QUALITY MANAGEMENT PLAN / QUALITY ASSURANCE PROJECT PLAN FOR CONTINUOUS AND FILTER BASED PARTICULATE (PM_{10}), VOLATILE ORGANIC COMPOUND, AND METEOROLOGICAL PARAMETERS AT SIMS METAL RECYCLING FACILITY

Metal Management Midwest, Inc.

TRINITY CONSULTANTS

1801 S. Meyers Road
Suite 350
Oakbrook Terrace, Illinois 60181

August 2022

Project 171401.0156

Trinity Consultants

A. PROJECT MANAGEMENT ELEMENTS

A.1 Approval Signatures

_____ Date:
SIMS Metal
Debbie Hays, EHS Partner

_____ Date:
SIMS Metal
George Malamis, Central Region General Manager

_____ Date:
SIMS Metal
Ryan Smith, Operations Manager

_____ Date:
US Environmental Protection Agency, Region 5
Christopher Grubb, EPA Regional Counsel

_____ Date:
US Environmental Protection Agency, Region 5
Nathan Frank, EPA Project Manager

_____ Date:
US Environmental Protection Agency, Region 5
Karina Kuc, Quality Assurance Officer

_____ Date:
Trinity Consultants
Casey Lenhart, Consultants Project Director

_____ Date:
Trinity Consultants
Linda Conger, Quality Assurance Director

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A.3 Distribution List

The following individuals will be provided a copy of the Quality Assurance Project Plan (QAPP) for continuous and filter based particulate matter less than 10 microns in size (PM_{10}) and hazardous air pollutant (HAP) metal analysis, meteorological monitoring, and periodic volatile organic compound (VOC) canister sampling to be performed around the Metal Management Midwest, Inc., aka SIMS Metal (SIMS) metal recycling facility located in Chicago, Illinois.

Table A-1 Distribution List for QAPP

Personnel	Organization	Email Address	Business Address	Telephone Number
Debbie Hays	SIMS Metal	Debbie.hays@simsmm.com	SIMS Metal 2500 S. Paulina Street Chicago, IL 60425	773-650-6495
George Malamis	SIMS Metal	George.malamis@simsmm.com	SIMS Metal 2500 S. Paulina Street Chicago, IL 60425	773-650-6440
Ryan Smith	SIMS Metal	Ryan.smith@simsmm.com	SIMS Metal 2500 S. Paulina Street Chicago, IL 60425	773-254-1200 203-499-8446 (cell)
Christopher Grubb	EPA Region 5	Grubb.christopher@epa.gov	US EPA Region 5 Ralph Metcalfe Fed. Bldg. 77 West Jackson Blvd. Chicago, IL 60604-3590	312-886-7187
Nathan Frank	EPA Region 5	Frank.nathan@epa.gov	US EPA Region 5 Ralph Metcalfe Fed. Bldg. 77 West Jackson Blvd. Chicago, IL 60604-3590	312-886-3850
Karina Kuc	EPA Region 5	Karina.kuc@epa.gov	US EPA Region 5 Air Enforcement and Compliance Assurance Branch Ralph Metcalfe Fed. Bldg. 77 West Jackson Blvd. Chicago, IL 60604-3590	312-353-5090
Casey Lenhart	Trinity Consultants	clenhart@trinityconsultants.com	Trinity 4525 Wasatch Blvd. Suite 200 Salt Lake City, Utah 84124	801-272-3000 Ext. 4501
Linda Conger	Trinity Consultants	lconger@trinityconsultants.com	Trinity 4525 Wasatch Blvd. Suite 200 Salt Lake City, Utah 84124	801-272-3000 Ext. 4516

A.4 Project/Task Organization

SIMS is committed to quality and the implementation of the procedures and practices found in this Quality Assurance Project Plan (QAPP). Quality Assurance (QA) is 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. 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 stated requirements established by the customer. The QC system includes the operational techniques and activities that are used to fulfill requirements for quality.

Quality control is largely implemented through the QA Program Plan. Each monitoring program has unique requirements, statutory guidelines, rules, and policies that must be followed. The QAPP incorporates the unique qualities that are specific to each monitoring program.

Implementation of a quality program requires a management system available to all Project Directors. SIMS and its subcontractors are committed to the principles and practices of its QA Program at the highest level. Senior management recognizes and accepts this responsibility to identify the quality requirements that will meet needs and expectations of the monitoring program. The QA Program developed for this program focuses on preventing quality problems.

The following sub-sections describe the project participants and the roles and responsibilities of each participant.

A.4.1 SIMS E.H.S. Partner

The SIMS EHS Partner is Debbie Hays who will be responsible for oversight of the continuous ambient air quality and meteorological monitoring program. Ms. Hays has overall responsibility for the project including contractor technical quality of work performed and budgets. The SIMS E.H.S. Partner has the authority to allocate funds and to issue stop/resume work orders. SIMS will coordinate with its contractor in the performance of the work needed to obtain quality and accurate data. Ms. Hays will ensure that the selected contractor is proficient and capable of performing the requested monitoring activities. This includes review of contractors standard operating procedures (SOPs), verifying contractor credentials to perform the required activities. The SIMS EHS Partner will also perform QA activities for this project in coordination with the Trinity QA Director.

A.4.2 SIMS Central Region General Manager

The SIMS representative, George Malamis is the Central Region General Manager who serves as the Plant General Manager who is responsible for overseeing operations at the facility.

A.4.3 SIMS Operations Manager

Ryan Smith is the Operations Manager for SIMS Midwest who will be responsible for operational management and implementation of the ambient air quality monitoring program. He will ensure compliance with the QAPP and will ensure that the data collected at the site meets all quality assurance requirements. Mr. Smith will inform the SIMS EHS Partner, SIMS Central Region General Manager, and Trinity Project Director via email or phone of any issues or any changes that may be needed to the monitoring operations.

A.4.4 Analytical Laboratories

Pace Analytical and EAS Labs will provide the sampling media and perform the analytical analyses for the monitoring project. Pace Analytical will provide the PM_{10} filters to be used for sampling and will determine lead concentrations by energy-dispersive X-ray fluorescence spectrometry (EDXRF) according to 40 CFR Part 50, Appendix Q, reference Method for the Determination of Lead in Particulate Matter as PM_{10} Collected from Ambient Air. Metal hazardous air pollutants (HAPs) (antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, and selenium) concentrations from the filter-based sampling will be determined by Pace Analytical utilizing Section 4.4.11 PM_{10} Metals Analysis by ICP/MS – EPA IO 3.5 within NATTS Program TAD Revision 3.

EAS Lab will provide, clean, evacuated 6-liter stainless steel SUMMA canisters for use in the 1-in-3-day VOC sampling. Canisters will be analyzed by EAS Lab using EPA Compendium Method TO-15A, Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters and Analyzed by Gas Chromatography–Mass Spectrometry (GC-MS).

A.4.5 Trinity Consultants Project Director

The Trinity Project Director is ultimately responsible for all quality-related functions. His QA responsibilities are to authorize the issuance of QA policy, direct implementation of QA objectives, plans, and policies, appointing of the QA Director who directs the QA program, and approves the QA implementation strategy. In addition, as Project Director, Mr. Lenhart has overall responsibility for the project, including client liaison, planning document preparation, technical quality of work performed, data acquisition, report preparation, as well as budget and schedule management. Mr. Lenhart will be responsible for determining the technical staff assigned to the project and will oversee their understanding and compliance with the QC procedures that apply to their activities. He will be responsible for overseeing the day-to-day operation, routine and preventive maintenance, data collection and data validation activities. He will communicate with the Trinity Consultants Field Technicians and the SIMS EHS Partner to ensure that project deliverables and activities are in accordance with project requirements.

Mr. Lenhart will determine the staff assigned to the project and will be responsible for training the field and data staff involved with this project. Documentation of training is maintained in the individual's personnel file which is kept at Trinity.

Mr. Lenhart's QA responsibilities are to authorize the issuance of QA policy, direct implementation of QA objectives, plans, and policies, appointing of the QA Director who directs the QA program, and approves the QA implementation strategy. Mr. Lenhart will coordinate with the Trinity quality assurance director with implementing the quality management program designed for this monitoring program. Mr. Lenhart will respond to corrective action requests by field and data management personnel and will assure that deficiencies are corrected in a timely manner.

Trinity's Monitoring Project Director is assisted by the technical staff. He will be assisted by Trinity instrumentation specialists, Mr. Mike Peterson, Mr. Tomy St. Laurent, Mr. Trey Denney, Mr. Isaac Legare, and/or Mr. Ryan Evans who will perform the site equipment installation, routine maintenance, and equipment decommissioning. Trinity monitoring field technicians have the requisite background and training needed to perform their assigned tasks.

Mr. Mike Peterson, a Trinity instrumentation specialist, will be responsible for sending standards to vendors or metrology labs for recertification. Mr. Peterson utilizes an electronic calendar that tracks all monitoring standards and when certifications of standards expire. Standards are sent out in advance of expiration to ensure all certifying equipment has current traceability. Standard certifications are archived in hard copy and electronically on Trinity's computer network.

A.4.6 Trinity Quality Assurance Director

The Trinity QA Director manages the QA Program and is responsible for the technical quality of all work products at Trinity and the development and maintenance of a sufficient level of technical resources to support the company's objectives. The QA Director reports to the Trinity Consultants Project Director and has the authority to halt the transmittal of any work product that in her opinion is not consistent with Trinity's quality standards. The Trinity QA Director will work with the SIMS EHS Partner to ensure the technical quality goals for this project are met.

The Trinity QA Director's primary duties are: (1) To provide a central point of responsibility for assessing company-wide technical strengths, needs and direction to maintain a consistent Company-wide quality of work that meets or exceeds the current standard of practice; (2) To develop Trinity QA policy; (3) Coordinate training; (4) Provide guidance to the QC reviewers in carrying out QC-related functions; (5) Inform project personnel of QA policies, procedures, and other guidance documents; (6) Prepare the QA Project Plan; (7) Audit selected projects and monitoring general compliance with the QA policy; (8) Advise Project Directors of deficiencies on peer reviews and identify corrective action needs; and (9) Maintain Trinity QA files, including audit reports.

The responsibility for planning, developing, and implementation the Quality System resides with Trinity's Quality Assurance (QA) director, Ms. Linda Conger. The QA director reports to the Trinity Consultants Project Director. Ms. Linda Conger is responsible for oversight of Trinity's quality assurance/quality control activities from field measurements to data validation, data reporting, and implementation of quality assurance policies and procedures. She is responsible for ensuring that each staff member involved with collecting or analyzing environmental data has the necessary technical, quality assurance, and project management training required for his or her assigned tasks and functions. Ms. Conger has over 30 years of on-the-job experience planning, developing, and implementing quality assurance/quality control activities associated with environmental monitoring projects. She has participated in Trinity QA training courses and worked with Battelle and other large firms in implementing QA programs under the oversight the Nuclear Regulatory Commission.

Ms. Conger will prepare and maintain the official approved QAPP. The SIMS EHS Partner will provide the final internal review after the Trinity Consultants Project Director has completed his review. The SIMS EHS Partner will approve prior to submitting the QAPP to EPA. The QAPP and standard operating procedures prepared and submitted with the plan are reviewed when significant changes are made to the monitoring equipment or methodologies utilized.

When approved by EPA, the QAPP will be signed and dated by program participants including monitoring personnel. All appropriate personnel in the organizations performing or reviewing work covered by the scope of the QAPP are notified through email, meeting, or via telephone of changes to the quality system so they are informed of current requirements. All communications will be documented, and documentation will be maintained in a trackable system.

Field or data issues, which will be documented, and documentation maintained, will be brought to the Trinity Consultants Project Director and SIMS Operations Manager who will inform the Trinity QA Director if changes are necessary. Oversight responsibilities for QA/QC may result in disagreements between the oversight group and the program reviewed. Such disputes may occur in situations involving technical issues (e.g., quality requirements, assessments, audits, surveillance, data, and technical information) and management issues. All parties will make every effort to resolve disputes through discussion and negotiation. If the parties are unable to resolve the dispute, resolution will be initiated by the Trinity QA Director with concurrence from the Trinity Consultants Project Director. The SIMS EHS Partner will be informed of all disputes.

Ms. Conger will be assisted in quality related activities by technical personnel who have knowledge in the quality assurance aspects of this project. These QA specialists, include Mr. Adam Lenkowski and Ms. Cara Keslar. Mr. Lenkowski or Ms. Keslar reports to the Trinity Consultants Project Director but also maintains a direct communication link and reporting relationship with the QA Director on quality-related matters. Any non-conformance with QC procedures identified by Mr. Lenkowski or Ms. Keslar will be reported to the Project Director and QA Director, along with recommended corrective measures for implementation.

Mr. Lenkowski or Ms. Keslar will be assigned to perform quality control of data collection, data validation and reporting. He/she will review all data with respect to QC criteria and will communicate review comments to project team members. Mr. Lenkowski or Ms. Keslar have the requisite background and training needed to perform QA/QC activities. They have participated in vendor provided QA courses and have taught quality assurance courses for ambient monitoring.

Documentation of all formal training is maintained in the individual's personnel file which is kept at Trinity. Training typically consists of "hands-on" training overseen by the Project Director or a supervisor proficient in the equipment or data collection and validation activities. Training resources may also be procured from outside of the company.

A.4.7 Trinity Field Technicians/Calibration

Mr. Mike Peterson, Mr. Ryan Evans, Mr. Isaac Legare, or Mr. Tomy St-Laurent will install the PM_{10}, meteorological, and VOC sampling equipment and perform routine maintenance, as necessary. The proposed field technicians have several years of field experience and on-the-job training and have demonstrated to the Trinity Consultants Project Director their capabilities to perform the required work. Documentation of training is maintained in Trinity's personnel folder for each employee.

A.4.8 Site Operators

Personnel from Trinity's Chicago and Salt Lake City offices will be the site operators for the SIMS monitoring project. The Trinity Chicago site operator will be the first responder responsible for routine maintenance and trouble-shooting activities for the PM_{10} samplers, meteorological sensors, and VOC canister sampling systems. The Trinity Chicago site operator will be trained by Trinity Salt Lake City monitoring staff in the proper operation and maintenance of the equipment. Trinity Salt Lake City personnel will provide remote technical support to the Trinity Chicago site operator to assist with any issues that might arise with the instrumentation and will travel to the site and rectify any issues that cannot be resolved by the first responder, as necessary. Trinity Chicago site operators will be responsible to conduct the monthly flow and leak check of the PM_{10} sampling equipment. Trinity Salt Lake City site operations will perform the quarterly and semi-annual calibrations of the PM_{10} samplers and meteorological sensors, respectively. Trinity has a monitoring staff of 10 who are available to assist with this project.

A.4.9 Trinity Data Management

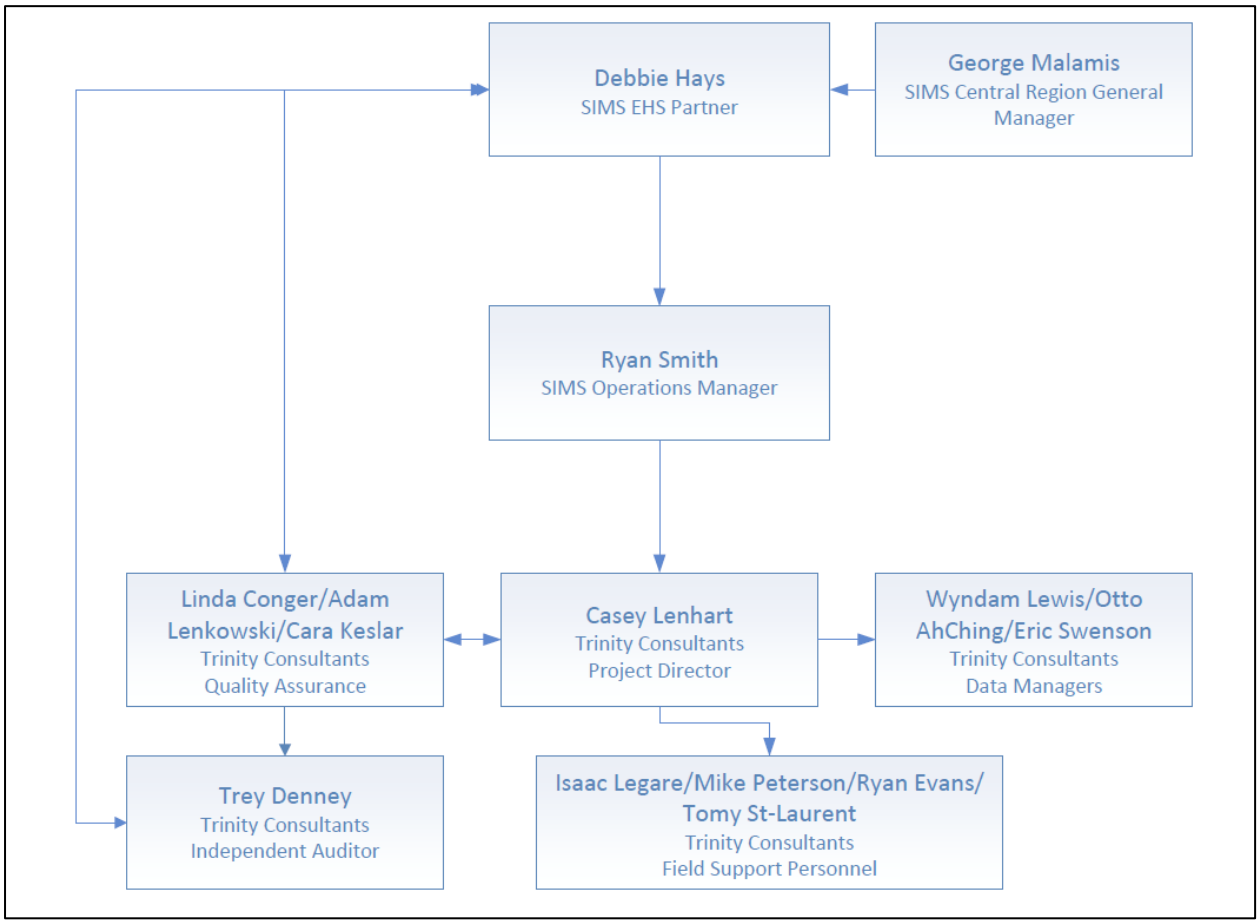
Trinity's data managers, Mr. Wyndam Lewis, Mr. Otto AhChing, or Mr. Eric Swenson will be responsible for ensuring timely data collection and data review, posting data, performing data verification activities including flagging data, data management, data validation, and preparation of data summaries for reports. Final data validation is the responsibility of Trinity data managers with concurrence by the Trinity Project Director, and the QA Director.

A.4.10 Trinity Independent Auditor

An independent auditor, Tret Denney, will conduct the quality assurance performance audits of the PM_{10} samplers and meteorological sensors. Mr. Denney has the requisite background and training needed to perform the independent quality assurance audits. The independent auditor will report audit findings to the SIMS E.H.S. Partner as well as the Trinity Consultants Project Director and QA Director. The independent auditor will conduct the performance audits with different NIST-traceable reference standards than those that are used to conduct the calibrations.

Figure A.1 presents the organizational chart that shows the lines of responsibility and information flow for activities under this project.

Figure A.1 Organizational Chart



A.5 Problem Definition/Background

This quality assurance project plan has been prepared on behalf of SIMS. Pursuant to a U.S. Environmental Protection Agency (EPA) Section 114 (a) information request, this QAPP details the proposed methodologies to collect accurate PM_{10}, meteorological, metal hazardous air pollutants (HAPs) in PM_{10}, lead (pb), VOC, and other specified compound monitoring data.

The procedures outlined in this QAPP have been developed to meet the goals and objectives of the monitoring project. Revisions to the QAPP are made, as necessary, to reflect changes to the regulations or goals of the monitoring project. As a minimum, the QAPP is reviewed annually, and revisions are made, as necessary.
The information collected for this monitoring program will meet the requirements as found in the following documents:

- EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Quality Monitoring Program, EPA-454/B-17-001, January 2017, Appendix D,
- 40 CFR Part 58, Appendix A Quality Assurance Requirements for Monitors used in Evaluations of National Ambient Air Quality Standards,
- 40 CFR Part 50, Appendix J, Reference Method for the Determination of Particulate Matter as PM_{10} in the Atmosphere,
- 40 CFR Part 50, Appendix Q, Reference Method for the Determination of Lead in Particulate Matter as PM_{10} Collected from Ambient Air,
- National Air Toxics Stations (NATTS) Program Technical Assistance Document (TAD) Revision 3,
- EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV: Meteorological Measurements Version 2.0 (Final),
- EPA Compendium Method TO-15A, and,
- Manufacturer's manuals.

The guidance presented in the above listed documents and to be followed for this monitoring program is intended to ensure that data and technical information that are measured are of documented and appropriate quality and usability.

A.5.1 Area Climate and Topography

The climate of the region and the topography of the area are presented below.

A.5.1.1 Climate

Chicago lies midway between the Continental Divide and the Atlantic Ocean and has four distinct seasons. Chicago's climate is typically continental with cold winters and warm summers. Lake Michigan provides a moderating influence on temperature while increasing to the amount of snowfall that is received in the city. The Chicago area sees many storms during all seasons. The polar jet stream is often located near or over Illinois which allows for the creation and movement of low-pressure storm systems that bring in clouds, wind, and precipitation. Since Chicago is an urban area, buildings, parking lots, roads, and industrial activities can make the urban climate warmer by 2°F, on average, especially at night. Since Chicago is located on the southwest corner of Lake Michigan, the city often experiences breezes off the lake.

Table A-2 presents the monthly climatic summary, based on meteorological data from 1981 through 2010 obtained from the O'Hare Airport.

