the equipment to authorized personnel only (see Section 22.5, "Control of Data"*)*.

Equipment that has been shown to be defective or outside specifications, has been subject to overloading or mishandling, or has given suspect results, is taken out of service. The equipment is isolated to prevent its use or clearly labeled as being out of service until it has been shown to function properly. In addition, it is the Technical Director's responsibility to notify all people within their domain, and all Project Managers, of the condition of the equipment. If it is shown that previous tests are affected, then procedures for non-conforming work are followed and results are documented (see Section 12, "Control of Non-conforming Environmental Testing Work" and Section 14, "Corrective Action").

No equipment outside of the permanent control of the laboratory is used.

Each item of equipment and software used for testing, and significant to the results, is uniquely identified. Records of equipment and software are maintained. This information includes the following:

a) identity of the equipment and its software;

b) manufacturer's name, type, identification, serial number or other unique identifier;

c) checks that equipment complies with specifications of applicable tests;

d) current location;

e) manufacturer's instructions, if available, or a reference to their location;

f) dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria and the due date of next calibration;

g) maintenance plan, where appropriate, and maintenance carried out to date;

h) documentation on all routine and non-routine maintenance activities and reference material verifications;

i) any damage, malfunction, modification or repair to the equipment;

j) date received and date placed into service (if available); and

k) condition when received, if available (new, used, reconditioned).

Table 23-1 Laboratory Equipment				
Name - Unique Identifier	**Location**	**Brand / Model**	**Serial Number** ***(Italicized numbers are capital equipment inventory numbers)***	**Month / Year Placed into Service**
Gravimetry Laboratory				
Cahn 1 microbalance (not in use since 2008)	Gravimetry Laboratory	Cahn C31	73139	2/1988
ME5 microbalance	Gravimetry Laboratory	Sartorius ME5	22006645	9/2007
Sartorius B120S balance	Gravimetry Laboratory	Sartorius B120S	38070080	6/1990
100 mg Class 1 Daily weight	Gravimetry Laboratory	unknown	69699	Date put into service changes annually
300 mg Class 1 Daily weight	Gravimetry Laboratory	unknown	69699	Date put into service changes annually
500 mg Class 1 Daily weight	Gravimetry Laboratory	unknown	69699	Date put into service changes annually
3.0000g Class 1 Daily weight	Gravimetry Laboratory	unknown	10139	Date put into service changes annually
5.0000g Class 1 Daily weight	Gravimetry Laboratory	unknown	10139	Date put into service changes annually
100.0000g Class 1 Daily weight	Gravimetry Laboratory	unknown	14091	Date put into service changes annually
100 mg Class 0 Monthly weight	Gravimetry Laboratory	unknown	1000155857	Date put into service changes annually
300 mg Class 0 Monthly weight	Gravimetry Laboratory	unknown	1000155856	Date put into service changes annually
500 mg Class 0 Monthly weight	Gravimetry Laboratory	unknown	1000155858	Date put into service changes annually
3.0000g Class 1 Monthly weight	Gravimetry Laboratory	unknown	10147	Date put into service changes annually

Table 23-1 Laboratory Equipment				
Name - Unique Identifier	**Location**	**Brand / Model**	**Serial Number** ***(Italicized numbers are capital equipment inventory numbers)***	**Month / Year Placed into Service**
5.0000g Class 1 Monthly weight	Gravimetry Laboratory	unknown	10147	Date put into service changes annually
Computer tracked thermometer/ hygrometer	Gravimetry Laboratory	Dickson TP125	(unreadable)	2/2007
Computer tracked thermometer/ hygrometer	Gravimetry Laboratory	Dickson TP125	16305824	5/2018
Secondary thermometer/ hygrometer	Gravimetry Laboratory	VWR Traceable	Serial number changes with expiration date	Date put into service changes annually
Max/Min thermometer	OC/EC Freezer #5	VWR 89094-770	Serial number changes with expiration date	Date put into service changes annually
Max/Min thermometer	Standards Refrigerator #5	VWR 89094-770	Serial number changes with expiration date	Date put into service changes annually
Max/Min thermometer	Conventional Lab Fridge/Freezer #6	VWR 89094-770	Serial number changes with expiration date	Date put into service changes annually
IR thermometer	Sample Receiving	VWR Traceable	Serial number changes with expiration date	Date put into service changes annually
Cabinet Desiccator (small)	Gravimetry Laboratory	Boekel	None	3/2011
Humidifier	Gravimetry Laboratory	LASKO 1128	225651	3/2013
Humidifier	XRF	Holmes HM3650	unknown	Pre-1992
Humidifier	SPM Laboratory	DuroCraft DH-836/837	unknown	Pre-1992
Humidifier	Gravimetry Laboratory	Kenmore 758.154120	08128	Pre-1992
Dehumidifier	Gravimetry Laboratory	Kenmore 106.57500790	QG1104204	8/1997
Dehumidifier	XRF	Whirlpool AD5OUSLI	QM1328295	8/2002
Dehumidifier	XRF	Hisense	KGFGHJA0439	6/2018

Table 23-1 Laboratory Equipment				
Name - Unique Identifier	**Location**	**Brand / Model**	**Serial Number** ***(Italicized numbers are capital equipment inventory numbers)***	**Month / Year Placed into Service**
Dehumidifier	SPM Laboratory	Whirlpool AD5OUSLI	QM3743639	8/2002
Dehumidifier	Gravimetry Laboratory	HISENSE IKD070	0113Z0PKK69Y5 A0174	6/2015
Laminar flow hood	Gravimetry Laboratory	ATMOSTECH Industries	*436*	Pre-1992
XRF & Resuspension Laboratory				
XRF 772	XRF Laboratory	Kevex 770	8003407A1327	8/2001
Pulse Processor for 772	XRF Laboratory	IXRF/4460	128	8/2001
Vacuum pump for 772	XRF Laboratory	Alcatel	164208	9/2015
X-ray tube chiller for 772	XRF Laboratory	Kevex	1349	8/2001
Quant'X XRF	XRF Laboratory	Thermo	1111540	(1/2012) Data generation started in 8/2012
Vacuum pump for Quant'X	XRF Laboratory	Edwards RV8	119493083	1/2012
Refrigerator (dorm sized)	XRF Laboratory	Sanyo	960825190	Pre-1992
Sieve Catch Pans (3)	Compressor Room	USA Standard	None	Pre-1986
#400 Sieve Pans (3)	Compressor Room	USA Standard	38 µm	Pre-1986
#200 Sieve Pans (3)	Compressor Room	USA Standard	75 µm	Pre-1986
#80 Sieve Pans (2)	Compressor Room	USA Standard	180 µm	Pre-1986
#60 Sieve Pans (3)	Compressor Room	USA Standard	250 µm	Pre-1986
#40 Sieve Pans (4)	Compressor Room	USA Standard	425 µm	Pre-1986
#18 Sieve Pans (3)	Compressor Room	USA Standard	1 mm	Pre-1986
#10 Sieve Pans (3)	Compressor Room	USA Standard	2 mm	Pre-1986
#6 Sieve Pans (2)	Compressor Room	USA Standard	3.35 mm	Pre-1986

Table 23-1 Laboratory Equipment				
Name - Unique Identifier	**Location**	**Brand / Model**	**Serial Number** ***(Italicized numbers are capital equipment inventory numbers)***	**Month / Year Placed into Service**
#5 Sieve Pans (3)	Compressor Room	USA Standard	4 mm	Pre-1986
Sieve Pan Lids (2)	Compressor Room	USA Standard	None	Pre-1986
Dicot inlet	SPM room	Sierra/Anderson	165	Pre-1986
Dicot pump	SPM room	11/244	165	Pre-1986
Pump	SPM room	Gast/0322-V103-G8DX	0784	Pre-1986
Digital flow meter	SPM room	Kurz/545-1-SP	NE2243	Pre-1986
Resuspension apparatus	SPM room	In-house	In-house	Pre-1986
Conventional Chemistry Laboratory				
Air compressor for ICP	Compressor room	PowerEx OTS015242	(H) 6/24/2002 1860271-02	8/2002
Microwave oven	Conventional Chemistry Lab	Westinghouse	WCM11100B16200400	12/2016
100.0000g Class 1 weight	XRF Laboratory	Troemner	20190106	Date put into service changes annually
Cabinet Desiccator 1	XRF Laboratory	Boekel	None	Pre-1992
Cabinet Desiccator 2	XRF Laboratory	Unknown	None	4/2011
Cabinet Desiccator 3	XRF Laboratory	Fisher	None	6/2011
Cabinet Desiccator 4	XRF Laboratory	Boekel	None	10/2011
Cabinet Desiccator 5	XRF Laboratory	Fisher	None	10/2011
Cabinet Desiccator 6	XRF Laboratory	Unknown	None	8/2016
Cabinet Desiccator 7	XRF Laboratory	Unknown	None	8/2016
Cabinet Desiccator 8	XRF Laboratory	Unknown	None	8/2016
Combination thermometer/ hygrometers	Laboratory Desiccators	VWR 36934-164	Serial numbers change with expiration date	Dates put into service change when expired

Table 23-1 Laboratory Equipment				
Name - Unique Identifier	**Location**	**Brand / Model**	**Serial Number** ***(Italicized numbers are capital equipment inventory numbers)***	**Month / Year Placed into Service**
Freezer/ Refrigerator #5	Conventional Chemistry Lab	Amana TR18KW	8810077163 *467*	Pre-1992
Freezer #10	SPM Laboratory	GE FP21DSCRWH	SL163859 *901*	11/1994
Freezer #11	SPM Laboratory	GE FP21DSCRWH	TL162281 *902*	11/1994
Freezer/ Refrigerator #6	Conventional Chemistry Lab	Frigidaire FFHT1814QW1	BA52800708	8/2015
AND static eliminator	XRF	AND AD1683	None	7/2009
Lab balance	Conventional Chemistry Lab	Sartorius CPA 224S	25650404	12/2010
SPM balance	XRF	Sartorius MSA 225S	33503396	12/2015
Milli-Q RO/DI Unit	Conventional Chemistry Lab	Millipore	F2KA41704D	1/2013
ICS-5000 (Anion & Cation IC)	Conventional Chemistry Lab	Thermo ICS-5000	17030857	1/2018
AS-AP Autosampler (ICS-3000)	Conventional Chemistry Lab	Thermo AS-AP	17030857	1/2018
AXP anion regenerant pump (ICS-5000)	Conventional Chemistry Lab	Thermo AXP	17030857	1/2018
AXP cation regenerant pump (ICS-5000)	Conventional Chemistry Lab	Thermo AXP	17030857	1/2018
pH/mV meter	SPM Laboratory	VWR SympHony	D04910	1/2011
pH/mV meter	Conventional Chemistry Lab	Orion 3 Star	B43712	2012
pH electrode	SPM Laboratory	Orion 8102BNUWP	(lot code) QX1	2/2013
Small sonicator	SPM Laboratory	AmericanBrand	48L5287	Pre-1992
Big Sonicator (8510)	Conventional Chemistry Lab	Branson	RPA02112447G	2/2011

Table 23-1 Laboratory Equipment				
Name - Unique Identifier	**Location**	**Brand / Model**	**Serial Number** ***(Italicized numbers are capital equipment inventory numbers)***	**Month / Year Placed into Service**
Big Sonicator (8800)	Conventional Chemistry Lab	Branson	BGQ041693220B	8/2016
Heated Sonicator	Conventional Chemistry Lab	Elmasonic P	102048034	4/2014
ICS-1100 (Cr6 IC)	Conventional Chemistry Lab	Dionex	11050922	8/2011
Autosampler (1100)	Conventional Chemistry Lab	Dionex AS-DV	11050840	8/2011
UV/Vis Cr6 detector (1100)	Conventional Chemistry Lab	Dionex VWD	11050494	8/2011
Auxiliary Pump (1100)	Conventional Chemistry Lab	Dionex	Z0042664	8/2011
Aquion IC (Cr6 IC)	Conventional Chemistry Lab	Thermo-Dionex	160540158	8/2016
Autosampler (Aquion)	Conventional Chemistry Lab	Thermo-Dionex AS-DV	160510939	8/2016
UV/Vis Cr6 detector (Aquion)	Conventional Chemistry Lab	Thermo-Dionex VWD	16031453	8/2016
Auxiliary Pump (Aquion)	Conventional Chemistry Lab	Thermo-Dionex	Z0055802	8/2016
OC/EC analyzer	Conventional Chemistry Lab	Sunset Labs	141A	1/2002
CVAA	Conventional Chemistry Lab	Nippon Instruments 3320A	08400784	8/2010
CVAA autosampler	Conventional Chemistry Lab	SC-3	09410401	8/2010
CVAA Reagent Dispenser	Conventional Chemistry Lab	RD-3	08420583	8/2010
Water bath	Conventional Chemistry Lab	Thermo 280 Series	206799-339	1/2009
Thermometer – electronic for sonicators	Conventional Chemistry Lab	VWR	140774934	9/2015
Oven – Forced Air	SPM room	Baxter DIV48	198002	Pre-1992
Oven – Forced Air	Conventional Chemistry Lab	Binder	13-21559	10/2014

Table 23-1 Laboratory Equipment				
Name - Unique Identifier	**Location**	**Brand / Model**	**Serial Number** ***(Italicized numbers are capital equipment inventory numbers)***	**Month / Year Placed into Service**
Oven – muffle	Conventional Chemistry Lab	American Scientific Products FP-41	132028	Pre-1992
Centrifuge	Conventional Chemistry Lab	Thermo CL2	42620462	4/2011
ICP	Conventional Chemistry Lab	PerkinElmer Optima 8300	078S1401204	4/2014
Chiller for ICP	Conventional Chemistry Lab	PolyScience	2F1411787	4/2014
ICP Autosampler	Conventional Chemistry Lab	PerkinElmer AS S10	102513020605	4/2014
ISE: fluoride	Conventional Chemistry Lab	Thermo	249030-A01	9/2008
Thermometers – Fluoride distillation	Conventional Chemistry Lab	Various	Various (considered consumable)	Various (considered consumable)
5mg Class 1 weight	Conventional Chemistry Lab	Troemner	31235	Dates put into service change when expired
5mg Class 1 weight	Conventional Chemistry Lab	Troemner	1000085970	Dates put into service change when expired
50g Class 1 weight	Conventional Chemistry Lab	Troemner	1000085970	Dates put into service change when expired
50g Class 1 weight	Conventional Chemistry Lab	Troemner	1000143398	Dates put into service change when expired
500mg Class 1 weight	Conventional Chemistry Lab	Troemner	1000137823	Dates put into service change when expired
500mg Class 1 weight	Conventional Chemistry Lab	Troemner	1000085970	Dates put into service change when expired
Separatory funnel shaker	Conventional Chemistry Lab	In-house	In-house	Pre-1994
Stirplate 663	SPM Laboratory	Thermolyne Syborn	30708171	Pre-1992
Stirplate 652	SPM Laboratory	Thermo Cimarec 1	46402186	Pre-1992
Stirplate	Conventional Chemistry Lab	Thermo Cimarec 2	63891800366 *1006*	1/2011

Table 23-1 Laboratory Equipment				
Name - Unique Identifier	**Location**	**Brand / Model**	**Serial Number** ***(Italicized numbers are capital equipment inventory numbers)***	**Month / Year Placed into Service**
Stirring Hotplate (Hotplate no longer functional)	Conventional Chemistry Lab	Thermo Cimarec 3	Unreadable	Pre-1992
Hotplate, 12" yellow	Conventional Chemistry Lab	Thermo Cimarec 3	Unreadable	Pre-1992
Hotplate, 12" yellow	Conventional Chemistry Lab	Thermo Cimarec 3	1073990872646	Pre-2006
Hotplate, 10" gray	Conventional Chemistry Lab	Corning	Unknown *948*	5/2005
Hotplate, 10" yellow digital	SPM Laboratory	Thermo Cimarec	C1757110206628 *1012*	4/2011
Hotplate, 10" yellow digital	SPM Laboratory	Thermo Cimarec	C1757110104596 *1008*	4/2011
Hotplate, 12" Gray	Conventional Chemistry Lab	Cole-Parmer HP11C-P	50002174	11/2015
Stirring Hotplate, 12"	Conventional Chemistry Lab	Thermo Cimarec 3	1072000428345	unknown
UV/Vis Spectrometer	Conventional Chemistry Lab	MiltonRoy Spec 20D	3321025001	Pre-1992
Orbital Shaker	Conventional Chemistry Lab	LabLine	3520	Pre-1992
Laminar Hood	Conventional Chemistry Lab	LabConco (unreadable model#)	195468	Pre-1992
Vortex Mixer	Conventional Chemistry Lab	Labnet	00017305042	2/2018

23.2 Support Equipment

Support Equipment includes but is not limited to: balances, ovens, refrigerators, freezers, water baths, chillers, temperature/humidity measuring devices, humidifiers, dehumidifiers, vacuum pumps where needed by instrumentation, and volumetric dispensing devices.

All support equipment is maintained in proper working order. Records are kept for all repair and maintenance activities including service calls. For NIST-traceable items (including weights, thermometers, and hygrometers), certifications are maintained near their point of use. For the reagent water system, cartridge replacement is noted on the daily control chart. All refrigerators containing samples are monitored with a max/min thermometer and recorded on each

business day. As water baths, ovens and sonicators tend to fail catastrophically, the equipment is usually replaced rather than repaired. One common maintenance log is maintained for the water bath, ovens, heated ultrasonicator, muffle furnace, lab thermometers (glass body), and other support equipment.

All raw data records are retained to document equipment performance where performance is an integral part of the method being performed. These records include primarily logbooks. Some records however, may take the form of certificates by certifying laboratories or invoices retained by the Laboratory Director.

23.2.1 Support Equipment Maintenance

Regular maintenance of support equipment, such as balances, ovens, water baths, furnaces, Class 0 and Class 1 weights and fume hoods, is conducted at least annually.

Maintenance of other support equipment, especially those with manufacturer's expiration dates, such as thermometers, thermometer/hygrometers, is conducted on an as-needed basis.

Records of maintenance to support equipment are documented in various locations depending on the department, as follows:

- For the gravimetry lab, maintenance is documented in the NIST certificates three ring binder, the balance maintenance log, or the temperature & Humidity log.
- For the XRF laboratory, maintenance is documented in the run log for each instrument or the QS control charts.
- For the Conventional Chemistry laboratory, maintenance is documented in the Support Equipment Maintenance Log.

Table 23-2 includes a summary of support equipment maintenance.

Table 23-2 Summary of Support Equipment Calibration And Maintenance

Instrument	Activity	Frequency	Documentation
Gravimetry Laboratory			
100 mg Class 1 Daily Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
300 mg Class 1 Daily Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate

Table 23-2 Summary of Support Equipment Calibration And Maintenance			
Instrument	**Activity**	**Frequency**	**Documentation**
500 mg Class 1 Daily Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
3.0000 g Class 1 Daily Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
5.0000 g Class 1 Daily Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
100.0000 g Class 1 Daily Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
100 mg Class 0 Monthly Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
300 mg Class 0 Monthly Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
500 mg Class 0 Monthly Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
3.0000 g Class 1 Monthly Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
5.0000 g Class 1 Monthly Weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
Computer Tracked NIST thermometer/hygrometer (Secondary standard)	Recertify when NIST traceability expires, calibrate against primary standard as needed.	Annually and as needed	Keep certificate. “as-needed” adjustments recorded in Daily Temp/RH logbook.
Primary NIST Thermometer/ hygrometer	Recertify or buy new when NIST traceability expires	As needed	Keep certificate
Max/Min thermometer(s)	Recertify when NIST traceability expires	As needed	Keep certificate
Cabinet Desiccator (small)	Verify seal maintaining	Day of use	Record percent humidity in raw data

Table 23-2 Summary of Support Equipment Calibration And Maintenance			
Instrument	**Activity**	**Frequency**	**Documentation**
IR thermometer	Recertify or buy new when NIST traceability expires	As needed	Keep certificate
Humidifier	Fill with water	As needed	N/A
Dehumidifiers	Empty water	As needed	N/A
Laminar Flow Hood	Check flow rate with vanometer	Annually	Label on side of hood
XRF Laboratory			
Pulse Processor for 772	Energy Calibration	As needed	QS control charts
Vacuum pump for 772	Maintain oil level	As needed	QS control charts
X-ray tube chiller for 772	Maintain water level	Weekly	Maintenance log
Vacuum pump for Quant'X	Maintain oil level	As needed	QS control charts
Refrigerator (dorm sized)	Check with IR gun thermometer	Day of use	Control chart
Sieve Catch Pans (3)	Clean with warm water and dry at 60 °C	After each use	none
#400 Sieve Pans (3)	Clean with warm water and dry at 60 °C	After each use	none
#200 Sieve Pans (3)	Clean with warm water and dry at 60 °C	After each use	none
#80 Sieve Pans (2)	Clean with warm water and dry at 60 °C	After each use	none
#60 Sieve Pans (3)	Clean with warm water and dry at 60 °C	After each use	none
#40 Sieve Pans (4)	Clean with warm water and dry at 60 °C	After each use	none
#18 Sieve Pans (3)	Clean with warm water and dry at 60 °C	After each use	none
#10 Sieve Pans (3)	Clean with warm water and dry at 60 °C	After each use	none
#6 Sieve Pans (2)	Clean with warm water and dry at 60 °C	After each use	none
#5 Sieve Pans (3)	Clean with warm water and dry at 60 °C	After each use	none
Sieve Pan Lids (2)	Clean with warm water and dry at 60 °C	After each use	none
Dicot pump	Calibrate with rotameters	As needed	None
Pump	Calibrate with digital flow meter	Each use	None

Table 23-2 Summary of Support Equipment Calibration And Maintenance			
Instrument	**Activity**	**Frequency**	**Documentation**
Digital flow meter	N/A	N/A	N/A
Cabinet Desiccator 1	Verify seal maintaining	Day of use	Record percent humidity in raw data
Cabinet Desiccator 2	Verify seal maintaining	Day of use	Record percent humidity in raw data
Cabinet Desiccator 3	Verify seal maintaining	Day of use	Record percent humidity in raw data
Cabinet Desiccator 4	Verify seal maintaining	Day of use	Record percent humidity in raw data
Cabinet Desiccator 5	Verify seal maintaining	Day of use	Record percent humidity in raw data
Cabinet Desiccator 6	Verify seal maintaining	Day of use	Record percent humidity in raw data
Cabinet Desiccator 7	Verify seal maintaining	Day of use	Record percent humidity in raw data
Cabinet Desiccator 8	Verify seal maintaining	Day of use	Record percent humidity in raw data
(Desiccator) Combination thermometer/ Hygrometers	Recertify when NIST traceability expires	As needed	Keep certificate
Conventional Chemistry Laboratory			
Microwave oven	Verify working by determining if digestion bombs are hot to the touch	Each use	none
5 mg Class 1 weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
500 mg Class 1 weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
50.0000g Class 1 weight	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
Millipore RO/DI Unit	Verify MΩ within control	Day of use	Control chart
Refrigerator #11	check temperature with NIST-traceable max/min thermometer	Daily	control chart
Refrigerator #12	check temperature with NIST-traceable max/min thermometer	Daily	control chart

Table 23-2 Summary of Support Equipment Calibration And Maintenance			
Instrument	**Activity**	**Frequency**	**Documentation**
Refrigerator #4	check temperature with NIST-traceable max/min thermometer	Daily	control chart
Freezer #4	check temperature with NIST-traceable max/min thermometer	Daily	control chart
Refrigerator #5	check temperature with NIST-traceable max/min thermometer	Daily	control chart
Freezer #5	check temperature with NIST-traceable max/min thermometer	Daily	control chart
Water bath	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
Thermometer – sonicator bath (electronic)	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
Ovens – Forced Air	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
Oven – muffle	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
Chiller for ICP	Keep fins clean of dust/debris Replace chiller fluid	As needed As needed	None Maintenance Log
Air compressor for ICP	Drain water from tank	Weekly	None
Thermometers – Fluoride distillation (glass)	Accuracy determined by A2LA-accredited weights and measurement laboratory	Annually	Keep certificate
Laminar Hood	Check flow rate with vanometer	Annually	Label on side of hood
Fume Hoods	Check flow rate with vanometer	Annually	Label on side of hood

23.2.2 Support Equipment Calibration

Calibration requirements for analytical support equipment are found in Tables 23-3 and 23-4.

All NIST-traceable support equipment is calibrated or verified annually, across the entire range of use, using NIST traceable references where available. If the results of the calibration or verification of support equipment are not within specifications: (1) the equipment is removed from service until repaired or (2) records are maintained of correction factors to correct all measurements. If correction factors are used, this information is clearly marked on or near the equipment.

Each day prior to use, support equipment such as balances, refrigerators and freezers are verified with an NIST traceable reference, if available, to ensure operation is within the expected range for the application for which the equipment is to be used. Analytical thermometers (e.g., Fluoride distillation thermometers), and water bath/oven thermistors are verified annually by an A2LA certified laboratory

Volumetric devices, including dispensing devices and fixed-volume devices (except Class A glassware), are checked for accuracy on a monthly basis. Plastic, disposable volumetrics, such as centrifuge tubes, are checked once per shipping box. Plastic volumetric ware such as volumetric flasks or graduated cylinders are checked quarterly. Class A glassware is verified upon receipt. All volumetric ware is given a unique ID and is traceable to each sample result for which it was used.

Table 23-3 Calibration Acceptance Criteria for Support Equipment

Equipment	Type of Calibration / Number of Standards	Frequency	Acceptance Limits	Corrective Action
Gravimetric Laboratory (Gravimetry Laboratory)				
100 mg Class 1 Daily Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.003 mg	Send back for recertification
300 mg Class 1 Daily Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.003 mg	Send back for recertification
500 mg Class 1 Daily Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.003 mg	Send back for recertification
3.0000 g Class 1 Daily Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.0005 g	Send back for recertification
5.0000 g Class 1 Daily Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.0005 g	Send back for recertification

Table 23-3 Calibration Acceptance Criteria for Support Equipment				
Equipment	**Type of Calibration / Number of Standards**	**Frequency**	**Acceptance Limits**	**Corrective Action**
100.0000 g Class 1 Daily Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.0000 g (used for calibration, must be exact)	Send back for recertification
100 mg Class 0 Monthly Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.005 mg	Send back for recertification
300 mg Class 0 Monthly Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.005 mg	Send back for recertification
500 mg Class 0 Monthly Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.005 mg	Send back for recertification
3.0000 g Class 1 Monthly Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.0005 g	Send back for recertification
5.0000 g Class 1 Monthly Weight	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.0005 g	Send back for recertification
Computer Tracked secondary thermometer/ hygrometer	Inspected and calibrated by A2LA accredited laboratory annually. Adjusted as needed against primary NIST-traceable thermometer/hygrometer	Annually or as needed	±0.5 °C from Primary	Recertify or replace
Primary Thermometer/ hygrometer(s)	Inspected and calibrated by A2LA accredited laboratory annually.	When NIST traceability expires	Per manufacturer	Recertify
Max/Min thermometer(s)	Inspected and calibrated by A2LA accredited laboratory annually.	When NIST traceability expires	Per manufacturer	Recertify
IR thermometer	Inspected and calibrated by A2LA accredited laboratory annually.	When NIST traceability expires	Per manufacturer	Recertify

Table 23-3 Calibration Acceptance Criteria for Support Equipment				
Equipment	**Type of Calibration / Number of Standards**	**Frequency**	**Acceptance Limits**	**Corrective Action**
Humidifier	Adjust controller	When humidity is out of acceptance	Refer to Gravimetry Laboratory SOPs	Adjust controller or replace unit.
Dehumidifier	Adjust controller	When humidity is out of acceptance	Refer to Gravimetry Laboratory SOPs	Adjust controller or replace unit.
Conventional Chemistry Laboratory				
Refrigerator #11	Adjust thermostat	When temperature is out of acceptance	0 - 6 °C (as verified by NIST traceable thermometer)	Adjust thermostat or replace
Refrigerator #12	Adjust thermostat	When temperature is out of acceptance	0 - 6 °C (as verified by NIST traceable thermometer)	Adjust thermostat or replace
Refrigerator #5	Adjust thermostat	When temperature is out of acceptance	≤4 °C (as verified by NIST traceable thermometer)	Adjust thermostat or replace
Freezer #5	Adjust thermostat	When temperature is out of acceptance	≤0 °C (as verified by NIST traceable thermometer)	Adjust thermostat or replace
Freezer #6	Adjust thermostat	When temperature is out of acceptance	≤0 °C (as verified by NIST traceable thermometer)	Adjust thermostat or replace
Refrigerator #6	Adjust thermostat	When temperature is out of acceptance	≤4 °C (as verified by NIST traceable thermometer)	Adjust thermostat or replace
5mg, 500mg, 50g Class 1 weights	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	± 0.00010 g or within Class 1 specifications	Send back for recertification or replace
Combination thermometer/ hygrometers	Inspected and calibrated by A2LA accredited laboratory annually.	When NIST traceability expires	Per NIST	Send back for recertification or replace

Table 23-3 Calibration Acceptance Criteria for Support Equipment				
Equipment	**Type of Calibration / Number of Standards**	**Frequency**	**Acceptance Limits**	**Corrective Action**
Water bath	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±2.0°C from set value	Have re-inspected and recalibrated by A2LA accredited laboratory.
Thermometer – sonicator bath (electronic)	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±0.5 °C from 15 - 30 °C	Have re-inspected and recalibrated by A2LA accredited laboratory.
Ovens – Forced Air	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±2.0°C from set value in range of 95 – 105 °C	Have re-inspected and recalibrated by A2LA accredited laboratory.
Oven – muffle	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±50°C from set value in range of 550 – 600 °C	Have re-inspected and recalibrated by A2LA accredited laboratory.
Thermometers – Fluoride distillation (glass)	Inspected and calibrated by A2LA accredited laboratory annually.	Annually	±2.0 °C from 180 °C	Have re-inspected by A2LA accredited laboratory.
Fume Hoods	N/A	Annually	100 fpm with sash in marked position	Service as needed.

Table 23-4 Acceptance Criteria for Support Equipment		
Equipment Identification	**Use**	**Acceptance Criteria**
Gravimetric Laboratory (Gravimetry Laboratory)		
100 mg Class 1 Daily Weight	Verification of ME5 balance	± 0.003 mg
300 mg Class 1 Daily Weight	Verification of ME5 balance	± 0.003 mg
500 mg Class 1 Daily Weight	Verification of ME5 balance	± 0.003 mg
3.0000 g Class 1 Daily Weight	Verification of B120S balance	± 0.0005 g
5.0000 g Class 1 Daily Weight	Verification of B120S balance	± 0.0005 g
100.0000 g Class 1 Daily Weight	Calibration of B120S balance	± 0.0000 g

Table 23-4 Acceptance Criteria for Support Equipment		
Equipment Identification	**Use**	**Acceptance Criteria**
100 mg Class 0 Monthly Weight	Verification of ME5 balance	± 0.005 mg
300 mg Class 0 Monthly Weight	Verification of ME5 balance	± 0.003 mg
500 mg Class 0 Monthly Weight	Verification of ME5 balance	± 0.005 mg
3.0000 g Class 1 Monthly Weight	Verification of B120S balance	± 0.0005 g
5.0000 g Class 1 Monthly Weight	Verification of B120S balance	± 0.0005 g
Computer Tracked thermometer/hygrometer	Daily verification of room temperature and humidity; weekly compilation of environmental data	Matches secondary thermometer/hygrometer
Secondary Thermometer/hygrometer	Verification of computer tracked thermometer/hygrometer	Within NIST expiry date
Max/Min thermometers	Daily freezer/refrigerator monitoring	Within NIST expiry date
IR thermometer	Sample receipt temperature	Within NIST expiry date
Cabinet Desiccator (small)	Desiccation	Maintains humidity ≤10%
Conventional Chemistry Laboratory		
Freezer #6	Sample storage	≤0 °C
Refrigerator #6	Sample storage	0 - 6 °C
Refrigerator #11	Sample storage	≤0 °C
Refrigerator #12	Sample storage	≤0 °C
Refrigerator #5	Sample storage	0 - 6 °C
Freezer #5	Sample storage	≤0 °C
5mg, 500mg, 50g, Class 1 weights	Verification of Lab balance calibration	± 0.00010 g or as given on certification whichever is greater
Cabinet Desiccator 1	Desiccating M5/M201/M202 samples	Maintains humidity ≤10%
Cabinet Desiccator 2	Desiccating M5/M201/M202 samples	Maintains humidity ≤10%
Cabinet Desiccator 3	Desiccating M5/M201/M202 samples	Maintains humidity ≤10%

Table 23-4 Acceptance Criteria for Support Equipment		
Equipment Identification	**Use**	**Acceptance Criteria**
Cabinet Desiccator 4	Desiccating M5/M201/M202 samples	Maintains humidity ≤10%
Cabinet Desiccator 5	Desiccating M5/M201/M202 samples	Maintains humidity ≤10%
Cabinet Desiccator 6	Desiccating M5/M201/M202 samples	Maintains humidity ≤10%
Cabinet Desiccator 7	Desiccating M5/M201/M202 samples	Maintains humidity ≤10%
Cabinet Desiccator 8	Desiccating M5/M201/M202 samples	Maintains humidity ≤10%
Combination thermometer/ hygrometers	Monitoring desiccators	Within NIST expiry date
Water bath	Digesting Hg and other metals (range 90 – 95 °C)	Within NIST certification expiry date
Thermometer – sonicator bath (electronic)	Monitoring sonicator water temperature	Within NIST certification expiry date
Ovens – Forced Air	Drying/Evaporating (range 30 – 300 °C)	Within NIST certification expiry date
Oven – muffle	Pre-firing quartz filters to remove carbon; Sodium fusion for M13B (range 500 – 600 °C)	Within NIST certification expiry date
Thermometers – Fluoride distillation (glass)	Monitoring distillation temperature (range 180°C)	Within NIST certification expiry date
Fume Hoods	Exhausts fumes from laboratory air	100 fpm draw when sash is at mark
RO/DI water unit (Millipore)	Making reagent water	≥18.0 MΩ

23.3 Analytical Equipment

23.3.1 Maintenance for Analytical Equipment

All equipment is properly maintained, inspected and cleaned.

Maintenance of analytical instruments and other equipment may include regularly scheduled preventative maintenance or maintenance on an as-needed basis. Instrument malfunction is documented in the pertinent instrument maintenance log, which becomes part of the laboratory's permanent records. A description of the problem, what was done to repair the malfunction, and proof of return to control are also documented in the log.

Table 23-5 Analytical Equipment Maintenance		
Instrument	**Procedure**	**Frequency**
Gravimetry Laboratory (Gravimetry Laboratory)		
ME5 microbalance	Check level of balance Clean surrounding area with air and Ethanol Clean inside chamber with air and Ethanol Calibrate Verify calibration Service and certify by A2LA lab	Day of use Day of use As needed Day of use Day of use Annually
B120S balance	Check level of balance Clean inside chamber and surrounding area with air and Ethanol Calibrate Verify calibration Service and certify by A2LA lab	Day of use Day of use Day of use Day of use Annually
XRF Laboratory		
XRF 772	Fill liquid Nitrogen dewar Clean excitation chamber	Weekly Weekly
Quant'X XRF	Perform energy calibration Clean excitation chamber	Weekly Weekly
Dicot inlet	Disassemble and clean	After each use
Resuspension apparatus	Disassemble and clean	After each use
Conventional Chemistry Laboratory		
Lab balances	Clean Check level Calibrate Verify calibration Service and certify by A2LA lab	As needed Day of use Day of use Day of use annually
ICS-5000 Anion/Cation IC	Check background pressure Check suppressor flow Check background conductivity Check all fluid levels (eluent & regenerant) Check autosampler water level Calibrate Replace columns & pump seals Replace suppressors Rebuild injector switches	Day of use Day of use Day of use Day of use Day of use As needed Annually or as needed Bi-annually or as needed Annually or as needed
pH/mV meter	Calibrate	Day of use

Table 23-5 Analytical Equipment Maintenance		
Instrument	**Procedure**	**Frequency**
pH electrode	Inspect frit for build-up	Day of use
	Calibrate with minimum of 2 standards	Day of use
	Verify Nernst slope	Day of use
ICS-1100 (Cr^{6+} IC)	Prime AXP	Day of use
	Clean colorimetric reagent carboy with acetone	Once per week of use
	Check waste container level	Day of use
	Check background pressure	Day of use
	Check all fluid levels (eluent & colorimetric)	Day of use
	Calibrate	Once per week of use
	Replace columns	Annually or as needed
	Rebuild injector switches	Annually or as needed
	Replace UV lamp	As needed
Aquion (Cr^{6+} IC)	Prime AXP	Day of use
	Clean colorimetric reagent carboy with acetone	Once per week of use
	Check waste container level	Day of use
	Check background pressure	Day of use
	Check all fluid levels (eluent & colorimetric)	Day of use
	Calibrate	Once per week of use
	Replace columns	Annually or as needed
	Rebuild injector switches	Annually or as needed
	Replace UV lamp	As needed
OC/EC analyzer	Clean oven by pre-firing	Day of use
	Monitor temperature steps during pre-fire	Day of use
	Clean surrounding area with ethanol	Day of use
	Record calibration area, psig, and transmittance for trend comparison	Day of use
CVAA	Check reagent delivery tubing	Annually or as needed
	Prime reagent delivery tubing three times	Day of use
	Rinse/drain tubing after use	Day of use
	Check level of/fill 1% HCl rinse solution	Day of use
ICP	Check torch for debris/dirt	Day of use
	Rebuild torch assembly	As needed
	Check waste container level, empty	Day of use, as needed
	Clean/replace windows	As needed
	Check tubing	Day of use
	Replace tubing	As needed
	Check Argon level in tank	Day of use
	Drain compressor	End of week of use
	Replace nebulizer	As needed
	Replace spray chamber	As needed
Reagent Water	Check conductivity	Day of use
	Replace cartridges	As needed
	Sanitize System	As needed
	Replace UV lamp	As needed

Table 23-5 Analytical Equipment Maintenance		
Instrument	**Procedure**	**Frequency**
Fluoride ISE (rarely used)	Empty/refill filling solution Verify Nernst slope	Day of use Day of use
UV/Vis spectrometer (has not been used in >10 years)	Check cell for cleanliness Zero out absorbance Set 100% T to 100 Check/replace light bulb	Day of use Day of use Day of use As needed

23.3.2 Instrument Calibration

Information on instrument calibration is located in Appendix H and the relevant SOPs for that instrument.

Initial instrument calibration verification and continuing instrument calibration verifications are an important part of ensuring data of known and documented quality. If more stringent calibration requirements are included in a mandated reference method or by regulation, those calibration requirements override any requirements outlined here or in laboratory SOPs, unless the method is archaic (see Appendix A of pertinent SOPs). Generally, procedures and criteria regarding instrument calibrations are provided in the laboratory SOPs.

Section 24

MEASUREMENT TRACEABILITY
(TNI V1:M2 – Section 5.6)

Measurement quality assurance comes in part from traceability of standards to certified materials.

All equipment used affecting the quality of test results are calibrated prior to being put into service and on a continuing basis (see Section 23, "Calibration Requirements"). These calibrations are traceable to national standards of measurement where available.

If traceability of measurements to NIST is not possible or not relevant, evidence for correlation of results through inter-laboratory comparisons, audit samples, or independent analysis is provided, if possible.

24.1 Reference Standards

Reference standards are standards of the highest quality available at a given location, from which measurements are derived. These standards are used to verify standards used on a daily basis such as weights used to check balance calibrations, or thermometers used to verify other thermometers. They are the standards by which other standards are verified.

Reference Standards, such as NIST Class 0 and Class 1 weights, are used for calibration and to verify other standards, unless it is shown that their performance as reference standards becomes invalidated by use.

Where possible, reference standards are calibrated by an A2LA certified reference lab that can provide traceability to national or international standards. An example of a situation in which this is not possible is the NIST thin film standards for XRF analysis. NIST no longer manufactures these standards and, although they are "expired," there is no other NIST traceable provider, and it is highly unlikely that the standard will degrade without visible signs of deterioration due to the physical nature of the standard and the limited use to which the standards are put.

The following reference standards are calibrated and traceable to a national standard as indicated in Section 23:

- Standard weights;
- reference thermometers;
- Max/Min thermometers;
- balances; and
- combination thermometer/hygrometers.

Note: For cost efficiency purposes, some thermometers and combination thermometer/hygrometers are simply replaced rather than recalibrated as recalibration is more expensive than replacing the unit with a new NIST traceable one. (See Table 23-3.)

24.2 Reference Materials

Reference materials are substances that have concentrations that are sufficiently well established to use for calibration or as a frame of reference.

Reference materials, where commercially available, are traceable to national standards of measurement, or to Certified Reference Materials, usually by a Certificate of Analysis. Purchased reference materials require a Certificate of Analysis.

Laboratory-generated reference materials, such as working standards or intermediate stock solutions, are checked as far as is technically and economically practical.

Where possible, working standards or intermediate stock solutions are checked against a second source at first time of use. When a second source is not available, a vendor-certified different lot is accepted as a second source. In most cases, the analysis of an Initial Calibration Verification (ICV) standard or a Laboratory Control Sample (LCS) can be used as a second source confirmation. Working standards and intermediate stock solutions are given unique IDs and expiration dates when they are prepared based on method requirements, regulatory requirements, Technical Director's knowledge of the method, or, where none exist, the earliest expiration date of the primary standards from which the working standards are prepared. These standards are used in their entirety or disposed of by the expiration date.

Additional working standards such as working Class 1 weights or internal thermometers are checked using the frequency summarized in Table 23-3.

24.3 Transport and Storage of Reference Standards and Materials

The laboratory handles and transports reference standards and materials in a manner that protects the integrity of the materials. Reference standards and material integrity is protected by separation from incompatible materials and/or minimizing exposure to degrading environments or materials.

Reference standards and materials are stored according to manufacturer's recommendations, method SOP requirements and separately from samples. See Table 24-1 below and SOP QA-011.

Table 24-1 Standard Storage and Preparation

Instrument	Stock Storage	Preparation	Intermediate Stock Solution or Working Standard Storage	Frequency of Preparation
ICP	Room Temperature, metals cabinet	Working Standards from Stock	Room Temperature	As needed
IC – Anions and Cations	Standards Refrigerator	Working Standards from Stock	Room Temperature	As needed
IC-PCD (Cr6)	Room Temperature, metals cabinet	Working Standards from Stock	Room Temperature	As needed
CVAA	Room Temperature, Hg hood	Working Standards from Stock	Room Temperature	Daily
OC/EC	Room Temperature (dry chemical)	Working Standard from dry chemical	Standards Refrigerator	Every 6 months
pH meter	Room Temperature, pH supplies	N/A	N/A	N/A
All Class 1 weights	Room Temperature, gravimetric areas	N/A	N/A	N/A
XRF	Room Temperature, XRF laboratory	N/A	N/A	N/A

24.4 Labeling of Reference Standards, Reagents and Reference Materials

The laboratory has procedures for purchase, receipt and storage of standards, reagents and reference materials. Purchase procedures are described in Section 9, "Purchasing Services and Supplies".

All standards and reagents are disposed of after their expiration date.

Reagent quality is verified upon receipt by examination of the Certificate of Analysis and again upon use for blank analysis.

24.4.1 Stock Standards, Reagents, Reference Materials and Media

Records, in the form of Certificates of Analysis, for all standards, reagents, reference materials and media* include:

- the manufacturer/vendor name and lot number (or traceability to purchased stocks or neat compounds);
- the manufacturer's Certificate of Analysis or purity (if available);

- the date of receipt (stamped or hand-written on the CoA and container); and
- recommended storage conditions (if available).

*Note: It is assumed that "media" here is referring to microbiological media. However, as the term is not well defined, ***CHESTER LabNet*** defines the term to mean "air filters and sorbent tubes." Most media for Air Quality do not have Certificates of Analysis available.

If the original container does not have an expiration date provided by the manufacturer or vendor, either on the container or on the Certificate of Analysis, "X NG" shall be written on the label in indelible ink to indicate that the expiration date was "none given". If an expiration date is provided on the Certificate of Analysis but not printed on the label, the expiration date as given on the Certificate shall be written on the label in indelible ink.

In methods where the purity of reagents is not specified, analytical reagent grade or better is used. If the purity is specified, that is the minimum acceptable grade. Purity is verified and documented according to Section 9, "Purchasing Services and Supplies". Certificates of Analysis are maintained in appropriate binders throughout the laboratory.

24.4.2 Prepared Standards, Reagents, Reference Materials and Media

Prepared standards and reagents are recorded in the applicable bound standards and reagents logbook. Records for standards and reagents preparation include:

- traceability to purchased stock compounds;
- reference to or description of the method of preparation;
- date of preparation;
- an expiration date after which the material shall not be used;
- preparer's initials; and
- unique standard ID.

Reagents used NEAT have the following information recorded with the raw data at time of usage:

- manufacturer and lot number; and
- expiration date.

All containers of prepared standards, reagents or materials are labeled with a unique ID and an expiration date. The unique ID is in the format of LLL-PPP-SS where:

LLL = laboratory logbook number as issued from the QA Officer;
PPP = page number within the logbook; and
SS = sequential number on the page.

Prepared reagents are verified to meet the requirements of the reference method through the blank analysis performed with each run (e.g., Cal blank, ICB, etc.). If the blank results are suspect, an investigation into the cause of the suspect results will be undertaken and the reagent shall be made fresh if deemed necessary, even if it is still within its expiry date. Prepared standards are verified against existing non-expired standards where possible.

Section 25

COLLECTION OF SAMPLES
(TNI V1:M2 – Section 5.7)

CHESTER LabNet does not provide sampling services and has no control over the actions or inactions of the client in the field or the reagents used by the client, unless the reagents are purchased directly from the laboratory. The laboratory does not supply the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, ice or packing materials. The nature of most air quality methods (ambient or source) makes doing so prohibitively expensive to the laboratory and the client. The laboratory does, upon request, supply clients with Chain of Custody forms and filter or sorbent media.

25.1 Sampling Containers

The 2009 TNI Standard QAMP Template states, *"…The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory."* The 2016 TNI Standard makes no references to the provision of sampling containers by the laboratory.

The nature of air quality sampling generally requires clients to provide their own coolers, reagent water, sample containers (as defined in the disclaimer at the beginning of this document), secondary sample containers, custody seals (if needed), ice and packing materials. The laboratory does not offer clean bottles for use by clients unless specifically requested by the client.

For ambient sampling, the laboratory may or may not be asked to provide filters or sorbent tubes in appropriate secondary containers. The laboratory does offer filters of various types and cassette rental for filters loaded in cassettes (not all filters are loaded prior to shipment, this occurs at the request of the client).

The laboratory also offers shipment of sorbent media, but makes no guarantees as to the cleanliness of the media (e.g., Anasorb tubes, acidified silica gel tubes). For sorbent media, the laboratory is acting as a middle-man and not a supplier.

For the vast majority of media used during sampling, attempting to clean the media prior to sampling would destroy the media. This situation is so well understood in the air quality industry that the majority of methods include processes for media blank subtraction prior to finalizing data. The client is responsible for any blank subtraction as the laboratory is blind to the field activities. The quality of the media rests solely on the manufacturer.

25.1.1 Preparing "Container" (Media) Orders

Filters and sorbent tubes are provided to the client upon request.

The Gravimetry Laboratory Technical Director is informed of the request by the Project Manager or personnel who accepted the order. The request is filled as soon as possible based upon availability, the type of analysis the client is performing, the need or lack thereof for cassettes, and the client's sampling schedule.

Once ready to be shipped, the media are given to the Project Manager. The Project Manager packages the media in a suitable shipping container (suitability depends upon the type of media and the reference method). Shipping is by common carrier (e.g., USPS, UPS, FedEx, DHL) and sometimes performed using the client's account number.

Media is packaged and shipped in such a manner as to prevent harm or breakage of the media under typical shipping conditions. The laboratory will ship to any address specified by the client and in the manner specified by the client. For example, shipping filters to Antarctica involves following very specific instructions from the client. Shipping to foreign countries involves following the customs regulations of that country carefully. All factors involving receipt of the media by the client must be taken into account during the shipping process.

25.1.2 Sampling Containers, Preservation Requirements, Holding Times

Sampling container, preservation and holding time requirements are documented in in the reference methods. ***CHESTER LabNet*** has no control over the activities of clients in the field, the sample containers used, impinger solutions used, whether or not the clients keep the samples at the temperature specified in the method, or any other activity which occurs prior to the samples being received at the laboratory. ***CHESTER LabNet*** has no control over the quality of filters or sorbent material produced by manufacturers, with the exception of attempting to find the least problematic media available on the open market.