Table A-2 Monthly Climatic Summary for Chicago, Illinois

	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Ann.
Avg. Max. Temp. (°F)	31.0	35.3	46.6	59.0	70.0	79.7	84.1	81.9	74.8	62.3	48.2	34.8	59.1
Avg. Min. Temp. (°F)	16.5	20.1	29.2	38.8	48.3	58.1	63.9	62.9	54.3	42.8	32.4	20.7	40.8
Avg. Total Prec. (in)	1.73	1.79	2.50	3.38	3.68	3.45	3.70	4.90	3.21	3.15	3.15	2.25	36.89
Avg. Total Snow. (in)	10.8	9.1	5.6	1.2	0.0	0.0	0.0	0.0	0.0	0.3	1.2	8.5	36.7

A.5.1.2 Topography

The topography of the area is flat. The average land elevation is 579 feet above sea level. Chicago's topography is almost entirely a product of glaciation.

A.6 Project/Task Description

The primary purpose of the monitoring program is to collect site-specific continuous and filter based PM_{10}, meteorological, Pb, VOC, and other speciated compound information to satisfy EPA's 114(a) data request. The proposed PM_{10}, meteorological, and VOC sampling equipment to be operated around the SIMS facility will fulfil the 114(a) monitoring requirements. The operating range of the continuous and filter based PM_{10} monitors, and VOC sampling equipment brackets the range of expected ambient air quality concentrations at the monitoring stations.

A.6.1 SIMS Air Quality Monitoring Stations

Five (5) perimeter near-reference method continuous PM_{10} air monitors will be deployed around the Facility. The perimeter air monitoring stations will monitor PM_{10} continuously, 24-hours per day, seven days per week. The proposed continuous PM_{10} air monitors are designed to agree with EPA Class I and Class III FRM/FEM particulate samplers and provides real-time particulate measurement through near-forward light scattering. In addition to the five PM_{10} monitoring stations, a meteorological station will be installed at the SIMS facility and operated to continuously monitor meteorological conditions.

Monitoring site selection guidance presented in 40 CFR Part 58 Appendices A and E was considered when selecting the placement of the PM_{10} monitors. The proposed sites were selected based on suitability of terrain and distance from obstructions to ensure that representative data are collected. Accessibility to each site was also an important consideration in choosing the placement of the monitors.

The five monitoring sites are identified as AQ1, AQ2, AQ3, AQ4, and AQ5 and are presented in Figure A.2 below. All five sites will be equipped with near-reference E-Sampler PM_{10} monitors. Monitoring instruments at locations AQ1, AQ2, and AQ5 will also collect continuous PM_{10}, 24-hour PM_{10} filter-based samples, and VOC samples in accordance with EPA's 1-in-3-day sampling schedule. The filter based PM_{10} samples will commence during business hours at approximately the same time of day for each sample collection day to allow for 24-hours of sampling. The typical midnight to midnight timeframe is not practical due to the type of instrument, staffing availability, and site safety concerns at midnight. Filter samples will be analyzed by the analytical laboratory for metal HAPs following Compendium Method IO-3.5. The VOC samples for compounds identified in Appendix C of the 114(a) request will be analyzed by an accredited laboratory, in accordance with EPA's Compendium Method TO-15A.

Small alterations in the exact location of the five monitoring sites may occur with operational implementation of the equipment based on operational interferences and/or obstructions.

Figure A.2 Proposed Ambient PM_{10} Monitors and Meteorological Station for SIMS

Four (4) monitors, AQ1, AQ2, AQ4, and AQ5 were selected to meet the requirement in the City of Chicago Department of Public Health (CDPH) Rules for Large Recycling Facilities (City Rules), Section 3.9.21.2(a) which calls for at least one monitor placed along the fence line, at each 45-degree direction from the center of the Facility where there is a "Sensitive Area"[1] within 660 feet of the Facility boundary.

[1] Per the City Rules, A "Sensitive Area" means any property with a residential use, a park, a hospital, a clinic, a church, a day-care center, or a school.

The latitude and longitude coordinates for each sampling site are presented in Table A-3.

Table A-3 Monitoring Station Locations

Station ID	Latitude	Longitude
AQ1 and Meteorological	41°51'4.27" N	-87°40'1.99" W
AQ2	41°50'54.88" N	-87°40'1.99" W
AQ3	41°50'54.49" N	-87°40'2.33" W
AQ4	41°50'41.69" N	-87°40'6.28" W
AQ5	41°50'37.57" N	-87°40'19.01" W

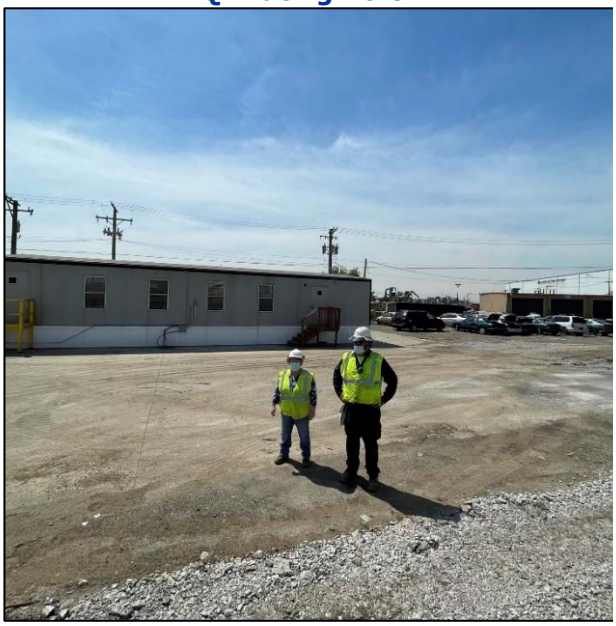
A.6.1.1 AQ1 Monitoring Station

The AQ1 station is proposed to be located at the north end of the Facility. The location is within 660 feet of residential areas located north of the Facility and within 660 feet from residential areas and other sensitive areas (as defined by the City.) This station site also is being proposed for the meteorological tower. The meteorological system will continuously monitor temperature and relative humidity at 10-meters, wind speed and wind direction at 10 meters, and precipitation at 1.5 meters. This station site meets U.S. EPA siting criteria and is generally free of obstructions. Figure A.3 presents photographs of the four cardinal directions surrounding the AQ1 site location.

Figure A.3 Photographs of Proposed AQ1 Monitoring Station Location

AQ1 Facing North

AQ1 Facing East



AQ1 Facing South

AQ1 Facing West

A.6.1.2 AQ2 Monitoring Station

The AQ2 station is proposed to be located at the northeast side of the Facility. Figure A.4 below presents photographs of the four cardinal directions surrounding the AQ2 site location. To limit the influence of obstructions adjacent to the site, this station will be placed on an elevated platform. The distance of the inlet of the PM samplers to the adjacent building will be at least twice the differential from the inlet to the top of the building, where possible.

Figure A.4 Photographs of Proposed AQ2 Monitoring Station Location

AQ2 Facing North

AQ2 Facing East

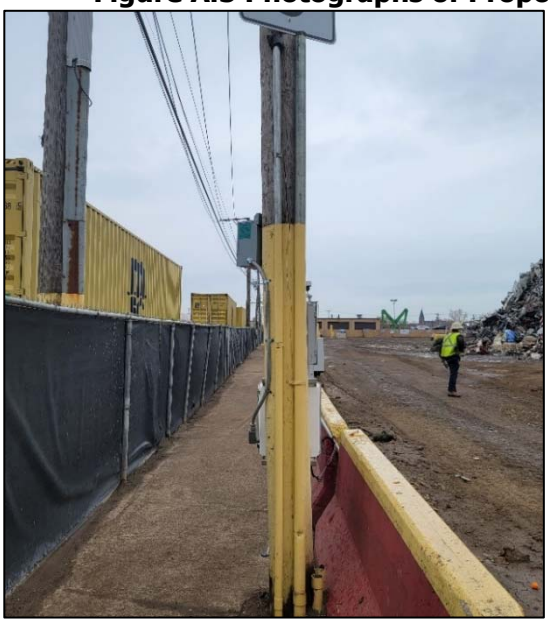
AQ2 Facing South

AQ2 Facing West

A.6.1.3 AQ3 Monitoring Station

The AQ3 monitoring station is proposed to be located along the west fence line of the Facility adjacent to Paulina Street. Figure A.5 below presents photographs of the four cardinal directions surrounding the AQ3 site location.

Figure A.5 Photographs of Proposed AQ3 Monitoring Station Location



AQ3 Facing North

AQ3 Facing East

AQ3 Facing South

AQ3 Facing West

A.6.1.4 AQ4 Monitoring Station

The AQ4 station, the east monitor, is proposed to be located at the southeast corner of the Facility. The location is within 660 feet to the nearest residential area to the east of the Facility. Figure A.6 below presents photographs of the four cardinal directions surrounding the AQ4 site location.

Figure A.6 Photographs of Proposed AQ4 Monitoring Station Location

AQ4 Facing North

AQ4 Facing East

AQ4 Facing South

AQ4 Facing West

A.6.1.5 AQ5 Monitoring Station

The AQ5 station is proposed to be located at the southwest corner of the Facility. The location is within 660 feet to the nearest residential area to the southwest of the Facility. Figure A.7 below presents photographs of the four cardinal directions surrounding the AQ5 site location.

Figure A.7 Photographs of Proposed AQ5 Monitoring Station Location

AQ5 Facing North

AQ5 Facing East

AQ5 Facing South

AQ5 Facing West

A summary of the PM_{10} and meteorological instrumentation to be installed at the SIMS monitoring stations is presented in A-4.

Table A-4 SIMS PM_{10} and Meteorological Monitoring Station Measurements

Parameter	Manufacturer/Model	Sensor Height (meters)
PM_{10} (AQ1)	Met One Instruments E Sampler-9800	2.0
PM_{10} (AQ2)	Met One Instruments E Sampler-9800	2.0
PM_{10} (AQ3)	Met One Instruments E Sampler-9800	2.0
PM_{10} (AQ4)	Met One Instruments E Sampler-9800	2.0
PM_{10} (AQ5)	Met One Instruments E Sampler-9800	2.0
Wind Direction Sensor	Wind Monitor Model 05305-AQ	10.0
Wind Speed Sensor	Wind Monitor Model 05305-AQ	10.0
Relative Humidity	Campbell Scientific HygroVUE 10	2.0
Temperature	Campbell Scientific HygroVUE 10	10.0
Precipitation	Texas Electronic TE525	1.5
Data Acquisition	Campbell Scientific CR1000x	NA

A.6.2 Sampling Frequency

At each monitoring station location, the E-Sampler will collect and store 1-minute and hourly average PM_{10} information which will be transmitted to a web-based collection platform. Five (5) minute and hourly averages of the meteorological data will be calculated and stored on a directly connected datalogger. A Sierra Wireless RV50 cellular modem will be utilized to transmit the meteorological data to Trinity's data center for further evaluation. Filter based PM_{10} and HAPS metal analysis, and VOC samples will be collected every 3 days in accordance with United States Environmental Protection Agency (US EPA) three-day sampling schedule.

A.6.3 Project Schedule

Personnel working on this project will be fully qualified, trained, and capable to perform their assigned duties. Work schedules include daily data review by a Trinity Data Manager; quarterly PM_{10} monitor calibrations; semi-annual meteorological sensor calibrations; monthly PM_{10} flow and leak checks, VOC canister sample event leak checks; data summaries within 30 days of month completion; and maintenance and corrective action, as needed. A PM_{10} and meteorological performance audit will also be conducted. Table A-5 presents the project schedule. Monitoring data collection will officially begin around the SIMS facility within 60 days of final QAPP approval by EPA.

Table A-5 Project Schedule

Task	Time
Quality Assurance Project Plan	Start of monitoring project and as needed to reflect changes in equipment or monitoring requirements.
Monitoring Operations	Calibrations – after installation, semi-annual for meteorological sensors, quarterly for particulate monitors, and whenever an instrument exceeds specified control limits or undergoes major maintenance or repair. The end of each monitoring period is quarterly. Monthly flow and leak checks on PM_{10} sampling equipment. VOC SUMMA canister leak checks prior to every sampling event. 1- in 3-day filter based PM_{10} samples and VOC canisters collected.
Quality Assurance	A PM_{10} sampler and meteorological performance audit will be conducted after the first flow check, and every 180 days, 5-7 months apart.

A.6.4 Project Reports

Table A-6 presents the reports that will be produced as part of this project.

Table A-6 Project Reports

Reports	Frequency	Content	Responsible Position	Distribution
QAPP	Prior to Start of Monitoring	Outlines the procedures the monitoring project will use to ensure the data it collects and analyzes meets project requirements	Trinity QA Manager	See Section A.3 Distribution list
Data Summary	Monthly within 30 days of preceding month end	Summarize PM_{10}, meteorological, VOC, HAP and Pb laboratory data following EPA guidelines.	Trinity Data Manager	See Section 1.3 Distribution list
Corrective Action Reports	As needed, but once initiated, resolved within 7 days	Summarizes corrective actions taken Example CAR form found in Appendix B.	Trinity Consultants Project Director	See Section 1.3 Distribution list
Response to Corrective Action Reports	As needed, but once initiated, resolved within 7 days	Reports the results of the corrective actions taken	Trinity Consultants Project Director	See Section 1.3 Distribution list

A.7 Quality Objectives and Criteria for Measurement of Data

Presented in this section are the Measurement Quality Objectives (MQOs) for the PM_{10}, meteorological, VOC, HAPs and other speciated compound measurements. 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 data quality objectives. MQO's can be defined in terms of the following data quality indicators: precision; accuracy; representativeness; detectability; completeness; and comparability. Monitoring results are assessed against these objectives to demonstrate the quality of the measurement data.

Precision is a measure of agreement among repeated measurements of the same property under identical, or substantially similar, conditions. This is the random component of error. Precision is estimated by various statistical techniques typically using some derivation of the standard deviation.

Bias is the systematic or persistent distortion of a measurement process which causes error in one direction. Bias will be determined by estimating the positive and negative deviation from the true value as a percentage of the true value.

Accuracy is a measure of the overall agreement of a measurement to a known value and includes a combination of random error (precision) and systematic error (bias) components of both sampling and analytical operations.

Representativeness refers to the degree to which data accurately and precisely represent a characteristic of a population, a parameter variation at a sampling point, a process condition, or a condition.

Detectability is the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated level of probability.

Completeness describes the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions.

Comparability is a measure of the confidence with which one data set or method can be compared to another, considering the units of measurement and applicability to standard statistical techniques. Comparability of datasets is critical to evaluating their measurement uncertainty and usefulness.

The EPA has developed a Data Quality Objective (DQO) seven-step process for use in environmental measurement projects. Table A-7 summarizes this process. The benefits of the DQO process are that it prompts a statement of the problem, identifies the decisions to be made and the inputs needed to make the decisions, and specifies a decision rule.

Table A-7 Data Quality Objectives Process for Ambient Monitoring Project

Step 1	Define the Problem	SIMS will collect continuous PM_{10} and meteorological data, and intermittent filter based PM_{10} and Pb, VOC, and HAP to satisfy EPA's 114(a) data request
Step 2	Identify the Goal of the Study	EPA will use data to determine emission levels around SIMS
Step 3	Identify Information Inputs	$PM_{10,}$ VOC, and HAP measurements will be made in accordance with EPA, NATTS TAD Revision 3, and Compendium Method TO-15A sampling methods
Step 4	Define the Study Boundaries	Sampling frequencies are discussed in Section A.6.2; sampling locations are presented in Section A.6.1
Step 5	Develop the Analytical Approach	The measured ambient concentration data will be used to determine emission levels on SIMS property
Step 6	Specify the Limits of the Decision	The site operator will demonstrate that the instrument system is capable of measuring air quality parameters with an accuracy that is consistent with the DQO's of the true value on a regular basis
Step 7	Develop the Plan for Obtaining Data	If the system does not conform to the required QA/QC protocols identified in this document, the site operator will initiate corrective action to bring into conformance

For the monitoring project, the PM_{10}, VOC, and meteorological MQOs are presented in Tables A-8 and A-9. PM_{10} sampler and meteorological sensor response characteristics are presented in Table A-10 and Table 2-11. Calibration and accuracy criteria are presented in Tables A-12 and A-13. Key criteria such as precision, accuracy, and completeness enable the project team to measure progress and success in attaining the quality goals for the monitoring effort. Representativeness and comparability are also used to evaluate quality. Representativeness is ensured through site selection including sample probe siting evaluations. Comparability is accomplished using standard measurement methods approved by EPA and by reporting measurement data in common units to facilitate comparison with other data sets generated by regulatory and private concerns.

PM_{10} and meteorological measurements recorded will be subject to and consistent with the quality assurance requirements as found in 40 CFR Part 58, Appendix A and EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volumes II and IV. Measurements that do not meet the MQO's may be invalidated unless justification can be identified for not doing so. In addition, data may be invalidated if a sampler fails a performance audit and further investigation confirms the audit results. These data may be invalidated back to the last good check or calibration of the equipment. Trinity will notify SIMS within two days of an issue and will provide an explanation of the issue and the corrective action being performed to remedy the issue.

Table A-8 PM_{10} and VOC Measurement Quality Objectives

Parameter	Requirement	Frequency	Acceptance Criteria	Reference	Data Completeness
PM_{10} E-Sampler (Continuous and Filter Based)	Average flow rate	Every 24 hours of operation	Average within <±5.1% of design	Recommendation	85%
	Temperature	Monthly	<±2.1 °C	40 CFR Part 50, App. L Sec. 9.3	
	Pressure	Monthly	<±10.1 mm Hg	40 CFR Part 58 App. A, Section 3.1	
	PM_{10} Calibration	Install and quarterly, and after major repair	3 of 4 calibration points within <±10.1% of design	40 CFR Part 50 App. L, Sec. 9.2	
	PM_{10} One-point Flow rate verification	Monthly	<±7.1% of transfer standard	40 CFR Part 58, App. A, Sec 3.3	
	PM_{10} Variability in Flow Rate	Every 24 hours of operation	CV ≤ 2%	40 CFR Part 50 App. L, Section 9.2.5	
	Flow Rate Performance Audit	Within 30 days of start of monitoring then every 180 days, 5-7 months apart	<±10.1% of audit standard	Part 58, App. A, Sec. 3.3.3	
	Sampling Period	All PM_{10} 1-in-3 day Filters		40 CFR Part 50, App. J Sec. 7.15	
VOC	Analyte Result	Each sample event	±25% accuracy of target value	NATTS	
	Canister Initial Vacuum	Prior to sample collection	>28 in Hg.	TO-15A	
	Sampling Period	24 hours per sample event	24 Hours±1 hour	NATTS	
	Precision	Every 24 hours of operation	CV ≤ 15%	NATTS	
	Leak Check	Every sample event	<0.2 psi over 5 minutes	NATTS	

Table A-9 Meteorological Measurement Quality Objectives

Parameter (Manufacturer/ Model)	Specified Accuracy	Required Accuracy	Sensor Resolution in System	Required Resolution	Data Completeness
Wind Speed RM Young Model 5303 Wind Monitor AQ	±0.2 m/s	±0.20 m/s	0.01 m/s	0.1 m/s	90%
Wind Direction RM Young Model 5303 Wind Monitor AQ	±3 degrees	±5 degrees	0.01	1.0	90%
Temperature CSI Hygrovue 10	±0.2°C (-40°C to 70°C)	±0.5°C	0.01°C	0.1°C	90%
Relative Humidity CSI Hygrovue 10	±1.5 (25°C, over 0-80% RH) ± 2.0% RH (25°C, over 80 - 100% RH)	±7% RH	0.1%	0.5%	90%
Precipitation Texas Electronics TR525	1.0% up to 2 in/hour (50 mm/hr)	±10% of input volume	±0.01 in.	0.01 in.	90%
Vector Data					
Wind Speed	±0.2 m/s	±0.20 m/s	0.01 m/s	0.1 m/s	90%
Wind Direction	±3 degrees	±5 degrees	0.01	1.0	90%
Sigma Theta	±5 degrees	±5 degrees	0.01 m/s	0.1 m/s	90%

Table A-10 PM_{10} Measurement Methods and Response Characteristics

Parameter	Measurement Method	Analyzer Response Characteristic
PM_{10} E-Sampler	Near forward-light scattering	Range: 0 - 65,530 µg/m^3 Nephelometer Accuracy: ±10% to gravimetric method typical when k-factored to local particulate type Gravimetric Accuracy: ±8% of NIOSH 0600 Precision: Greater of 3 µg/m^3 or 2% Data Storage Resolution: 1 µg/m^3 Flow rate: 2.0 lpm ±0.1 lpm Operating Temperature: 0 to 50°C Approvals: Designed to agree with EPA Class I and EPA Class III FRM/FEM PM samplers

Table A-11 Meteorological Measurement Methods and Response Characteristics

Parameter	Measurement Method	Sensor Response Characteristic	EPA-Required Response Characteristics
Wind Speed – RM Young Model 05305-AQ	Propeller rotation produces AC signal with frequency output proportional to wind speed	Starting Threshold = 0.4m/s Distance Constant =2.1m	Starting Threshold = ≤0.5m/s Distance Const. ≤5m
Wind Direction – RM Young Model 05305-AQ	Precision potentiometer	Starting Threshold = 0.5m/s @10° displacement Delay Distance = 1.2 m Damping Ratio = 0.45	Starting Threshold ≤0.5m/s Delay Distance ≤5m Damping Ratio=0.4 to 0.7
Temperature – CSI Hygrovue 10	SHT35 Modified by CSI	Time Constant = <130 sec	Time Constant ≤1 min.
Relative Humidity – CSI Hygrovue 10	SHT35 Modified by CSI	Response Time = <8 sec	≤30 minutes
Precipitation – Texas Electronic TE525	Tipping Bucket	4.73 ml/tip (0.16 fl. oz/tip)	Time Constant - 5 min

Table A-12 PM_{10} Measurement Accuracy Criteria

Parameter	Accuracy		
	Type	Acceptance Criteria	Frequency
PM_{10} - continuous	Flow rate verification with flow standard Temperature Pressure	<±10.1% of transfer standard <±2.1°C <±10.1 mm Hg	Within 60 days of startup and 3-month intervals
PM_{10} filter based	Verification with reference standards Flow rate Temperature Pressure	<±4.1% of audit standard <±5.1% of design flow rate <±2.1°C <±10.1 mm Hg	Within 60 days of startup and 3-month intervals

Table A-13 Meteorological Measurement Accuracy Criteria

Parameter	Calibration			Accuracy		
	Type	Acceptance Criteria	Frequency	Type	Acceptance Criteria	Frequency
Wind Speed	NIST-traceable synchronous motor	±.20 m/s	6-month intervals	NIST-traceable synchronous motor	±.20 m/s	Within 60 days of startup and 6-month intervals
Wind Direction	Compass System Orientation plus linearity	±5°includes orientation error	6-month intervals	Compass System Orientation plus linearity	±5° includes orientation error	Within 60 days of startup and 6-month intervals
Temperature	3 pt. water bath with NIST-traceable thermometer	±0.5°C	6-month intervals	3 pt. water bath with NIST-traceable thermometer	±0.5°C	Within 60 days of startup and 6-month intervals
Relative Humidity	Collocated NIST-certified RH sensor	±7%RH	6-month intervals	NIST- certified RH sensor	±7% RH	Within 60 days of startup and 6-month intervals
Precipitation	Separatory funnel and graduated cylinder	±10% of input volume	6-month intervals	Separatory funnel and graduated cylinder	±10% of input volume	Within 60 days of startup and 6-month intervals

A.7.1 Representativeness of Air Quality Measurements

Site selection and probe placement followed guidelines in the following U.S. EPA documents to assure that measurements are representative of air quality and meteorological monitoring conditions at the SIMS facility:

- EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I: Principles, Volume II: Ambient Air Specific Method,
- EPA's Quality Assurance Handbook for Air Pollution Measurement Systems Volume IV, Meteorological Measurements, March 2008,
- EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Quality Monitoring Program, EPA-454/B-17-001, January 2017,
- 40 CFR 58, Appendices A, C, and E,
- 40 CFR Part 50, Appendices J
- 40 CFR Part 50, Appendix Q, Reference Method for the Determination of Lead in Particulate Matter as PM_{10} Collected from Ambient Air,
- National Air Toxics Stations (NATTS) Program Technical Assistance Document (TAD) Revision 3, and,
- EPA Compendium TO-15A.

The goal in locating monitors is to correctly match the spatial scale represented by the sample of monitored air with the spatial scale most appropriate for the monitoring site type, air pollutant to be measured, and the monitoring objective. Thus, spatial scale of representativeness is described in terms of the physical dimensions of the air parcel nearest to a monitoring site throughout which actual pollutant concentrations are reasonably similar. The scale of representativeness of most interest for the SIMS monitoring sites is microscale.

A.7.2 Data Quality Indicators

Data quality indicators provide the structure for ensuring that the project data are quality data and meet the data quality objectives presented in Section B of this QAPP. Data quality indicators are also the DQOs as defined in Part 58, Appendix A, Section 2.3. These indicators include precision, accuracy, bias, completeness, and comparability of the measurements. The calculations used to determine quality control data assessment statistics (precision, accuracy, and bias) are presented in B.5.8.1 of this QAPP. Bias, the systematic or persistent distortion of a measurement process which causes errors in one direction, is determined based on the results of the monthly flow rate verifications for PM_{10}.

The quality control data assessment statistics are calculated during the final data validation step. Concentration data are not corrected based on the results of the data assessment statistics.

The data recovery goal for the monitoring project is 85% per calendar quarter for PM_{10} and VOC samples and 90% for meteorological measurements. Higher data recovery rates are anticipated. The calculation of percent valid is based on the number of valid measurements as compared to the number of possible measurements.

The SIMS monitoring sites were selected to be as representative as possible to the general region of interest. Placement of the monitor considered local interferences, distance to structures, trees, and roadways, and height of probe above ground, and available line power. The stations will be set up in accordance with EPA-defined ambient air quality and meteorological siting criteria.

For comparability purposes, siting, equipment specifications, monitoring methods, and data validation and reporting procedures will be in accordance with EPA guidelines. Data will be reported in standard units. PM_{10} data will be reported at standard temperature and pressure.

A.7.3 Special Training/Certifications

Trinity and SIMS personnel assigned to the ambient air quality and meteorological monitoring activities will be thoroughly trained by the Trinity Consultants Project Director or his designee in the proper operation, calibration, and maintenance of the equipment to ensure continued collection of valid, representative data. Training typically consists of "hands-on" training overseen by the Trinity Consultants Project Director or a supervisor proficient in the equipment or data collection and validation activities. Training resources may also be procured from outside of the company, EPA's AMTIC website, or the Air Pollution Training Institute (APTI). Trinity and SIMS staff members are encouraged to seek additional training that is relevant to their work.

The Trinity Consultants Project Director will document the type of training conducted and when the training was performed. This documentation is kept in Trinity's personnel file by employee. These personnel have met the educational, work experience, responsibility, and training requirements for their position. Ambient air monitoring professionals with several years of experience will have responsibility for conducting the significant quality control and quality assurance activities on site.

Should a change in quality assurance requirements be identified, the Trinity QA Director will schedule a meeting with the project staff and will review the proposed QA changes to be made. The Trinity Consultants Project Director will be responsible for overseeing the implementation of any QA-related changes. If additional training is required, the Trinity Consultants Project Director or his designee will perform the training or will authorize project participants to obtain the training from an outside vendor, if needed. In these meetings, an agenda will be prepared and will include the QA requirement changes, differences between current practices, and a discussion on the QA requirement implementation. All QA-related training will be documented, implemented, and communicated to all team members as changes are made.

When any technical changes to the project are made, the SIMS Environmental Manager will be notified in writing. An SOP will be prepared by Trinity Consultants addressing the change. The SIMS Environmental Manager will distribute the SIMS staff personnel involved with the project. Trinity Consultants Project Director will send change to EPA notifying of intent to modify the QAPP. EPA will review and accept comments, as desired.

Trinity Consultants air quality monitoring professionals will have the responsibility for conducting the significant quality control and quality assurance activities on site. Sim's personnel will be trained by Trinity monitoring personnel in the proper operation, flow/leak checks, and maintenance of the equipment. All project personnel will be required to read this QAPP and applicable Standard Operating Procedures (SOPs) prior to the start of the sampling program as part of their training.

A.8 Documents and Records

The ambient air quality and meteorological monitoring program is committed to fully documenting all activities related to data collection, analysis, validation, and reporting. Table A-14 contains a list of the records maintained by the air monitoring program. An electronic logbook (E-logbook) will be used for this monitoring program. All originals and finished products will be retained by Trinity Consultants including QC forms, corrective action forms, and email exchanges used to validate data. Electronic records will be stored on Trinity's redundant network file system and archived in a mirrored data center in Dallas. Copies of the electronic logbook will be kept by Trinity QA personnel and will be included as part of Trinity's project specific file.

Table A-14 Documentation and Reports

Category	Record/Document Types	Archive Location	Retention Period
Management and Organization	Personnel qual. and training Training certifications Support contracts	Trinity Salt Lake City and Dallas Offices, SIMS	> 5 years
Site Information	Site Description Site Maps Site Pictures	Trinity Salt Lake City and Dallas Offices, SIMS	> 5 years
Data Operations	QA Project Plan Standard Operating Procedures Electronic Logbook (One-Note) Email Records Related to Data Collection Maintenance and Repair Records	Trinity Salt Lake City and Dallas Offices, SIMS	> 5 years
Raw Data	Routine and QC Data Data spreadsheets	Trinity Salt Lake City and Dallas Offices	> 5 years
Data Reporting	Summary Data Reports	Trinity Salt Lake City and Dallas Offices, SIMS	> 5 years
Data Management	Data Archiving Equipment Repair Records (E-Logbook) Data Validation Notes	Trinity Salt Lake City and Dallas Offices	> 5 years
Quality Assurance	Corrective Action Reports/Response Performance Audit Reports	Trinity Salt Lake City and Dallas Offices, SIMS	> 5 years

As mentioned above, an electronic logbook will be used for this project. This logbook system has adequate levels of security and administration to ensure e-logbook data cannot be tampered with and have adequate levels of backup (i.e., frequency and multiple storage locations). Personnel entering or editing information are uniquely identified and have been given authority to enter/edit. Every logbook entry/edit is date/time stamped and the entry person identified. Original entries are recorded and archived. Initial entries are not erased when revisions (edits to previous entries in a different entry session) are made. This ensures an audit trail is available for all entries. This logbook conforms to EPA guidance as found in the Technical Note - Use of Electronic Logbooks for Ambient Air Monitoring, April 20, 2016.

Electronic records will be stored on SIMS and Trinity password protected computer networks. Copies of the data will also be archived at Trinity's Salt Lake City office. At Trinity, all project files are backed up daily. In addition, at Trinity, weekly network backup occurs. The weekly backup network files are replicated onto cloud-based storage devices at Trinity's Dallas data center. All original data will be maintained by Trinity Consultants.

This monitoring QAPP will be made available to all concerned parties via a PDF attachment to an email. Trinity's QA Officer will be responsible for distribution and revisions to the QAPP, when necessary. The QAPP and SOPs are archived in the project-specific and SOP directories which are backed up daily and weekly with copies stored in Salt Lake and at Trinity headquarters in Dallas, Texas. The Trinity assigned project data manager is the records custodian. Access to project files and data are restricted to only Trinity and SIMS personnel involved with the project.

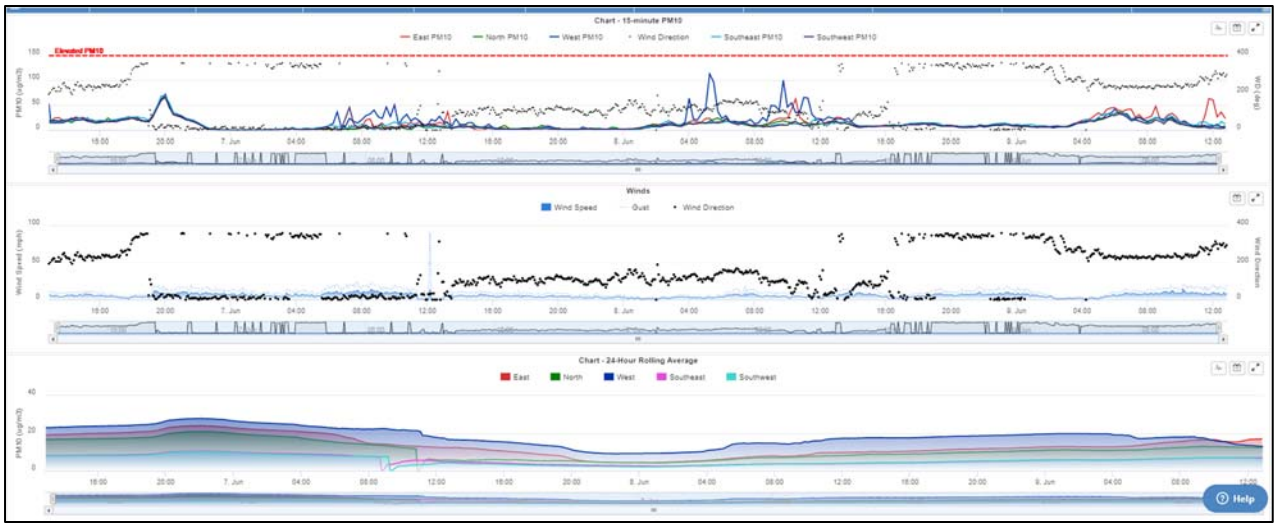
Primary data collection for the air quality and meteorological monitoring equipment will be accomplished using CSI CR1000x data loggers. Continuous PM_{10} and meteorological data will be stored in the data logger's memories as 1 minute, 5 minute, 15 minute, and hourly averages in $\mu g/m^3$ for PM_{10}. Data management will be accomplished by remotely interrogating each monitoring site from Trinity's Salt Lake City office at least every five minutes or more often to maximize data recovery and identify problems in a timely manner.

Trinity will host a password-protected project website which would be updated after every successful download. This website will only be accessible to limited project personnel. The site will contain current PM_{10} sampler and meteorological chart graphics, daily minimum, maximums, and averages, and quality assurance station notes. Historical data will also be available for review from the web site.

Stacked parameter plots and other informative graphics will be generated which consist of every data point and reviewed by a Trinity Consultants Data Manager for consistency and possible problems. These data will be reviewed daily to determine if the measurements appear normal as well as identify instrumentation problems in a timely manner. Examples of the website graphics are presented as Figures A8 and A9.

Figure A.8 Example Website PM_{10} Graphic with Wind and Pollutant Roses

Figure A.9 Example Website Stacked Parameter Plot



A summary report will be compiled by Trinity and submitted to SIMS with written and electronic data submittal no later than 21 days of the end of each month of monitoring and SIMS will coordinate reporting to EPA within 30 days. The following data and quality assurance results will be contained in the summary report:

- Monthly printouts with valid hourly and daily averages.
- Reports on performance audits
- Monthly percent data recovery by parameter.

- Results of calibration (by make, model, and serial number for the analyzer and reference calibration equipment) and quality control checks.
- Problems and corrective actions/resolved.
- Analytical laboratory results.

The QA Project Plan is a key component of the quality system developed for a project. The QA Project Plan is intended to ensure that the data an agency uses in its decision-making process has known and documented quality. The QA Project Plan and standard operating procedures (SOPs) prepared and submitted with the plan are updated when significant changes are made to the monitoring equipment or methodologies utilized by the QA Director. The most current QA Project Plan and SOPs, as evidenced by the revision number and date of each document, will be reviewed, and updated. Each original document will be maintained by Trinity Consultants; revised versions will be updated with the next sequential revision number and current date.

The QA project plan, as well as all contract required documents, are reviewed for accuracy and completeness by the Trinity Consultants Project Director and SIMS personnel. Sim's personnel will review and approve all contract deliverables before deliverables are submitted to the appropriate agency as final documents. This review ensures that the contract technical and quality goals are met.

QAPP revisions will be forwarded to the individuals on the distribution list in electronic or hard-copy format. Trinity's QA officer will be responsible for QAPP distribution. Trinity Consultants and SIMS will retain all records for a minimum of five (5) years.

B. MEASUREMENT AND DATA ACQUISITION

This section describes the project design and implementation of the SIMS ambient air quality and meteorological monitoring project, including sampling methods, sample collection, data handling and analysis, quality control requirements, equipment testing, inspection, maintenance, and managing and validating the data.

B.1 Sampling Process Design

The primary purpose of the monitoring program is to collect site-specific continuous, and filter based PM_{10}, meteorological, Pb, VOC, and other speciated compound information to satisfy EPA's 114(a) data request. The proposed PM_{10}, meteorological, and VOC sampling equipment to be operated at the SIMS facility will fulfil the 114(a) monitoring requirements.

SIMS will ensure that monitoring is performed according to regulatory and project requirements by contracting with experienced and knowledgeable contractors, reviewing the deliverables from the contractor, and ensuring the QAPP is followed.

Probe siting information and site configuration for the monitoring are in accordance with 40 CFR Part 58, Appendix E where possible. Each sampler and sensor produce a signal transmitted to the data acquisition system where it is digitized and converted to engineering units and stored in electronic memory. Accessibility and site security were also considered in the placement of the monitors. All measurements described in this QAPP are critical to achieve project objectives.

Site information with a map of the monitoring site is presented in Section A.6.

B.2 Monitoring Equipment and Methods Description

This section summarizes the instrumentation to be used at the SIMS monitoring sites. The operating range of the monitor brackets the range of environmental conditions expected at the site. The proposed equipment manufacturer and model numbers are outlined in Table A-4. The standard operating procedures followed to calibrate and operate the equipment listed in Table A-4 are presented in Table B-2.

There will be one data acquisition system (DAS) at each monitoring station (AQ2 – AQ5) to collect the PM_{10} data and one DAS at AQ1 to store the PM_{10} and meteorological data. The DAS' at AQ2 – AQ5 will be mounted to each PM_{10} tripod in an enclosure. At AQ1, the DAS will be housed in an enclosure on the meteorological tower. A precipitation gauge will be installed near the base of the meteorological tower. Lightning protection measures are also installed on the meteorological tower.

Each PM_{10} analyzers intake will be located approximately 2 meters or 6.5 feet above ground level. The distance of the inlet of the PM samplers to the adjacent buildings will be at least twice the differential from the inlet to the top of the building *where possible*.

B.2.1 Particulate Monitoring Equipment Description

Continuous PM_{10} monitoring will be conducted at five locations using near-reference monitors. At three locations, the PM_{10} samplers will be configured to collect filter-based samples in accordance with EPA's 1-in-3-day sampling schedule. A description of the air quality monitoring equipment to be utilized at the SIMS monitoring stations is discussed below. Full specifications for the monitoring equipment can be found in Appendix A.

B.2.1.1 Near-Reference PM_{10} Monitors

Continuous PM_{10} monitoring will be conducted at five monitoring sites (AQ1, AQ2, AQ3, AQ4, and AQ5) at the SIMS Facility utilizing Met One Instruments, Inc. E-Sampler compact monitoring stations. The E-Sampler is a dual technology instrument that combines the unequaled real-time measurement of light scatter with the accuracy standard of filter methods. The simple filter loading process is a seamless blending of both technologies. Filters can be extracted and replaced in less than one minute, and the filter medium can be selected based on laboratory analysis. Particulate loading on the filter does not reduce performance due to the Met One actual flow control protocol. Ambient temperature and pressure are measured, and actual flow is calculated and controlled by the E-Sampler microprocessor independent of filter loading change.

The E-Sampler is a near-reference monitor which provides real-time particulate measurement through near-forward light scattering. An internal rotary vane pump draws air at 2 liters per minute (LPM) into the sensing chamber, where it passes through visible laser light. Particles in the air scatter light in proportion to the particle load in the air. The scattered light is collected by precise glass optics and focused on a PIN diode. Rugged state-of-the-art electronics measure the intensity of the focused light and output a signal to the CPU. The output is linear to concentrations greater than 65,000 micrograms per cubic meter ($\mu g/m^3$).

The sharp cut cyclone is a precision engineered component fitted to the dust meter inlet that physically selects particles 10 microns in diameter and below. This ensures precise measurement of only the PM_{10} size fraction. The inlet is fitted with a heater that is used to remove moisture from the incoming sample. Moisture can reduce the accuracy of optical measurement, so for best results the inlet heater is activated in the event of high humidity.

Each E-Sampler will be equipped with a Campbell Scientific CR1000X data logger to collect and store PM_{10} and flow information as well as other important instrument status readings. All PM_{10} data will be collected and stored in the units of $\mu g/m^3$, which is consistent with units for the 24-hour National Ambient Air Quality Standards (NAAQS) for PM_{10}.

B.2.1.2 Filter-Based Sampling

At three locations (AQ1, AQ2, AQ5), PM_{10} E-samplers will be operated utilizing the second channel of the sampler specifically designed to accept 47-mm filters. The sampler accommodates a single quartz fiber 47-mm diameter filter and requires manual operation. There is no timer feature to start and stop the sample, therefore field technicians will exchange filters on a routine basis during daylight operating hours. The filter samples will be collected in accordance with EPA's 1-in-3-day sampling schedule and PM_{10} filters will be collected for gravimetric mass and laboratory metals evaluation.

Exposed 47-mm quartz fiber samples will be retrieved, placed in cold storage, and shipped to the laboratory for post-weighing and further metal HAP analysis. In the E-Samper, a sample stream passes through filter cassettes containing a 47 mm diameter sample filter. A mass flow controller downstream of the filter controls the flow rate at a constant volumetric level. The sampler is configured to collect samples continuously, and field personnel will install and retrieve sample filters at a specific time each sample day to provide for a 24-hour sample every three days in accordance with the schedule adopted by EPA.

B.2.2 Volatile Organic Compound Monitoring Equipment

Speciated VOC measurements will be collected using six-liter SUMMA® canisters connected to a flow controller and analyzed for specific compounds using EPA Compendium Method TO-15A. Each sampler will be outfitted with a silonite coated flow control valve to control the sample flow rate into the canister for a 24-hour integrated sample with some negative pressure remaining in the canister at the end of the period. Canister samples will be collected intermittently, according to EPA's 1-in-3-day monitoring schedule at three sites (AQ1, AQ2, AQ5), which were considered priority locations due to the proximity of nearest residential areas.

B.2.3 Meteorological Monitoring Equipment

SIMS will install a 10-meter tower to continuously measure the following parameters at the site:

- Wind speed at 10 meters,
- Wind direction at 10 meters,
- Temperature at 10 meters,
- Relative humidity at 10 meters, and
- Precipitation (near ground-level).

A brief description of each meteorological sensor is presented below. Specification sheets for each meteorological sensor is presented in Appendix A.

B.2.3.1 Wind Speed and Wind Direction

The R.M. Young Model 05305 Wind Monitor AQ, to be used at the 10-meter level, is made of UV-stabilized plastic with stainless steel and anodized aluminum fittings. Precision grade, stainless steel ball bearings are used. Transient protection and cable terminations are in a convenient junction box.

The wind speed sensor is a four-blade helicoid propeller. Propeller rotation produces an AC sine wave voltage signal with frequency directly proportional to wind speed. Slip rings and brushes are eliminated for increased reliability. The starting threshold is 0.4 m/s.

The wind direction sensor is a rugged yet lightweight vane with a sufficiently low aspect ratio to assure good fidelity in fluctuating wind conditions. Vane angle is sensed by a precision potentiometer housed in a sealed chamber. With a known excitation voltage applied to the potentiometer, the output voltage is directly proportional to vane angle. A mounting orientation ring assures correct alignment of the wind direction reference when the instrument is removed for maintenance. The vane starting threshold is 0.5 m/s at 10 degrees displacement.