For instance, a client may request sodium bicarbonate impregnated acid hardened cellulose filters for the purposes of sampling for Hexavalent Chromium in air. The laboratory will impregnate the filters, load them in cassettes, and store the filters (loaded or unloaded) frozen. The client then may take the filters out to the field where sampling may take 24 hours at ambient temperatures, and the samples may not be retrieved from the sampler for up to 3 days after the sampler has shut off, thereby allowing the sample 4 days at ambient outdoor temperatures (ranging from below freezing to over 100 °F). After the samples are collected, they may or may not be stored frozen prior to the samples being returned to the laboratory, where the samples will again be stored frozen. ***CHESTER LabNet*** has no control over the actions or inactions of the clients once the filters have left the laboratory's possession. This lack of control is true for all filters, impinger solutions and sorbent media.

Since air samples cannot be collected in the same manner as water or soil samples, "containers" is an inappropriate term for sample collection devices and solutions; and the collection itself is an integral part of the "preservation". Below is a table listing the Analyte(s), Reference Method, type of "container", "Preservation" and Holding Time as given in the reference method.

If preservation or holding time requirements are not met, and the cause of not meeting the requirements is under the laboratory's control, the procedures in Section 12, "Control of Non-conforming Environmental Testing Work" are followed. If samples are received by the laboratory after the hold time has expired, this shall be noted in the final report.

Table 25-1 Summary of Sampling Container, Preservation and Holding Time Requirements

Analyte(s)	Reference Method	Method specified "Container"	Preservation	Holding Time
Ambient Air (note: filters or sorbent tubes are the "container" for ambient air samples)				
TSP	40 CFR 50, Appendix B	8"x10" Glass Fiber or Quartz Filter	None Given	None Given
PM_{10}	40 CFR 50, Appendix J	8"x10" Glass Fiber or Quartz Filter	None Given	None Given
PM_{10} Dichotomous	IO 2.2	37mm Teflon or Quartz Filter	None Given	None Given
$PM_{2.5}$	40 CFR 50, Appendix L	47mm Teflon filter	None **OR** <4 °C	10 days with no refrigeration, **OR** 30 days if stored at <4 °C
Total Metals (ambient air)	40 CFR 50, Appendix G; IO 3.2; IO 3.4	8"x10" Glass Fiber; IO 3.0 specifies a filter "meeting specifications" but not the actual matrix or size	None Given	None Given
Mercury	EPA 7471, by reference in other methods	Any type of filter the client uses	None Given for ambient air samples	None Given for ambient air
Total Metals	IO 3.3 (XRF)	IO 3.0 specifies a filter "meeting specifications" but not the actual matrix or size	None Given	None Given
Anions & Cations	IO 4.2	Teflon filter & denuder rinses	None Given	"analyze as soon as possible after collection"; also "analyze on day of extraction"
Organic Carbon/Elemental Carbon	NIOSH 5040; IMPROVE A Method	Pre-fired Quartz filters (37mm or 47mm)	"frozen"	None Given
Hexavalent Chromium	CARB SOP MLD 039 (not reference)	Bicarb impregnated cellulose	"frozen"	90 days prior to extraction, 24 hours after extraction
Hexavalent Chromium	ASTM D7614-12	Bicarb impregnated cellulose	"frozen"	None for filters/samples. "…extraction should be performed immediately prior to analysis."
Total Nuisance Dust	NIOSH 0500	37mm Teflon or PVC filter	None Given	None Given
Respirable Particles	NIOSH 0600	37mm Teflon or PVC filter	None Given	None Given

Table 25-1 Summary of Sampling Container, Preservation and Holding Time Requirements				
Analyte(s)	**Reference Method**	**Method specified "Container"**	**Preservation**	**Holding Time**
Arsine	NIOSH 6001	Coconut Charcoal sorbent Tube	None Given	6 days
Phosphine	NIOSH 6002	$Hg(CN)_2$-coated silica gel sorbent tube	None Given	7 days
SO_2	NIOSH 6004	0.8µm cellulose ester membrane filter FOLLOWED BY bicarb impregnated cellulose fiber filter	None Given	None Given
Diborane	NIOSH 6006	PTFE membrane filter, 13-mm diameter, 1-µm pore size FOLLOWED BY oxidizer-impregnated charcoal sorbent tube	None Given	7 days
Mercury	NIOSH 6009	Hopcalite Tube (no longer manufactured)	None Given	30 days
Br_2 & Cl_2	NIOSH 6011	Teflon filter with 0.5-µm pore size FOLLOWED BY a 25mm silver membrane filter with 0.45-µm pore size	None Given	30 days
NO_2	NIOSH 6014	7mm sorbent tube containing 400 mg TEA-coated molecular sieve (type 13x, 30-40 mesh) FOLLOWED BY 7mm sorbent tube containing 800 mg oxidizer (chromate) FOLLOWED BY 7mm sorbent tube containing 400 mg TEA-coated molecular sieve (type 13x, 30-40 mesh)	None Given	7 days
Ammonia	NIOSH 6016	Acidified silica gel sorbent tube	None Given	35 days
Ammonia	OSHA ID188	H_2SO_4 acidified carbon bead sorbent tube	None Given	29 days
Elements by ICP [metals]	NIOSH 7302	37mm mixed cellulose ester	None Given	None Given
Elements by ICP [metals]	NIOSH 7304	37mm PVC filter [unimpregnated]	None Given	None Given
Chromium (VI)	NIOSH 7605	37mm PVC filter [unimpregnated]	None Given	14 days at room temp; 28 days "refrigerated"

Table 25-1 Summary of Sampling Container, Preservation and Holding Time Requirements				
Analyte(s)	**Reference Method**	**Method specified "Container"**	**Preservation**	**Holding Time**
Fluorides	NIOSH 7902	37mm cellulose ester membrane filter 0.8-µm pore size FOLLOWED BY bicarb impregnated cellulose pad	None Given	None Given
Volatile Acids	NIOSH 7907	Quartz filter FOLLOWED BY bicarb impregnated quartz filter	None Given	7 days at 20° C, 28 days at 4° C
Non-volatile Acids	NIOSH 7908	37mm quartz or Teflon filter	None Given	7 days at 20° C, 28 days at 4° C
Source Emissions Sampling (note: impinger solutions are the "container" for source emission gases)				
Particulates	40 CFR 60, Appendix A, Methods 5 – 5F	Glass or polyethylene petri dishes for filter (filter type not specified); 500 or 100mL glass bottles for Acetone	None Given	None Given
SO_2	40 CFR 60, Appendix A, Method 6	100 mL polyethylene bottles for H_2O_2 solution	None Given	None Given
NO_x	40 CFR 60, Appendix A, Method 7A	Polyethylene bottles for H_2SO_4/H_2O_2 solution	None Given	None Given
NO_x	40 CFR 60, Appendix A, Method 7D	Polyethylene bottles for $NaOH/KMnO_4$ solution	None Given	None Given
H_2SO_4 & SO_2	40 CFR 60, Appendix A, Method 8	1L polyethylene bottles, 1 each for IPA solution and H_2O_2 solution	None Given	None Given
Pb	40 CFR 60, Appendix A, Method 12	1000 mL borosilicate glass bottles for 0.1N HNO_3 solution	None Given	None Given
Total Fluoride	40 CFR 60, Appendix A, Method 13B	1L wide mouth HDPE bottles for impinger water and filter	None Given	None Given
HX & X_2	40 CFR 60, Appendix A, Method 26	100- or 250-mL HDPE bottles with Teflon screw cap liners for both H_2SO_4 and NaOH fractions	None Given	None Given
HX & X_2	40 CFR 60, Appendix A, Method 26A	1L HDPE bottles with Teflon screw cap liners for both H_2SO_4 and NaOH fractions	None Given	None Given

Table 25-1 Summary of Sampling Container, Preservation and Holding Time Requirements				
Analyte(s)	**Reference Method**	**Method specified "Container"**	**Preservation**	**Holding Time**
Multi-metals	40 CFR 60, Appendix A, Method 29	500 - 1000 mL glass for $KMnO_4/H_2SO_4$ impinger solution; HDPE or glass for all other solutions; Glass or plastic petri dishes for filter.	None Given	None given for any metal except Hg. Hg has a "suggested maximum" hold time of 28 days by reference to SW-846 method 7470.
Hg	40 CFR 61, Appendix B, Method 101	100 mL & 1000 mL glass with Teflon-lined caps for ICl solution	None Given	None Given
Hg	40 CFR 61, Appendix B, Method 101A	100 mL & 1000 mL glass with Teflon-lined caps for $KMnO_4/H_2SO_4$ solution	None Given	"suggested maximum" hold time of 28 days by reference to SW-846 method 7470
Hg	40 CFR 61, Appendix B, Method 102	100 mL & 1000 mL glass with Teflon-lined caps for ICl solution	None Given	None Given
Be	40 CFR 61, Appendix B, Method 103	Glass bottles for filter and acetone washes	None Given	None Given
Be	40 CFR 61, Appendix B, Method 104	1L glass bottles with Teflon-lined lids for water impinger solution combined with acetone rinses; Glass or plastic petri dishes for filter	None Given	None Given
As	40 CFR 61, Appendix B, Method 108	500mL – 1000mL polyethylene or polypropylene for water impinger solution combined with NaOH rinse solutions	None Given	None Given
Particulates 201A	40 CFR 51, Appendix M, Method 201A	Any leak-proof container for acetone rinses; Glass or plastic petri dishes for filter	None Given	None Given
Particulates 202	40 CFR 51, Appendix M, Method 202	500 mL amber glass bottles for water impinger solutions; and Hexane/Acetone rinses	None Given	None Given
Hexavalent and Total Chromium	40 CFR 63, Appendix A, Method 306	250 mL, 500 mL or 1,000 mL polyethylene, with leak-free screw cap for 0.1N NaOH or 0.1N $NaHCO_3$ impinger solution	4 °C for Cr(VI); None Given for total Cr	14 days at 4 °C for Cr(VI); 60 days at room temperature for total Cr

Table 25-1 Summary of Sampling Container, Preservation and Holding Time Requirements				
Analyte(s)	**Reference Method**	**Method specified "Container"**	**Preservation**	**Holding Time**
Hexavalent Chromium	SW-846, Method 0061	250 mL, 500 mL or 1,000 mL polyethylene, with leak-free screw cap for 0.1M KOH impinger solution	None Given	14 days
$H2SO_4$ & SO_2 (Titration)	CTM-013 (aka NCASI 8A)	None Given for water rinse or H_2O_2 impinger solution	None Given	None Given
$H2SO_4$ & SO_2 (IC)	CTM-013A	125 mL Nalgene bottles for water rinse or H_2O_2 impinger solution	None Given	None Given
Ammonia	EPA CTM-027 ("draft" as of 1997, not yet given reference method status)	250 mL – 500 mL HDPE bottles for 0.1N H_2SO_4 impinger solutions	4 °C	"2 weeks"
HF/F_2 & HCl/Cl_2	CARB 421	"…borosilicate glass bottles for impinger solutions and washes, 1000 mL. Teflon or high-density polyethylene or polypropylene bottles may be used. Use screw-cap liners that are either rubber-backed Teflon or leak-free" for impinger solution of 1.7 mM Sodium Bicarbonate and 1.8 mM Sodium Carbonate.	None Given	None Given
Total and Hexavalent Chromium	CARB 425	500 ml or 1000 ml borosilicate glass bottles, screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free. Alternatively, polyethylene bottles may be used for 0.1N NaOH impinger solutions.	None Given	None Given

Table 25-1 Summary of Sampling Container, Preservation and Holding Time Requirements				
Analyte(s)	**Reference Method**	**Method specified "Container"**	**Preservation**	**Holding Time**
Multi-metals	CARB 436	Glass or polyethylene petri dishes for Quartz fiber or glass fiber filters without organic binders; 500 ml or 1000 ml borosilicate glass bottles, screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free. Alternatively, polyethylene bottles may be used for HNO_3/H_2O_2 impinger solutions, $KMnO_4/H_2SO_4$ impinger solutions, 0.1N HNO_3 rinse solutions and 8N HCl rinse solutions.	None Given	None Given
Ammonia	BAAQMD ST-1A & ST-1B	250 mL – 500 mL HDPE bottles for 0.1N HCl impinger solutions	"store them in the Refrigerator"	"within five days of their submission to the laboratory."
Particulates	Oregon DEQ 5	[By reference to EPA Method 5] Glass or polyethylene petri dishes for filter (filter type not specified); 500 or 100mL glass bottles each for Acetone rinse, water impinger solution and Dichloromethane	None Given	None Given
Particulates	Oregon DEQ 8	Acetone or Methanol is given as the rinse solution; Glass fiber filters are given as the filter matrix; No guidance is given for containers for the rinse solution or filters.	None Given	None Given

25.2 Sampling Plan

The laboratory does not perform sampling. For purposes of remaining independent from the sampling group and the ultimate client, the laboratory does not act as a consultant for determining sampling plans.

25.3 Sampling Records

Sampling records are maintained by the client. If sampling records are given to the laboratory, they will be used as directed by the client and retained in the client's job folder.

Section 26

HANDLING SAMPLES AND TEST ITEMS
(TNI V1:M2 – Section 5.8 and Section 1.7 of Technical Modules TNI V1:M 3-7)

26.1 Sample Receipt

When samples are received at the laboratory, chain-of-custody is reviewed, sample condition is documented, samples are given unique identifiers, and they are logged into the sample tracking system.

26.1.1 Chain of Custody

The chain of custody is reviewed. The chain of custody form provides information on what type of testing is being requested and can act as an order for laboratory services in the absence of a formal contract. An example chain of custody form is located in Figure 26-1. Chain of custody and any additional records received at the time of sample submission are maintained by the laboratory in each client's job file.

26.1.1.1 Legal Chain of Custody

The laboratory does not *knowingly* receive samples for evidentiary purposes.

26.2 Sample Acceptance

Procedures for opening shipping containers and examining samples are provided in SOP AD-008. Samples received outside normal business hours are handled in the same manner as those received during normal business hours.

The 2009 and 2016 TNI Standard QAMP template states, "*The laboratory has a sample acceptance policy that is made available to sample collection personnel. An example is provided in Figure 26-2. It emphasizes the need for use of water resistant ink, providing proper documentation (to include sample ID, location, date and time of collection, collector's name, preservation type, sample type and any special remarks about the sample), labeling of sample containers to include a unique sample ID, use of appropriate containers, adherence to holding times, and sample volume requirements. In addition the laboratory has nonconformance/corrective action procedures to handle samples that don't meet the requirements above or show signs of damage, contamination or inadequate preservation. Data will be appropriately qualified where samples are reported that do not meet sample acceptance requirements.*" This list is a required element of the QAMP by the 2009 and 2016 TNI Standard, although not typically applicable to air quality samples.

The laboratory has a sample acceptance policy provided in SOP AD-008 and below in Figure 26-2. As the laboratory does not perform sampling, the policy is not provided to sample collection personnel.

The incompatibilities between ambient/source gas stream sampling and water/soil sampling encountered with the above requirements are as follows:

- water resistant ink is rarely needed as the vast majority of samples are filters, and these samples are destroyed if they become wet, or the client labels the samples in the field. The laboratory is not responsible for the actions of the client in the field;
- sample location may not be provided to the laboratory by the client;
- date of sampling is usually known and often provided to the laboratory; however, many samples may have a collection "time" of 2 hours to 14 days and this timing is rarely reported to the laboratory;
- collector's name is rarely reported to the laboratory, and more than one person may be involved in collecting the sample, particularly if the sample has a long collection time;
- samples do not have "preservation" except in a few cases where thermal preservation is called for. Preservation is typically achieved by the media on/in which the sample is collected;
- sample type is usually identified by method number and is understood within the air quality industry;
- filters sent to clients for ambient air sampling have unique IDs assigned to them prior to leaving the laboratory, however, source samples rarely have a unique ID as each project/job site constitutes a unique identifying characteristic and the laboratory has no control over the actions, including identification of containers, of the client in the field;
- appropriate containers are the responsibility of the client, and as they often must travel to extremely remote locations, by necessity, they may be forced to use what they have on-hand;
- very few methods utilized by the laboratory have holding times required within the reference method; and,
- sample "volume" may mean either the gas volume pulled during sampling or the liquid volume of impinger contents post-sampling and is dictated by the amount of gas volume pulled by the clients in the field, the method utilized to capture said gasses, and in the case of source emission samples, the moisture content of the source.

The laboratory has non-conformance/corrective action procedures to handle samples that don't meet the requirements of the reference method, or that show signs of damage or contamination. Data will be appropriately qualified where samples are reported that do not meet sample acceptance requirements.

The laboratory checks samples for the following to evaluate sample acceptance:

- samples are intact;
- samples are not damaged; and
- samples appear to be submitted in good faith and are not, to the laboratory's knowledge, fraudulent.

Criteria regarding holding time, sample matrix, and sample containers is located in Table 25-1 of this document. If these conditions are not met, the client is contacted prior to any further processing, then 1) the sample is rejected as agreed with the client, 2) the decision to proceed is documented and agreed upon with the client, 3) the condition is noted on the Chain of Custody form and/or lab receipt documents, and/or 4) the data are qualified in the Case Narrative of the report. Samples are never rejected without the consent of the client.

26.2.1 Preservation Checks

The following preservation checks are performed and documented upon receipt:

26.2.1.1 *Thermal preservation:*

a) For temperature preservation, the temperature must be within the guidelines specified by the reference method. Note that some methods merely say "ship with blue ice" and do not specify a temperature.

b) The 2009 and 2016 TNI Standard states, "*Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of [thermal preservation]. In these cases, the samples shall be considered acceptable if the samples were received on ice.*" The laboratory very rarely receives samples on the same day they are collected due to the complexity and length of duration of sample collection for air quality methods.

c) Where applicable, record the received temperature on the Chain of Custody and note if ice ("wet" or dry) is present.

Chlorine checks:

CHESTER LabNet performs no methods requiring Chlorine checks.

pH checks:

CHESTER LabNet performs no methods requiring pH checks; however, the laboratory performs pH checks on a few select methods. These checks are typically performed after analyses of the samples to prevent contamination of the sample.

26.3 Sample Identification

The 2009 and 2016 TNI Standard states, "*The laboratory shall have a documented system for uniquely identifying the sample containers that hold samples to be tested, to ensure that there can be no confusion regarding the identity of such samples at any time. This system shall include identification for all samples, sub-samples, preservations, sample containers, tests, and subsequent extracts and/or digestates.*"

See SOP AD-008, "Sample Receipt and Log-in".

Samples are uniquely identified, based on the needs of the client and the reference method requested in the laboratory's LIMS. The LIMS maintains an unequivocal link with the field ID code assigned to each sample by the client. Digests, subsamples, and multi-fraction samples are assigned separate unique IDs, depending on the reference method. The laboratory ID is placed as a durable mark on the sample container in the form of an adhesive label or indelible ink.

Samples are assigned sequential numbers that reference more detailed information electronically stored in the LIMS. Refer to SOPs AD-007 and AD-008 for the laboratory's sample identification procedure.

The following information is included in the LIMS:

- client and, where known, project name;
- date and time of receipt at the lab;
- unique laboratory identification number; and
- initials of the person making the entries.

In addition, the following information, where known, is maintained and linked to the log-in record:

- date and time of sampling;
- unique field identification number linked to the laboratory sample ID *(note: the laboratory has no control over the Field ID. Field ID's may not be unique. Assignation of Field ID's is the responsibility of the client)*;
- analyses requested (including applicable approved method numbers) linked to the laboratory sample ID; and
- comments regarding rejection or other issues (if any).

All documentation received regarding the sample, such as memos or chain of custody, are retained in the job file.

26.4 Sample Aliquots / Subsampling

In order for analysis results to be representative of the sample collected in the field, the laboratory has subsampling procedures. Note that the vast majority of subsampled samples consist of 8" x 10" filters. Refer to SOP ME-008 for subsampling of 8" x 10" filters. Very few other samples are subsampled as most other methods are either non-destructive (gravimetry and XRF analyses) or the entire sample is consumed.

26.5 Sample Storage

Storage conditions are monitored for any required criteria, verified, and the verification recorded where appropriate.

Samples that require thermal preservation are stored in a manner compliant with the reference method. For samples with a specified storage temperature of 4 °C, storage at a temperature above the freezing point of water to 6 °C is acceptable. For samples required to be kept "frozen", any temperature below 0 °C is acceptable.

Samples are held secure, as required. Samples are accessible only to laboratory personnel.

Samples are stored apart from standards, reagents, food or potentially contaminating sources, and in a manner that minimizes cross-contamination. All portions of samples, including extracts, digestates, leachates or any product of the sample is maintained according to the required conditions.

The majority of samples, based upon reference method requirements, are stored at room temperature, or in a temperature- and humidity-controlled room.

26.6 Sample Disposal

Samples are retained for a minimum of 60 days after the report is sent unless other arrangements have been made with the client.

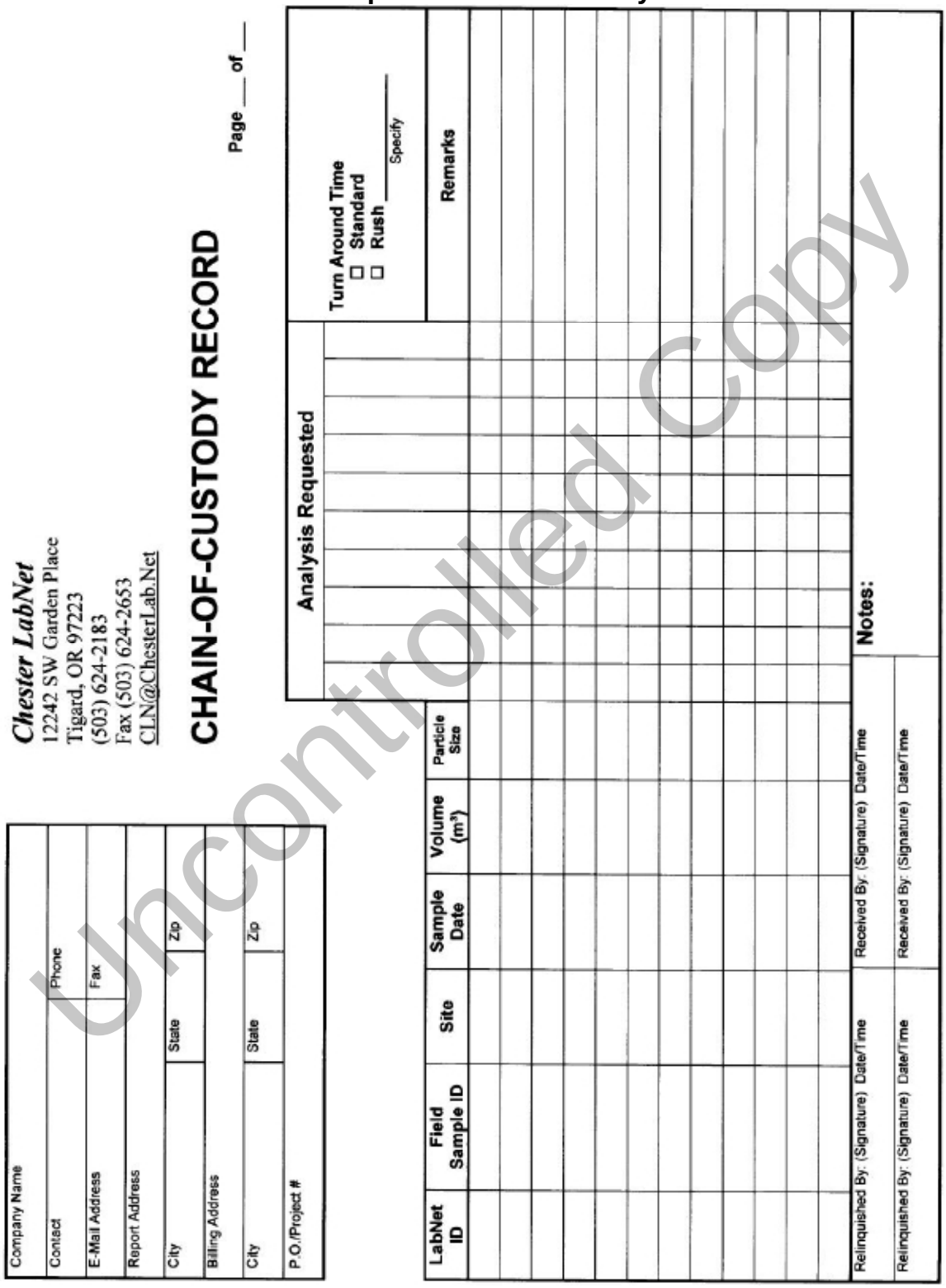
Samples are disposed of according to Federal, State and local regulations. Procedures are described in SOP AD-002 for the disposal of samples, digestates, leachates and extracts.

26.7 Sample Transport

Samples transported under the responsibility of the laboratory, where necessary, are transported safely and according to storage conditions. This includes moving samples within the laboratory. Specific safety operations are addressed outside of this document in method specific SOPs and the laboratory's Chemical Hygiene Plan. Samples are not shipped by the laboratory.

Figure 26-1

Example Chain-of-Custody



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CHAIN-OF-CUSTODY RECORD

Page ___ of ___

Company Name | Phone
Contact | Fax
E-Mail Address
Report Address
City | State | Zip
Billing Address
City | State | Zip
P.O./Project #

Analysis Requested

Turn Around Time
☐ Standard
☐ Rush ____ Specify

LabNet ID	Field Sample ID	Site	Sample Date	Volume (m^3)	Particle Size	Remarks

Relinquished By: (Signature) Date/Time | Received By: (Signature) Date/Time | Notes:
Relinquished By: (Signature) Date/Time | Received By: (Signature) Date/Time

Figure 26-2

Example Sample Acceptance Policy

Disclaimer: The 2009 and 2016 TNI Standard have a number of requirements for sample acceptance. As these requirements are designed for water or soil samples, most are not generally applicable to the analysis of ambient air or source samples. For example, the laboratory rarely knows the name of the sample collector, there may be two or three people involved in sample collection, and the "time of collection" may range from 2 hours to 2 weeks.

CHESTER LabNet will not reject any sample except at the request of the client, unless the acceptance of the sample would be fraudulent. The laboratory will note its opinion if the data are suspected to be of dubious usefulness to the client or to the client's client or regulator. The laboratory will notify clients if their samples are received in such a state as to make analysis impracticable or lead to suspect data. Notification will occur, wherever possible, upon receipt of the samples

To demonstrate the laboratory's good-faith effort to fulfill the 2009 and 2016 TNI requirements, a checklist is completed at the time of sample receipt. The checklist is maintained with the job file. It is not, however, reported to the client.

CHESTER LABNET

SAMPLE RECEIPT CHECKLIST FOR NELAC REQUIREMENTS

Client _____ Method _____ Date _____

Archaic methods or methods that can't be analyzed as written: M26/26A; M202; M12; M101/101A/102 (This is not a complete listing)

NELAC Required Sample Condition	(circle one)			
Received in condition required by method? [1]	Y	N	N/A	
Samples in appropriate containers? [1]	Y	N	N/A	
Correct temperature?	Y	N	N/A	*
Within hold time?	Y	N	N/A	*
Broken/damaged?	Y	N	N/A	!!
Sufficient sample present to perform analysis? [1]	Y	N	N/A	!!
Preserved appropriately? [1]	Y	N	N/A	

Chain of Custody	(circle one)			
Chain of Custody present?	Y	N	N/A	!!
Client contact information present on CoC? [1]	Y	N	N/A	
All requested analyses definitively identified? [2]	Y	N	N/A	!!
If no, is this a long-standing project with understood analyses?	Y	N	N/A	

Additional NELAC Requirements				
All samples identified uniquely? [1]	Y	N	N/A	!!
Labels water resistant? [1]	Y	N	N/A	
Indelible ink used on labels? [1]	Y	N	N/A	
Location of Sample collection listed? [1]	Y	N	N/A	
Sample collector's name listed? [1]	Y	N	N/A	
Preservation type listed? [1]	Y	N	N/A	
Sample type listed? [1]	Y	N	N/A	

NELAC required Method information			
Method requested the latest valid edition? [3]	Y	N	N/A
Method appropriate for the analyses requested? [3]	Y	N	N/A
Is it possible to use the method requested? [3]	Y	N	N/A
Method out of date (wrong revision number)? [3]	Y	N	N/A
Is the method archaic? [3]	Y	N	N/A
Can the method be performed as written? [3]	Y	N	N/A

[1] ***CHESTER LabNet*** will not notify clients of these "deficiencies" to avoid alienating clients by perpetual contact.

[2] may be identified by Method number if method contains no room for doubt as to analytes of interest.

[3] Many Air Quality/Soure Emission methods are archaic, contradict themselves or are inappropriate. Some regulators require the use of older versions of a method. ***CHESTER*** will follow, to the best of their ability, the method requested by the client.

!! address ***prior to any analytical work being started*** . *** **note in case narrative** *upon reporting of results to client.*

Signed _____

Notes _____

Note: NELAC requirements are designed for Water/Soils, and are often incompatible with Ambient or Source Air Promulgated Methods.

For Source Sampling, a second, more specific and applicable checklist (below) was created prior to the advent of TNI or ORELAP and has been in use by the laboratory for many years. This checklist is reported to the client.

CHESTER LABNET
SOURCE SAMPLE RECEIPT CHECKLIST

Client _____ Date _____

Runs _____ Time _____

Custody Seals Inspected, If Present	☐	
Chain-of-Custody Form Inspected	☐	
CoC present with samples?	☐	*
CoC indicate analytical methodology to be used? (eg M29 etc)	☐	!!
CoC indicate if compliance testing? (esp. M26)	☐	!!
M26 samples have Thiosulfate added in field?	☐	!!
M29 indicate FH/BH separate or combined?	☐	!!
Has Form Been Signed?	☐	
Have Date and Time Custody Released Been Noted on Form?	☐	
All Sample Containers Inspected	☐	
Does Number of Samples Match Number on CoC Form?	☐	!!
Do All Sample ID Numbers Match Those on the CoC Form?	☐	!!
Did client mark sample volumes prior to shipment?	☐	*
If required by method, did client vent samples prior to shipment?	☐	
Are the Sample Containers Intact?	☐	!!
Are signs of leakage present?	☐	*
Chain-of-Custody Form Signed and Dated by CLN	☐	
Corrective Actions	☐	
Client Contacted Due to Mismatching Sample ID Numbers	☐	
Client Contacted Due to Broken Sample Container(s)	☐	
Client Contacted Due to Leaking Sample Container(s)	☐	
Client contacted for verification of methodology?	☐	
Corrective Actions Documented?	☐	
Corrective Actions Accomplished?	☐	

*Items marked !! shall be addressed **prior to any analytical work being started**.*
*Items marked * shall be **noted in case narrative** upon reporting of results to client.*

Signed _____

Notes _____

Section 27

QUALITY ASSURANCE FOR ENVIRONMENTAL TESTING*(TNI V1:M1, V1:M2 – Section 5.9 and Section 1.7 of Technical Modules TNI V1:M 3-7)*

CHESTER LabNet has procedures for monitoring the validity of the testing it performs. The qualities of test results are recorded in such a way that trends are detectable, and where practicable, are statistically evaluated. To evaluate the quality of test results, the laboratory utilizes certified reference materials, audit samples (where available), interlaboratory comparisons (where available), replicate testing using different methods (e.g., Methods 6 and 8) and results for non-sample-based QC elements (e.g., ICV's, LCS's, etc.).

In addition to procedures for calibration, the laboratory monitors quality control measurements such as blanks, laboratory control samples (LCS), matrix spikes (MS), duplicates and certified Class 1 weights to assess precision and accuracy. Proficiency Testing samples are required by the 2009 and 2016 TNI Standard, however there are none available from a Proficiency Testing Provider Accreditor approved Proficiency Test Provider for any *accredited* method performed by ***CHESTER LabNet***. Audit Samples are required by the EPA with every set of compliance samples sent to the laboratory for which audit samples are available.

Quality control data are analyzed and, when found to be outside pre-defined criteria, action is taken to correct the problem and to prevent incorrect results from being reported. Data associated with quality control data outside of criteria and still deemed reportable will be qualified such that the end user of the data may make a determination of the usability of the data (see Section 28, "Reporting of Results").

27.1 Essential Quality Control Procedures

When not archaic, the quality control procedures specified in reference methods are followed by laboratory personnel. The 2009 and 2016 TNI Standard requires that "*When it is not apparent which is more stringent, the QC in the mandated method or regulations is to be followed."* The most stringent of control procedures is used in cases where multiple controls are offered. If it is not clear which is the most stringent, that mandated by reference method or listed in the 2009 and 2016 TNI Standard (Appendix H) is followed. Often a hybrid of the TNI, CLP and method specific QC elements and control limits is used. The laboratory rarely knows what the applicable regulation is for any set of samples, as each source and project may have different regulatory limits, regulatory agencies and QC requirements. In such situations, the laboratory defaults to its own internally-generated quality control procedures or defers to client request.

For reference methods that do not provide acceptance criteria for an essential quality control element or where no regulatory criteria exist, acceptance criteria are developed. The criteria used vary from method to method and are documented in in the method-specific SOPs.

For the Gravimetry Laboratory, criteria are set based upon the narrowest ranges of quality control which meet all of the methods being utilized in that department.

For the XRF lab, criteria are set based upon the determination of uncertainties for the instruments. Static limits are set for the percent recovery of the quality control standard analyzed with each run and the replicate analyses of samples.

The conventional chemistry lab generally relies on CLP guidelines for static limits on most quality control elements. Where CLP guidelines are not applicable, other means of determining limits for quality control elements are defined.

Written procedures to monitor routine quality controls, including acceptance criteria, are located in the method SOPs, except where noted, and include such procedures as:

- positive and negative controls to monitor tests such as blanks and matrix spikes;
- tests to define the variability and/or repeatability of the laboratory results such as replicates;
- measures to assure the accuracy of the method including calibration and/or continuing calibrations, used of certified reference materials, audit samples, or other measures;
- measures to evaluate method capability, such as detection limit and limit of quantitation or range of applicability, such as linearity;
- selection of appropriate formulae to reduce raw data to final results such as regression analysis, comparison to internal/external standard calculations, and statistical analyses;
- selection and use of reagents and standards of appropriate quality;
- measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the method such as temperature, humidity, light or specific instrument conditions; and
- " … *measures to assure the selectivity of the test method for its intended purpose*" as required by the 2009 and 2016 TNI Standard. However, it is rare that the laboratory has any discretion when it comes to the method utilized. Method selection lies with the laboratory's clients, their clients and/or their clients' regulatory agencies. The laboratory attempts to follow the method chosen by the aforementioned entities to achieve results which are, at a minimum, useable, and preferably accurate.

27.2 Internal Quality Control Practices

Analytical data generated with QC samples that fall within all prescribed acceptance limits indicate that the method is deemed to be in control.

QC samples that fall outside QC limits indicate that the method is out of control (non-conforming) and corrective action is required, and/or that the data need to be

qualified (see Section 12, “Control of Non-conforming Environmental Testing Work” and Section 14, “Corrective Actions”).

Detailed QC procedures and QC limits are included in method standard operating procedures (SOPs).

All QC measures are assessed and evaluated on an on-going basis so that trends are detected.

27.2.1 General Controls

The following general controls are used, where possible:

27.2.1.1 Positive and Negative Controls such as:

a) blanks (negative); and,

b) laboratory control samples (positive).

27.2.1.2 Selectivity is assured through:

a) absolute and relative retention times in chromatographic analyses;
b) use of the correct method according to its scope assessed during method validation;
c) use of qualitative spikes where sample matrices may “push” peaks around;
d) presence of mass for gravimetric analyses;
e) use of element specific wavelengths for analysis by ICP;
f) use of element specific KeV excitation lines for analysis by XRF; and,
g) use of temperature and transmittance/reflectance for analysis by OCEC.

27.2.1.3 Consistency, Variability, Repeatability and Accuracy are assured through:

a) proper installation and operation of instruments according to manufacturer’s recommendations or the processes used during method validation;
b) monitoring and controlling environmental conditions (temperature, access and proximity to potential contaminants);
c) selection and use of reagents and standards of appropriate quality;
d) cleaning glassware appropriate to the level required by the analysis as demonstrated with Method Blanks (See SOP AD-004);
e) following SOPs and documenting any deviation, assessing for impact, and treating data appropriately;

f) testing to define the variability and/or repeatability of the laboratory results, such as replicates; and

g) use of measures to assure the accuracy of the method, including calibration and/or continuing calibrations, use of certified reference materials, audit samples, or other measures.

27.2.1.4 Method Capability (also see Section 22, "Environmental Methods and Method Validation") is assured through:

a) establishment of the detection limit where applicable;

b) establishment of the limit of quantitation or reporting level where applicable; and/or

c) establishment of the acceptable concentration range where applicable, such as linearity.

27.2.1.5 Data reduction is assured to be accurate by:

a) selecting appropriate formulae to reduce raw data to final results, such as regression;

b) following specific procedures for data reduction, such as manual integration procedures; and

c) reviewing data reduction processes periodically to assure applicability.

27.2.1.6 Sample specific controls are used, where possible, to evaluate the effect of sample matrix on the performance of the selected analytical method (not a measure of laboratory performance). For example:

a) Matrix Spike and Matrix Spike Duplicate (MS/MSD)

b) Sample Duplicates

c) Post digestion/extraction Replicates

d) Post digestion/extraction Spikes

27.2.1.7 The following tables summarize the key elements of a quality control system for a laboratory performing chemical testing. Note that many air methods consume the entirety of the sample and do not allow for redigestion or reanalysis. Many also are not compatible with some of the QC elements listed below (e.g., Method 5 and Method 202 cannot have an LCS). Some instrumentation used in air quality analyses does not lend itself to all of the elements listed below (e.g., LCS or blanks for XRF analysis, spikes for $PM_{2.5}$ analysis). All QC elements defined in Table 27-1 are understood to be "where applicable."

Table 27-1 Essential Quality Control Elements for Chemistry			
Item	**Frequency**	**Acceptance Criteria**	**Corrective action**
Initial Calibration	As required by method or technology	Laboratory defined based on 2009 and 2016 TNI Standard, Volume 1, Module 4.	Recalibrate.
Initial Calibration Verification - ICV	Immediately after successful calibration	Method specific or 90% - 110% Recovery	Corrective action as given in SOP
Initial Calibration Blank - ICB	Immediately after ICV	Method specific or <DL	Corrective action as given in SOP
Low Level Calibration Verification – LL-CCV	1/analytical run where instrument is calibrated with single point standard (ICP).	Method specific or 60% - 140% Recovery	Corrective action as given in SOP
Negative Control (Method Blank)	1/digestion batch	Laboratory defined	Reprocess, reanalyze, or qualify data.
Negative Control (Sample Media Blank)	1/digestion batch	Method specific, client specific, regulatory specific, contract specific or as determined by the laboratory	Reprocess, reanalyze, or qualify data.
Positive Control (Laboratory Control Sample - LCS)	1/digestion batch	Method specific or 80% - 120% Recovery	Reprocess, reanalyze, or qualify data.
Positive Control (Laboratory Control Sample Duplicate – LCS-D)	1/digestion batch	Method specific or 80% - 120% Recovery	Reprocess, reanalyze, or qualify data.
Positive Control (Low Level Laboratory Control Sample – LL-LCS)	1/digestion batch	Method specific or 50% - 150% Recovery	Reprocess, reanalyze, or qualify data.
Matrix Spike; Matrix Spike Duplicates (pre - digestion) *Note: Spiked samples are designed as data quality indicators for a specific sample using the designated method. These controls alone are not used to judge a laboratory's performance.*	Minimum of 1/digestion batch where possible. Per method requirement, client request, or laboratory discretion.	Method specific or 75% - 125% Recovery	Reprocess, reanalyze, or qualify data.

Table 27-1 Essential Quality Control Elements for Chemistry			
Item	**Frequency**	**Acceptance Criteria**	**Corrective action**
Matrix Duplicates/Replicates *See note above.*	Minimum of 1/digestion batch where possible. Per method requirement, client request, or laboratory discretion.	Method specific or ±20% RPD	Reprocess, reanalyze, or qualify data.
Continuing Calibration Verification - CCV	1 every 10 analytical readings	Method specific or 90% - 110% Recovery	Corrective action as given in SOP
Continuing Calibration Blank - CCB	Immediately after every CCV	Method specific or <DL	Corrective action as given in SOP

Note1: Control Limit for LL-CCV based on twice the LCS control limit, with a larger allowable tolerance due to the LL-CCV being at the LoQ.

Note 2: Control Limit for LL-LCS based on the LL-CCV control limit plus an additional 10%, due to the concentration of the LL-LCS being below the LoQ.

27.2.2 Specific Controls

27.2.2.1 Blanks

Laboratory blanks (Lab Blanks) are used for Air Quality Methods requiring evaporation and/or gravimetry (Appendix B, J and L; Methods 5, 5A – 5F, 201A, 202, and similar). Laboratory blanks consist of an empty weighing vessel (e.g., beaker, pan, or filter) which follows the other weighing vessels through the process, to the extent possible, and are then gross weighed with the rest of the weighing vessels. A lab blank must be analyzed at a minimum of one per sampling batch, which may exceed 20 samples although it rarely does. For Appendices B, J and L analyses, a lab blank is a blank filter which has been tare weighed, but has never left the confines of the laboratory. It is gross weighed along with its associated samples and must meet the same QC criteria as those samples. Control limits exist only for $PM_{2.5}$ Lab Blanks (± 15µg). The other gravimetric methods have no controls for lab blanks. Per the reference method, the data is reported "as-is" since even negative numbers have interpretive value to the client. Filter Laboratory Blanks are only performed at the request of the client.

Method Blanks are processed along with and under the same conditions as the associated samples to include all steps in the method and all reagents used. They do not, however, contain any of the matrices on or in which a sample is captured (e.g., no filter or sorbent material). A

Method Blank must be analyzed at a minimum of one per preparation batch.

When no separate preparation method is used, the batch is defined as the environmental samples that are analyzed with the same method and personnel, using the same lots of reagents, not to exceed the analysis of twenty environmental samples, not including Sample Media Blanks, LCS, matrix spikes and matrix duplicates. In the case of no separate preparation method, an ICB or CCB is considered equivalent to a Method Blank.

Sample Media Blanks are processed along with and under the same conditions as the associated samples to include all steps and matrices (e.g., filter type, sorbent material) in the method. A Sample Media Blank must be analyzed at a minimum of one per preparation batch, whenever the laboratory has media available from the same manufacturer's lot number as used during sampling. If no media from the same manufacturer's lot number as used during sampling is available, reagent water is used for QC elements and no Media Blank is analyzed. Nearly all media used in the sampling of ambient or source air have some background contamination from the manufacturing or sampling processes. Due to the complex nature of many source sampling methods, Sample Media Blanks may have detectable levels of analytes of interest present.

The Sample Media Blank results should be lower than the lowest sample result, or within 20% of the sample result in cases where the result is greater than five times the detection limit. Sample Media Blanks are not required for some analyses such as XRF, OC/EC and pH.

Contaminated blanks are identified according to the acceptance limits in the method SOPs. Note that all filter media are not created equal and all filter media may have some form of contamination present. The laboratory has no control over filter manufacturing or the choice of filter matrix and, thus, it is expected that some filters for some analyses will show detectable quantities for some analytes.

The laboratory identifies a media blank as contaminated based upon the method being used. For Method 29 Mercury analysis, for example, a Method Blank is considered contaminated if the result is higher than the detection limit. For Glass Fiber filter analysis, the list of commonly seen contaminants is quite long, thus the performance of the laboratory would not be considered insufficient if the Sample Media Blank were to have analytes of interest present. For Quartz filters, the list is somewhat shorter. Cellulose filters will always be very high in Organic Carbon if analyzed by OC/EC as cellulose is very high in Carbon. Teflon filters are generally very clean for most analytes, but if used for Method 13B, will yield high amounts of fluoride. Each matrix, analyte and method must be taken into account when determining if a Sample Media Blank is considered contaminated enough to induce corrective actions.

When a blank is determined to be contaminated, the cause must be investigated and measures taken to minimize or eliminate the problem.

Data that are unaffected by the blank are reported unqualified. Data is considered unaffected if the result for the analyte of interest in the blank is <DL or <10% of the lowest sample value obtained.

Sample data that are suspect due to the presence of a contaminated blank are reanalyzed if possible, or noted in the Case Narrative of the final report. If the blank results are less than the sample results and the contaminant is common in the media being analyzed, no notation is made in the report.

Regardless of whether the blank(s) affect the sample results or not, all Method Blank and Sample Media Blank results are reported to the client in the form of a QC report. It is the policy of the laboratory to never blank subtract results submitted to the client. The client is responsible for performing or not performing any blank subtraction. Most reference methods using filter media have blank subtraction protocols.

Client-provided Field Blanks, Trip Blanks, Proof Blanks, Recovery Blanks, Train Blanks and Reagent Blanks have no control limits and are treated as samples.

27.2.2.2 Laboratory Control Samples

Laboratory Control Samples (LCS), Duplicate Laboratory Control Samples (LCS-D), and Low Level Laboratory Control Samples (LL-LCS) are prepared from analyte-free water and spiked with verified and known amounts of analytes for the purpose of establishing precision or bias measurements and for collection of data to perform DL verification studies (See Appendix H). Not all methods are amenable to LCS's (e.g., OCEC, XRF, PM_{10}, $PM_{2.5}$, Method 202, etc.).

Laboratory Control Samples (LCS and LCS-D) are analyzed at a frequency mandated by method, regulation, laboratory discretion or client request, whichever is more stringent. The standard frequency of LCS preparation and analysis is one per preparation batch of 20 or fewer samples, or as otherwise stated in a laboratory SOP. LCS-Dups are only prepared when the method or sample size prevents the laboratory from analyzing a Sample Duplicate. Low Level Laboratory Control Samples (LL-LCS) are analyzed once per preparation batch of 20 or fewer samples, or as otherwise stated in a laboratory SOP. Exceptions would be for those analytes or methods where no spiking solution is available, such as Method 202, OCEC and XRF analysis, PM_{10} or $PM_{2.5}$ analysis, etc.

When no separate preparation method is used, the batch is defined as the environmental samples that are analyzed with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples. In the case of no separate preparation

method, an ICV or CCV is considered equivalent to an LCS/LCS-D.

When there is no separate preparation method, a Low Level Laboratory Control Standard (LL-LCS) is prepared and analyzed in a similar manner as a CCV standard. Results from the LL-LCS are used to verify the low end of the calibration curve (LoQ) and to gather data for verification of the detection limit. No reference method in use by the laboratory as of this writing has a requirement for an LL-LCS.

The analytes to be spiked in the LCS are the same as the analytes specified by the client or in the method SOP. The client's requests take precedence over the reference method.

The results of laboratory control samples (LCS, LCS-D and LL-LCS) are calculated in percent recovery. The calculation for percent recovery is given below:

$$\%R = (AV/KV) \times 100$$

Where:
AV = Analyzed Value
KV = "known value" (True Value Spiked)

The individual LCS percent recovery is compared to the acceptance criteria as published in the reference method, or the laboratory established limits when there are no established criteria, as described above. Where no established criteria exist, the laboratory defaults to ±20% of the true value for LCS's and LCS-D's, per CLP guidelines, and to 50% - 150% for LL-LCS's. See Note 2, Table 27-1.

27.2.2.3 Matrix Spikes and Matrix Spike Duplicates

Matrix Spikes and Matrix Spike Duplicates (MS/MSD) are a second/third aliquot of an environmental sample fortified with a known amount of analyte to help assess the effect of the matrix on method performance. ***CHESTER LabNet*** rarely performs Matrix Spike Duplicates. In some cases, a second aliquot of the sample may not be possible, in which case a second aliquot of the extract/digestate will be utilized (called a Post-Digestion Spike or "post spike" to differentiate it from a true spiked analysis).

The laboratory procedure for a matrix spike includes spiking appropriate analytes at appropriate concentrations, calculating percent recoveries, and evaluating and reporting the results. The procedure is documented in in the pertinent method SOPs. The calculation for percent recovery is given below:

$$\%R = \frac{(AV - SV) \times 100}{KV}$$

Where:
AV = Analyzed Value
SV = Sample Value
KV = "known value" (True Value Spiked)

The individual spike percent recovery is compared to the acceptance criteria as published in the mandated reference method, or the laboratory established limits when there are no established criteria. Where there are no established criteria, the laboratory defaults to the CLP guidelines of 75% - 125% Recovery.

For spike results outside established criteria, the data are reported with annotations in the report. For some methods, such as Fluoride by Method 26, it is common to have matrix interferences which cause the spike to fall out of control. For other methods, it is not possible to spike the sample (e.g., OCEC, XRF, Method 202, $PM_{2.5}$, PM_{10}).