B.2.3.2 Relative Humidity and Temperature

The Campbell Scientific Hygrovue relative humidity (RH)/temperature probe is designed for rugged, accurate air long-term, unattended applications. It includes a proprietary coating on the RH element that increases the life of the element and protects it from dirt, dust, salt, or other contaminants. The relative humidity sensor has an accuracy of ±1.8% from 0 to 80% RH and± 3.0% RH, from 90 to 100% RH.

B.2.3.3 Precipitation

For precipitation measurements, a Texas Electronic model TE525 tipping bucket rain gage is proposed. The precipitation is funneled into a bucket mechanism that tips when filled to a calibrated level. A magnet attached to the tipping mechanism actuates a switch as the bucket tips. The momentary switch closure is counted by the pulse-counting circuitry of a Campbell Scientific datalogger. The accuracy of the gauge is 1.0% up to 2 inches per hour.

B.2.3.4 10-Meter Tower

All proposed meteorological sensors will be secured to a Campbell Scientific Model UT30 10-meter guyed aluminum tower. The UT30 includes a mounting base secured in concrete. Lightning protection will be mounted to the tower. The tower tilts down to ground level which eliminates the need to climb the tower for servicing.

B.2.4 Telecommunications

Sierra Wireless RV50x modems with Verizon wireless 5G connections will be utilized for remote access to the equipment to download data and check the status of on-site equipment. The site will be securely available via a password protected static IP address and be available for data collection and interrogation 24/7/365. A fiberglass, NEMA4 enclosure will house the dataloggers and affiliated communication equipment.

B.2.5 Data Acquisition System

PM_{10} and meteorological data will be stored on Campbell Scientific Inc. Model CR1000x data acquisition systems. The CR1000x is a versatile data acquisition system which offers the following features: 1) user programmable options; 2) built in data instruction set; 3) large internal data storage; 4) solid state data memory modules; 5) Ethernet interface; 6) PC compatibility; 7) built-in surge protection; and 8) ability to control external devices. The DAS is capable of being polled locally through a RS232 connector or remotely via an internet connection. An on-site display will allow users to view current values of the parameters being measured.

B.2.6 Standard Operating Procedures

Standard Operating Procedures (SOPs) have been developed to provide instructions to the site operators regarding routine operation of the air quality and meteorological monitoring equipment. These SOP's range from inventory of equipment, equipment inspection and acceptance testing, visual inspections, preventive maintenance, and sampler operation. These SOPs were developed from the information presented in the manufacturer's manuals. The original and copies of the SOPs will reside on Trinity's servers.

Routine maintenance will be performed on the PM_{10} and meteorological monitoring equipment to ensure that the monitoring equipment is operating properly, and data are accurate. The type and frequency of the maintenance to be performed will be in accordance with manufacturer recommendations. Supplies will be retained and stored in Trinity's ambient laboratory in a secure area or at the SIMS facility.

Conditions adverse to quality will be identified promptly by the Trinity Data Manager or site operators and the Trinity Consultants Project Director will be notified. Once an issue that is averse to quality has been identified, the Trinity Consultants Project Director or his designate will troubleshoot the issue to identify the cause and the issue will be corrected as soon as possible. A Corrective Action Report (CAR) (Appendix B) which includes the date and time when the problem was identified, the proposed corrective action to resolve the issue, and the date and time of the results of the proposed action will be initiated. The Trinity Consultants Project Director will copy the CAR to the Trinity Consultants QA Director and SIMS Environmental Manager. The final CAR, indicating how a problem was verified resolved, will be provided to SIMS Environmental Manager and Trinity Consultants QA Director. The Trinity Consultants QA director and SIMS Environmental Manager are responsible for verifying that the corrective action that was taken was satisfactory.

Table B-1 presents the SOPs used for this program. Copies of these SOP's can be found in Appendix C.

Table B-1 Standard Operating Procedures

SOP No.	Revision No. and Date	SOP Title	Regulatory Citation
SOP 69	Rev 6. 07/28/2022	Calibration and Audit Equipment Certification	[1]
SOP 106	Rev 3. 12/07/2020	In-House Calibration of Test Equipment	[1]
SOP 107	Rev 2. 12/07/2020	Equipment Inventory Procedure	NA
SOP 117	Rev 4. 10/26/2020	Computer Program Validation	NA
SOP 193	Rev 1. 06/09/2022	Sub-atmospheric Pressure Canister Sampling – Time-Integrated Samples Using Simple Timer	[5]
SOP 222	Rev 0. 06/10/2022	Met One E-Sampler Operation	[1,2,3]
SOP M11	Rev 0. 10/27/2020	Wind Direction and Wind Speed Sensor Operation	[4]
SOP M13	Rev. 0 10/27/2020	Temperature Sensor Operation	[4]
SOP M14	Rev. 0 12/11/2020	Precipitation Gauge Operation	[4]
SOP M15	Rev. 0 10/27/2020	Relative Humidity Sensor Operation	[4]

[1] Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. II: Ambient Air Quality Monitoring Program.

[2] 40 CFR Part 50, Appendix J.

[3] Manufacturer equipment manual.

[4] Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. IV: Meteorological Measurements Version 2.0 (Final).

[5] Technical Assistance Document for the National Air Toxics Trends Stations Program, Revision 3

B.3 Sample Handling and Custody

PM_{10} filters to be used for sampling will be provided by Pace Analytical. These filters will be numbered, and pre-weighed at Pace prior to shipment. After weighing, the filters will be shipped to the field accompanied by a Pace Analytical chain-of-custody (COC) form. The site technician will receive the filters and do a visual inspection of the filters to ensure there are no pinholes, chaff, loose material, separation, discoloration, or filter non-uniformity Any filters that do not pass the visual inspection check will be set aside and not utilized for sampling. A quality control check of all data related to the filters will be performed, and relevant data will be input into the sample run log sheets for post-processing of the data.

Filters will be deployed within 30 days of pre-sample weighing date. Filters will be chilled upon retrieval from the sampler(s) and shipped to the analytical laboratory by the site technicians for analysis. All filters will be shipped and maintained below average ambient temperature (or at 4°C or below if average ambient sampling temperatures are <4°C) from sample end date to arrive at the analytical laboratory for pre-weigh conditioning within 30 days of exposure. The analytical laboratory will record the temperature of the thermometer provided by the laboratory with the shipment when received prior to post-sampling analysis.

The filter analysis procedures will take place in a controlled lab setting at Pace for both pre- and post-sample weight determinations. Each filter will have a specific serial number which can be found on both the glassine and outer filter envelopes.

Measurement of these VOCs is based on the techniques described in EPA Compendium Method TO-15A, which describe collection of whole air samples into evacuated stainless-steel canisters followed by preconcentration of the volatiles for analysis via GC/MS. EAS Lab will provide, clean, evacuated 6-L stainless steel SUMMA canisters for use in the 1-in-3-day VOC sampling. The canisters will be accompanied by a COC form and initial pressure readings will be taken by the site technician and documented prior to a sampling event. The site technicians will fill out the provided COC forms and ship the exposed canisters directly back to the laboratory within two weeks after sampling for analysis.

Copies of a Pace Analytical (formerly IML) and EAS chain-of-custody (COC) forms can be found in Appendix D. Pace Analytical PM_{10} laboratory analysis and COC procedures are discussed in full detail in its Quality Assurance Project Plan for Laboratory and Data Management Support of the Determination of Fine Particulate Matter as PM_{10} and $PM_{2.5}$ in the Atmosphere. The Pace QAPPs are included in Appendix D. A copy of the EAS Laboratory QAPP and laboratory procedures is also presented in Appendix D.

Continuous PM_{10} and meteorological data will be remotely accessed and downloaded via internet connection.

B.4 Analytical Methods

As described in Section B.2, the sampling and analytical methods to determine ambient concentrations of PM_{10} and meet the requirements of 40 CFR Part 50 and 53.

The analytical balance(s) to be used by Pace Analytical for the gravimetric determination of filter based PM_{10} concentrations will meet the specifications identified in 40 CFR Part 50, Appendices J and L. Temperature and humidity conditions in the weighing room will be monitored and recorded. Laboratory QC checks, frequencies, acceptance criteria, and sample filter conditioning procedures are outlined in the analytical laboratories QAPP.

Lead concentrations from the filter-based sampling will be determined according to 40 CFR Part 50, Appendix Q. Lead concentrations will be determined per the requirements of 40 CFR Part 50, Appendix Q, reference Method for the Determination of Lead in Particulate Matter as PM_{10} Collected from Ambient Air. The Pb content of the PM_{10} sample will be analyzed by energy-dispersive X-ray fluorescence spectrometry (EDXRF) by Pace.

Metal HAP concentrations from the filter-based sampling will be determined utilizing Section 4.4.11 PM_{10} Metals Analysis by ICP/MS – EPA IO 3.5 within NATTS Program TAD Revision 3. The 1-in-3-day samples collected will undergo laboratory analysis by Pace for the determination of metal HAPs (antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, and selenium) following Compendium Method IO-3.5.

VOC samples will also be collected at the AQ1, AQ2, and AQ5 monitoring locations in accordance with EPA's 1-in-3-day sampling schedule. Canisters will be analyzed using EPA Compendium Method TO-15A. The VOCs and other specified compounds to be analyzed for by EAS are presented in Table B-2. Laboratory QC checks, frequencies, acceptance criteria, and sample conditioning procedures are outlined in the analytical laboratories QAPPs.

Table B-2 VOCs and Other Specified Compounds to be Determined

Compounds	
Acrolein	Methanol
Acrylonitrile	Methyl bromide (bromomethane)
Benzene	Methyl chloride (chloromethane)
Benzyl Chloride	Methyl isobutyl ketone (4-methyl-2-pentanone)
Bromoform (tribromomethane)	Methyl methacrylate
1,3-Butadiene	Methyl tert-butyl ether
Carbon disulfide	Methylene chloride (dichloromethane)
Carbon tetrachloride (tetrachloromethane)	Styrene
Chlorobenzene	1,1,2,2-tetrachloroethane
Chloroform (trichloromethane)	Tetrachloroethene
1,2-dibromoethane	Toluene
1,4-dichlorobenzene	1,2,4-trichlorobenzene
Dichlorodifluoromethane (Freon 12)	1,1,1-trichloroethane
1,1-dichloroethane	1,1,2-trichloroethane
1,2-dichloroethane	Trichlorofluoromethane (Freon 11)
1,1-dichloroethene	1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)
1,2-dichloropropane	Trichloroethene
1,2-dichlorotetrafluoroethane (Freon-114)	Vinyl acetate
1,4-dioxane	Vinyl bromide
Ethyl chloride (chloroethane)	Vinyl chloride (chloroethene)
Ethylbenzene	m-xylene
Hexachloro-1,3-butadiene	p-xylene
Hexane	o-xylene

B.5 Quality Control Requirements

This section describes the routine quality control procedures used for the ambient air quality and meteorological monitoring program. All procedures have been specifically designed to provide the appropriate quality control and ensure that valid data recovery meets or exceeds the data recovery requirements of 85 percent of PM_{10} concentrations and 90% for meteorological data.

The air quality monitoring program will follow the quality control guidelines as stated in the following documents:

- EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I: Principles, Volume II: Ambient Air Specific Method,
- EPA's Quality Assurance Handbook for Air Pollution Measurement Systems Volume IV, Meteorological Measurements, March 2008,
- EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Quality Monitoring Program, EPA-454/B-17-001, January 2017,
- 40 CFR 58, Appendices A, C, and E,
- 40 CFR Part 50, Appendices J
- 40 CFR Part 50, Appendix Q, Reference Method for the Determination of Lead in Particulate Matter as PM_{10} Collected from Ambient Air,
- National Air Toxics Stations (NATTS) Program Technical Assistance Document (TAD) Revision 3, and,
- EPA Compendium TO-15A.

Table B-3 presents quality control procedures and frequency.

Table B-3 Quality Control Procedures

Procedure	Frequency	Requirement
1. Visual Inspection of Equipment	Routine or Emergency Site Visits; Monthly by site operator	Meets MQO
2. Remote interrogation of monitoring station and inspection of data	Daily	QC Checks for data screening
3. Routine calibration	PM_{10} – Quarterly Meteorological – Semi-Annually	Meets MQO
4. Calibration reference standard certification	Annually for meteorological and PM_{10}	NIST-traceable or A2LA if applicable
5. Flow/leak checks	Monthly	Meets MQO
6. Equipment Maintenance	Monthly	Section B.6
7. Personnel Training	On-going	Trinity SOP 109
8. Data Validation	Daily and monthly Monthly	Electronic data screening Time/Parameter Plot visual check Data processing calculation check Missing data periods confirmed. Off-line periods confirmed. Data validation checklist
9. Performance Audits	Within 30 days of monitoring start and then every 180 days, 5-6 months apart	Section C.1
10. Filter-Based PM_{10} and VOC Samples	1-in-3-day	Meet MQO

B.5.1 Visual Inspection of Equipment

A site technician will visit the sites as least every 3 days to check that the PM_{10} monitors are operational and recording concentrations typical for the environment. Flow checks will be performed monthly. Visual inspection of the meteorological tower will also be performed monthly. Maintenance will be performed as needed. A site check form will be completed during each site visit. A sample Site Check Form is found in Appendix E.

B.5.2 Remote Interrogation of Monitoring Station and Inspection of Data

The DAS' at the SIMS monitoring sites will be interrogated daily via internet connection to download and process the data. Abnormal data values or problems will be reported as soon as possible to the SIMS Environmental Manager and Trinity Consultants Project Director who will initiate corrective action and determine if a special site visit is required.

Computerized and visual inspection of the PM_{10} and meteorological data will be performed daily using an outlier program. Values that fall outside of prescribed limits will be evaluated by a Trinity Consultants Data Manager and corrections to data will be documented for use in data validation. Abnormal data values or problems will be reported as soon as possible to the Trinity Consultants Project Director who will initiate corrective action and determine if a special site visit is required. Should corrective action be necessary, the Consultants Project Director and QA Director may initiate the process by preparing a Corrective Action Form to document the issue, time of discovery, the affected measured parameters, and the recommended course of action. The Consultants Project Director will notify project participants via email or telecommunications of any planned corrective action that cannot be immediately resolved and may result in data loss.

The Trinity Consultants Project Director will be responsible for verifying that corrective actions are appropriate and were performed correctly and in a timely manner. The Trinity Consultants Project Director is responsible for maintaining and tracking the Corrective Action Form to document completion of work. All final Corrective Actions Forms will be signed by the Consultants Project Director once all work is completed. The QA Director will review these forms to ensure corrective action was satisfactorily completed. Copies of the completed Corrective Action Forms will be included in the monthly data summaries prepared for this monitoring program.

B.5.3 Equipment Calibration

The purpose of a calibration is to establish a relationship between standard conditions and an instruments response. The term "calibration" means an adjustment – in either the instrument or software. Multi-point verifications are considered "checks without correction" (i.e., no adjustment) and are used to ensure the instruments are within their respective calibration tolerances. Generally, if the instrument is within its established calibration tolerances, adjustments do not need to be made.

The PM_{10} sampler will be calibrated at installation, quarterly, when changes are made to an analyzer or monitor, anytime the equipment is moved or repaired, or when other maintenance procedures require it. The calibration criteria for PM_{10} are 3 of 4 calibration points within $<\pm 10.1\%$ of design. Meteorological equipment will be calibrated semi-annually.

B.5.4 Calibration Reference Standard Certification

Reference standards used for calibration of the PM_{10} sampler and meteorological sensors will be certified annually and will be traceable to National Institute of Standards and Technology (NIST) standards. Calibration certificates are kept on file at Trinity's office and are included with each calibration report. Reference standards will be certified over the ambient measurement range expected at the SIMS monitoring stations.

B.5.5 Establishing Gravimetric Correction K-Factor for E-Samplers

One of the most important uses for the 47 mm filter system is determination of a gravimetric K-factor (slope multiplier) to correct the E-Sampler signal to compensate for local particulate characteristics. To establish the K-factor, a filter disc will be carefully weighed on a microbalance scale, and then placed into the E-Sampler filter holder and run for approximately 3 days. The filter is then reweighed by the analytical lab, and the resulting total mass of the dust on the filter is correlated with the volume of air sampled and compared with the concentrations that the E-Sampler recorded over the same time period.

To calculate the K-Factor, the following equation will be utilized:

$$\text{K-Factor} = \frac{47\ mm\ filter\ total\ concentration}{E\text{–}Sampler\ Total\ Light\ Scatter\ Concentration}$$

The K-factor will be determined prior to commencement of sampling and annually on all E-samplers.

B.5.6 Automatic Zero and Span-Tests for the Optical System of E-Sampler

To assure stable concentration data, the E-Sampler performs automated optical system zero and span self-tests on a daily schedule. A separate zero air pump activates and circulates clean air through the optical system. The E-Sampler filters the air through a 0.2-micron pore size, 99.99% efficient filter element before it enters the sensor. This is the purge filter located in the front panel of the instrument. The E-Sampler zeroes itself based on this clean air condition. Next, the E-Sampler activates a solenoid shutter which allows a small amount of laser light from the light trap to feed back into the detector using fiber optics. This span level is used to check the response of the detector and related electronics.

B.5.7 Flow and Leak Checks

Monthly, each continuous PM_{10} E-Sampler at sites AQ1, AQ2, AQ3, AQ4, and AQ5 will be leak tested by the site technician with a NIST-traceable reference standard to check for airflow system leaks which could affect the accuracy of the flow measurements or cause unwanted measurement biases. During each monthly flow check on the continuous E-Sampler, temperature and pressure quality control checks will also be performed with a NIST-traceable reference standard. Agreement between the measured PM_{10} flow rates and the NIST-traceable flow standard values should be within <± 7.1% of transfer standard.

For filter-based sampling at Sites AQ1, AQ2, and AQ5 which will utilize the second channel on the PM_{10} E-samplers, agreement between the measured PM_{10} flow rates and the NIST-traceable flow standard should be within <±4% of the transfer standard and <±5% of flow rate design value. If the continuous and filter-based E-Sampler flow and leak checks are not within the above listed tolerances, the site technician will alert the Trinity Data Manager and the affected data will be flagged or invalidated, and the monitor recalibrated.

For canister sampling, once vacuum is verified, a leak check is performed. A leak check may be performed by quickly opening and closing the valve of the canister to generate a vacuum in the sampling unit. The vacuum/pressure gauge in the sampling unit should be observed for a minimum of 5 minutes to ensure that the vacuum does not change by more than 0.2 psi.

B.5.8 SUMMA Canister Vacuum Pressure

EAS will send closed, capped SUMMA canisters to be used for sampling. Before the stainless-steel cap on the canisters is removed, the site technician will ensure the canister valve is closed. The site technician will remove the brass cap, attach a vacuum gauge, open, and close the valve quickly, and will document the initial canister pressure prior to each sampling event. The initial vacuum pressure must be greater than 25 inches of mercury (in Hg) for the canister to be used for sampling. Canisters with pressures less than 25 in Hg will not be used for sampling. At the end of each sampling event, the final canister vacuum will also be documented.

B.5.9 PM_{10} Filter Field Blank

At a frequency of 10% of the scheduled sampling runs, a filter field blank will be taken to the field which will be open and closed but not installed on the sampler or exposed. The filter field blank will be returned with the exposed filter samples. Each filter field blank will be labeled.

B.5.10 PM_{10} Filter Trip Blank

A trip blank, which is a PM_{10} filter that is treated exactly as a field blank but is never open or closed or placed into the sampler or exposed, will be collected at a frequency of 10% of the scheduled sampling runs. The trip blank will be used to assess possible contamination to PM_{10} filters during packing and transport to and from the laboratory to the sampling location. Each filter trip blank will be labeled.

B.5.11 Duplicate VOC Samples

Duplicate VOC samples will be collected at a 10% frequency at each VOC monitoring location. Duplicate samples will be collected for the required 24-hour sampling period and will be compared to the primary sample to determine the precision. VOC duplicate sample precision will follow NATTS recommendations of ≤25% relative percent difference for target compounds ≥five-fold the laboratory minimum detection limit (MDL).

B.5.12 Equipment Maintenance

Manufacturer's recommendations for maintenance of the PM_{10} E-sampler and meteorological sensors will be followed. Instrument instruction manuals will be available for reference of preventive and remedial maintenance procedures. Preventive and corrective maintenance will be documented on the calibration forms after any maintenance. See Section B.6.3 for equipment maintenance procedures.

B.5.13 Personnel Training

Personnel operating the ambient PM_{10}, VOC, and meteorological monitoring equipment will be thoroughly trained in the proper operation, calibration, and maintenance of the equipment to ensure continued collection of valid, representative data.

B.5.14 Data Validation

Table B-4 outlines the criteria deemed critical for PM_{10}. Data that do not meet each criterion on the critical table will be invalidated unless compelling reason or justification exists for not doing so. The samples for which one or more of these criteria are not met are invalid unless proven otherwise. The cause for not operating within the acceptable range for each violated criterion will be investigated and corrective action taken to remedy the problem such that additional data will be invalidated. The Consultants Project Director will be alerted by the Data Manager or site technician when critical criteria are exceeded. The Consultants Project Director will notify SIMS personnel when critical criteria are exceeded causing data to be invalidated.

Table B-5 presents the criteria that are important for maintaining and evaluating the quality of the data collection system. Violation of a criterion or several criteria may be reason to invalidate data. The decision to invalidate should consider other control information that may or may not indicate that these data are acceptable. Data that does not meet the criteria in Table B-4 are suspect unless other quality control information demonstrates otherwise. The Trinity Consultants Project Director will be alerted by the Trinity Data Manager or site technician when the operation criteria are not being met and the issue will be investigated, mitigated, or justified.

Table B-6 presents the systematic issues that are important for the correct interpretation of the data. These issues, however, usually do not impact the validity of the data. See Section D of this QAPP has specifics on the data validation procedures.

Table B-4 Critical Criteria for PM_{10}

Requirement	Frequency	Accept. Criteria	Reference	Action
Sampling Period	Every 24 hours of operation	1440 minutes ±60 minutes midnight to midnight	40 CFR Part 50, App. J. Section 7.1.5	Invalidate data to last acceptable check.
PM_{10} Average Flow Rate	Every 24 hours of operation	Average within <±5.1% of design	Recommendation	Invalidate data to last acceptable check.
Filter based PM_{10} one-pt. flow rate ver. (Sites AQ1, AQ2, AQ5)	Every month	±4% of transfer standard ±5% of flow rate design	40 CFR Part 50, App. L, Sec. 9.2.5	Invalidate data to last acceptable check.
Continuous and Filter based PM_{10} Variability in Flow Rate	Every 24 hours of operation	CV ≤2%	40 CFR Part 50, App. L Section 7.4.3.2	Invalidate data to last acceptable check.
Pre-sampling Filter Hold Times (filter-based samples at Sites AQ1, AQ2, AQ5)	All Filters	≤30 days before sampling	40 Part 50, App. L, Section 8.3.5	Void Sample
Sample Recovery (Filter-based samples at Sites AQ1, AQ2, AQ5)	All Filters	≤7 days 9 hours from sample end date	40 Part 50, App. L, Section 10.10	Invalidate sample.
Filter Based PM_{10} at Sites AQ1, AQ2, AQ5	Every Sample Period	1380 - 1500 minutes	40 CFR Part 50 App J Section 7.1.5; 40 CFR Part 50, App. L Section 3.3	Invalidate data to last acceptable check.
Filter Based PM_{10} Average Flow Rate at Sites AQ1, AQ2, AQ5	Every 24 hours of operation	Average within ≤5.1%	Part 50 App. L Section 7.4.3.1	Invalidate data to last acceptable check.

Table B-4 Continued Critical Criteria for PM_{10}

Requirement	Frequency	Accept. Criteria	Reference	Action
Filter Based PM_{10} One-pt. flow rate ver. at Sites AQ1, AQ2, AQ5	Every 30 days	<±4.1% of transfer standard <±5.1% of flow rate design	40 CFR Part 50, App. L Section 9.2.5	Invalidate data to last acceptable check.
Individual Flow Rates	Every 24 hours of operation	No flow rate excursions > ±5% for > 5 minutes	40 CFR Part 50, App. L Section 7.4.3.1	Invalidate data to last acceptable check.
Filter Temp. Sensor	Every 24 hours of operation	No excursions >5˚C for > 30 minutes	40 CFR Part 50, App. L Section 7.4.11.4	Invalidate data to last acceptable check.
External Leak Check	Before each flow rate verification and before and after PM_{10} separator maintenance	<80.1 mL/min	Part 50 App. L, Section 7.4.6.1	Invalidate data to last acceptable check.
Internal Leak Check	If failure of external leak check	<80.1 mL/min	Part 50 App. L, Section 7.4.6.2	Invalidate data to last acceptable check.
Continuous PM_{10} Average Flow Rate	Every 24 hours of operation	Average within <±5.1% of design	Recommendation	Invalidate data to last acceptable check.

Table B-4 Continued Critical Criteria for PM_{10}

Requirement	Frequency	Acceptance Criteria	Reference	Action
Post-sampling weighing	All filters	Protected from exposure to temperatures above 25˚C from sample retrieval to conditioning <10 days from sample end date if shipped at ambient temp, or < 30 days if shipped below avg ambient (or 4˚ C or below for avg. sampling temps < 4˚C) from sample end date	40 CFR Part 50, App. L, Section 8.3.6	No weighing conducted
Filter visual defect check	All filters	Correct type & size and for pinholes, particles or imperfections	40 CFR Part 50, App. L, Section 10.2	Flag or invalidate filter
Filters (Equilibration)	All filters	24 hours (minimum) 24-hour Temp mean: 20 to 23°C	40 CFR Part 50, App. L, Section 8.2	No weighing conducted
Temp. Control	All filters	<2.1°C SD over 24 hrs	40 CFR Part 50, App. L, Section 8.2	No weighing conducted
Humidity Range	All filters	30-40% RH or within ±5% sampling RH but ≥20.0% RH	40 CFR Part 50, App. L, Section 8.2	No weighing conducted
Humid. Control	All filters	<5.1% SD over 24 hours	40 CFR Part 50, App. L, Section 8.2	No weighing conducted
Pre-and Post-Sampling RH	All filters	≤±5% RH (mean) difference in 24 hours	40 CFR Part 50, App. L, Section 8.3.3	No weighing conducted
Balance	All filters	Located in filter conditioning environment	40 CFR Part 50, App. L, Section 8.3.2	No weighing conducted
Microbalance auto-calibration	Prior to each weighing session	Manufacturer's specification	40 CFR Part 50, App. L, Section 8.1	No weighing conducted

Table B-5 Operational Criteria for PM_{10}

Requirement	Frequency	Acceptance Criteria	Reference	Action
PM_{10} Flow Rate Calibration	Install, major maintenance/ repair, failure of criteria, or every 3 months	3 of 4 points within <±10.1% of design	40 CFR Part 50, App. L Section 9.2	Flag or invalidate data
PM_{10} Performance Audit	Within 30 days and then every 180 days, 5-7 months apart	<±10.1% of audit standard	40 CFR Part 58, App. A, Section 3.3.3	Flag or invalidate data
Inlet/Downtube Cleaning	Every 30 days	Cleaned	Method 2.10 Section 6.1.2	Flag or invalidate data
One-point temperature verification	Every 30 days	Continuous and Filter based PM_{10} <±2.1˚C	40 CFR Part 50 App. L, Section 9.3	Recalibrate
Pressure Verification	Every 30 days	Continuous and Filter based PM_{10}: <±10.1 mmHg	40 CFR Part 50 App. L, Section 9.3	Recalibrate
Lot Blanks	9 filters per lot	< ±15.1 µg Change between Weighing	Method 2.12 Section 7.7	Filters reweighed or discarded
Exposure Lot Blanks	3 filters per lot	< ±15.1 µg Change between Weighing	Method 2.12 Section 7.7	Filters reweighed or discarded
Filter Integrity (Exposed)	Each filter	No visual defects	Method 2.12 Section 10.7	Filters flagged or invalidated
Field Filter Blank	10% or 1 per weigh session	<±30.1 µg Change between Weighing	40 CFR Part 50, Appendix L, Sec. 8.3.7.1	Equilibrate and Reweigh batch
Lab Filter Blank	10% or 1 per weigh session	<±15.1 µg Change between Weighing	40 CFR Part 50, Appendix L, Sec. 8.3.7.2	Equilibrate and Reweigh batch
Balance Check	Beginning, every 10, and at the end	≤3.1 µg from Certified Value	Method 2.12 Sec. 7.9 Method 2.12 Sec. 4.5	Balance Serviced
Routine Filter Re-weighing	1 per weighing session	<±15.1 µg Change between Weighing	Method 2.12 Sec. 10.8	Reweigh Batch

Table B-5 Continued Operational Criteria for PM_{10}

Requirement	Frequency	Acceptance Criteria	Reference	Action
Microbalance Audit	Annual	<±0.003 mg or Manufacturer Specs., whichever is tighter	Method 2.12 Sec. 11.2.7	Balanced Serviced
Lab Temp. Check	Every 90 Days	<±2.1°C	Method 2.12 Sec. 4.3.8	Reweigh Batch
Lab Humidity Check	Every 90 Days	<±2.1%	Method 2.12 Sec. 4.3.8	Reweigh Batch

Table B-6 Systematic Criteria for PM_{10}

Requirement	Frequency	Acceptance Criteria	Reference	Action
Siting	1/year	Meets siting criteria	40 CFR Part 58 App. E, Sec. 2.3b&c	----
Completeness	24-hour (PM_{10})	≥75% of scheduled sampling days	40 CFR Part 50 App. K Sec. 4.1 & 4.2	----
Precision	Every 90 days	CV<10.1% for values ≥ 3.0 µg/m^3	40 CFR Part 58, App. A, Sec. 4.2.1	----
Reporting	All data	PM_{10} – ambient /STP conditions Metal HAPs – corrected to local conditions	40 CFR Part 50 App N Sec. 3.0 (PM_{10})	----
Flow Rate Transfer Standard	Annually	<±2.1% of NIST transfer standard	40 CFR Part 50, App. L, Section 9.1 & 9.2	----
Thermometer	Annually	±0.1˚C resolution, ±0.5˚C accuracy	Method 2.12 Section 4.2.2	----
Barometer	Annually	±1 mmHg resolution, ±5 mmHg accuracy	Method 2.12 Section 4.2.2	----
Lower DL	All filters	≤2 µg/m^3 (filter based PM_{10})	40 CFR Part 50 App. L Section 3.1	-----

B.5.14.1 Particulate Monitor Precision and Bias

For the particulate monitors, the Data Manger will calculate monthly precision and bias from the monthly flow checks conducted. For each precision check, the percent difference (d_i) will be calculated according to the following:

$$d_i = \frac{Y_i - X_i}{X_i} \cdot 100$$

Where: X_i is the concentration of the reference flow rate standard
Y_i is the flow rate measured by PM monitor

The bias estimate is calculated using the monthly flow checks for the particulate monitors. Bias will be calculated using the following formula:

$$|bias| = AB + t_{0.95,\ n-1} \cdot \frac{AS}{\sqrt{n}}$$

Where: n is number of monthly flow checks
$t_{0.95,\ n-1}$ is the 95th quantile of a t-distribution with n-1 degrees of freedom
AB = Mean of absolute values of the d_i's (below)

AB is calculated with the following equation:

$$\mathrm{AB} = \frac{1}{n}\sum_{i=1}^{n} \left| d_i \right|$$

The quantity AS or standard deviation of the absolute values of the d_i's is calculated as:

$$AS = \sqrt{\frac{n\ x\ \sum_{i=1}^{n}|d_i|^2\ - \left(\sum_{i=1}^{n}|d_i|\right)^2}{n\ (n-1)}}$$

Precision is estimated via duplicate measurements from the continuous and filter-based measurements using the following equation:

$$d_i\ = \frac{X_i\ -\ Y_i}{(X_i + Y_i)/2} \times 100$$

Where: X_i is the concentration from the continuous channel
Y_i is the concentration value from the filter-based sample

The coefficient of variation upper bound is calculated using the following equation:

$$CV = \sqrt{n\ x\ \sum_{i=1}^{n} d_i^2 - \left(\sum_{i-1}^{d} d_i\right)^2} \times \sqrt{\frac{n-1}{x^2_{0.1,n-1}}}$$

Where: X_i is the concentration from the continuous channel
Y_i is the concentration value from the filter-based sample

B.6 Instrument/Equipment Testing, Inspection and Maintenance

A description of the equipment testing and integration, inspection, and maintenance to be performed at the monitoring site is presented below.

B.6.1 Acceptance Testing of Instrumentation and Equipment Integration

Prior to installation, all equipment will be visually inspected to ensure there is no physical damage and will be calibrated before use. Acceptance testing of instrumentation will be performed to verify that the instruments meet the required performance specifications and anytime the instrument undergoes maintenance, repair, or any type of movement. Any equipment that fails to meet specifications will be returned to the manufacturer. Inspection testing results, calibration and operating manual checks will be documented. After installation, the particulate monitors will be checked according to the procedures presented in the operating manual for the instrument. To ensure that the monitoring equipment continues to operate properly, checks of the instruments will be conducted during each site visit or, at a minimum, monthly. Preventive maintenance and quality assurance procedures will be conducted on a routine basis. Inspection testing results, calibrations and operating manual checks will be documented.

B.6.2 Site Surveillance and System Check Procedures

At least monthly at sites AQ3 and AQ4 and weekly at sites AQ1, AQ2, and AQ5, the site technician will visit the monitoring stations to inspect and verify proper operation of the particulate sampling equipment. A visual inspection of the meteorological tower and sensors will be made monthly. The site operator will conduct monthly flow checks. During each site visit, entries will be made in the site logbook documenting anytime the site technician visits the site, all site activities conducted, and any observations while on site. If changes are made to the equipment or configuration of the system, these changes will also be entered in the site logbook. Entries will be made any time there is a change or modification in the way a sample is obtained, or the station configuration altered.

Entries will be made anytime the site technician visits the site, recording activities performed and any observations made while at the site. Any problems identified will be reported to the Consultants Project Director.

If the site technician encounters a problem which cannot be rectified, he will contact the Consultants Project Director who will be responsible for resolving the issue. The Project Director will initiate a plan for corrective action and will dispatch one of Trinity's field technicians as soon as possible.

B.6.3 Site and Equipment Maintenance

Manufacturer's recommendations for maintenance of the PM_{10} monitoring equipment will be followed. Instrument instruction manuals will be available for reference of preventative and remedial maintenance procedures. Preventive and corrective maintenance will be documented on the calibration forms after any maintenance.

Per the manufacturer's equipment specific operation manual, the maintenance activities to be performed for the particulate monitors are presented in Table B-7. Maintenance will be performed prior to calibrations, and equipment repair. Generally, calibration is performed quarterly and after instrument movement, repair, or maintenance. Calibration will include an "as found" calibration and "as left" calibration.

Table B-7 PM_{10} E-Sampler Monitoring Equipment Maintenance Activities

Maintenance Activity	Frequency
System leak check	Monthly
Flow, temperature, and pressure calibrations	Monthly
Clean sharp-cut cyclone and particle trap	Monthly
Check digital alarm log	Monthly
Clean 47 mm filter holder screen	Monthly
Check filter RH sensors	6 Months
Replace Pump filter and Purge filter	12 Months
Factory service, recalibration, and optical system cleaning	24 Months
Replace lithium backup battery, as needed	5 Years

B.6.4 Spare Parts or Supplies

Spare parts or supplies for the PM_{10} and meteorological monitoring equipment will be stored at the Chicago Trinity office and will be used as needed. These spare parts include, but are not limited to, sample line filters. Other parts are available to be delivered via overnight air express, if needed.

B.7 Instrument/Equipment Calibration and Frequency

Descriptions of the PM_{10} and meteorological monitoring equipment calibration procedures and frequency is presented below. All calibration standards used to calibrate, and check instruments are separate and distinct from those used to perform the performance audits.

B.7.1 Air Quality Equipment

Quality control checks performed on particulate monitors includes calibrations and flow checks every month. For the PM_{10} monitor, quality control checks will be performed monthly. Maintenance will be performed on the PM_{10} monitor whenever the following conditions occur:

1) Flow rate verifications are greater than ±7.1% of transfer standard for PM_{10},
2) PM_{10} temperature difference is <±2.1°C,
3) PM_{10} pressure difference is <±10 mmHg,
4) After repair or significant maintenance activities are performed on the sampler, and,
5) Every three months.

Calibration of the continuous PM_{10} samplers will be conducted at installation, every three months, after major maintenance or repair, and failure of criteria. In addition, for the continuous PM_{10}, multi-point calibrations will be performed when the monthly flow check is greater than ±7.1% of transfer standard. For the filter based PM_{10} samplers, multi-point calibrations will be performed when the monthly flow check is greater than 4.1% of the transfer standard and greater than 5.1% percent of the flow rate design value.

Calibration of the sampler consists of several procedures which include measuring the flow with a certified flow transfer standard and calculating the deviations from the inlet design and the set point flowrates. In addition to the flow check, a leak test is also performed. The "as found" and "as left" flow and leak checks will be recorded. The ambient temperature and pressure sensors are compared to calibrated reference sensors during calibration. All calibration equipment will be traceable to NIST standards which will be recertified annually against NIST traceable standards. PM_{10} calibration procedures and calibration forms are found in Appendix C.

B.7.2 Meteorological Equipment

The meteorological parameters presented below will be calibrated in accordance with guidance found in EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV: Meteorological Measurements Version 2.0 (Final). Meteorological equipment SOPs and calibration forms are presented in Appendix C. Meteorological equipment calibrations will be performed semi-annually with equipment that is in current calibration and is traceable to NIST or A2LA standards. Sensors which do not meet calibration specifications or fail a performance audit will be repaired and re-calibrated. Calibration certifications and records remain on file at Trinity's Salt Lake City office. Calibration equipment will be certified to NIST or A2LA standards annually.

B.7.2.1 Wind Direction

The cross-arm orientation will be checked using a professional compass. The wind vane will be aligned with the cross arm and set to true north. True north is distinguished from magnetic north by reading a magnetic compass and applying a correction factor for the magnetic declination. The declination will be determined from a declination calculation computer program. If the overall wind direction error (orientation plus linearity) exceeds ±5 degrees from true North, the sensor will be re-calibrated.

The wind direction sensor starting threshold will be checked using a torque gauge. The torque gauge is placed on the sensor shaft and the torque is measured. If the sensor starting threshold is greater than 0.5 meters per second (m/s), the bearings will be replaced, and the sensor will be re-calibrated.

The wind direction linearity will be checked using a direction template. The sensor response will be checked at a minimum at 30-degree increments in both clockwise and counterclockwise rotations and compared with the DAS readings. If the indicated wind direction linearity plus orientation error exceeds ±5 degrees, the sensor will be repaired and re-calibrated.

B.7.2.2 Wind Speed

Horizontal wind speed response checks will be performed using a synchronous motor. Sensor readings taken from the DAS will be compared to calibration values obtained from transfer functions provided in the sensor manufacturer's specifications. If the wind speed error exceeds ±0.2 m/s, then the instrument will be recalibrated.

The horizontal wind speed sensor starting threshold will be checked using a torque gauge or a torque disc. The torque device is placed on the sensor shaft and the torque is measured. If the measured torque exceeds manufacturer's tolerance specifications for wind speed sensor starting threshold of 0.5 m/s, then the bearings will be replaced, and the instrument will be recalibrated.

B.7.2.3 Temperature

Temperature sensor calibration will be verified by direct comparison of sensor outputs to a collocated calibrated reference standard thermometer encompassing the measurement range expected at the site. If the sensor output is more than 0.5 degrees Centigrade (°C) different from the reference, the sensor will be repaired and re-calibrated.

B.7.2.4 Relative Humidity

The relative humidity sensor calibration will be verified by comparison of station sensor outputs with a relative humidity reference sensor collocated at ambient conditions. If the site sensor output differs by more than ±7 percent relative humidity from the reference, the sensor will be recalibrated.

B.7.2.5 Precipitation

Precipitation sensor output will be verified using a standard graduated burette to add water to the gauge simulating rainfall. The volume of water required to produce ten tips will be recorded for each of three runs. This volume will be divided by the area of the gauge opening to determine the calculated amount of simulated rainfall. This amount will be compared with amounts reported by the station data logger. If the sensor differs by more than ±10% from the reference input, the sensor will be recalibrated. During calibration verification, the technician will conform that both sides of the tip bucket have similar sensitivity and provide similar, balance results.

B.8 Inspection/Acceptance of Supplies and Consumables

Field equipment supplies and consumables will be obtained either directly from the original equipment vendor, or from a scientific equipment/materials vendor whose products are proven to be equivalent in quality. Spare parts and supplies will be purchased by the Trinity Consultants Project Director. They will be inspected by a site technician for shipping damage upon receipt. Spare parts and supplies will be kept by Trinity for use when needed. The use of spare parts or supplies will be documented on calibration forms. After checking for acceptability, each item will be labeled with a date received and the item will be tracked when used by Trinity in an equipment inventory system. Invoices for the supplies/consumables will be maintained. Supplies and consumables will be used earliest in, first out. Expiration dates of supplies/consumables will be tracked where necessary. When supplies are deployed/consumed, notations will be recorded I the inventory system.

B.9 Non-direct Measurements

SUMMA canister sampling is a non-direct measurement since the VOCs are subsequently separated by gas chromatography (GC) and measured by mass-selective detector or multidetector techniques. Metal concentrations will be determined following Compendium IO-3.5 using inductively coupled plasma/mass spectrometer (ICP/MS).

The PM_{10} data collected from this monitoring program will be a direct measurement which will be used for comparison to the National Ambient Air Quality Standard (NAAQS). The current NAAQS for PM_{10} are as follows:

Pollutant	Averaging Period	NAAQS	Form
PM_{10}	24-hour	150 $\mu g/m^3$	Not to be exceeded more than once per year on average over 3 years

B.10 Data Management

The proper management of all data is critical to assuring the quality and usability of the monitoring results. As such, procedures have been implemented to ensure robust data acquisition, validation, reduction, reporting, and storage of electronic data. Air quality and meteorological monitoring data will be retrieved from the monitoring site via internet connection daily and data will be stored on CSI CR1000x data loggers. The monitoring site can be called from any computer having the correct software and the IP address. Raw monitoring data will be posted on a password-protected website provided by Trinity that will be available SIMS. This website will present the raw PM_{10} concentration data from all monitoring locations in a graphical and tabular form. Data will be available at https://mytrinitydata.com.

All electronic calculations and statistical analyses will be performed using standard software (Microsoft Excel) that can be easily verified. Formulas and equation fields will be locked so that these fields will not be overwritten. Manual calculations are routinely performed by QA personnel designated by the QA Director to verify Excel results are correct. Manual calculations will be performed when software is updated to verify that the software is working as intended and producing the intended and accurate results. All project documentation, records, data, and reports will be stored for at least five years following project completion. Data will be stored on Trinity's data center servers which are backed up nightly and are archived on and offsite to Trinity's corporate headquarters in Dallas, Texas.