27.2.2.4 Matrix Duplicates

Matrix Duplicates ("dups") are a second aliquot of an environmental sample processed along with and under the same conditions as the associated sample to include all steps in the method. In some cases, a second aliquot of the sample may not be possible, in which case a second aliquot of the extract/digestate will be utilized (called a Replicate or "rep" to differentiate it from a true duplicate analysis).

The laboratory procedure for a matrix duplicate/replicate includes calculating the relative percent difference (RPD) and evaluating and reporting the results. The calculation for Relative Percent Difference is given below:

$$\%RPD = \frac{|(SV - DV)| \times 100}{\overline{X}_{SV,DV}}$$

Where:
SV = Sample Value
DV = Duplicate Value
$\overline{X}_{SV,DV}$ = average of sample and duplicate values

Some methods, notably Method 26/26A and most Source Mercury methods, require all samples to be analyzed in duplicate. These methods stipulate that the RPD of a given sample analysis be within 5% RPD or 3% RPD (method dependent). Method 12 requires triplicate analyses, with no more than 5% relative standard deviation (RSD) between data points for the same sample. That calculation is not shown here. Where no established criteria exist, the laboratory defaults to ±20% RPD, per CLP guidelines.

27.2.2.5 Calibration

All instruments requiring a multi-point calibration must use a minimum of 5 calibration points for a linear curve, and six calibration points for a quadratic curve. The correlation coefficient of the curve must be greater than 0.995.

The accuracy of all calibration points is verified by determining the Percent Relative Error of each standard. Standards with concentrations at or below the LoQ must have a Relative Error ≤50%. Standards above the LoQ must have a Relative Error ≤10%.

The calculation for Relative Error (expressed as a percent) is given below:

$$RE \text{ (or } \%RE) = 100 - \frac{(AV - KV) \times 100}{KV}$$

Where:
AV = Analyzed Value
KV = "known value" (True Value Spiked)
%RE is presented as an absolute value

27.3 Proficiency Test Samples or Interlaboratory Comparisons

27.3.1 Compliance with Accreditation Requirements

The 2009 and 2016 TNI Standard states, "*The laboratory shall analyze and report a PT study at least twice per year for each accreditation FoPT for which it seeks to maintain accreditation…*" No FoPT exists for the TNI defined Quality Matrix of "Air".

In June, 2013, the US EPA required all source samples analyzed for regulatory or compliance purposes be analyzed with an audit sample, where an audit sample is available. In 2017, the laboratory analyzed 100 audit samples for Method 26/26A compliance sampling events, or approximately two audit samples per week.

The SSAS expert committee of NELAC applies to Source Emissions samples. An FoPT does not exist for source emissions or ambient air sampling. At this time, the only method for which the laboratory is accredited and for which an audit sample exists is 40 CFR 60 Method 26A. Audit samples are also available for Method 29, Method 101A, Method 6 and Method 8.

The client is responsible for providing the audit sample to the laboratory, as the regulator is responsible for requesting a specific concentration range of the audit sample for the facility from which the client is collecting samples. Based upon client demand, the laboratory may run audit samples for a given method once per year or in excess of once per week. All SSAS-related TNI laboratory requirements are followed.

27.3.2 Audit Sample Handling, Analysis and Reporting

Audit samples are obtained by the client and sent to the laboratory with the client's associated samples. The laboratory does not share audit samples with other laboratories, except at the request of the client, and does not communicate with other laboratories regarding current audit sample results. In cases where audit or inter-laboratory samples are provided by the client, the laboratory does not attempt to obtain the assigned value of any audit sample from the audit provider or client prior to submission of results.

Per the SSAS Module, which is functionally parallel to the water/soil TNI PT programs, audit samples for source samples are to be reported simultaneously and directly to the audit Provider, the regulator and the client. To determine whether the laboratory's method is in control, the laboratory determines the assigned value of the audit sample from the audit provider (agency, client or manufacturer). If the assigned value cannot be obtained, the laboratory makes a good faith effort to determine, at the very least, whether the laboratory passed or failed the audit sample.

It is highly unusual for the laboratory to request an audit sample from a third party. In the rare cases where *the laboratory* requests an audit from a third-party provider, the laboratory may give the audit provider a concentration range to ensure that the audit samples are neither higher than the high end of the calibration curve nor below the detection limit. The laboratory may request a "trip blank" audit sample, in addition to the usual audit samples requested, to prove that no contamination occurred during the shipping and handling of the sample media.

Audit samples are treated in the same manner as regular samples in the normal production process where possible, including the same Analysts, preparation, calibration, quality control and acceptance criteria, sequence of analytical steps, number of replicates, and sample log-in. Audit samples are not analyzed multiple times unless routine samples are analyzed multiple times. Where audit samples present special problems in the analysis process, they will be treated as laboratory samples where samples present special problems.

The type, composition, concentration and frequency of quality control samples analyzed with the audit samples are the same as with typical samples.

Prior to the reporting of an audit sample, laboratory personnel do not:

- subcontract analysis of an audit sample to another laboratory being run for compliance purposes;
- knowingly receive and analyze an audit for another laboratory being run for compliance purposes, unless at the request of the client;
- communicate with an individual from another laboratory concerning the analysis of the audit sample; or
- attempt to find out the assigned value of an audit from the audit provider.

The laboratory's procedure for handling low level audit samples is to treat the audit sample in exactly the same manner as it would treat any other sample of low concentration. This may include the reporting of "< [DL]" as a result.

As there are no FoPT PT or PE or audit samples available for *source emissions or ambient air* sampling methods, the laboratory cannot participate in the TNI FoPT PT or PE study for ambient air methods.

For interlaboratory comparison samples submitted with other samples (e.g., "Round Robin" samples), the laboratory reports the data alongside the data for the samples received in conjunction with the interlaboratory comparison.

Any result at or above the detection limit is reported as the resultant value. Any result less than the detection limit is reported as < [DL]. No reference methods performed by the laboratory have LoQ requirements.

The laboratory institutes corrective action procedures for failed audit samples following the guidelines in Section 14, "Corrective Action". Following the TNI SSAS Volume 1, Module 3, corrective action for a failed audit sample is the responsibility of the regulator, not the laboratory or field tester.

Retention of audit records is similar to that maintained for typical analytical records, and audit results and all documentation pertinent to those results are maintained in the report file containing the samples to which the audit sample pertains. In addition, the Laboratory Director maintains a 3-ring binder containing all audit results.

Note: It has been the laboratory's experience to date that most audit providers do not understand the methods for which the audit is supplied, and consequently, it is very common for the laboratory to need to heavily modify the instructions and calculations in order to meet the criterion of treating the audit sample in the same manner as the samples. For example, a Method 29 audit sample's instructions may say "dilute 5 mL to 1000 mL and analyze by ICP" where the method requires the entire impinger catch to be evaporated down to 50 mL at sub-boiling temperatures. The audit instructions to 'dilute and shoot' do not reflect the preparation process that the actual samples undergo. The laboratory will, to the best of its ability, treat the audit sample as closely as possible to the reference method.

27.4 Data Review

The laboratory reviews all data generated in the laboratory for compliance with method, laboratory, and, where appropriate, client requirements.

Three levels of data validation are performed. Levels 0 and I are performed on all samples received by ***CHESTER LabNet***. Level II data validation is only performed when required by contractual obligation. The purpose of data validation is to ensure that the reported data are free from transcription and calculation errors (manual or electronic), and that all quality control measures are reviewed and evaluated prior to data being reported.

27.4.1 Level 0

Level 0 validation occurs at the sample receipt and log in stage of sample analysis. Elements of Level 0 validation include:

- examining the integrity of custody seals, if present;
- taking the temperature of the transit temperature bottle, if present, and record on chain of custody;
- examining integrity of shipping bottles or containers;
- examining the chain of custody (COC) form(s) for the presence of all required information and signatures;
- verifying the number of samples and sample IDs against those listed on the COC form(s);
- Completing the Sample Receipt Checklist(s) appropriate to the samples received;
- retaining Sample Receipt Checklist(s) in the job file;
- contacting the appropriate authority upon finding irregularities;
- documenting and performing corrective actions; and,
- completing the project specific checklist confirming Level 0 validation, and place in the project file (for CLP or CLP-like reports only).

For projects requiring additional documentation of the level 0 validation process, ***CHESTER LabNet*** provides a written checklist covering the above steps. This checklist is filled out, signed, and dated by the Sample Custodian or designated alternate. The completed checklist is added to the project file.

27.4.2 Level I

Level I data validation begins during sample analysis and is carried out at the instrument by the Analyst. This phase of level I validation involves performing and maintaining instrument calibration and assessing precision and accuracy of the data via the analysis of all of the appropriate QC checks, as discussed in Section 27.1 and 27.2. The Analyst ensures that the QC statistics are within control limits and takes appropriate corrective actions during analysis, if needed.

For projects requiring additional documentation of the level I validation process, the laboratory provides a written Analyst's checklist. This checklist is filled out, signed and dated by the Analyst. The completed checklist is added to the project file.

The second phase of level I data validation is performed by the QA Officer or Technical Director for that particular department. During this phase, raw data is verified as being in control with the appropriate QC parameters, worklists are checked for accuracy against the raw data, raw data is checked for any discrepancies which may have been missed by the Analyst (e.g., spike lot numbers or expiration dates) and any corrective actions are taken to remedy deficiencies prior to the data being submitted to the Project Manager.

The third phase of level I data validation is performed by the QA Officer or Project Manager, who confirms all keyboard entries and electronic data entries into the LIMS, then confirms that the correct analyses have been completed on the correct samples. The Project Manager then reviews all of the data and QC results for a given project or report and, for certain clients, prepares QC summary tables and data assessments. Problem data discovered during this review are annotated in the report.

If any analytical errors are found in any of these stages of data review, and there is enough sample extract remaining, and holding times have not been exceeded, the preparation and/or analysis will be repeated and the new results will be subjected to the same QC/validation.

The final report is reviewed by the Laboratory Director, who signs the report prior to its release to the client. SOPs QA-002 and AD-007 are relevant to this stage of review.

27.4.3 Level II (by client request only)

Level II data validation is only performed for CLP style reports, is carried out by the QA Officer, and occurs after the data package has been correctly assembled. The first step is to recalculate, by hand, the final result for a randomly chosen sample. This is accomplished by first taking the raw calibration data and recalculating the appropriate calibration statistics (i.e., slope, intercept and correlation coefficient). Next, using the raw instrument response, the instrument concentration result is recalculated. Finally, the sample preparation data (i.e., digestate volume, filter aliquot size, etc.) are used to recalculate the final result as reported to the client. All of these steps are documented on a Sample Calculation form, which is signed and dated by the reviewer and included in the final data report.

The second step is to review all QC statistics and raw data for compliance with control limits, frequency of application, and correct sequences. In addition, flagging is checked as well as reporting units, holding times and the correct use of significant figures. Finally, corrective actions (if applied) are noted. The review is aided by following a preprinted checklist, which is signed and dated by the QA Officer and placed in the data report. Results for all data review, verification and cross checking procedures are documented within

each data package, to the extent that is required for each particular client's needs. At a very minimum, documentation shall consist of at least one person's signature or initials attesting to the performance of data review.

Section 28

REPORTING THE RESULTS
(TNI V1:M2 – Section 5.10)

The result of each test performed is reported accurately, clearly, unambiguously and objectively, and complies with all specific instructions contained in the reference method and/or required by client or regulator.

The 2009 and 2016 TNI Standard states, "*The results … shall be reported accurately, clearly, unambiguously and objectively, and in accordance with any specific instructions in the test or calibration methods. The results shall be reported, usually in a test … and shall include all the information requested by the customer and necessary for the interpretation of … results and all information required by the method used.*" For Aqueous samples, this usually includes both sampling and analysis. As the laboratory's clients perform the sampling from collection to shipment (see Disclaimer prior to Section 3), the laboratory has no knowledge of the sampling methods nor control over the actions or inactions of the clients in the field. Accordingly, the laboratory cannot include in its report information which it is neither responsible for nor privy to. Thus, if a method requires samples be reported in µg/dscm, but the client does not provide air volumes, temperature or humidity data, it is impossible for the laboratory to report all information required by the method used. The laboratory reports do include all the information requested by the client and necessary for the interpretation of the test results where possible.

Data are reported without qualification if they are greater than the LoQ, lower than the highest calibration standard, and without compromised sample or method integrity. The detection limit (DL) is reported with each sample/analyte for all reports where the method is amenable to a detection limit study. The LoQ is defined as five-times the detection limit. Data falling between the DL and the LoQ are reported as qualified data. Note: For air quality methods, meeting the requirement of reporting the LoQ places the lab in violation of reporting data "clearly, concisely and *unambiguously*" as air quality methods report to the detection limit, and consequently most of the laboratory's clients do not understand the meaning of an LoQ, and the inclusion of LoQs on the report causes confusion to the client.

28.1 Test Reports

The report formats have been designed to accommodate each type of test performed and to minimize the potential for misunderstanding or misuse. The laboratory does not issue multiple reports for the same samples where there is different information on each report unless requested to do so by the client (e.g., one report for regulatory purposes, a second report for engineering purposes).

A typical test report contains the following elements:

- a Cover Page/Title Page;
- a Case Narrative page;
- qualification of results with values outside the calibration range as appropriate (usually in the Case Narrative);

- data summary sheets;
- a QA/QC summary section (does not contain qualifiers, see Note below);
- a Chain of Custody form; and,
- a page stating that raw data are available upon request.

Note: The LL-LCS is set at approximately three-times the detection limit, while the LoQ is set at five-times the detection limit. Consequently, all QA/QC summary sections would require flagging of the LL-LCS. This would create added confusion for the client which would violate the need to report results "*clearly and unambiguously*", thus, no flagging is performed in the QA/QC summary.

Raw data are not included in a report unless specifically requested by the client. If requested, raw data are then reported as an Appendix.

Each test report generated, electronic or hardcopy, contains the following information:

a) a title, in the format of [Client Name; Project Name or Number*, Client Number, Report Number] (*if the project name or number has been provided by the client);

b) the name and address of the laboratory, the laboratory's phone number and name of a laboratory contact person;

c) unique identification of the test report, in the form of a report number, on each page and a pagination system that ensures that each page is recognized as part of the test report and a clear identification of the end of the report, such as "page 3 of 10";

d) the name and address of the client (typically on the CoC), where provided by the client;

e) the identification of the method used including revision number where there is one. Revision numbers may be the date of online promulgation if none is given in the method. Not all air quality methods have revision numbers;

f) the date of sampling for each sample, where provided by the client;

g) a description of, the condition of, and unambiguous identification of the sample(s) tested, including the client identification code;

h) the date of sample receipt, the time of sample preparation and analysis if the required holding time for either activity is less than or equal to 72 hours and, date(s) of analysis;

i) procedures used by the laboratory where these are relevant to the validity or application of the results;

The 2009 and 2016 TNI Standard requires the reports to include a reference to

"the location of sampling, including any diagrams, sketches or photographs;" and *"a reference to the sampling plan and procedures used,"* and *"details of any environmental conditions during sampling that may affect the interpretation of the test results,"* and *"any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned."* As the client - not the laboratory - performs sampling, the laboratory does not include this in test reports. It is the responsibility of the client collecting the samples to have a copy of their own sampling plan. It is exceedingly rare that the laboratory has a copy of the client's sampling plan. For some samples, a sampling plan may not even exist, and the laboratory would have no means of discerning this fact based upon the samples at time of receipt.

j) the test results, units of measurement, DL and LoQs for methods where DL studies are possible, failures identified (See Appendix F for a list of laboratory qualifiers);

k) the name, function and signature or an equivalent electronic identification of the person authorizing the test report and the date of issue;

l) a statement to the effect that the results relate only to the samples;

m) any non-accredited tests or parameters are clearly identified as such to the client when claims of accreditation to this Standard are made in the analytical report or in the supporting electronic or hardcopy deliverables; and

n) A statement that the report shall not be reproduced, except in full, without written approval of the laboratory.

28.2 Supplemental Test Report Information

When necessary for interpretation of the results or when requested by the client, test reports include the following additional information, usually found in the Case Narrative:

a) deviations from, additions to, or exclusions from the method SOP, information on specific test conditions such as environmental conditions, any non-standard conditions that may have affected the quality of the results, and any information on the use and definitions of data qualifiers;

b) where relevant, a statement of compliance/non-compliance with requirements and/or specifications;

c) where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a customer's instruction so requires, or when the uncertainty affects compliance to a specification limit;

d) opinions and interpretations, where appropriate and needed (When opinions and interpretations are included, the basis of those opinions and interpretations is documented. Opinions and interpretations are clearly marked as such in the test report.);

e) additional information which may be required by specific methods or client(s); and

f) qualification of results with values outside the calibration range as appropriate.

28.3 Environmental Testing Obtained from Subcontractors

Test results obtained from tests performed by subcontractors are most commonly sent directly to the client by the subcontractor and are not included in the laboratory's test report. Where the laboratory submits subcontracted data to the client, the entire subcontractor's report is submitted to the client, unless otherwise requested by the client.

28.4 Electronic Transmission of Results

All test results transmitted by e-mail or other electronic means comply with the requirements of the 2009 and 2016 TNI Standard and associated procedures to protect the confidentiality and proprietary rights of the client (see Section 22, "Environmental Methods and Method Validation").

CHESTER LabNet provides electronic data deliverables in a variety of formats on a client specific basis. E-reports are most commonly sent as email attachments. Files have been formatted as: CSV files, MS Excel files, fixed width column files, Adobe Acrobat files (.pdf), spreadsheet files, text files or proprietary client software files. ***CHESTER LabNet*** works closely with the client to ensure that e-reports are in a useable and acceptable format, to include the use of password protected delivery where requested by client. Preparation of electronic deliverables is highly specific to the client and/or project (some clients may have more than one project in progress, and each project may have a different electronic reporting format requirement).

28.5 Amendments to Test Reports

Material amendments to a test report after it has been issued are made only in the form of another document or data transfer. All supplemental reports meet all the requirements for the initial report and the requirements of this *Quality Manual*.

Amended test reports are re-issued and the re-issuance is documented by appending a chronological revision number to the report number (i.e., "Report #12-421 revision 1").

When it is necessary to issue a complete new report, the new report is uniquely identified and contains a reference to the original that it replaces, using the same nomenclature as above (i.e., "Report #12-421 revision 1"). The only exception to this is editorial changes, such as typographical errors, to a single page in the report.

28.6 Exceptions

Due to the highly complex nature of air quality testing, the wide variances between each regulatory jurisdiction, the wide variances between each individual client's preferences, attempting to list exceptions to the reporting procedures above would be a monumental undertaking.

Reporting shall meet the needs of the client first, then, whenever possible, the requirements of the 2009 and 2016 TNI standard.

Appendix A

Personal Ethics and Data Integrity Policy

A.1 Introduction

CHESTER LabNet's goal is to provide the most informed and accurate inorganic analysis of air quality samples possible from a commercial laboratory. ***CHESTER LabNet***'s management is committed to good professional practice and to the quality of its environmental testing in servicing its clients.

To achieve this goal, it is critical that all employees understand:

- the need for honesty and full disclosure of variances in all areas of analyses performed;
- when and how to report data integrity issues; and,
- the documentation of such issues when they arise.

Data integrity is defined as data of known (traceable and documented) quality, analyzed by documented procedures, fulfilling all Quality Control requirements established by those procedures, and meeting the requirements of the client. Inherent in the concept of data integrity is that no false manipulations of data or samples or omissions of pertinent information be performed to meet the Quality Control criteria. This inherent need is governed by the personal ethics of each employee and the overall corporate culture of ***CHESTER LabNet***.

The personal ethics of each and every employee results in the laboratory's ability to:

- Produce accurate results, which include QA/QC information that meets the client's or method's pre-defined Data Quality Objectives (DQOs);
- Present services in a confidential, honest and forthright manner;
- Provide employees with guidelines and an understanding of the ethical and quality standards of our industry;
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same;
- Educate clients as to the extent and kinds of services available;
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made; and
- Promote the status of environmental laboratories, their employees and the value of services rendered by them.

A.2 Management Responsibilities

Management responsibilities are many and begin with creating a culture of trust and honesty within the organization. Technical directors of each department understand that employees are human and do make mistakes. Honest mistakes are corrected and discussed with the employee. Technical Directors are charged with upholding the intent of this policy and implementing the specific requirements not only of this policy, but also of each documented procedure practiced by the laboratory. In addition, Technical Directors must perform their oversight duties with a positive attitude, maintaining focus on the goal of producing high quality data, and without personal attacks or negative attitudes which might lower morale or decrease the likelihood of employees being open and honest. Managers and Technical Directors must do their utmost to encourage a corporate culture of honesty and security for each employee, such that no employee is ever afraid to bring forth issues or problems they might encounter.

Technical Directors monitor the adherence of this document by supervising their employees and the data and/or reports produced by their employees. Prior to initiating an investigation into unethical behavior, Managers are required to challenge the evidence against Hanlon's Razor. If the challenge is passed, evidence of unethical behavior such as improper manipulations of data, clock rolling, inappropriate changes in concentrations of standards, failure to follow written procedures to bypass Quality Control checks, insufficient documentation, etc., are addressed to the employee and are documented via the annual employee review or addenda to the annual employee review, which are kept in the employee's personnel file. Technical Directors are charged with monitoring the breach after such a discussion to ensure that the employee's behavior has changed. If no change has occurred, the Technical Director and Laboratory Director shall decide upon the appropriate action to be taken. Actions may include termination of the employee, moving the employee to a different department, revoking some of the employee's duties or other actions to resolve the issue and prevent its further occurrence. The worst-case scenario may result in criminal or civil prosecution of the individual employee, fines or possible prison sentences. The laboratory does not and will not defend any employee charged in a court of law who, despite management's best efforts, knowingly submits false, incomplete or undocumented flawed data.

The QA Officer is responsible for initial data integrity and ethics training for all new personnel. In addition, the QA Officer performs annual refresher training for all staff. The QA Officer is responsible for maintaining documentation of the Data Integrity and Ethics training of all personnel in the form of signatures in this appendix.

In addition, the QA Officer is responsible for performing in depth data monitoring on a regular basis. This monitoring occurs during data review, periodic and random logbook checks, periodic and random standards checks, the internal audit process, review of corrective action reports, and investigations of any issues brought up by other employees.

Management has a zero tolerance policy for unethical behavior. ***CHESTER LabNet*** does not tolerate unethical behavior of any sort by its employees, whether said behavior is related or unrelated to data production. If a breach of ethics is found to be supported by evidence, the employee may expect to be terminated.

A.3 Employee Responsibilities

"Employees" include both managerial and non-managerial staff. Employee responsibilities include following written procedures and known scientific principles to produce data of the highest degree of scientific defensibility possible within the limitations of the sample matrices and currently available instrumentation. Each employee is responsible for ensuring that the data and/or reports they produce are accurate and complete (to include observations), and meet the Quality Control criteria described in the method or written procedure for the task.

An employee's personal ethics play a large role in maintaining data integrity. While ethics are more difficult to define and certainly more difficult to instill and enforce, for the purposes of this document, the most fundamental ethic required by ***CHESTER LabNet*** is honesty. Intent to deceive, either by data manipulation, verbal falsification of procedures followed, or by omission, is not supported in any way by ***CHESTER LabNet***.

If the issue is not addressed in the relevant SOP, all employees are charged with reporting any data integrity issue, be it their own or that of others to their Technical Director or supervisor in a timely manner. If that person seems unresponsive, employees should report their concern to the QA Officer or to the Laboratory Director. Non-reporting of known breaches of ethics is considered equally as damaging as having performed the breach oneself, and is subject to the same consequences as described in Section A.4. A Corrective Action Report is initiated by the person discovering the issue.

Breaches of ethical behavior include, but are not limited to:

- blatant falsification of data;
- improper data manipulations, such as questionable hand integrations, peak shaving, undocumented blank subtractions, not following established rounding rules in order to meet quality control criteria, etc.;
- changing computer clocks to show a different time in order to meet holding time criteria;
- changing standard or QC sample concentrations to force them to meet QC criteria (e.g., diluting or spiking LCSs);
- intentional failure to record information as described in the relevant SOPs (e.g., not recording balance calibration data or temperature/humidity during gravimetric analysis, etc.); and
- intentional failure to record information which may be of value to the client in interpreting results (e.g., "filter corner missing" for negative mass filters, "Acetone fraction shipped in plastic bottles" for gravimetric analysis, "non-homogenous sample deposit" for chemical analysis of filters, etc.).