Where software that is not considered public domain is used, quality control software verification is performed during software development and after software changes to ensure that the software adequately and correctly performs all intended functions. The verification tests performed demonstrate the capability of the computer program to produce valid results for test problems encompassing the range of permitted usage defined by the program documentation. Acceptable test problem solutions are:

a) Hand calculations,
b) Calculations using comparable proven programs, or

c) Empirical data and information from technical literature.

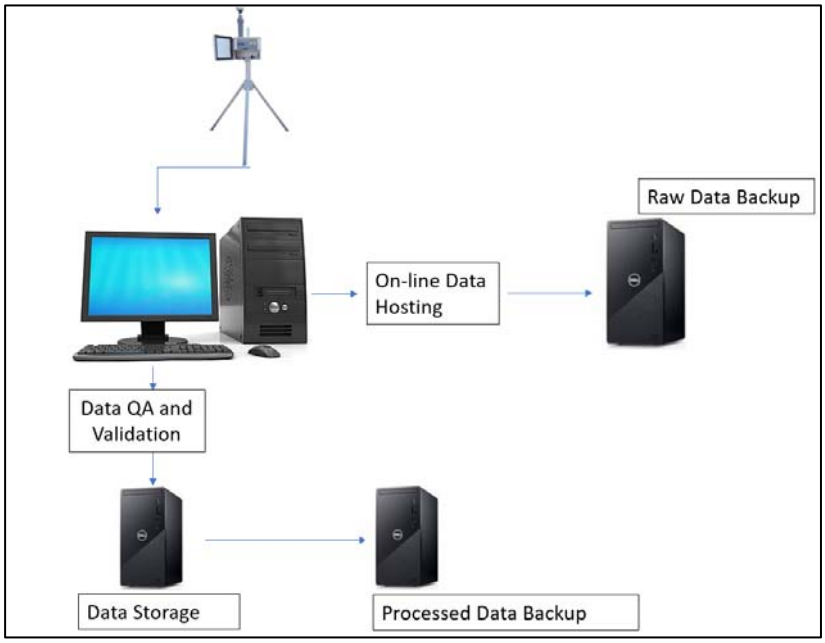
Depending on the complexity of the computer program being tested, testing can range from a single test of the completed computer program to a series of tests performed at various stages of computer program development to verify correct translation between stages and proper working order of individual modules, followed by an overall computer program test. All test results developed over the testing of the software are thoroughly documented by Trinity Consultants QA Director or designated QA staff member. Software and modifications to the software by a Trinity Consultants Data Manager are backed up on Trinity's server. Testing will be performed after software modifications/upgrades and will be made available to SIMS upon request.

Checks for data transformation, reduction, and transmission accuracy, including final transmission accuracy checks will be performed monthly. Transformation refers to analog to digital or human interpretable information (e.g., concentration data is interpretable and may be compared against NAAQS by reviewer). Reduction refers to the data management systems reducing minute, 5 minutes into hourly average, hourly data into 24-hour average. This will be accomplished by taking the average of minute/five minute/hourly data and whether it agrees with computer generated average.

PM_{10} and meteorological data will be reviewed daily by a Trinity Consultants Data Manager. These data will be subjected to several levels of quality control, validation and quality assurance as discussed in Section D.

The Trinity Data Manager will archive data on Trinity's network and on storage hard drives which are stored off-site. The overall flow of data management is illustrated in Figure B.1. The continuous and filter based PM_{10} data, metals, and VOC data will be summarized in monthly tables and reported monthly.

Figure B.1 Data Management Flow Chart



Trinity has developed and will utilize software which conducts live inspection of continuous monitoring site data. The program is called Trinity Data Scanning and Alert System (DSAS) and is used as a tool to assist Trinity data management staff identify data outliers, problems with site equipment, or site communication issues. Once every minute, Trinity DSAS scans all raw data files downloaded from the monitoring site to see if new data have arrived for interrogation.

After the data collection, Trinity DSAS conducts a computerized inspection of the data using pre-defined quality control checks. The quality control checks include data outliers, spikes in data, and data constancy. If parameters fail these tests, the parameter is flagged in the software and Trinity's staff is alerted.

A user interface for the Trinity DSAS software was developed for visual and audio cues to alert Trinity's staff when a parameter fails a quality control check. The user interface is displayed prominently as a primary tool for Trinity's data management section. The user interface displays a matrix listing all the meteorological and air quality stations in rows as well as associated parameters listed in columns. Each cell represents a parameter being measured at a monitoring site and will be colored with green, yellow, orange, or red depending on the severity of the failure. For example, a green cell indicates no problems were detected.

The outlier program does not invalidate data or erase file records based on these outlier tests. Outliers may be a onetime event but may significantly impact results (e.g, exceptional dust event). Outlier reviews will take place by the Trinity Data Manager monthly. To assist in data validation, a copy of the site logbook and the note section of the project site software will be examined to confirm periods when instrumentation may have been off-line due to power outages, maintenance or repair, or other quality assurance activities.

Continuous PM_{10} and meteorological data will be reviewed by using time-series plots. The graphs quickly reveal any data abnormalities due to power failure or instrument malfunction. Significant events will be checked against the graphs for consistency. Calibration data will be reviewed to assess the precision of the data. If the calibrations indicate invalid or low precision, data values may be invalidated, and the appropriate flags will be applied. Means, maxima, and minima for the month are computed. Especially high values will be checked to be sure that calibration data were not inadvertently included. Suspect data will be reported but flagged using AQS qualifier codes.

Laboratory analytical results received from Pace and EAS will be reviewed by a Trinity Consultants Data Manager. The lab data, provided in Microsoft Excel spreadsheets, will be compiled into a Microsoft Excel database. The review of the data packages will include an evaluation of the information provided on the analytical data sheets and support documentation for all sample analysis, and the supporting sample collection documentation, including chain-of-custody. Field data such as volume sampled, local condition parameters will be reviewed to effectively determine gravimetric and metals concentrations. The QA review will also examine adherence to the procedures as described in the requested analytical methods. Sample reanalysis may be requested of the laboratory, as needed.

Validated data are compiled into the final database for further analysis and report preparation. The final database is processed and stored on a personal computer and then archived on various storage media and maintained in duplicate in more than one location for protection.

B.10.1 Data Retrieval

Data are retrieved from the site by connecting to the DAS' via remote telemetry.

B.10.2 Raw Data

Raw data are records, notes, memoranda, worksheets, or exact copies and are the result of original observations and activities of the monitoring project. Raw data include data from the DAS' and data entered directly into a system. Raw data will be stored at each laboratory according to their respective QA manual.

B.10.3 Data Transfer

The continuous PM_{10} monitors, and meteorological tower will have an IP modem installed and a connection can be made via a cellular network. Data provided by the analytical laboratories will be in electronic and hard copy form.

B.10.4 DAS Data Review

Initial data review will be performed by the Trinity Data Manager. Daily, the average flow rate will be reviewed to verify that the CV≤2% for PM_{10}. Other items to be reviewed includes monthly flow checks, maintenance logs, hourly data, and any other information that might be vital to proper validation of the data. Information used in the review that may be used to invalidate data are input to Excel spreadsheet which contains data and time of suspect data by parameter, potential reason for data being suspect, and any pertinent comments that relate to this data value. Raw data files are never modified and are archived. Final data approval will be performed by EPA Region 5.

B.10.5 Data Validation

Data validation ensures that data processing operations have been carried out correctly and that the quality of field operations has been performed properly and in accordance with written procedures. Once data validation has identified problems, the data are flagged, or invalidated, and correction actions can be taken when necessary. In the event of an out-of-range calibration, the data manager will be responsible for checking or invalidating data. Data validation procedures are described in detail in Section D. Data will be invalidated if the critical criteria presented in Table B-3 above are not met. Data may also be invalidated if the criteria in Tables B-4 and B-5 are also not met. Data validation will be performed by Trinity QA personnel.

B.10.6 Data Transmittal

Data transmittal occurs when data are transferred from one location to another or from one person or group to another. An example of data transfer is the electronic transfer of data over a telephone or computer network. Data will be verified at regular intervals by the Trinity Data Manager to verify the operational status of the monitoring equipment. The IP address of the data capture system is verified against the IP address of the data logger. Actual checks of the data will occur to verify the data has not changed from data capture to data logger.

B.10.7 Data Processing

Data processing includes the aggregating and summarizing of results so they can be easily understood and interpreted in various ways. EPA regulations require certain summary data be computed and reported on a regular basis such as precision, accuracy, bias, etc.

B.10.8 Data Analyses

Data summary and analysis requirements, as presented in 40 CFR Part 58, Appendix A will be followed for this program. Single analyzer precision, bias, and data completeness will be tracked and reported for each monitoring station.

B.10.9 Data Flagging

Data will be flagged if a numeric result was available, but it has been qualified in some respect related to the validity of the result. Null data codes will be generated for invalid data.

B.10.10 Data Storage and Retrieval

Electronic copies of the original and processed data will be stored by at Trinity's Salt Lake City office and in Trinity's Dallas, Texas data center.

An electronic logbook system package will be utilized for the monitoring project. The e-logbook package features Microsoft One-Note. The e-logbook will keep track of all station activities including calibrations, maintenance, or any item that in the past would have been entered into a site paper logbook. The entries are automatically time stamped and cannot be deleted. Original entries are recorded and archived. The original record will be retained by One-Note. Initial entries are not erased when revisions (edits to previous entries in a different entry session) are made. Personnel entering or editing information are uniquely identified and have been given authority to enter/edit. Changes made to One-Note are traceable to the individual making the change and date stamped. A list of the personnel, their authority and access privileges are maintained by the Project Director. Personnel using the e-logbook system must be logged into the system with a unique ID so that all entries can be tracked definitively. All changes are recorded. One Note is accessible from anywhere by logging into Trinity's Microsoft SharePoint System.

E-logbook data are stored on personal computers at SIMS and at Trinity on Trinity's SharePoint server. Both the SIMS and Trinity's networks are backed up to a network storage unit daily. Data are also archived and stored at Trinity's data center in Dallas, Texas.

C. ASSESSMENTS AND OVERSIGHT

This section describes the quality assurance assessments conducted to provide oversight to the quality control protocols and ensure that the quality goals for the data are being met.

C.1 Assessments and Response Actions

For this monitoring program, performance audits will be performed after the first flow check and once every 180 days. 5 to 7 months apart. Audit results will be documented. Performance audit results will be determined using the following methodology: The sampler's flow rate accuracy (A) should be within ±4 percent of the audit value. Furthermore, the audit measured flow accuracy (A_D) should be within ±5 percent of the sampler's design inlet flow rate. The sampler's flow rate accuracies (A and A_D) are calculated as follows:

$$A\ (\%) = \frac{Q_{sampler} - Q_{Audit}}{Q_{audit}} \times 100$$

$$A_D\ (\%) = 100 \times (Q_{audit} - 2.0)/2.0$$

Where:

A = flow rate accuracy (percent)
AD = flow rate accuracy (percent) versus design flow rate
$Q_{sampler}$ = flow rate as measured by sampler (L/min)
Q_{audit} = flow rate measured by the flow rate transfer standard (L/min)
2.00 = design flow rate (L/min)

The auditor will also verify the flow rates, temperature, and pressure readings of the continuous and filter based PM_{10} samplers following EPA audit guidelines.

Pace Analytical will be contracted to provide the filters and filter analysis for the PM samplers, and EAS Labs will be contracted to provide the VOC canisters and TO-15A analysis. Both are accredited laboratories. Trinity is not qualified to audit them; however, their QAPP, laboratory SOPs, and monthly filter data reports will be reviewed to ensure the analyses being performed meet the requirements of 40 CFR 50 Appendices J and Q, and EPA Compendium Methods IO-3.5 and TO-15A.

C.1.1 Data Quality Audits

Data review is conducted daily utilizing electronic and visual scanning to identify outliers and determine whether data are reasonable and representative. QC checks, including NIST traceable certifications, flow checks, calibration and audits will be included in the review.

C.1.2 Corrective Actions

All deficiencies identified during routine data surveillance or site surveillances will be documented and reported to SIMS and Trinity Consultants Project Director no later than one working day of discovery and, depending on the nature of the deficiency, corrective action will be made no later than seven working days of the notification. Corrective actions to deficiencies will be addressed and documented in the station logbook and on a Corrective Action Report which will be provided to the SIMS and Trinity Consultants Project Director. Follow-up action shall be taken to verify implementation of the corrective action. A corrective action report form will be filled out that identified the problem or deficiency, the proposed corrective action, and the results of the corrective action. A copy of a Corrective Action Report is presented in Appendix B.

C.1.3 QAPP and SOP Revisions

If revisions to the SIMS QAPP or SOPs are needed, any modifications will be approved by SIMS and EPA. Only the part(s) of the affected section in the QAPP will be submitted. Any revisions to SOP's will be submitted in entirety. Document control will be maintained and updated, as appropriate. A revised edition will be distributed to all appropriate individuals on the distribution list. Updates will be made when there are:

- Changes in equipment and personnel,
- Addition of parameters to the monitoring program, and/or,
- Revisions to EPA technical guidelines.

C.2 Reports to Management

A summary of the reports to be generated is presented in Table C-1.

Table C-1 Reports to Management

Reports	Frequency	Content	Responsible Position/ Individual	Distribution
Monthly Data Summaries	Each month	Summarize Data including QC check results	Trinity Data Manager Wyndam Lewis	See Section A.3 Distribution List
Corrective Action Reports	As Needed	Summarizes Corrective Actions Taken to return the Monitoring Station into compliant status	Trinity Consultants Project Director Casey Lenhart	See Section A.3 Distribution List
Response to Corrective Action Reports	As Needed	Reports the results of the Corrective Actions Taken	Trinity Consultants Project Director Casey Lenhart	See Section A.3 Distribution List
Performance Audit Summary	Each audit	Summarize results of performance audit	Independent auditor	See Section A.3 Distribution List

A data summary will be submitted to SIMS within 21 days of the end of the previous month and SIMS will be submitting to EPA within 30 days of the end of the previous month. Corrective action reports will be submitted as needed within one week of identifying a deficiency. These reports will contain data summaries, a summary of any problems encountered in the monitoring project and the status of any current problems, a summary of any meetings or correspondence dealing with the monitoring program, a synopsis of percent recovery including brief explanations of missing data, overall data recovery, quality control, and all quality assurance documentation. Copies of corrective action forms will be included in each report as an appendix. Monthly flow checks and invalid data period summaries will also be provided in appendices. The summary report will be provided electronically, as an Adobe Acrobat PDF copy.

D. DATA VALIDITY AND USEABILITY

D.1 Data Review, Validation, and Verification Requirements

The purpose of this section is to identify the procedures and responsible parties who will perform data validation and 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. Data validation is an analyte and sample specific process that extends the evaluation of data beyond method, procedural, or contractual compliance to determine the analytical quality of a specific data set.

The air quality data validation criteria are based on manufacturer specifications and US EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volumes I and II. The data validation templates that are presented in the Quality Assurance Handbook for Air Pollution Measurement Systems - Volume II have been adopted for this project. These templates are comprised of critical criteria, operational evaluations, and systematic issues. Data that do not meet each criterion on the Critical Criteria Table should be invalidated unless there is a compelling justification for not doing so. Violation of a criterion on the Operational and Systemic Evaluations Table may be cause for invalidation or qualification and the reason for not meeting the criterion must be investigated, mitigated, or justified. See Section B.5.8 for further information.

Exceptional field events may occur, and field activities may negatively affect the validity of monitoring results. In addition, some of the QC checks may fail to meet acceptance criteria. Information on problems that affect the integrity of data is identified in the form of flags. QC checks will bracket the data to ensure validity. The review of routine data and associated QC data will be verified and validated monthly. It is assumed that if measurement uncertainty can be controlled within acceptance criteria, then the overall measurement uncertainty will be maintained within the precision and bias DQOs.

On a monthly basis, a thorough review of the data will be conducted for completeness and accuracy. Unacceptable or questionable data will be flagged appropriately. Trinity data management personnel will use the checks and procedures outlined in this QAPP in combination with EPA's data validation templates and other data quality information to determine verify the accuracy and validity of the data.

The Trinity Consultants Project Director and site technicians are responsible for verifying proper operation of the air quality monitoring equipment. The Trinity Data Manager and Project Director will review the incoming data to the standards discussed in Section B.5 of this document. Daily, the data will be reviewed again by the Trinity Data Manager to ensure that the data are complete, accurate, and representative. Data will be determined to be invalid whenever documented evidence exists demonstrating that the air quality monitoring equipment is not collecting data under representative conditions, or the instrumentation was malfunctioning.

The Trinity Data Manager will routinely check for irregularities during the daily data review. Data review includes evaluation of the raw data, monthly flow checks, and maintenance records. Any abnormalities in the data will be flagged and noted on the appropriate checklists. Any suspect data will be brought to the attention of the Project Director as soon as possible. All other documentation pertaining to the project (i.e., station logs, field notes, and calibration sheets, emails, correction actions) will be reviewed to ensure that erroneous data are identified and removed, as necessary from the final data set.

D.1.1 Data Acceptance Limits for PM_{10} Based on Audit

The continuous PM_{10} data will be valid and acceptable if the performance flow rate audit shows results are<±10.1% of the audit standard and no other known discrepancy is found. For the filter based PM_{10}, data will be valid and acceptable if the performance flow rate audit shows results are<±4.1% of the transfer standard and <±5.1% of flow rate design value of the audit standard and no other known discrepancy is found. Temperature and pressure data will be valid and acceptable if the temperature and pressure performance audit show results that are <±2.1°C and <±10.1 mmHg.

The acceptance limits for the HAP metals on the PM filters are as follows:

- Antimony – 1000 ng/filter
- Arsenic – 650 ng/filter
- Beryllium – 150 ng/filter
- Cadmium – 1000 ng/filter
- Chromium – 1500 ng/filter
- Cobalt – 125 ng/filter
- Lead – 100 ng/filter
- Manganese – 600 ng/filter
- Nickel – 1300 ng/filter
- Selenium – 1750 ng/filter

D.1.2 Data Acceptance Limits for VOC

Detection limits achieved for TO-15A are less than 0.02 ppbv for most target compounds. Reporting limits are less than 1 ppbv for all TO-15A compounds.

D.1.3 Data Acceptance Limits for Meteorological Parameters Based on Audit

In accordance with data acceptance criteria established by the EPA, data will be acceptable if quality assurance performance audit show the following results for accuracy:

- The wind direction error (orientation plus linearity) does not exceed ±5 degrees from true north, and the sensor starting threshold is less than 0.50 m/s wind speed,
- The horizontal wind speed average absolute error does not exceed ±0.20 m/s. The sensor starting threshold must be less than 0.50 m/s wind speed for horizontal wind speed,
- The ambient absolute temperature sensor average absolute error does not exceed ±0.5°C,
- The precipitation gauge mean percent difference does not exceed the acceptable tolerance of ±10%, and,

- Relative humidity sensor absolute average percent difference does not exceed the acceptable tolerance of ±7% relative humidity.

D.1.4 Data Validation and Verification Methods

Air quality data validation will be based on the critical criteria presented in QA Handbook Volume II, Appendix D and summarized in Table B-3 through B-5 above. Continuous PM_{10} concentration data will be recorded every one, five, and 15 minutes, and hourly by the CR1000x data loggers. An hourly average will be computed when at least nine five-minute averages are available for the hour. Minutely data will also be collected for resolution and saved per the project's retention schedule (i.e., > 5 years). Data validation will be performed on the hourly average data.

The Project Director, Data Manager and QA Manager are responsible for verifying the proper operation of the air quality monitoring equipment by reviewing the monthly flow checks, calibration records, performance audit results, and field notes from the site technicians prior to formal acceptance of these data. Precision and bias calculations will also be reviewed. The Project Director and Data Manager will use the validation templates (Section B.5) to ensure that the reported data meets the appropriate data quality objectives.

D.1.5 Level 0 Data Validation

Level 0 data validation is essentially raw data obtained directly from the sampling equipment or data acquisition systems in the field. These data have not received any adjustments for known biases or problems that may have been identified during preventive maintenance checks. Level 0 data validation is accomplished by:

- Collecting data via modem, and
- Initially screening the daily data for anomalies using Trinity's QC software. See Section D.2.2 for further details.

Stacked parameter plots will be generated which consist of every data point downloaded since the last site interrogation and reviewed by the Trinity Data Manager for consistency and possible problems. This redundancy assures that problems that might go unnoticed by the software will always be caught by the reviewer.

To aid in data validation, a password-protected project website will be hosted which will be updated daily. The site contains PM_{10} and meteorological chart graphics, daily minimum, maximums, and averages, quality assurance reports. Historical data can also be reviewed at this website. Figures D.1 and D.2 present examples of these graphics. By using this approach, data collection percentages are greatly enhanced, and data management personnel can quickly note and resolve any potential instrumentation problems.

Figure D.1 Login Page to Web Site

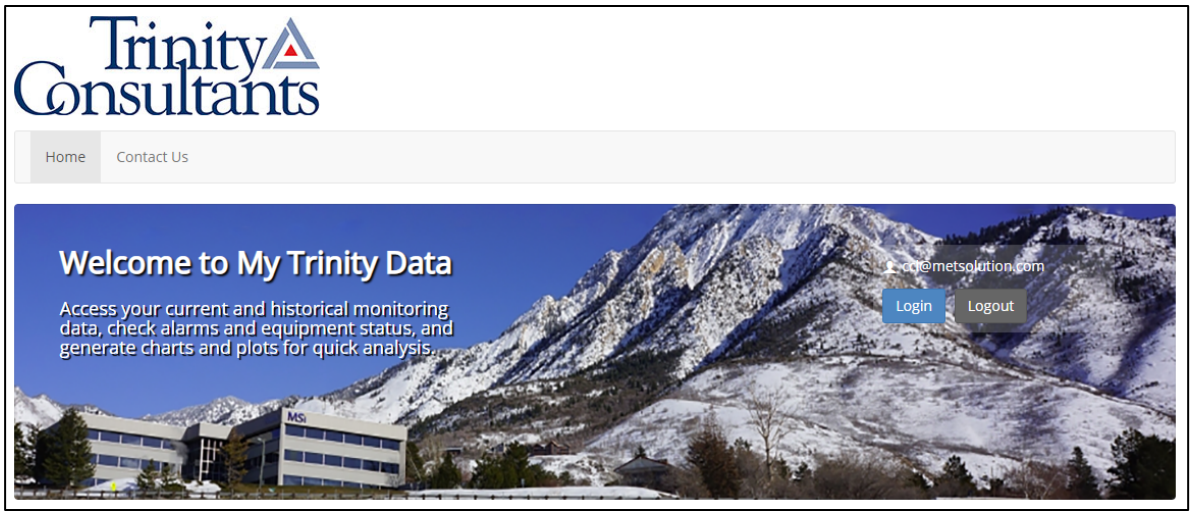
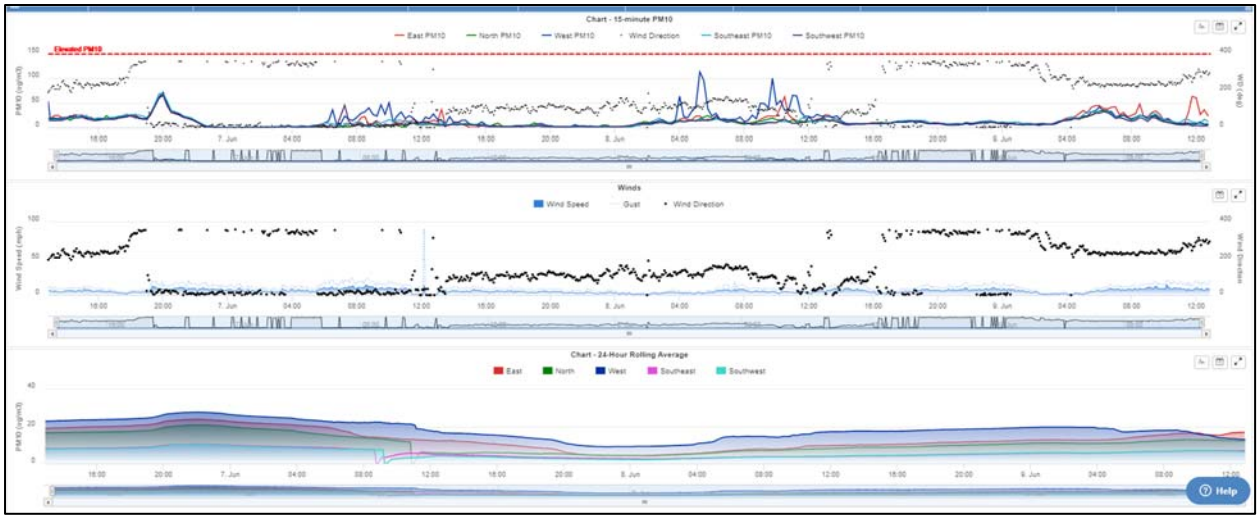


Figure D.2 Real-Time PM_{10} Display



D.1.6 Quality Control Checks for Data Validation

Once data are downloaded via modem, they will be subjected to a series of quality control checks by a software package. The software package performs extensive quality control checks of the data, generates a data summary report which lists means, maximums, minimums, time of occurrence, data values which fall outside of prescribed ranges, periods of constant values, and periods of rapid value changes. This software uses selected data flagging criteria. Example criteria that will cause a data flag in the air quality and meteorological data are presented in Table D-1.

Table D-1 Data Flagging Criteria

Parameter	Less Than	Greater Than	Constant For
Wind Speed	0 m/s	35 m/s	2 hours
Wind Direction	0 deg	360 deg	2 hours
Sigma Theta	0 deg	150 deg	2 hours
Temperature	-30 deg C	45 deg C	30 minutes
Relative Humidity	3%	102%	16 hours
Precipitation	0 inches	1 inch in 24 hours	N/A
PM_{10}	-5 ug/m^3	150 ug/m^3	4 hours
Battery Voltage	11.5 V	16 V	N/A

These criteria may be adjusted as data are collected to encompass site-specific conditions more accurately. The quality assurance software is used to generate flags or warnings that the parameter value is outside of a normally acceptable range. The outlier program does not invalidate data or erase file records based on these outlier tests. Raw data files are never modified and are archived. It will be left to the Trinity Data Manager to review the results of the outlier program in conjunction with the data parameter plots and initiate corrective actions if warranted (site visit or data invalidation). Data will be validated in accordance with Appendix D of EPA's Quality Assurance Handbook for Air Pollution Measurement Systems Volume II.

D.1.7 Level 1 Data Validation

After the QC software is run, visual inspection of the data are performed to identify suspect data values that warrant further investigation. These values will be flagged. Data validation will be performed by Trinity QA personnel who are independent of data production.

Per EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Quality Monitoring Program, EPA recommends the use of flags or result qualifiers to identify potential problems with data (or a sample). According to EPA, a flag is an indicator of the fact and the reason that a data value (a) did not produce a numeric result, (b) produced a numeric result but it is qualified in some respect relating to the type or validity of the result, or (c) produced a numeric result but for administrative reasons is not to be reported outside the organization.

Thus, quality control flags and codes, consisting of a letter and value will be assigned to each datum to indicate its quality. Multiple flags will be applied to each invalid data point such as data invalid due to calibration. Table D-2 presents the data flags and codes that will be applied to the data.

Table D-2 Data Flags

Flag[1]	Code	Description
V	0	Valid
C	1	Corrected or Estimated
S	7	Suspect: data appears to be a data spike or outside normal data range
I	8	Invalid data
M	9999	Missing data: measurement not taken
BJ	9963	Operator Error
AC	9969	Construction in Area
AE	9971	Shelter Temperature Outside Limits
AH	9974	Sample Flow Rate Out of Limits
AL	9978	Voided by Operator
AM	9979	Miscellaneous Void
AN	9980	Instrument Malfunction
AP	9982	Vandalism
AQ	9983	Collection Failure
AS	9985	Poor QA Results
AT	9986	Calibration
AV	9988	Power Failure
AW	9989	Wildlife Damage
AX	9990	Precision Check
AY	9991	QC Control Points (Zero/Span)
AZ	9992	QC Audit
BA	9993	Maintenance
BB	9994	Unable to Reach Site
BC	9995	Multi-Point Calibration
BD	9996	Auto Calibration
BF	9998	ZPS

[1] https://aqs.epa.gov/aqsweb/documents/codetables/qualifiers.html.

The URL listed above on Table D-1 identifies whether the flag is a NULL flag used to invalidate the data, QA flag for QA related issues where the results may be usable, or other type of identifier.

To assist in data validation, a copy of the site logbook and corrective action reports will be examined to confirm periods when instrumentation may have been off-line due to power outages, maintenance or repair, or other quality assurance activities. Significant events will be checked against the graphs for consistency.

Especially high values will be checked. Suspect data will be reported but flagged as suspect. Missing data will be left missing. Reasoning for flagging will be documented. Data will **not** be post-processed. The justification for all data invalidations will be permanently documented in a data validation summary spreadsheet.

For reporting purposes, hourly continuous PM_{10} data will be presented as monthly tables in the summary report. Continuous PM_{10} concentrations less than -3 $\mu g/m^3$ will be set to 0 and the source of the problem will be investigated. The means, maxima, and minima for the month will also be computed. The reset of -3 $\mu g/m^3$ to 0 will not apply to the means, maxima, or minima.

D.1.8 Minimum Acceptable Data Recovery Percentage

The data recovery goal for the air quality parameters will be at least 85 percent per month and 90% for meteorological parameters.

D.2 Reconciliation with User Requirements

The PM_{10} monitoring system will operate according to established protocols by the EPA to provide scientifically defensible air quality data. Data are expected to provide a true representation of PM_{10}, Pb, HAP metals, and VOC at the SIMS facility and fulfill the goals and objectives of the monitoring program which are defined in Section A.5. Reconciliation with the DQOs will involve reviewing both routine and QA/QC data to determine whether the DQOs have been attained and that the data are adequate for their intended use. Following the procedures described in this QAPP, Trinity monitoring personnel will ensure that the data quality objectives are met, and the data will be of acceptable quality, accuracy, precision, and completeness.

APPENDIX A. PM_{10} AND METEOROLOGICAL SAMPLING EQUIPMENT SPECIFICATIONS

Met One Instruments, Inc.

E-Sampler Dual Ambient Monitor/Sampler

The E-SAMPLER is the most feature-packed light-scatter Aerosol Monitor available. Whatever your monitoring needs, the E-sampler will provide accurate, dependable and relevant data.

The E-SAMPLER is a dual technology instrument that combines the unequaled realtime measurement of light scatter with the accuracy standard of filter methods. The simple filter loading process testifies to the seamless blending of both technologies. Filters can be extracted and replaced in less than one minute. Filter medium can be selected based on laboratory analysis requirements.

Particulate loading on the filter does not reduce performance due to the Met One actual flow control protocol. Ambient temperature and pressure are measured and actual flow is calculated and controlled by the E-SAMPLER microprocessor, independent of filter loading change.

The E-SAMPLER provides real-time particulate measurement through near-forward light scattering. An internal rotary vane pump draws air at 2 LPM into the sensing chamber where it passes through visible laser light. Aerosols in the air scatter light in proportion to the particulate load in the air. Scattered light is collected by precise glass optics and focused on a PIN diode.

Rugged state of the art electronics measure the intensity of the focused light and output a signal to the CPU. The output is linear to concentrations greater than 65,000 ug/m^3. Every E-SAMPLER is factory calibrated using polystyrene latex spheres of known index of refraction and diameter at multiple points to validate linearity.

Features:

- Programmable Auto-Zero
- Programmable Auto-Span
- Auto-ranging (1 to 65000 µm/m^3)
- Automatic Flow Control
- Protocol
- Internal Battery (30 Hours Operation without heater & 10 Hours with heater.)
- Laser-Diode Precise Optical Engine
- Integral 47mm Analysis Filter
- Ambient Pressure and Temperature
- Internal Data-logger
- PM_{10}, $PM_{2.5}$, PM_1, TSP Monitoring
- Aluminum Weatherproof Enclosure
- Purge-Air protected Optics
- Completely Self-Contained
- No Tools Filter Replacement

Applications:

- Ambient Air Monitoring
- Remediation Site Perimeter Monitoring
- Indoor Air Quality Monitoring
- Source Monitoring
- Visibility Monitoring
- Mobile Monitoring

Measurement Principles:	Light Scatter and 47mm low flow gravimetric filter sampler.
Available Cut Points:	TSP Inlet Standard. PM_{10}, $PM_{2.5}$, and PM_1 sharp-cut cyclone inlets available.
Measurement Range:	0 to 65 mg/m^3 (0 to 65,530 µg/m^3) dynamic range. 16 bit digital range.
Nephelometer Accuracy:	± 10% to gravimetric method typical when K-factored to local particulate type.
Gravimetric Accuracy:	± 8% of NIOSH 0600.
Precision:	Greater of 3 µg/m^3 or 2%.
Data Storage Resolution:	1 µg/m^3
Data Storage Intervals:	User-Selectable 1, 5, 10, 15, 30, or 60 minute averages.
Nephelometer Interval:	1-second measurements, available on analog output and display.
Sample Cycles:	Continuous operation or programmable scheduled sample runs.
Particle Size Sensitivity:	0.1 to 100 micron. Optimal sensitivity 0.5 to 10 micron particles.
Laser Type:	Diode Laser, 5 mW, 670nm. Visible red.
Long Term Stability:	5% with clean optics.
Flow Rate:	2.0 liters/minute ± 0.1 lpm. Actual volumetric flow.
Pump Type:	10,000 hour brushless diaphragm sample pump and secondary purge pump.
Gravimetric Filter Type:	47mm disc filters (not included). Accepts standard FRM filter holder cartridges.
Automatic Zero and Span:	User-selectable 15 min, 1 hour, 2 hour, 12 hour, or 24 hour intervals. 2.8 min cycle.
Internal Battery:	12V, 12 Amp-Hour. Yuasa NP12-12 or equivalent, Optional lead acid battery.
Internal Battery Run Time:	Up to 30 hours with inlet heater off. Up to 10 hours with inlet heater on.
Power Supply:	Universal 100-240 VAC input, 15 VDC output power supply included. Compatible with solar power kits or external batteries using optional DC power cable.
Power Consumption:	1.1 amps @ 12 VDC (15 Watts) max continuous draw, running with inlet heater on. 0.35 amps (4.2 Watts) running with inlet heater off.
Operating Temperature:	0 to +50°C . (Ambient Temperature Sensor Range -30 to +50°C).
Barometric Pressure:	60,000 to 104,000 Pascal pressure sensor range.
Ambient Humidity Range:	0 to 90% RH, non-condensing.
Humidity Control:	Automatic 10 Watt inlet heater module controlled to sample RH setpoint. Sample RH sensor standard. Optional EX-593 ambient RH sensor available.
Approvals:	CE, ISO-9001. Designed to agree with EPA Class I and Class III FRM/FEM particulate samplers and monitors. Not an EPA-designated equivalent method.
User Interface:	Menu-driven interface with 4x20 character LCD display and dynamic keypad.
Analog Voltage Output:	0-1, 0-2.5, or 0-5 volt DC output. User-set range with 1-second real-time output.
Serial Interface:	RS-232 duplex serial port for PC, datalogger, or modem communications.
Alarm Contact Closure:	Normally closed contact closure relay output. Contact rating 0.5A @ 100V DC max.
Compatible Software:	Comet™ (included), Air Plus™, terminal programs such as HyperTerminal®
Alarm Reporting:	Available through serial port data files, display, and relay output.
Memory:	4369 data logger records (182 days @ 1 record/hr, 3 days @ 1 record/min).
Factory Service Interval:	24 Months typical, under continuous use in normal ambient air.
Mounting Options:	Pole or wall mount bracket standard. Optional EX-905 tripod recommended.
Unit Weight:	6.4 kg (14 lbs) without tripod, battery, or optional accessories.
Unit Dimensions:	65cm high, 27cm wide, 16.5cm deep. (25.5" x 10.5" x 6.5"). With inlet assembly

Specifications are subject to change at any time.

Met One Instruments, Inc.

1600 Washington Blvd. Grants Pass, Oregon 97526 **Phone:** 541.471.7111

Sales: sales@metone.com | **Service:** service@metone.com | **Website:** www.metone.com

August 2019

wind

High Performance Wind Sensor for Air Quality Applications

YOUNG

Model 05305

Wind Monitor-AQ

The Wind Monitor-AQ is a high resolution wind sensor designed specifically for air quality applications. It combines simple, corrosion-resistant construction with low threshhold, fast response and excellent fidelity.

The Wind Monitor-AQ meets the requirements of the following regulatory agencies:

U.S. Environmental Protection Agency – Ambient Monitoring Guidelines for Prevention of Significant Deterioration (PSD).

U.S. Nuclear Regulatory Agency – NRC Regulatory Guide 1.23 Meteorological Programs in Support of Nuclear Power Plants.

American Nuclear Society – Standard for Determining Meteorological Information at Power Plants.

Wind speed is sensed by a lightweight, carbon fiber thermoplastic (CFT), helicoid propeller. Propeller rotation produces an AC sine wave voltage signal with frequency directly proportional to wind speed. Slip rings and brushes are not used.

The wind direction sensor is a lightweight vane with performance characteristics that assure excellent fidelity in fluctuating wind conditions. Vane position is sensed by a precision potentiometer. Output is a DC voltage directly proportional to vane angle.

The instrument body is UV stabilized plastic with stainless steel and anodized aluminum fittings. Precision grade, stainless steel ball bearings are used throughout. Transient protection and cable terminations are located in a convenient junction box. The instrument mounts on standard 1 inch pipe.

The Wind Monitor-AQ is available with two additional output signal options. **Model 05305V** offers calibrated voltage outputs, convenient for use with many dataloggers. **Model 05305L** provides a calibrated 4-20 mA current signal for each channel, useful in high noise areas or for long cables (up to several kilometers). Signal conditioning electronics are integrated into the sensor junction box.

Ordering Information — MODEL

WIND MONITOR-AQ	**05305**
WIND MONITOR-AQ VOLTAGE OUTPUTS	**05305V**
WIND MONITOR-AQ 4-20mA OUTPUTS	**05305L**

Specifications

Range:
Wind speed: 0-50 m/s (112 mph)
Azimuth: 360° mechanical, 355° electrical (5° open)

Accuracy:
Wind speed: ±0.2 m/s (0.4 mph) or 1% of reading
Wind direction: ±3 degrees

Threshold:*
Propeller: 0.4 m/s (.9 mph)
Vane: 0.5 m/s (1.0 mph) at 10° displacement

Dynamic Response:*
Propeller distance constant (63% recovery) 2.1 m (6.9 ft)
Vane delay distance (50% recovery) 1.2 m (3.9 ft)
Damping ratio: 0.45
Damped natural wavelength: 4.9 m (16.1 ft)
Undamped natural wavelength: 4.4 m (14.4 ft)

Signal Output:
Wind speed: magnetically induced AC voltage, 3 pulses per revolution. 1800 rpm (90 Hz) = 9.2 m/s (20.6 mph)
Azimuth: analog DC voltage from conductive plastic potentiometer – resistance 10K Ω, linearity 0.25%, life expectancy – 50 million revolutions

Power Requirement:
Potentiometer excitation: 15 VDC maximum

Dimensions:
Overall height: 38 cm (15.0 in)
Overall length: 65 cm (25.6 in)
Propeller: 20 cm (7.9 in) diameter
Mounting: 34 mm (1.34 in) diameter (standard 1 inch pipe)

Weight:
Sensor weight: 0.7 kg (1.5 lbs)
Shipping weight: 2.3 kg (5 lbs)

**Nominal values, determined in accordance with ASTM standard procedures. Shielded bearings lubricated with Type LO-1 light General Purpose Instrument Oil.*

MODEL 05305V **Voltage outputs**

Power Requirement:
8-24 VDC (5 mA @ 12 VDC)

Operating Temperature:
-50 to 50° C

Output Signals:
WS: 0-2.5 VDC (0-50 m/s)
WD: 0-5 VDC (0-360°)

MODEL 05305L **4-20 mA outputs**

Power Requirement:
8-30 VDC (40 mA max.)

Operating Temperature:
-50 to 50° C

Output Signals:
4-20 mA full scale

CE *Complies with applicable CE directives.*
Specifications subject to change without notice.

R.M. YOUNG COMPANY
2801 Aero Park Drive
Traverse City, Michigan 49686 USA
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E-mail: met.sales@youngusa.com
Web Site: www.youngusa.com

CAMPBELL SCIENTIFIC®
WHEN MEASUREMENTS MATTER

HygroVUE10

Digital Temperature and Relative Humidity Sensor with M12 Connector

Rugged, Reliable, and Flexible

Simple to use and easy to maintain

Overview

The HygroVUE™10 offers a combined temperature and relative humidity element in an advanced digital sensor that is ideal for weather networks. The electronics within the sensor provide accurate measurements, and the sensor is easy to use. The digital SDI-12 output allows a simple connection and measurement by many data logging systems. Another benefit is that this digital output avoids the extra errors associated with measuring analog sensors.

A stainless-steel mesh filter on the HygroVUE™10 minimizes the effects of dust and dirt on the sensor while allowing air exchange around the sensor element and reducing the chances that condensation remains inside the filter cap. A small PTFE membrane filter is bonded to the surface of the element, which prevents any finer dust or mold from directly influencing the measurement.

Because the sensor housing is designed to withstand permanent exposure to various weather conditions and to fit inside a range of radiation shields (including compact shields), the HygroVUE™10 is truly suitable for a wide range of monitoring applications.

The HygroVUE™10 utilizes a latest-generation, Swiss-made, combined relative humidity and temperature element based on CMOSens® technology that offers good measurements, accuracy, and stability. Each element of the HygroVUE™10 is individually calibrated with the calibration corrections stored on the chip. You can easily change the sensor element in the field, which reduces your downtime and calibration costs.

Benefits and Features

- Uses a combined, pre-calibrated digital humidity and temperature element
- Field-changeable element for fast, on-site recalibration
- Digital SDI-12 output, allowing long cables with no added errors
- Simple data logger programming
- Low power consumption
- Wide operating voltage
- Rugged design with potted electronics
- Standard M12 connector with IP67 sealing rating

Detailed Description

Mounting

When you use the HygroVUE™10 outdoors, it is standard practice to install the sensor within a housing, known as a shield. The shield prevents solar radiation from heating the sensor and creating measurement errors. The radiation shield also provides a degree of protection from adverse weather, such as hail or driving rain. The most common type of shield is a relatively small, naturally ventilated screen that is low maintenance and requires no power.

The HygroVUE™10 is specifically designed for field use with dimensions to suit common radiation shields. (Campbell Scientific recommends the RAD10E 10-Plate Solar Radiation Shield.) You can mount the RAD10E on vertical or horizontal poles.

Field Calibration

Calibration is easy to carry out by simply changing the sensor element. As each sensor element is individually calibrated, no further adjustments of the sensor are required. This means that when you change the element, it returns the sensor to the factory calibration state for both temperature and humidity—without interrupting your measurement collection for long periods.