Given the wide variety of matrices, sampling methods, background contaminants and physical states of samples analyzed at ***CHESTER LabNet***, it is to be expected that Quality Control criteria will occasionally fail. It is the responsibility of the employee to properly document the failure, attempt to meet the Quality Control criteria, where possible, and ensure the client is informed of such deviations from normal protocol.

Any errors must be lined out with a single line, such that the original entry is still legible. The line out must be dated and initialed by the employee correcting the mistake. With the exception of obvious typographical or handwritten errors, the reason for the correction or redaction of the entry is noted.

In cases where non-conforming data is submitted to a client anyway, the client must be notified, usually in the Case Narrative, as to the nature of the non-conformance and the reason(s) and/or opinions as to why the non-conformances could not be rectified. In addition, any opinions about the possible value of the data are included in the Case Narrative.

Other observations of samples, such as possible interfering peaks, mass changes as a result of filter defects, precipitation occurring during sample preparation which are out of the norm for a given method or any other observance which is not typical for a particular method must be noted in the raw data and/or the Case Narrative. Any opinions of the laboratory concerning data quality, integrity, accuracy or legal defensibility must be clearly documented, and must be noted to be the opinion of the laboratory. This documentation must be contained in the Case Narrative or conveyed to the client by some written means.

A.4 Ramifications of Unethical Behavior

All employees of ***CHESTER LabNet*** understand the ramifications of unethical behavior, and by their signatures on this document, attest to knowing the possible outcomes of such behavior. By their signatures on this document, the employees also attest that they are free from any undue pressures or influences which may adversely affect the quality of their work, and will avoid involvement in activities that would diminish confidence in their competency, impartiality, judgment or operational integrity.

A.5 Summary

CHESTER LabNet endeavors to foster an open and non-retaliative corporate atmosphere where all employees are not only encouraged, but also expected, to bring any data integrity issues to the notice of the appropriate personnel. All employees understand the need to produce the highest quality data possible. While management holds the ultimate responsibility for data integrity issues, it is the personal ethics of every employee that support the production of high quality data, and, thereby, the reputation of the laboratory.

Personal Ethics and Data Integrity Agreement

The following employees, by their signature, attest to having read, understood, and agreed to the most current version of the Personal Ethics and Data Integrity Policy for ***CHESTER LabNet***:

Name	Title/Responsibility	Signature	Date
Paul Duda	President Laboratory Director LIMS Administrator Client Services Technical Director		3/27/19
Sheri Heldstab	QA Officer Conventional Chemistry Technical Director Lead Analyst		2-21-19
Rick Sarver	XRF Technical Director Lead XRF Analyst		3.1.19
Lisa Ball	Project Manager Sample Custodian		3.4.19
Jennifer Schleis	Gravimetry Laboratory Technical Director XRF Analyst Lead Grav. Lab. Tech. Analyst		3.03.19
T. Mike May	Analyst Gravimetry Laboratory Technician		3.03.19
Julie Delarue	Analyst XRF Analyst Gravimetry Laboratory Technician		3.5.19
Theodore ("Ted") Perry	Analyst Gravimetry Laboratory Technician Chemical Hygiene Officer Health & Safety Officer		2.27.19
Kevin Healey	Gravimetry Laboratory Technician		3.4.19

Appendix B

CHESTER LabNet Standard Operating Procedures

SOP #	SOP Title
AD-001.06	Laboratory Safety and Security Procedures
AD-002.04	Waste and Sample Disposal
AD-003.05	Refrigerated Storage Monitoring
AD-004.04	Glassware Cleaning for Inorganics Laboratory
AD-005.06	Reagent Procurement and Control
AD-006.06	Laboratory Deionized Water Supply
AD-007.05	Laboratory Information Management System (LIMS)
AD-008.06	Sample Receipt and Log In
AD-009.02	Electronic Data Backup and Recovery
GR-001.08	8x10 Quartz & Glass Fiber Filter Inspection and Gravimetry
GR-001a.02	Punching of Exposed 8x10" Quartz or Glass Fiber Filters (renamed ME-008)
GR-002.06	80-125 mm Filter Inspection and Gravimetry **DEACTIVATED 7/14**
GR-003.02	Gravimetric Processing of 25-47mm Quartz Filters **DEACTIVATED 2/02**
GR-004.04	Chemical Impregnation of Cellulose Filters
GR-005.02	Impregnation of Cellulose Filters with Sodium Carbonate **DEACTIVATED 1/02**
GR-006.09	Filter Cassette Loading and Unloading
GR-007.02	Inspection & Preparation of 25-47mm Teflon Filters **MERGED W/GR-010, 5/03**
GR-008.04	Oil Coating of Teflon Filters
GR-009.01	Inspection & Preparation of 82.6-125mm Quartz & Glass Fiber Filters **DEACT 3/02**
GR-010.05	Teflon & Quartz Fiber Filter … Cahn I - **SUSPENDED 1/2013 **
GR-011.02	Inspection and Preparation of … Carbon Impregnated Filters **SUSPENDED 6/05**
GR-012.01	Inspection & Preparation of 102mm Teflon Filters **DEACTIVATED 3/02**
GR-013.02	Inspection & Preparation of 8x10" Pallflex Weave Filters **DEACTIVATED 1/02**
GR-014.01	Gross Weighing of 25-47mm Teflon Filters **DEACTIVATED 3/02**
GR-015.04	Quartz Filter Preparation for Carbon Analysis
GR-016.07	Preparation & Use of Control Charts for Gravimetric Analysis **DEACTVATED 7/18**
GR-017.03	Acceptance Testing of 47mm Teflon Filters, CFR 50 Part 50 App. L (Drop Test)
GR-018.03	Dickson Temperature and Humidity Data Logger
GR-019.04	Sartorius ME5 Microbalance: Teflon and Quartz fiber filter preparation and gravimetry.
GR-020.03	Use of Balance Run Logbooks and Filter Tracking Logbooks

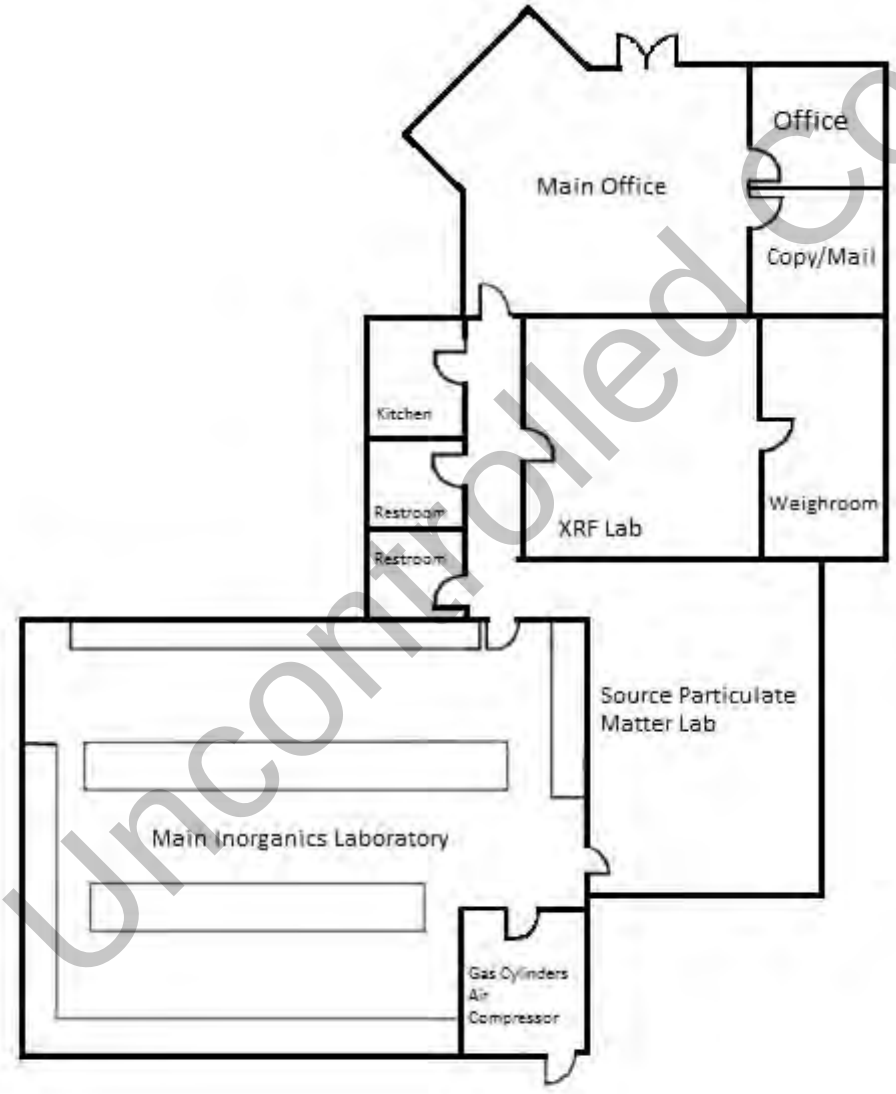
SOP #	SOP Title
IC-001.02	Borate Eluant Anions ** DEACTIVATED 4/2000 **
IC-002.02	Preparation of Air Filters for Fluoride Analysis ** DEACTIVATED 4/2000 **
IC-003.06	Extraction of Media for Ion Chromatographic Analysis
IC-004.02	Clean-Up of Anion Columns **DEACTIVATED 1/2002**
IC-005.04	Ion Chromatography: Anions **DEACTIVATED 4/2008**
IC-006.04	Ion Chromatography: Cations **DEACTIVATED 4/2008**
IC-007.02	Clean-Up of Cation Columns **DEACTIVATED 1/2002**
IC-008.04	Hexavalent Chromium by IC-PCD (Air filters) **DEACTIVATED 8/2011**
IC-009.02	Ion Chromatography: Anions & Cations **DEACTIVATED 12/2017**
IC-010.06	Hexavalent Chromium by IC-PCD (Thermo-Dionex ICS-1100 and Aquion instruments)
IC-011.03	Ion Chromatography: Anions & Cations ICS-5000
IC-012.01	Preparation & Extraction of Filters for Cr6+ by IC-PCD
ME-001.03	Analysis of Elements by ICP-AES (P40) **DEACTIVATED 3/2003**
ME-002.05	Analysis of Elements by Graphite Furnace **DEACTIVATED 10/2014**
ME-003.06	Sample Digestion for Analysis of Elements by ICP (EPA Method 3050)
ME-004.02	Analysis of Mercury in Aqueous Samples **DEACTIVATED 9/2010**
ME-005.02	Analysis of Mercury in Solid Samples **DEACTIVATED 9/2010**
ME-006.03	Analysis of Mercury in Hopcalite Sorbent Tubes **DEACTIVATED 9/2010**
ME-007.02	Analysis of Elements by ICP (Optima 2000) **DEACTIVATED 4/7/14**
ME-008.05	Subsectioning of Exposed 8x10" ... Filters Modified 40CFR50 Appendix G
ME-009.02	Analysis of Mercury in Aqueous and Solid Samples EPA 7470 & 7471 (Nippon 3320A)
ME-010.03	Analysis of Mercury by NIOSH 6009 (Nippon 3320A)
ME-011.02	Analysis of Elements by Inductively-Coupled Plasma Emission (Optima 8300)
ME-012.01	Digestion of Filters for Metals Analysis (Appendix G - Hot Sonication)
OC-001.06	Organic & Elemental Carbon by the Thermal-Optical Method (NIOSH 5040 & IMPROVE_A)
QA-001.06	Laboratory Training
QA-002.05	Laboratory Data and Report Validation
QA-003.06	Implementation, Distribution, & Control of Std. Operating Procedures
QA-004.04	Distribution and Control of Laboratory Logbooks
QA-005.02	Control of Laboratory QA/QC Records **DEACTIVATED 9/11/01**
QA-006.07	Determination of Detection Limits, Precision & Bias, & DoC
QA-007.05	Calibration of Variable Volumetric Dispensing Devices
QA-008.04	Assembly and Preparation of Data Reports (Original QA-008 merged with QA-002)
QA-009.03	Internal Auditing
QA-010.03	Laboratory Balance Calibration and Verification
QA-011.02	Control and Handling of Standards and Reference Materials

SOP #	SOP Title
QA-012.01	Manual Integrations of Chromatographs, Thermographs or Spectrographs
QA-013.01	Removal or Replacement of Calibration Points
ST-001.03	Halide & Hydrogen Halide Emissions from Stationary Sources (M26/26a)
ST-002.04	Particulate Emissions from Stationary Sources (M5-5F, ODEQ M5, "old" M202))
ST-003.05	Sulfur Dioxide Emissions from Stationary Sources (M6)
ST-004.04	Nitrogen Oxide (NOx) Emissions from Stationary Sources (M7D)
ST-005.03	Sulfuric Acid Mist & Sulfur Dioxide Emissions from Stationary Sources (M8)
ST-006.03	Elements by EPA Method 29 or CARB Method 436 (M29)
ST-007.01	Hydrogen Sulfide Content of Fuel Gas Streams **SUSPENDED 3/2006**
ST-008.02	Inorganic Lead Emissions from Stationary Sources (M12)
ST-009.03	Total Fluoride Emissions (From Source Samples) (M13B)
ST-010.04	Total Reduced Sulfur Emissions (M15/16) **DEACTIVATED 5/2015**
ST-011.02	Total Reduced Sulfur ** DEACTIVATED** 6/05 (merged w/ ST010 6/14/05)
ST-012.01	Sulfur Dioxide Emissions **DEACTIVATED** 8/2005
ST-013.04	Particulate & Gaseous Mercury (M101, 101A, 102)
ST-014.04	Beryllium Screening (M103)
ST-015.03	Beryllium Emissions from Stationary Sources (M104)
ST-016.01	Mercury in Sewage Sludge **SUSPENDED** 6/2005
ST-017.01	Particulate & Gaseous Arsenic Emissions **SUSPENDED** 6/2005
ST-018.03	Ammonia in Stationary Sources (CTM027 & ST-1B)
ST-019.06	Condensable Particulate Matter Method 202 & NCASI modification (M202)
ST-020.04	Determination of PM10 & PM2.5 from Stationary Sources (M201A)
ST-021.01	Use of BERT Proprietary Software for Particulate Matter Analysis
ST-022.01	Sample Preparation for Total Chromium (M306, SW846 0061, CARB 425)
WC-001.01	Chemical Oxygen Demand (COD) **DEACTIVATED** 9/11/09
WC-002.01	Specific Conductance **DEACTIVATED 9/11/09**
WC-003.02	Fluoride by Ion Selective Electrode **SUSPENDED 3/20/18**
WC-004.01	Ammonia-Nitrogen by Ion Selective Electrode **DEACTIVATED** 10/2005
WC-005.01	Gravimetric Oil & Grease in Liquids ** DEACTIVATED ** 4/2005
WC-006.01	Soil pH **SUSPENDED** 1/2005
WC-007.01	Gravimetric TPH. ** DEACTIVATED ** 4/2000
WC-008.01	Alkalinity. **SUSPENDED** 1/2005
WC-009.01	Cation Exchange Capacity. **SUSPENDED** 1/2005, DEACTIVATED 1/2019
WC-010.01	Redox Potential (eH). **DEACTIVATED**9/11/09
WC-011.01	Hardness. **SUSPENDED** 1/2005, DEACTIVATED 1/2019
WC-012.01	Nitrite-Nitrogen. **SUSPENDED** 1/2005, DEACTIVATED 1/2019
WC-013.01	Organic Matter. Walkley-Black Method **DEACTIVATED** 9/11/09
WC-014.005	pH in Aqueous Solutions. (Unsuspended 1/11/10)
WC-015.01	Phosphorous/Phosphate, All Species **SUSPENDED** 1/2005, DEACTIVATED 1/2019
WC-016.01	Total Dissolved Solids. **SUSPENDED** 1/2005, DEACTIVATED 1/2019

SOP #	SOP Title
WC-017.01	Total Kjeldahl Nitrogen. **DEACTIVATED** 10/2005
WC-018.01	Total Suspended Solids. **SUSPENDED** 1/2005, DEACTIVATED 1/2019
WC-019.01	Turbidity. **DEACTIVATED** 9/11/09
WC-020.01	Hexavalent Chromium. **SUSPENDED** 1/2005, DEACTIVATED 1/2019
WC-021.03	Alkalinity in Teflon Filters
WC-022.01	Dustfall - Suspended and Dissolved Particulate Matter (ASTM D1739-98)
XR-001.03	Resuspension of Particulate Matter onto Filter Media
XR-002.07	Analysis of Elements in Air Particulates by X-Ray Fluorescence (Kevex 770)
XR-003.03	Preparation of Samples for Resuspension
XR-004.04	Kevex XRF Spectrometer Calibration
XR-005.03	Kevex Spectrometer Data Generation, Interpretation and Reporting
XR-006.02	X-Ray Fluorescence (Kevex-771) **DEACTIVATED 1/2009**
XR-007.03	Analysis of Elements in Air Particulates by X-Ray Fluorescence (Thermo ARL QUANT'X)

Appendix C

Laboratory Floor Plan



Appendix D

CHESTER LabNet Most Commonly Utilized* Methods (by Issuing Authority and Method Number)

*Note: The laboratory has the capability to perform many other methods. This listing contains the laboratory's most commonly performed methods.

NELAC Accredited Method(s)

Method Number	Analyte/Element	Instrumentation
CARB MLD 039	Hexavalent Chromium (ambient air)	IC-PCD
ASTM D7614-12	Hexavalent Chromium (ambient air)	IC-PCD
40 CFR 60 Method 202	Condensable Particulate Matter (source emissions)	Balance (gravimetry)
40 CFR 50 Appendix J	PM_{10} (ambient air)	Balance (gravimetry)
40 CFR 50 Appendix L	$PM_{2.5}$ (ambient air)	Balance (gravimetry)
NIOSH 5040	Diesel PM (elemental Carbon)	OC/EC
DRI SOP#2-216r2 (IMPROVE_A)	Organic/Elemental Carbon	OC/EC
40 CFR 60 Method 26A	HF, HCl, HBr, Cl2, Br2	IC

US EPA IO Methods

Method Number	Analyte/Element	Instrumentation
3.1	Gravimetry and metals prep.	Balance and wet chemical
3.3	metals	XRF
3.4	metals	ICP
4.2	Anions and cations	IC

EPA Water/Wastewater Methods (by reference from other methods)

Method Number	Analyte/Element	Instrumentation
EPA 300.0	Anions	IC
ASTM Method D6919-03	Cations	IC
EPA 340.2	F	Ion Selective Electrode

EPA SW-846 Methods (by reference from other methods)

Method Number	Analyte/Element	Instrumentation
3050	metals prep.	wet chemical
6010	metals	ICP-OES
7470/7471	Hg	CVAA
0061	Hexavalent & Total Cr	IC-PCD and ICP

ODEQ Methods

Method Number	Analyte/Element	Instrumentation
5	Particulates	Balance (gravimetry)

40 CFR 50, 51 & 60 Promulgated Source Testing Methods

Method Number	Analyte/Element	Instrumentation
5	Particulates	Balance (gravimetry)
6	SO_2	titrimetric
7 (A & D)	NO_x	IC
8	H_2SO_4/SO_2	titrimetric
12	Pb	ICP
13B	F	IC or ISE
26/26A	H_X, H_X & X_2	IC
29	Multi-metals (inc. Hg)	ICP (& CVAA if Hg requested)
101A	Hg	CVAA
201A	Particulates	Balance (gravimetry)
202	Particulates	Balance (gravimetry)
306	Hexavalent and Total Cr	IC-PCD and ICP
CTM 027*	NH_4	IC
CTM 013 & 013A*	H2SO4/SO2	IC or titration

*CTM: Conditional Test Method

NIOSH Methods

Method Number	Analyte/Element	Instrumentation
5040	Elemental Carbon	OC/EC
6009 (revoked)	Hg	CVAA
7907	Volatile Acids (Anions)	IC
7908	Non-volatile Acids (Anions)	IC

CARB Methods

Method Number	Analyte/Element	Instrumentation
421	HF & HCl	IC
425	Hexavalent and Total Cr	IC-PCD and ICP-OES
436	multiple metals	ICP (& CVAA if Hg requested)
SOP MLD039	Hexavalent Cr (ambient air)	IC-PCD

40 CFR 50, Ambient Air Methods

Method Number	Analyte/Element	Instrumentation
Appendix B	TSP	Balance (gravimetry)
Appendix G	Metals prep (hot sonication)	wet chemical
Appendix J	PM_{10}	Balance (gravimetry)
Appendix L	$PM_{2.5}$	Balance (gravimetry)
Appendix Q	Pb	XRF

ASTM Ambient Air Methods

Method Number	Analyte/Element	Instrumentation
ASTM D7614-12	Hexavalent Chromium	IC-PCD
ASTM 1739-98	Dustfall	Balance (gravimetry)

Appendix E

Laboratory Accreditation / Certification / Recognition

CHESTER LabNet maintains the following certifications and accreditations:

Organization	Certificate Number	Parameter List
ORELAP	OR100051-010	PM_{10}; $PM_{2.5}$; CARB SOP MLD039 - CrVI (ambient air); ASTM D7614-12 - CrVI (ambient air); Carbon (Improve_A); Carbon (NIOSH 5040); Condensable PM (Method 202); Hydrogen Halides and Halides (Method 26A).

The certificates and parameter lists (which may differ) for each organization is located in files maintained by the Laboratory Director.

If accreditation is terminated or suspended, the laboratory will immediately cease to use the certificate number reference in any way and inform clients impacted by the change.

Appendix F

Data Qualifiers

List of CHESTER LabNet Data Qualifiers		
Location	Qualifier	Meaning
Standard Reports		
Data Summary (on request)	U	Non-detect
Data Summary	B	Greater than DL, less than LoQ
QC Summary	N/C	Duplicate RPD can't be calculated as one or both data points are less than the DL
QC Summary	#	Duplicate RPD control limits do not apply as one or both of the data points is less than the LoQ
QC Summary	*	Spike recovery limits do not apply where spiked amount is less than ¼ sample result.
Special "CLP-like" Reports		
Data Summary (Form I)	J	Concentration greater than DL but less than contract required detection limit
Data Summary (Form I)	U	Concentration less than DL
Data Summary (Form I)	E	Concentration is estimated based upon interferents
Data Summary (Form I)	N	Spike recovery not in control
Data Summary (Form I)	*	Duplicate analysis not in control
Data Summary (Form I)	D	Reported value is from a dilution
Data Summary (Form I)	P	Analyzed by ICP-AES
Data Summary (Form I)	MS	Analyzed by ICP-MS
Data Summary (Form I)	CV	Analyzed by CVAAS
Data Summary (Form I)	C	Analyzed by manual spectrophotometric equipment
Data Summary (Form I)	" "	Where no data has been entered
Data Summary (Form I)	NR	Analyte is not required
QC Summary (Form II – ICV/CCV)	M	Analyzed for analyte
QC Summary (Form II – ICV/CCV)	NR	Analyte is not required
QC Summary (Form II – CRI/CRA)	J	Concentration greater than DL but less than contract required detection limit
QC Summary (Form II – CRI/CRA)	U	Concentration less than DL
QC Summary (Form III – blanks)	J	Concentration greater than DL but

List of CHESTER LabNet Data Qualifiers		
Location	Qualifier	Meaning
		less than contract required detection limit
QC Summary (Form III – blanks)	U	Concentration less than DL
QC Summary (Form III – blanks)	P	Analyzed by ICP-AES
QC Summary (Form III – blanks)	MS	Analyzed by ICP-MS
QC Summary (Form III – blanks)	CV	Analyzed by CVAAS
QC Summary (Form III – blanks)	C	Analyzed by manual spectrophotometric equipment
QC Summary (Form V – spikes)	N	Spike amount is less than ¼ sample result
QC Summary (Form V – spikes)	P	Analyzed by ICP-AES
QC Summary (Form V – spikes)	MS	Analyzed by ICP-MS
QC Summary (Form V – spikes)	CV	Analyzed by CVAAS
QC Summary (Form V – spikes)	C	Analyzed by manual spectrophotometric equipment
QC Summary (Form V – spikes)	NR	Analyte is not required
QC Summary (Form V – post-spikes)	P	Analyzed by ICP-AES
QC Summary (Form V – post	MS	Analyzed by ICP-MS
QC Summary (Form V – post	CV	Analyzed by CVAAS
QC Summary (Form V – post	C	Analyzed by manual spectrophotometric equipment
QC Summary (Form V – post	NR	Analyte is not required
QC Summary (Form VI – duplicates)	*	Both sample results are greater than the LoQ, AND the RPD is out of control
QC Summary (Form VI – duplicates)	P	Analyzed by ICP-AES
QC Summary (Form VI – duplicates)	MS	Analyzed by ICP-MS
QC Summary (Form VI – duplicates)	CV	Analyzed by CVAAS
QC Summary (Form VI – duplicates)	C	Analyzed by manual spectrophotometric equipment
QC Summary (Form VII – LCS)	J	Concentration greater than DL but less than contract required detection limit
QC Summary (Form VII – LCS)	U	Concentration less than DL
QC Summary (Form VIII – Serial Dilutions)	E	RPD greater than 10% and original sample concentration is greater than 50 times the DL.
QC Summary (Form VIII – Serial Dilutions)	P	Analyzed by ICP-AES
QC Summary (Form VIII – Serial Dilutions)	MS	Analyzed by ICP-MS
QC Summary (Form VIII – Serial Dilutions)	CV	Analyzed by CVAAS
QC Summary (Form VIII – Serial Dilutions)	C	Analyzed by manual spectrophotometric equipment

Note: many of the larger projects will have their own reporting guidelines, Contract Required Quantitation Limits, Action Limits and Data Qualifier Codes. The laboratory will use the guidelines issued by the client in all regards when reporting data, to include reported Quantitation limits and Qualifier Codes.

List of Common Sample Observations	
Comment	Meaning
Filter Gravimetry Laboratory	
Loose deposit/PM	Material in deposit not adhering to filter
GPM (gross particulate matter)	Insect, plastic, other non-suspended particulate present that does not resemble the rest of the deposit.
Tear in filter	Tear in the filter that may have occurred before, during or after the sampling event.
Scratch on deposit	Filter is intact, but deposit is marred by something sharp, leaving a small scratch. Loss of deposit possible.
Deposit on back of filter	Filter loaded into sampler backwards
[insect/object] on filter (removed or not?)	Notes presence of non-deposit object, notes whether object was removed prior to weighing.
PM on/in cassette	Particulate in or on the cassette in addition to being on the filter. May indicate loss of deposit.
PM on support ring	Particulate in or on the support ring in addition to being on the filter. May indicate loss of deposit.
Scrape on deposit	Filter is intact, but deposit is marred by something blunt, leaving a scraped area on the deposit. Loss of deposit possible.
Hole in membrane	Hole in the filter that may have occurred before, during or after the sampling event.
Corner torn/tattered	Filter corner torn, abraded. Possible loss of filter material during sampling.
Edge torn/tattered	Filter edge torn, abraded. Possible loss of filter material during sampling.

List of Common Sample Observations	
Comment	Meaning
Non-uniform deposit	Deposit is not uniform across deposit area. Could mean sampler was operating incorrectly, sample was composed of coarse particulate that would not adhere to filter well, too much particulate was loaded on the filter, or any number of issues. Loss of deposit is possible.
Desiccated	Filters arrived wet or damp and were desiccated to dryness before equilibrating in weighroom.
Indentation	Dent in filter. May show mishandling of filter.
Received wet	Filter received damp or wet. Deliquescence possible.
PM in folder/glassine	Particulate present inside filter container, indicating loss of deposit in container.
Unloading error	May mean filter was dropped, scratched or otherwise damaged during removal from cassette. Possible loss or gain of mass as a result.
Deposit in margin	Deposit in margins of filter where it should have been covered by the cassette or filter holder. Indicates misloading of filter.
Filter punching/preparation for digestion/extraction	
loose deposit	Particulate matter shifts on surface of filter
heavy deposit	Heavy loading of particulate on filter
non-homogeneous deposit	Part of deposit is visibly different than other areas of the deposit
deposit off filter edge	Deposit area is off the edge of the filter, indicating incorrect loading into sampler. Probably indicates loss of capture efficiency due to gas flowing around the filter rather than through it.
skewed deposit	Deposit not square on filter. Filter margins vary from one side of the filter to the other. Probably indicates incorrect loading into

List of Common Sample Observations	
Comment	Meaning
	sampler.
fold skewed	Filter folded at an angle, causing deposit to rub off on the margins of the filter.
spots w/appearance of water droplets on deposit	Part(s) of deposit look like drops of a liquid fell onto it (may be rain water).
deposit on back of filter	Filter loaded into sampler backwards. May or may not affect capture efficiency.
[identifiable thing] on deposit	Object on filter that is visibly and obviously not part of the air shed suspended particulate (e.g., moths, boot prints, mud, etc.)
filter not folded	Deposit exposed to filter container in such a manner that the deposit may have transferred to the container, possibly decreasing the amount of particulate remaining on the filter.
filter in [inappropriate container]	Filter arrived in a container that may affect the results, such as filters being wrapped in Aluminum foil prior to being tested for aluminum.
Source Particulate Matter (M5/M202)	
particles with the appearance of glass shards/glass dust in sx deposit	Glass shards or dust present in sample, adding mass to the sample. Probably from loading/unloading the sampling train.
[object] in sx	Object that is visibly and obviously not part of the condensable particulate present in sample. This could be pieces of O-Ring, fluff from clothing, dead insects, etc. Presence will bias results high.
edge(s) of filter(s) frayed	Filter edge torn, abraded. Possible loss of filter material during sampling.
thick deposit falling off filter/deposit inside petri dish	Loss of deposit from filter, which will bias results low.
pieces of filter missing	Piece of filter visibly missing and not included in the filter container at the time of receipt by the laboratory.
deposit on back of filter	

List of Common Sample Observations	
Comment	Meaning
	Filter loaded into filter assembly backwards. May or may not affect capture efficiency.
IC analysis of impinger solutions	
doublet/triplet/quadruplet [analyte] peak in spiked sx	Some species of target analyte form complexes in solution that do not reach equilibration. The most commonly encountered is Fluorosilicates and Phosphofluorosilicates, which can cause triplet or quadruplet peaks to form, all of which are integrated as a single peak.
interfering peak riding on/merged with [analyte] peak	A non-target analyte is riding on, merged with or interfering with the ability of the software to accurately determine a baseline. The Analyst will note how the sample or chromatogram was modified to attempt to resolve the interference.

Appendix G

Employee Resumés

Paul D. Duda

President, Laboratory Director, Lead Project Manager, LIMS Administrator

Background:

Hire date: 1989. Experience in air quality filter analysis by X-Ray Fluorescence; experience as Project Manager; experience with SAS and CLP data package requirements. Experience as Laboratory Information Management System (LIMS) administrator, coordinating all LIMS activities; special expertise in interfacing laboratory data to client-specific databases and end-user data programs.

Career Chronology:

Employment Information	Responsibilities and Duties
President; Laboratory Director; Lead Project Manager; LIMS Administrator; ***CHESTER LabNet*, Tigard, OR** **2001 – Present**	Corporate affairs for laboratory, including proposal writing, marketing and sales, program and Project Management, overall profit/loss for company, all accounting/payroll and purchasing; report production for all projects requiring EPA, CLP deliverables. Oversee all procedures, QA/QC and corrective actions associated with sample receipt, log-in, chain-of-custody and storage; Project Management, and general and specialized report production; client management; oversees operation and maintenance of laboratory information management system (LIMS), including all software and hardware, general data entry, QA/QC, coordination with other Project Managers and technical staff, training of new users.
Project Manager, LIMS Administrator, Sample Custodian, ***CHESTER LabNet*, Tigard, OR** **1992 - 2001.**	Project management, all accounting/payroll and purchasing; report production for all projects requiring EPA, CLP deliverables. Oversees all procedures, QA/QC and corrective actions associated with sample receipt, log-in, chain-of-custody and storage; Project Management, and general and specialized report production; client management; oversees operation of laboratory information management system (LIMS), including all software and hardware, general data entry, QA/QC, coordination with other Project Managers and technical staff, training of new users.
Gravimetry Laboratory and XRF Analyst, ***CHESTER LabNet*, Tigard, OR** **1989 - 1992.**	Performed all operations of the filter gravimetry laboratory, including maintaining supplies, filter media acceptance testing, gravimetric analysis of filter media following EPA protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation. Also served as XRF technician, including preparation of samples for analysis, instrument operation, interpretation of spectral results, QA/QC.
1987 - 1988	Miscellaneous employment.

Education:

- Graduate Studies, Business Administration, Portland State University, Portland, OR, 1991-1992.
- B.S., Engineering Management, University of Portland, Portland, OR, 1987.

Sheri Heldstab
QA Officer, Conventional Chemistry Laboratory Technical Director, Lead Analyst

Background:
Hire date: 1992. Experience in inorganic analysis of environmental samples, method development for unusual sample matrices, and data interpretation and validation; experience with SAS and CLP data package preparation and requirements; experience in technical writing of Standard Operating Procedures, QA/QC project plans; experience with TNI/ORELAP accreditation requirements and documentation.

Career Chronology:

Employment Information	Responsibilities and Duties
QA/QC Officer, Conventional Chemistry Technical Director, Lead Analyst, H&SO and CHO (until 2019) ***CHESTER LabNet*,** **Tigard, OR** **1999 – Present** Member: TNI (2017) Vice Chair TNI SSAS committee (2019) Voting member: ASTM D22 (2018)	Oversee all operations of the conventional chemistry laboratory; analyze samples; ensure data meets QA/QC requirements; general technical guidance for clients and staff; manage the flow of samples and data through the laboratory; oversee and train other Analysts; oversee day to day operation of the laboratory; ensure meeting of due dates, proper maintenance of instruments, and adherence to all QA/QC protocols. Oversight of standard operating procedure and program document production and implementation, oversight of accreditation specific requirements, QC review of data reporting, technical guidance on general QA issues, report production for all projects requiring EPA CLP deliverables.
Account Manager, Lab Support, Portland, OR 1998 - 1999	Performed all duties required to run a one person branch office, including service calls, resolution of client disputes, marketing to new clients, filling of orders and recordkeeping.
Chemist, ChemTrace, Portland, OR 1997 – 1998	Primary operator for IC and GFAA. Performed analysis on high purity water for various nutrients, microbiological testing and silica content.
Lead Analyst, ***CHESTER LabNet*, Tigard, OR** **1994 – 1997**	Primary operator for IC, ICP, GFAA, CVAA. Analyzed variety of air quality samples using primarily CFR methods. Supervised one Analyst. Generated CLP QC reports. Managed sample throughput and Level I data validation of laboratory.
Associate Chemist, ***CHESTER LabNet*, Tigard, OR** **1992 – 1994**	Primary operator for IC and performance of bench methods. Analyzed variety of environmental samples using CFR, SW846, DW, SM, NIOSH, OSHA, and a variety of other methods.
Laboratory Technician, ASiMI, Washougal, WA 1991 – 1992	Analyzed high purity raw silicon for contaminants utilizing specialized equipment. Generated QC reports to be used in the preparation of Certificates of Lot Analysis.
Chemist, Coffey Laboratories, Portland, OR 1990 – 1991	Analyzed a variety of environmental samples for inorganic constituents using DW, SW846 and SM methods.

Education:
- B.S., Biology (Chemistry minor), University of Oregon, 1989
- Secondary Teaching Certification, University of Oregon School of Education, 1990

Richard H. Sarver
XRF Technical Director, Lead XRF Analyst

Background:

Hire date: 1986. Experience in analytical chemistry, including biochemical applications and environmental air quality analysis, specializing in the analysis of air particulates by x-ray fluorescence.

Career Chronology:

Employment Information	Responsibilities and Duties
XRF Technical Director, Lead XRF Analyst, ***CHESTER LabNet*,** **Tigard, OR** **1986 - Present.**	Coordinate all XRF activities with Project Managers as needed; train XRF technicians and oversee all XRF operations; market XRF capabilities to outside clients; responsible for maintenance and repair of instruments, supervise sample flow, data interpretation, QA/QC and report generation from XRF analysis; perform highly specialized sample preparation for non-deposit samples, including size fraction and resuspension; technical guidance for clients and in-house staff. Perform XRF analysis and provide technical assistance for state and federal agencies, industrial, consulting and university clients. Ensure adherence to all QA/QC protocols. Awarded EPA equivalency method EQL-0589-072, "Determination of Lead Concentration in Ambient Particulate Matter by EDXRF Spectrometry in May 1989. Principal scientist for the XRF analysis of air particulates for the U.S. EPA national PM2.5 Chemical Speciation Program. Developed XRF method of analysis for the Hazardous Element Sampling Train (HEST), which used activated carbon to trap volatile metals. Continues to participate in the ongoing effort to obtain equivalent method status to EPA Method 29.
Analytical Chemist, Pioneer Hi-Bred International Portland, OR 1980-1986.	Utilized FID/GC analysis to determine metabolic pathways of resident microorganisms in the digestive tract of stressed mice. Handled animals and performed analytical work. Developed SOPs for in house use.

Education:

A.A.S., Chemical Technology, Chemeketa Community College, Salem, OR 1980.

Selected Publications and Presentations:

Sarver, R. H. 1996. Aerosolization as a Means of Sample Preparation of Geological Materials for XRF Analysis and its Validity Compared to EPA Method 3050A Digestion. Journal of the Air & Waste Management Association. 46: 234-240.

Sarver, R.H. and Lytle, C.R. 2000. Parameter optimization for the analysis of PM2.5 by energy dispersive x-ray fluorescence (EDXRF). Presented at PM2000: Particulate Matter and Health, Air & Waste Management Association Specialty Conference, Charleston, SC, January 24-28, 2000.

Sarver, R.H., Mace, J.C. and Duda, P.D. 2002. XRF: Inter-Excitation Quality Assurance and Deposit Uniformity. Presented at Symposium on Air Quality Measurement Methods & Technology, Air & Waste Management Association Conference, San Francisco, CA, November 13–15, 2002.

Jennifer Schleis

Gravimetry Laboratory Technical Director, Lead Gravimetry Laboratory Technician, XRF Analyst, Analyst

Background:

Hire date: 2007. General laboratory experience. Prior experience in NELAC accredited laboratory. Experience in filter weighing using CFR methods. Instrument experience including IC-PCR, ICP, IC, OC/EC, CVAA. Experience with inorganic analytical methods including CFR, NIOSH, and OSHA methods.

Career Chronology:

Employment Information	Responsibilities and Duties
Gravimetry Laboratory Technical Director; XRF Analyst Lead Gravimetry Laboratory Technician; Analyst (inactived 2019); ***CHESTER LabNet*,** **Tigard, OR** **2010 - Present**	Oversees and performs all operations of the filter gravimetry laboratory to include acceptance testing, QA/QC, inventory and archives. Ensures data meets QA/QC requirements; provides general technical guidance for clients and staff; manages the flow of samples and data through the laboratory; oversees and trains other technicians; oversees day to day operation of the laboratory; manages flow of samples and data through the laboratory; ensures meeting of due dates, proper maintenance of instruments and adherence to all QA/QC protocols. Performs XRF analyses, ensures adherence to all QA/QC protocols. Performs other analytical duties in the Conventional Chemistry lab (as needed).
Analyst; Gravimetry Laboratory Technician ***CHESTER LabNet*,** **Tigard, OR** **2007 - 2010**	Analyzed a variety of air quality samples using primarily CFR methods, performed level I data review in real time. Analyzed samples for metal constituents by ICP, CVAA; analyzed samples by IC, IC-PCD and wet chemical methods. Performed all operations of the filter gravimetry laboratory, including filter media acceptance testing, gravimetric analysis of filter media following CFR protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation.
Laboratory Technician Aquatic Biotech & Environmental Lab, Athens, GA 2005 – 2006	Analyzed mutant strains of transgenic fish via specialized assays, gel electrophoresis, PCR, sequencing, DNA isolation; performed in-vitro fertilization and dissections; maintained aquaria and stocks of fresh and saltwater fish.
Laboratory Technician The University of Georgia, Athens, GA 2004 – 2005	Performed high-throughput DNA sequencing; maintained and operated all laboratory equipment; provided weekly presentation of data summaries and quality assurance; provided technical assistance to students and research staff.

Education:

B.S. Environmental Economics and Management, The University of Georgia, 2003
Continuing Education (Chemistry), Portland Community College & Portland State University, 2015 - 2016

Lisa Ball
Project Manager, Sample Custodian

Background:

Hire date: 1997. Experience as Project Manager. Experience as environmental Analyst performing wet chemical analysis.

Career Chronology:

Employment Information	Responsibilities and Duties
Project Manager, Sample Custodian, ***CHESTER LabNet*,** **Tigard, OR** **2003 - Present.**	Project management. Performs all procedures, QA/QC and corrective actions associated with sample receipt, log-in and chain-of-custody, Project Management and general and specialized report production; client management; general data entry; coordination with other Project Managers and technical staff; training of new users. Provides technical guidance to clients.
Project Manager, Sample Custodian, Gravimetry Laboratory Coordinator, ***CHESTER LabNet*,** **Tigard, OR** **2001 - 2003.**	Project management. Performs all procedures, QA/QC and corrective actions associated with sample receipt, log-in and chain-of-custody, Project Management and general and specialized report production; client management; general data entry; coordination with other Project Managers and technical staff; training of new users. Provides technical guidance to clients. Oversee and perform all operations of the filter gravimetry laboratory.
Analyst, Gravimetry Laboratory Coordinator, ***CHESTER LabNet*,** **Tigard, OR** **1997 - 2001.**	Performed all operations of the filter gravimetry laboratory, including maintaining supplies, filter media acceptance testing, gravimetric analysis of filter media following EPA protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation. Analyzed air quality samples using primarily CFR methods, including: sample preparation, and digestion and analysis of samples. Principal Operator of IC, ICP, CVAA. Responsible for Level I data review and reporting.
Extraction Chemist, Oregon Analytical Laboratory, Beaverton, OR, 1997 (full-time, temporary).	Performed extractions for total petroleum hydrocarbons (TPH and TPHD), hydrocarbon identification (HCID), PAHs and oil and grease. Analyzed water and soil samples for a variety of inorganic constituents including: CODs, pHs, alkalinity, flashpoints, TKN analysis, and Cyanide analysis.
Chemist, American Environmental Network, Durham, OR, 1996-1997.	Primary wet chemist. Brought new wet chemistry methods on line and wrote corresponding SOPs for wet chemistry methods.

Education:

B.S., Integrated Science, Portland State University, 1996. OSHA 1910.120: 24-hour, 1996.

T. Mike May
Analyst, Gravimetry Laboratory Technician

Background:

Hire date: 2009 – 2011 and 2017. Prior experience as an analytical chemist for production facility.

Career Chronology:

Employment Information	Responsibilities and Duties
Analyst Gravimetry Laboratory Technician ***CHESTER LabNet,*** **Tigard, OR** **2017 - Present**	Analyzes a variety of air quality samples using conventional chemistry techniques utilizing primarily CFR methods; performs instrumental analysis using IC, IC-PCD, OC/EC, ICP and CVAA; performs level I data review in real time. Performs all operations of the filter gravimetry laboratory, including filter media acceptance testing, gravimetric analysis of filter media following CFR protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation.
Sales/Applications Engineer MCAM NW Oregon City, OR 2011 – 2017	Acted as primary sales and support for Quality programs; implemented and improved overall clients' shop Quality; trained client employees in gathering and reporting reliable quality data; supported clients in troubleshooting and expanding operation to new projects.
Analyst Gravimetry Laboratory Technician ***CHESTER LabNet,*** **Tigard, OR** **2008 - 2011**	Analyzed a variety of air quality samples using primarily CFR methods; performed instrumental analysis using ICP, IC, IC-PCD, OC/EC and CVAA; performed level I data review in real time. Performed all operations of the filter gravimetry laboratory, including filter media acceptance testing, gravimetric analysis of filter media following CFR protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation.
Chemist Bodycote Materials Testing, Portland, OR 2006 - 2008	Analyzed samples by ICP-MS, GFAA XRF, IR and Thermal Conductivity gas analyzers, and ICP-OES; developed and wrote procedure for digestion and analysis of trace metals; trained new employees; analyzed chemical composition of various metals and coatings,

Education:

B.S., Biology, Portland State University, 2005

Julie Delarue

Analyst, Gravimetry Laboratory Technician, XRF Analyst

Background:

Hire date: 2015. Prior experience as an analytical chemist for production facility. General laboratory experience, instrumental experience including ICP, UV/Vis and IR spectrophotometer, GC, and XRF.

Career Chronology:

Employment Information	Responsibilities and Duties
Analyst Gravimetry Laboratory Technician ***CHESTER LabNet,*** **Tigard, OR** **2015 - Present**	Analyzes a variety of air quality samples using conventional chemistry techniques utilizing primarily CFR methods; performs instrumental analysis using IC, IC-PCD, OC/EC, ICP and CVAA; performs level I data review in real time. Performs all operations of the filter gravimetry laboratory, including filter media acceptance testing, gravimetric analysis of filter media following CFR protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation.
Laboratory Technologist, Husky Energy, Lloydminster, SK (CAN) 2010 – 2014	Performed troubleshooting and method development; analyzed solid, liquid and gaseous samples by GC and XRF; prepared diesel fuel certification for sale. Responsible for all analytical documentation.
QA Laboratory Technologist, Nestle Purina Petcare, Innisfail, AB (CAN) 2009 - 2010	Performed Protein, Moisture and Fat analyses on pet food; responsible for formal release of product. Responsible for all analytical documentation.
Analytical Laboratory Technologist, Nova Chemicals, Calgary, AB (CAN) 2008 - 2009	Analyzed solid, liquid and gas samples by GC; performed troubleshooting and method development. Responsible for all analytical documentation.

Education:

- B.S., Environmental Sciences, University of Alberta (CAN), 1999
- A.S., Chemical Technology, SAIT Polytechnic (CAN), 2009

Theodore ("Ted") Perry

Analyst, Gravimetry Laboratory Technician, Chemical Hygiene Officer, Health and Safety Officer

Background: Hire date: 2015. Prior experience as an analytical chemist.

Career Chronology:

Employment Information	Responsibilities and Duties
Analyst Gravimetry Laboratory Technician Chemical Hygiene Officer Health & Safety Officer ***CHESTER LabNet,*** **Tigard, OR** **2019 - Present**	Analyzes a variety of air quality samples using conventional chemistry techniques utilizing primarily CFR methods; performs instrumental analysis using IC, IC-PCD, OC/EC, ICP and CVAA; performs level I data review in real time. Performs all operations of the filter gravimetry laboratory. Ensures safe and proper storage and handling of Chemicals. Ensures safety and laboratory hygiene protocols are followed.
Analyst Gravimetry Laboratory Technician ***CHESTER LabNet,*** **Tigard, OR** **2015 - 2018**	Analyzes a variety of air quality samples using conventional chemistry techniques utilizing primarily CFR methods; performs instrumental analysis using IC, IC-PCD, OC/EC, ICP and CVAA; performs level I data review in real time. Performs all operations of the filter gravimetry laboratory.
Chemist, Thornton Laboratory, Tampa, FL 2013 - 2015	Prepared and analyzed fertilizer samples for Nitrogen content, including TKN, Ammonium content, insoluble nitrogen, Nitrate and slow release nitrogen. Responsible for all analytical documentation.
Chemist Mission Mountain Labs Arlee, MT 2012 – 2013	Prepared and analyzed primarily nutraceutical samples for metals analysis by GFAA, FAA and FTIR. Responsible for all analytical documentation.
Work Study Oregon State University Corvallis, OR 2011 -2012	Moved surplus supplies and equipment from buildings or storage into other buildings or storage.
2008 - 2010	Miscellaneous employment.

Education:

B.S., Chemistry (Environmental Chemistry option), Oregon State University, 2012

Kevin Healey
Gravimetry Laboratory Technician

Background:

Hire date: 2019. Prior experience in Plant Tissue Culturing, Field Biodiversity studies, Agricultural field studies.

Career Chronology:

Employment Information	Responsibilities and Duties
Analyst Gravimetry Laboratory Technician ***CHESTER LabNet,*** **Tigard, OR** **2019 – Present**	Performs all operations of the filter gravimetry laboratory, including filter media acceptance testing, gravimetric analysis of filter media following CFR protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation.
Technician, Phytelligence, Tigard, OR 2018 - 2019	Performed *in vitro* propagation of apple species, trained new employees in aseptic laboratory technique, trained new employees in lab support duties.
Research Assistant Colorado State University Pueblo, CO 2017 - 2018	Developed protocol for field plant biodiversity survey; identified plants and grasses; pressed and cataloged field specimens; supervised and trained undergraduate field research assistants.
Tissue Culture Technician CSS Farms Colorado City, CO 2016 (summer)	Performed *in vitro* micropropagation of *Solanum tubersum*; sterilized equipment; identified in vitro pathogens; made Murashige and Skoog gel media; disposed of genetically modified plant materials.
Research Assistant CSS Farms Pueblo, CO 2016	Performed confidential experiment with genetically modified potato tubers; managed greenhouse irrigation system; created weekly reports.

Education:

B.S., Biology (Chemistry Minor), Colorado State University, 2018.
A.A., English, Pueblo Community College, 2015.

Appendix H

Chemistry

H.1 Method Validation

Reference methods are validated using the following criteria where possible:

- DL study;
- Precision and Bias study; and
- Evaluation of Selectivity.

H.1.1 a) Detection limit (DL)

The Detection limit (DL) is the laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can detect with 99% confidence that the result is not a false positive. It does not grant any confidence that the result is not a false negative. None of the reference methods performed at ***CHESTER LabNet*** require DL studies.

DLs are not required for any component for which spiking solutions or quality control samples are not available. These include XRF analysis, gravimetric analysis (both Gravimetry Laboratory and Conventional Chemistry), and OC/EC analysis.

The 2009 and 2016 TNI Standard states, *"the DL determination shall incorporate the entire analytical process;"* and "*the DL shall be determined for the analytes of interest in each test method in the quality system matrix of interest in which there are neither target analytes nor interferences at a concentration that would impact the results, or the DL shall be performed in the sample matrix of interest.*"

The quality system matrix is "Air", in the case of all work performed at ***CHESTER LabNet***. Due to the fact that sampling is part of the reference method, it is not possible for the laboratory to perform a DL study incorporating *"the entire analytical process."* Also, due to the need for sampling media, the large variety of types of media, the large variation between manufacturer's lots of the same type and size of media, it is not possible to find a sampling matrix "*in which there are neither target analytes nor interferences at a concentration that would impact the results.*" The laboratory determines the DL for each reference method where a DL is possible to be determined, using a matrix free from the target analytes of interest or interfering analytes that impact the DL (e.g. reagent water). Sampling media, to include filters, sorbent tubes, passive sampling materials, and impinger solutions, are not included in the detection limit study, as including said media would determine the variability of the media provided by vendors, rather than the laboratory's detection limit.

The 2009 and 2016 TNI Standard states, *"If a mandated test method or applicable regulation includes protocols for determining detection limits, they shall be followed. The laboratory shall document the procedure used for determining the DL."*

No method performed at ***CHESTER LabNet*** requires a DL study. DL studies or DL verifications are performed annually for each method/analyte combination for which samples have been analyzed in the previous year. The procedure can be found in SOP QA-006. The DL/LoQ procedures utilized at the laboratory are based on the EPA method "*Definition and Procedure for the Determination of the Method Detection Limit, Revision 2"* (EPA 821-R-16-006, December 2016).

H.1.1 b) Initial Detection Limit Study:

Briefly, the initial Detection Limit Study is performed as follows: Three sets of three method blanks and three low level standards with a concentration of three-times the estimated Detection Limit are prepared on three separate days and analyzed on three separate days. All preparatory and analytical steps of the method are followed. For methods in which no preparation exists, the requirement to prepare three standards shall hold, however, standards may be prepared on the same day as analysis.

Example Timeline for Initial DL study		
	Prepare:	Analyze:
Day 1	Set 1	N/A
Day 2	Set 2	Set 1
Day 3	Set 3	Set 2
Day 4	N/A	Set 3

(where a "Set" consists of three Method Blanks and three low level standards at a concentration approximately three times the estimated DL)

The DL study is performed on all instruments, if more than one instrument is in operation that analyzes for the same analyte using the same technologies.

Two detection limits are determined, one from the blanks' results and one from the standards' results. The higher of the two calculated DLs is utilized as the claimed DL. The DL determined from the standard data is derived by calculating the standard deviation of the standards and multiplying this by the Student *t* test multiplier for the appropriate degrees of freedom of the data set (typically 8, must be at least 6). The DL determined from the blank data is derived by adding the standard deviation of the blanks multiplied by the Student *t* test multiplier for the appropriate degrees of freedom to the average of the blanks (or zero, if the average is <0).

Example calculations:

DL_{std} = stdev*Student *t* for 99% confidence

DL_{blk} = $Average_{blk}$‡ + stdev*Student *t* for 99% confidence
‡(if $Average_{blk}$ is <0, then 0 is used as the average)

Refer to SOP QA-006 for treatment of blank result data sets where some or all results are non-numerical.

H.1.1 c) Ongoing Verification of the Detection Limit:

A Method Blank (MB) and a Low Level Laboratory Control Standard (LL-LCS) spiked at approximately three times the DL are prepared and analyzed with every batch of samples for a given method and analyte. The results are collated in a proprietary Detection Limit spreadsheet that has the ability to sort by dates, analytes, instruments, methods and more. Once every 12 – 14 months, the previous data, up to the previous 24 months and including the initial DL study data if applicable, are processed through the same calculations as the initial DL study. A minimum of seven data points must exist. If there are not seven results in the data set, then additional runs are performed to reach the seven-data-point threshold.

If the calculated DL from the verification data is with ½ to 2 times the Initial Detection Limit as determined above, the DL is considered verified and will not be changed. If it is not within ½ to 2 times the Initial Detection Limit as determined, a new Initial Detection Limit study is performed within 30 days of the finding.

If the method has not been used in the previous twelve months, no verification is necessary, however, another Initial Detection Limit study must be performed prior to reporting any data from that method.

Air quality sampling media are often used to capture the samples (filter, sorbent tube, impinger solution, etc.). These media are considered to be part of the samples and are not included in the blanks or standards used in the detection limit study or verification.

H.1.2 a) Limit of Quantitation

No reference method performed by the laboratory has a requirement for a Limit of Quantitation, by any name. All methods are assumed to report to the Detection Limit, excepting those that specify that the absolute result shall be reported, even if negative. The 2009 and 2016 TNI Standard states *"The laboratory shall select an LoQ for each analyte, consistent with the needs of its clients, and greater than the DL."* The laboratory sets the LoQ at a level five-times the DL, based on CLP guidelines. The LoQ is used for the following:

- Setting control limits for duplicate analysis, where not specified in the method;
- Setting the concentration for the LL-CCV, and the LL-LCS; and,
- Setting a concentration which can be utilized in the DL and LoQ verification process.

LoQs are not required for components or properties for which spiking solutions or QC samples are not available. These include XRF analysis, gravimetric analysis (both Gravimetry Laboratory and Conventional Chemistry), and OC/EC analysis.

The laboratory verifies the LoQ by the analysis of a verification sample consisting of a spiked method blank at or below the concentration of the selected LoQ. The initial verification sample(s) is typically the same sample(s) used in determining the DL.

H.1.2 b) Initial LoQ Verification:

Existing data may be used if the data was generated within a minimum of three batches generated within the past two years. The LoQ is verified if the following is met:

- All results are greater than zero
- All results meet the qualitative identification criteria of the method (e.g., peak present)
- Recovery of each analyte is within the laboratory established accuracy acceptance criteria (50% - 150%)
- The LoQ is greater than the DL (by default, this will always be true)
- The LoQ is at or above the spiking concentration of the verification sample.

H.1.2 c) Ongoing verification of the LoQ:

A Low Level Laboratory Control Standard (LL-LCS) is prepared and analyzed with each batch of samples for a given method and analyte. The LL-LCS is spiked at the same concentration as the initial LoQ verification (approximately three times the DL).

Each LL-LCS (LoQ verification sample) is evaluated at the time of testing. The ongoing verification of the LoQ is met when:

- The results meet the qualitative identification criteria of the method (e.g., peak present);
- Recovery of each analyte is within the laboratory established accuracy acceptance criteria (50% - 150%); and
- The LoQ verification sample (LL-LCS) result is greater than the DL.

If an LoQ verification result does not meet the above requirements, the laboratory:

- Corrects the method of instrument performance and repeats testing, where possible;
- Evaluates the laboratory established control limits to ensure they reflect current performance; or,
- Raises the spiking level and repeats the Initial LoQ verification study within 30 calendar days of the initial failure.

Any samples analyzed with a failing LoQ verification are reported as qualified data.

H.1.3) Precision and Bias

Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms.

Bias is the systematic error that contributes to the difference between the mean of a significant number of test results and the accepted reference value.

Precision and bias using non-reference, modified-reference or laboratory-developed methods are established using the procedure outlined below and compared to the criteria established by the laboratory. The Precision and Bias study is only performed when the method is first brought online or when a change in instrumentation gives cause to believe that precision and bias of the method may have changed.

The 2009 and 2016 TNI Standard states that the laboratory shall "*process the samples through the entire measurement system.*"

Due to the fact that sampling is part of the reference method and therefore part of the measurement system, it is not possible for the laboratory to perform a Precision and Bias study by *"[processing] the samples through the entire measurement system."*

The laboratory uses the same matrices as described in subsection "a)" above (DLs) in performing Precision and Bias studies. Refer to SOP QA-006 for further detail. Briefly, the Precision and Bias study is as follows:

- A Method Blank and three standards are prepared following all analytical preparatory steps contained in the method (if any). Standards are prepared at the following concentrations: the LoQ, the mid-point of the calibration curve, and at 80% of the highest standard. In instances where no preparation is performed on the samples (samples are run as received), the standard is prepared in a clean matrix (e.g., reagent water). Following the steps in the appropriate standard operating procedure, measure the Method Blank and one standard at each concentration level together in one analytical run.
- Calculate the mean recovery for each of the three results.
- On a second, non-consecutive day, repeat above for the second set.
- On a third, non-consecutive day, repeat steps above for the third set.
- Calculate mean recovery for each concentration level over the three days, and for all nine samples.
- Calculate the relative standard deviation of each of the separate mean recoveries obtained.

- Compare the standard deviations for the different days and different concentrations with established criteria; compare the overall mean and standard deviation with established criteria.

H.1.4) Selectivity

Selectivity is the capability of a method or instrument to respond to a target substance or constituent in the presence of non-target substances (EPA-QAD).

The laboratory evaluates selectivity through the use of sample spikes, where possible. With many source emissions samples, the chemistry occurring in one source can be wildly different from another source. Often, the chemistry occurring in the gas stream of the source includes pollution control device emissions containing non-target substances specifically designed to remove the target substance. Selectivity using any substance other than a sample from the source is moot.

Some methods are incompatible with Selectivity tests. Gravimetric analyses, both on filters and source emission samples, do not lend themselves to selectivity.

H.2 Demonstration of Capability

Demonstration of Capability (DoC): A procedure to establish the ability of the Analyst to generate analytical results of acceptable accuracy and precision.

Before reporting any data with a given method, a satisfactory DoC is performed. Thereafter, each Analyst demonstrates continuing proficiency through the procedures outlined in Ongoing Demonstration of Capability.

H.2.1) Initial Demonstration of Capability (IDoC)

An IDoC is performed:

- before using any method;
- when an Analyst learns a method new to the Analyst;
- each time there is a change in instrument type, personnel or method; and
- if the laboratory or Analyst has not performed the method in a twelve-month period.

The IDoC(s) for each Analyst is documented on a DoC form retained in the Analyst's DoC folder maintained by the QA Officer. The document identifies the Analyst(s) involved in preparation and/or analysis; matrix; analyte(s); the method(s) performed; the laboratory-specific SOP used for analysis (including revision number); the date(s) of analysis; and a summary of the results used to calculate the mean recovery and standard deviations.

All raw data, preparation records and calculations for each IDoC are retained either in hardcopy or electronically and are available for review.

When the method specifies a DoC procedure to be followed, only those procedures will be used. If no procedures are specified, the laboratory uses its own procedure. No reference methods utilized by the laboratory require or reference an LoQ by any name. The laboratory uses the same matrices as described in subsection H.1.1.a above (DLs) in performing IDoC studies. Refer to SOP QA-006 for further detail. Briefly, the IDoC study is as follows:

- Prepare four samples in a clean matrix following the entire procedure described in the associated SOP (including any preparatory steps), spiking the clean matrix at a level one to four times the LoQ.
- Following the steps in the appropriate standard operating procedure, measure the Method Blank and the low level standards.
- Using all of the standard results, calculate the mean recovery and the standard deviation of the samples in the same units as the reporting units for samples.
- Compare the recovery and standard deviation to the corresponding acceptance criteria in the method.
- Complete the Demonstration of Capability Certification Statement and place in the appropriate employee's QA file.

H.2.2) Ongoing Demonstration of Capability

After the demonstration of capability is completed, on-going proficiency is maintained and demonstrated at least annually. Each Analyst is expected to consistently meet the QC requirements of the method, the laboratory SOP, client requirements and/or the 2009 and 2016 TNI Standard. Ongoing DoC's for each Analyst are documented on a DoC form retained in the Analyst's DoC folder maintained by the QA Officer, and all records related to the demonstration are retained.

The laboratory follows SOP QA-006 to demonstrate ongoing DOC. The same process as IDoC is used for ongoing DoC's with the exception that ongoing DoC's typically utilize the most recent four consecutive second source standards (e.g., ICV's, LCS's) in the calculation of the statistics, rather than a low level standard. Note that these standards must only be consecutive, not necessarily on the same day or in the same run.

H.3 Calibration

Section 23.2.2 includes information on calibration of support equipment. This Section covers calibration of analytical equipment.

Initial instrument calibration and continuing instrument calibration verification are an important part of ensuring data of known and documented quality. If more stringent calibration requirements are included in a mandated reference method or by

regulation, those calibration requirements override any requirements outlined here or in laboratory SOPs. Generally, procedures and criteria regarding instrument calibrations are provided in the SOPs governing the instruments. XRF has a separate SOP discussing calibration of the instruments due to the complexity of the calibration.

H.3.1 Initial Instrument Calibration

- Records:

  Initial instrument calibration includes calculations, integrations, acceptance criteria and associated statistics referenced in the pertinent SOPs.

  Sufficient raw data records are collected to allow reconstruction of the initial instrument calibration. These include, at a minimum, calibration date, instrument, analysis date, analyte names, Analyst's signature or initials, concentration and response, calibration curve or response factor, or unique equation or coefficient used to reduce instrument responses to concentration.

  Calibration date and expiration date (when recalibration is due) is documented for equipment requiring calibration, where practicable (see Section 23.1).

  Calibration standards are traceable to NIST, when commercially available, and are of a manufacturer's lot different from standards used in all other applications (e.g., spiking, ICVs, etc.).

- Number of Standards and Concentrations:

  If the reference or mandated reference method does not specify the number of calibration standards to use, the minimum number is five for a linear fit curve and six for a quadratic fit curve, not including blanks or a zero standard.

  The 2009 and 2016 TNI Standard states that *"the lowest calibration standard shall be at or below the lowest concentration for which quantitative data are to be reported without qualification."* However, reference methods performed by the laboratory report to the DL without qualification, therefore, this is not possible for all instruments. The lowest calibration standard is at or above the DL and below the Limit of Quantitation (LoQ).

  The highest calibration standard is the highest concentration for which quantitative results can be reported. Data reported exceeding the highest calibration standard *without dilutions* is considered to have increased uncertainty and are reported with an explanation of the reason for the reporting of said data in the Case Narrative (e.g., re-analysis not possible). This does not apply to gravimetric data, XRF results or OC/EC results, where the technologies do not permit a detection limit study as described previously.

  For instrumentation where a single point calibration is recommended by manufacturer's instructions, such as with ICP (with a zero and single point calibration), the following apply:

a) For single point plus zero blank calibrations, the zero point and the single point standard are analyzed prior to the analysis of samples and the linear range of the instrument is established by analyzing a series of standards, one of which is at the lowest quantitation level.

b) Zero blank and single point calibration standards are analyzed with each analytical batch.

c) A standard corresponding to the limit of quantitation (LL-CCV) is analyzed with each analytical batch and must meet established acceptance criteria (60% - 140% Recovery) when using single point plus zero blank calibrations.

d) The linearity of single point plus zero blank calibrations is verified at a frequency established by the method or the manufacturer. Linearity may also be verified with each analytical batch by not reporting any data higher than the calibration standard.

Note that the 2009 and 2016 TNI Standards do not address either thin-film XRF or OC/EC analyses, both of which are used almost solely for air quality analyses. Both of these instrumentations also retain their calibrations for extended periods of time (over 12 months, sometimes several years), and both do not have detection limits, instead reporting to their uncertainties.

- Evaluation, Verification and Corrective Action:

All initial instrument calibrations are verified with a standard made from a second source standard traceable to NIST, when commercially available. The concentration of the calibration verification standard is less than or equal to half that of the highest calibration standard. If a second source is not available, a standard prepared from a different lot may be used. If no standard is commercially available, the laboratory will make a standard in-house, where possible, or find alternate means of ensuring the accuracy of the calibration, where possible.

Criteria for the acceptance of an initial instrument calibration are established and defined in the pertinent SOPs. The criteria used are appropriate to the calibration technique. For instruments with multipoint calibration curves, all calibration standard results are assessed using percent Relative Error (RE or %RE, see Section 27.2.2.5) and correlation coefficients.

Where appropriate, the laboratory has manual integration procedures that are adhered to when evaluating calibration data. These procedures are documented in SOP QA-012.

Any samples that are analyzed after an unacceptable initial calibration are re-analyzed where possible, or the data are reported with qualifiers appropriate to the scope of the unacceptable condition (see Section 12, “Control of Non-conforming Environmental Testing”).

Quantitation is always determined from the most recent initial calibration unless the test or reference method or applicable regulations require quantitation from the continuing instrument calibration verification, except in the case of OC/EC

analysis. OC/EC analysis includes an internal single point calibration standard with every sample analysis, which is necessary to compensate for minor fluctuations in gas flows, furnace temperatures, and FID functioning.

Corrective actions are performed when the initial calibration results are outside acceptance criteria. Calibration data points are never removed from a calibration. If a calibration fails to pass acceptance criteria, the problem is corrected and the calibration standards are re-analyzed to generate a completely new calibration curve.

H.3.2 Instrument Calibration Verification

- Records:

  Sufficient raw data records are collected to allow reconstruction of the continuing instrument calibration verification. These include, at a minimum, method, instrument, analysis date, analyte names, Analyst's signature or initials, concentration and response, calibration curve or response factor, or unique equation or coefficient used to reduce instrument responses to concentration. The Quant'X XRF and all balances used for gravimetric analyses are an exception as the instruments/software do not show the raw counts used during calibration.

  The acceptance criteria, calculations and associated statistics for continuing instrument calibration verification are documented in in the pertinent SOPs.

  Where appropriate, the laboratory has manual integration procedures that are adhered to when evaluating calibration verification data. Refer to SOP QA-012.

- Frequency:

  Calibration is verified for each compound, element, or other analyte being reported, to include mass.

  Calibration verifications are performed:

  - at the beginning and end of each analytical batch. Many methods require the CCV to be analyzed every 10 measurements. Some methods have more frequent CCV requirements (see pertinent SOPs).
  - whenever it is expected that the analytical system may be out of calibration or might not meet verification acceptance criteria.
  - when the time period for calibration or the most recent calibration verification has expired.
  - for all analytical systems that have a calibration verification requirement. Requirements are documented in in the pertinent SOPs.

- Evaluation, Verification and Corrective Actions:

  The validity of the initial calibration is verified prior to sample analysis by use of an initial instrument calibration verification (ICV) standard, and throughout the

run by use of a continuing calibration verification standard (CCV). Thin film XRF verifies the calibration once at the end of a run of 10 samples for the Quant'X or 15 samples for the Kevex 770. Source Particulate Matter balances are verified once, after initial calibration, and prior to every weighing batch thereafter.

The acceptance criteria and corrective actions are documented in in the pertinent SOPs.

Corrective action is initiated for ICV/CCV results that are outside of acceptance criteria (see Section 12, "Control of Non-Conforming Environmental Testing").

H.3.3 Unacceptable Continuing Instrument Calibration Verifications

If routine corrective action for continuing instrument calibration verification fails to produce a second consecutive (immediate) calibration verification within acceptance criteria, then a new calibration is performed.

For any samples analyzed on a system with an unacceptable calibration, some results may be useable if qualified and under the following conditions:

a) If the acceptance criteria are exceeded high (high bias) and the associated samples are below detection, then those sample results that are non-detects may be reported as non-detects.

b) If the acceptance criteria are exceeded low (low bias) and there are samples that exceed the maximum regulatory limit, then those exceeding the regulatory limit may be reported.

CHESTER LabNet only reports data associated with failed ICVs/CCVs/ICBs/CCBs if there is no other option. The data reported under such conditions is heavily annotated.

Appendix I

References

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Appendix J

Employee Competency Reviews

CHESTER LabNet

Technical Employee Performance Review

Employee Name: _____

Title: _____

Date: _____

Reason for review: _____ Annual Performance Review
_____ End of Probationary Period
_____ Critical Incident Review

		Unsatisfactory	Fair	Good	Excellent	Not Applicable
Technical Competency						
Procedures	Follows SOPs, documents necessary deviations.					
Documentation	Completes all pertinent documentation in a timely manner, records all supporting information.					
Technical Knowledge	Understands the technical basis of methods and instrumentation (chemistry, physics, mechanics).					
Mathematics	Correctly performs all mathematical operations necessary for the performance of assigned tasks.					
Computers/Software	Performs computer/peripherals/software troubleshooting.					
Troubleshooting	Able to locate and understand user's manuals, follow verbal troubleshooting instructions, troubleshoot on basis of knowledge.					
Precision	Performs tasks following the same protocols, without deviations, consistently over time.					
Accuracy	Routinely passes batch QC elements; failing QC elements are rare or traceable to issues unrelated to analyst proficiency.					
Corrective Actions	Takes appropriate and technically justified corrective actions. Seeks input from others if necessary.					
Data Reporting	Reports data in correct units, in a timely manner, accurately and completely.					
Reading Comprehension	Able to read, understand, and follow reference documents, including reference methods.					

CHESTER LabNet

Administrative Employee Performance Review

Employee Name: _____

Title: _____

Date: _____

Reason for review: _____ Annual Performance Review
_____ End of Probationary Period
_____ Critical Incident Review

		Unsatisfactory	Fair	Good	Excellent	Not Applicable
Administrative Competency						
Procedures	Follows SOPs, documents necessary deviations. Fills in all required checklists accurately and completely.					
Documentation - log in	Verifies CoC's complete and accurate, logs into LIMS and documents client communications completely and accurately.					
Documentation - internal	Creates appropriate and accurate internal worklists, records requested methods/analytes clearly and legibly.					
Method Knowledge	Understands the necessary parts of each method such that samples are logged in correctly for each method.					
Computers/Software	Performs computer/peripherals/software troubleshooting.					
Troubleshooting	Able to locate and understand user's manuals, follow verbal troubleshooting instructions, troubleshoot on basis of knowledge.					
Client Communication 1	Communicates in a timely and professional manner with clients.					
Client Communication 2	Ensures client's requirements, including methods to be used and desired analytes, are clearly defined, documented and understood.					
Internal Communication	Communicates clients' needs accurately and completely to involved personnel, including changes to work in progress.					
Corrective Actions	Takes appropriate and technically justified corrective actions. Seeks input from others if necessary.					
Data Reporting	Follows SOPs for data reporting, communicates with client for reporting needs, reports data following NELAC requirements.					
Reading Comprehension	Able to read, understand, and follow written communication from clients, analysts or other client services personnel.					

CHESTER LabNet

Manager Performance Review

Employee Name: _____

Title: _____

Date: _____

Reason for review: _____ Annual Performance Review
_____ End of Probationary Period
_____ Critical Incident Review

		Unsatisfactory	Fair	Good	Excellent	Not Applicable
Management Competency						
Procedures	Follows NELAC requirements, QAMP requirements and SOPs where applicable.					
Documentation - annual	Completes all annual documentation requirements, such as ORELAP application, internal document review, and others.					
Documentation - internal	Maintains all internal documentation, including SOPs, DOC's, employee files, training logs, etc.					
Training	Performs and documents company-wide training as needed (e.g., Safety training, Data Integrity and Ethics training, etc.)					
Computers/Software	Performs computer/peripherals/software troubleshooting.					
Client Communication	Communicates in a timely and professional manner with clients, as needed.					
Internal Communication	Communicates company expectations and requirements clearly, concisely, and unambiguously to employees.					
Corrective Actions	Reviews Corrective Action Reports and performs root cause analysis as needed.					
Human Resources	Follows all pertinent employment laws, performs hiring and firing processes in a professional manner.					
Morale	Fosters open, non-retaliative environment in the workplace. Is receptive to feedback from employees.					
Reading Comprehension	Able to read, understand, follow, and respond to written communication from clients and employees.					