Specifications

Sensing Element	SHT35 modified by Campbell Scientific
Communication Standard	SDI-12 V1.4 (responds to a subset of commands)
Supply Voltage	7 to 28 Vdc
EMC Compliance	Tested and conforms to IEC61326:2013.
Standard Operating Temperature Range	-40° to +70°C
Main Housing Material	UV stable, white PET-P
Electronics Sealing Classification	IP67
Sensor Protection	Outer glass-filled polypropylene cap fitted with a stainless-steel mesh dust filter with nominal pore size of < 30 µm. The sensor element has a PTFE protective film with a filtration efficiency of > 99.99% for particles of 200 nm or larger size.
Sensor Connector	M12, male, 4-pole, A-coded
Cable	Polyurethane sheathed, screened cable, nominal diameter 4.8 mm (0.19 in.)
Field-Replaceable Chip or Recalibrate	Field-replaceable chip
Sensor Cap Diameter	12.5 mm (0.5 in.)
Body Diameter at Connector	18 mm (0.7 in.)
Length	180 mm (7.1 in.) without cable fitted
Sensor Body Weight	50 g (1.8 oz)
Weight	250 g (8.8 oz) with 5 m (16.4 ft) cable

Relative Humidity

Measurement Range	0 to 100% RH
Accuracy	⟩ ±2% (at 25°C, over the range 80 to 100% RH) ⟩ *-NOTE- The accuracy figures quoted are the 95% confidence limits relative to factory standards.* ⟩ ±1.5% (at 25°C, over the range 0 to 80% RH)
Short-Term Hysteresis	< ±1% RH
Additional Errors at Other Temperatures	< ±1% RH (over -40° to +60°C)
Long-Term Stability	±0.5% per year (maximum drift in clean air conditions)
Reported Resolution	0.001% RH
Repeatability	0.05% RH (3σ noise level)
Response Time with Filter	8 s (63% response time in air moving at 1 m/s)

Air Temperature

Measurement Range	-40°C to +70°C
-NOTE-	*The accuracy figures quoted are the 95% confidence limits relative to factory standards.*
Accuracy	⟩ ±0.1°C (over the range -20 to +60°C)

	±0.2°C (over the range -40 to +70°C)
Long-Term Drift	< 0.03°C per year
Reported Resolution	0.001°C
Repeatability	0.04°C (3σ noise level)

Response Time with Filter	< 130 s (63% response time in air moving at 1 m/s)
Calibration Traceability	NIST and NPL standards

Maximum Current Drain

Quiescent	50 µA
During Measurement	0.6 mA (takes 0.5 s)

For comprehensive details, visit: www.campbellsci.com/hygrovue10

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CAMPBELL SCIENTIFIC®
WHEN MEASUREMENTS MATTER

CR1000X

Measurement and Control Datalogger

All CR1000X dataloggers are tested and guaranteed to meet electrical specifications in a standard -40° to +70°C or extended -55° to +85°C non-condensing environment. Datalogger recalibration is recommended every three years. System configuration and critical specifications should be confirmed with Campbell Scientific before purchase.

ANALOG (SE1 – SE16, DIFF1 – DIFF8)

16 single-ended (SE) or 8 Differential (DIFF) inputs individually configurable for voltage, thermocouple, ratiometric, and period average measurements, using a 24-bit ADC. One channel at a time is measured in numeric succession.

VOLTAGE MEASUREMENTS

INPUT RESISTANCE: 20 GΩ typical

INPUT LIMITS: ±5 V

SUSTAINED INPUT VOLTAGE WITHOUT DAMAGE: ±20 Vdc

DC COMMON MODE REJECTION: > 120 dB with input reversal (≥ 86 dB without input reversal)

NORMAL MODE REJECTION: > 70 dB @ 60 Hz

INPUT CURRENT: ±1 nA typical @ 25°C

FILTER FIRST NOTCH FREQUENCY (f_{N1}) RANGE: 0.5 Hz to 31.25 kHz

RANGE AND TYPICAL EFFECTIVE RESOLUTION:

Notch Frequency (f_{N1})[1] (Hz)	Range[2] (mv)	Typical Resolution, µV RMS (DIFF w/Input Reversal)	Typical Resolution, µV RMS (SE or DIFF w/o Input Reversal)
15000	±5000	8.2	11.8
	±1000	1.9	2.6
	±200	0.75	1.0
50/60[3]	±5000	0.6	0.88
	±1000	0.14	0.2
	±200	0.05	0.08
5	±5000	0.18	0.28
	±1000	0.04	0.07
	±200	0.02	0.03

ACCURACY (does not include sensor or measurement noise):

0° to 40°C	-40° to 70°C	-55° to 85°C
±(0.04% of measurement + offset)	±(0.06% of measurement + offset)	±(0.08% of measurement + offset)

OFFSETS:

Range (mV)	DIFF w/Input Reversal (µV)	SE or DIFF w/o Input Reversal (µV)
±5000	±0.5	±2
±1000	±0.25	±1
±200	±0.15	±0.5

MULTIPLEXED MEASUREMENT TIME: (450 µs + settling time + ($1/f_{N1}$)) * reps

Example f_{N1} (Hz)[4]	Multiplexed Measurement Time (ms) w/ 500 µs settling time	
	DIFF w/Input Reversal	SE or DIFF w/o Input Reversal
15000	2.04	1.02
60	35.24	17.62
50	41.9	20.95
5	401.9	200.95

MEASUREMENT SETTLING TIME: 20 µs to 600 ms; 500 µs default

RESISTANCE MEASUREMENTS

Resistance measurements for four- and six-wire full bridge and two-, three-, and four-wire half bridge using voltage excitation. Excitation polarity reversal available to minimize dc error.

ACCURACY:[5,6]

±(0.01% of voltage measurement + offset) , 0° to 40°C
±(0.015% of voltage measurement + offset), -40° to 70°C
±(0.02% of voltage measurement + offset , -55° to 85°C

PERIOD AVERAGE MEASUREMENTS

Up to 16 analog inputs can be used for period averaging.

ACCURACY: ±(0.01% of measurement + resolution), where resolution is 0.13 µs divided by the specified number of cycles to be measured.

RANGE DEPENDENT ON INPUT:

Gain Code	Minimum Peak-to-Peak Signal (mV)[7]	Maximum Peak-to-Peak Signal (V)[8]	Minimum Pulse Width (µs)	Maximum Frequency (kHz)[9]
0	500	10	2.5	200
1	50	2	10	50
2	10	2	62	8
3	2	2	100	5

VOLTAGE EXCITATION (VX1 – VX4)

4 independently configurable voltage sources that can operate in one of two modes: Switched Excitation mode or Switched Regulated Voltage Supply. In Switched Excitation mode, a single 16-bit digital-to-analog converter (DAC) shared by all VX outputs produces a user-specified voltage during measurement only. In Switched Regulated Voltage Supply mode, the port can continuously provide either 3.3 Vdc or 5 Vdc.

	Range (V)	Resolution	Accuracy[5,10]	Maximum Source/Sink Current (mA)[11]
Voltage Excitation	±4	0.06 mV	±(0.1% of setting + 2 mV)	±40
Switched, Regulated	+3.3 or 5	+3.3 or 5 V	±5%	50

PULSE COUNTING (P1, P2)

2 inputs individually configurable for switch closure, high-frequency pulse, or low-level AC measurements. Independent 32-bit counter for each input. See also C1 - C8 for additional switch closure and high-frequency measurement inputs.

MAXIMUM INPUT VOLTAGE: ±20 Vdc

MAXIMUM COUNT PER SCAN: 2^{32}

INPUT RESISTANCE: 5 kΩ

ACCURACY: ±(0.02% of reading +1/scan)

SWITCH CLOSURE INPUTS

PULL-UP RESISTANCE: 100 kΩ to 5 V

EVENT: Low (<0.8 V) to High (>2.5 V)

MINIMUM SWITCH CLOSED TIME: 5 ms

MINIMUM SWITCH OPEN TIME: 6 ms

MAXIMUM BOUNCE TIME: 1 ms open without being counted

HIGH-FREQUENCY INPUTS

PULL-UP RESISTANCE: 100 kΩ to 5 V

EVENT: Low (<0.8 V) to High (>2.5 V)

MAXIMUM INPUT FREQUENCY: 250 kHz

LOW-LEVEL AC INPUTS

MINIMUM RESISTANCE: 10 kΩ to G

DC-OFFSET REJECTION: Internal AC coupling eliminates DC-offset voltages up to ±0.5 Vdc

INPUT HYSTERESIS: 12 mV @ 1 Hz

RANGE:

Sine Wave (mV RMS)	Input Frequency Range(Hz)
20	1.0 to 20
200	0.5 to 200
2000	0.3 to 10,000
5000	0.3 to 20,000

DIGITAL I/O (C1 – C8)

8 ports configurable for digital input and output including status high/low, pulse width modulation, external interrupt, edge timing, switch closure pulse counting, high-frequency pulse counting, UART, RS-232, RS-485, SDM, SDI-12, I2C, and SPI function. Ports are configurable in pairs for 5 V or 3.3 V logic for some functions.

MAXIMUM INPUT VOLTAGE: ±20 Vdc

LOGIC LEVELS AND DRIVE CURRENT:

Terminal Pair Configuration	Logic Low	Logic High	Current Source
5 V	≤ 1.5 V	≥ 3.5 V	10 mA @ 3.5 V
3.3 V	≤ 0.8 V	≥ 2.5 V	10 mA @ 1.85 V

SWITCH CLOSURE INPUTS

ACCURACY: ±(0.02% of reading + 1/scan)
RESISTANCE: Port pair configurable with 100 kΩ pull-up or pull-down
SOFTWARE DEBOUNCE TIME: 3 ms
MAXIMUM BOUNCE TIME: 1 ms open without being counted
MAXIMUM INPUT FREQUENCY: 150 Hz

HIGH-FREQUENCY INPUTS

ACCURACY: ±(0.02% of reading + 1/scan)
RESISTANCE: Port pair configurable with 100 kΩ pull-up or pull-down
MAXIMUM INPUT FREQUENCY: 1 MHz

EDGE TIMING

MAXIMUM INPUT FREQUENCY: ≤ 2.3 KHz
RESOLUTION: 500 ns

RESISTIVE GROUND (RG1 – RG2)

2 resistance-to-ground inputs that can be used for non-isolated 0-20 mA and 4-20 mA current loop measurements or for terminating the ground reference of an RS-485 serial connection.

MAXIMUM INPUT VOLTAGE: ±16 V
RESISTANCE TO GROUND: 101 Ω
CURRENT MEASUREMENT SHUNT RESISTANCE: 10 Ω
MAXIMUM CURRENT MEASUREMENT RANGE: ±80 mA
ABSOLUTE MAXIMUM CURRENT: ±160 mA
CURRENT MEASUREMENT RESOLUTION: ≤ 20 nA
CURRENT MEASUREMENT ACCURACY: ±(0.1% of reading + 100 nA) @ -40° to 70°C

5V OUTPUT (5 V)

1 regulated 5 V output (±5%) with a current limit of 230 mA. Output is shared with CS I/O DB9 5V output. See also VX1 – VX4 for additional regulated voltage outputs.

12V OUTPUT (12V, SW12-1, SW12-2)

3 unregulated 12 Vdc outputs with voltage equal to the Power Input supply voltage. Two levels of thermal fuses regulate current sourcing. In total (12V + SW12-1 + SW12-2) the hold current is limited to 2.68 A @ -40°C, 0.96 A @ 80°C. SW12-1 and SW12-2 can be independently set under program control. Each SW12 has a hold current limited to 1.3 A @ -40°C, 0.47 A @ 80°C.

DEDICATED COMMUNICATION INTERFACES

USB: Micro-B device for computer connectivity

CS I/O: 9-pin D-sub multidrop interface to Campbell Scientific CS I/O peripherals

RS-232/CPI: A single RJ-45 interface that can operate in one of two modes, RS-232 or CPI. RS-232 connects to computer, sensor, or communication devices serially. CPI interfaces with Campbell Scientific CDM measurement expansion modules and sensors.

ETHERNET PORT: RJ-45, 10/100 Mbps, full or half duplex, Auto-MDIX, magnetic isolation and TVS surge protection

PROTOCOLS

INTERNET PROTOCOLS: Ethernet, PPP, CS I/O IP, RNDIS, ICMP/Ping, Auto-IP(APIPA), IPv4, IPv6, UDP, TCP, TLS, DNS, DHCP, SLAAC, SNMPv2, NTP, Telnet, HTTP(S), FTP(S), SMTP/TLS, POP3/TLS

ADDITIONAL PROTOCOLS: PakBus, PakBus Encryption, CPI, SDM, SDI-12, Modbus RTU / ASCII / TCP, DNP3, NTCIP, NMEA 0183, I2C, SPI, custom user definable over serial, TCP, and UDP

DATA FILE FORMATS: CSV, XML, JSON, binary, encrypted, custom user definable

POWER REQUIREMENTS

PROTECTION: Reverse polarity protected; overvoltage protected up to 30 Vdc

VOLTAGE INPUT: 10 to 16 Vdc

INPUT CURRENT LIMIT @ 12 VDC: 4.35 A @ -40°C, 1.56 A @ 85°C

AVERAGE CURRENT DRAIN @ 12 VDC:
IDLE: <1 mA
ACTIVE 1 HZ SCAN: 1 mA
ACTIVE 20 HZ SCAN: 55 mA
SERIAL ACTIVE (RS-232/RS-485): Active + 25 mA
ETHERNET POWER MODE 1 MINUTE: Active + 1 mA
ETHERNET LINK ACTIVE: Active + 48 mA

SYSTEM

PROCESSOR: Renesas RX63N (32-bit with hardware FPU, running at 100 MHz)

MEMORY: 128 MB Flash + 4 MB SRAM (battery backed)
DATA STORAGE: 4 MB SRAM + 72 MB flash
DATA STORAGE EXPANSION: Removable microSD flash memory; up to 8 GB

PROGRAM EXECUTION: 1 ms to one day

REAL-TIME CLOCK: Battery backed while external power is disconnected
RESOLUTION: 1 ms
ACCURACY: ±3 min. per year. Optional GPS correction to (±10 µs) using GPS, PakBus, or NTP

INTERNAL LITHIUM BATTERY: AA, 2.4 Ah, 3.6 Vdc (Tadiran TL 5903/S) for battery-backed memory and clock only. 3 year life with no external power source

WIRING PANEL TEMPERATURE: A 10K3A1A BetaTHERM thermistor, located between the two rows of analog input channels, is measured when reporting wiring panel temperature.

COMPLIANCE INFORMATION

VIEW EU DECLARATION OF CONFORMITY AT:

www.campbellsci.com/cr1000x

PHYSICAL

DIMENSIONS: 23.8 cm x 10.1 cm x 6.2 cm (9.4 in x 4.0 in x 2.4 in); additional clearance required for cables and leads

WEIGHT/MASS: 0.86 kg (1.9 lb)

WARRANTY

3 years against defects in materials and workmanship.

---

[1] Valid notch frequencies: 0.5 Hz to 31.25 kHz.

[2] Range overhead of ~5% on all ranges guarantees that full-scale values will not cause over range.

[3] 50/60 correspond to rejection of 50 and 60 Hz ac power mains noise.

[4] Notch frequency (1/integration time).

[5] Assumes input reversal for differential measurements along with excitation reversal for excitation voltage <1000 mV, not including bridge resistor errors or sensor and measurement noise.

[6] Resistance accuracy, rather than absolute accuracy, determines overall measurement accuracy of ratiometric resistance measurements.

[7] Minimum signal centered around specified period average threshold.

[8] Maximum signal centered around datalogger ground.

[9] The maximum frequency = 1/(twice minimum pulse width) for 50% duty cycle signals.

[10] Valid over -55 to +85 °C temperature range.

[11] Exceeding current limits causes voltage output to become unstable. Voltage should stabilize when current is reduced to within stated limits.

TERMINALS

Analog Input	SE1	SE2	SE3	SE4	SE5	SE6	SE7	SE8	SE9	SE10	SE11	SE12	SE13	SE14	SE15	SE16	RG1	RG2	Max
Single Ended	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓			16
Differential	H	L	H	L	H	L	H	L	H	L	H	L	H	L	H	L			8
Ratiometric Bridge	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓			16
Thermocouple	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓			16
Current Loop																	✓	✓	2
Period Average	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓			16

Analog Output	VX1	VX2	VX3	VX4	Max
Switched-Voltage Excitation	✓	✓	✓	✓	4

Voltage Output[12]	C1	C2	C3	C4	C5	C6	C7	C8	VX1	VX2	VX3	VX4	5 V	12V	SW12V-1	SW12V-2	Max
5 V	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓				9
3.3 V	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓					8
12 V														✓	✓	✓	3

Communication[13]	C1	C2	C3	C4	C5	C6	C7	C8	RS-232/CPI	USB	Ethernet	CS I/O	Max
SDI-12	✓		✓		✓		✓						4
GPS	PPS	Rx	Tx	Rx	Tx	Rx	Tx	Rx					1
TTL (0 to 5 V)	Tx	Rx	Tx	Rx	Tx	Rx	Tx	Rx					4
LVTTL (0 to 3.3 V)	Tx	Rx	Tx	Rx	Tx	Rx	Tx	Rx					4
RS-232					Tx	Rx	Tx	Rx	✓				3
RS-485 (Half Duplex)					A(-)	B(+)	A(-)	B(+)					2
RS-485 (Full Duplex)					Tx-	Tx+	Rx-	Rx+					1
I2C	SDA	SCL	SDA	SCL	SDA	SCL	SDA	SCL					4
SPI	MOSI	SCLK	MISO		MOSI	SCLK	MISO						2
SDM[14]	DATA	CLK	ENABLE		DATA	CLK	ENABLE						1
CPI/CDM									✓				1
USB										✓			1
Ethernet											✓		1
CS I/O												✓	1

Digital I/O[13]	C1	C2	C3	C4	C5	C6	C7	C8	Max
General I/O Pair	✓	✓	✓	✓	✓	✓	✓	✓	8
Pulse-Width Modulation Output	✓	✓	✓	✓	✓	✓	✓	✓	8
Timer Input	✓	✓	✓	✓	✓	✓	✓	✓	8
Interrupt	✓	✓	✓	✓	✓	✓	✓	✓	8

Pulse Counting[13]	C1	C2	C3	C4	C5	C6	C7	C8	P1	P2	Max
Switch Closure	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	10
High Frequency	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	10
Low Level AC									✓	✓	2

[12] For the Voltage Outputs, the C terminals have limited drive capacity and the voltage levels are configured in pairs.

[13] Triggering conflicts can occur when companion control ports are used for different triggering instructions (TimerInput, PulseCount, SDI12Recorder, WaitDigTrig). For example, if C3 is used for the SDI12Recorder instruction, C4 cannot be used in the TimerInput, PulseCount, or WaitDigTrig instructions.

[14] SDM can be on either C1-C3 or C5-C7, but not both at the same time.

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Global Sales & Support Network

A worldwide network of companies to help meet your needs

Australia
Location: Garbutt, QLD Australia
Phone: 61.7.4401.7700
Email: info@campbellsci.com.au
Website: www.campbellsci.com.au

Brazil
Location: São Paulo, SP Brazil
Phone: 11.3732.3399
Email: vendas@campbellsci.com.br
Website: www.campbellsci.com.br

Canada
Location: Edmonton, AB Canada
Phone: 780.454.2505
Email: dataloggers@campbellsci.ca
Website: www.campbellsci.ca

China
Location: Beijing, P. R. China
Phone: 86.10.6561.0080
Email: info@campbellsci.com.cn
Website: www.campbellsci.com

Costa Rica
Location: San Pedro, Costa Rica
Phone: 506.2280.1564
Email: info@campbellsci.cc
Website: www.campbellsci.cc

France
Location: Antony, France
Phone: 0033.0.1.56.45.15.20
Email: info@campbellsci.fr
Website: www.campbellsci.fr

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Location: Bremen, Germany
Phone: 49.0.421.460974.0
Email: info@campbellsci.de
Website: www.campbellsci.de

South Africa
Location: Somerset West, South Africa
Phone: 27.21.8800885
Email: cleroux@csafrica.co.za
Website: www.csafrica.co.za

Southeast Asia
Location: Bangkok, Thailand
Phone: 66.2.719.3399
Email: thitipongc@campbellsci.asia
Website: www.campbellsci.asia

Spain
Location: Barcelona, Spain
Phone: 34.93.2323938
Email: info@campbellsci.es
Website: www.campbellsci.es

UK
Location: Shepshed, Loughborough, UK
Phone: 44.0.1509.601141
Email: sales@campbellsci.co.uk
Website: www.campbellsci.co.uk

USA
Location: Logan, UT USA
Phone: 435.227.9120
Email: info@campbellsci.com
Website: www.campbellsci.com

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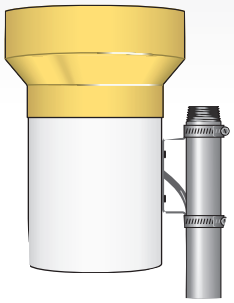
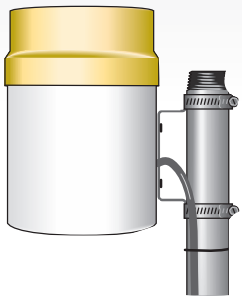
TE525, TE525WS, and TE525MM

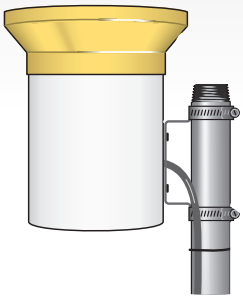
Texas Electronics Tipping Bucket Rain Gages



The TE525WS conforms to the National Weather Service recommendation for an 8-inch funnel orifice.



The TE525 is widely used in environmental monitoring applications.



The TE525MM measures rainfall in metric rather than US units.

Overview

The TE525-series tipping bucket rain gages are manufactured by Texas Electronics. They funnel precipitation into a bucket mechanism that tips when filled to a calibrated level. A magnet attached to the tipping mechanism actuates a switch as the bucket tips. The momentary switch closure is counted by the pulse-counting circuitry of Campbell Scientific dataloggers.

Benefits and Features

- High precision
- Integral bubble level
- Compatible with all Campbell Scientific dataloggers (including the CR200(X) series)
- Compatible with the CWS900-series interfaces, allowing it to be used in a wireless sensor network

Mounting

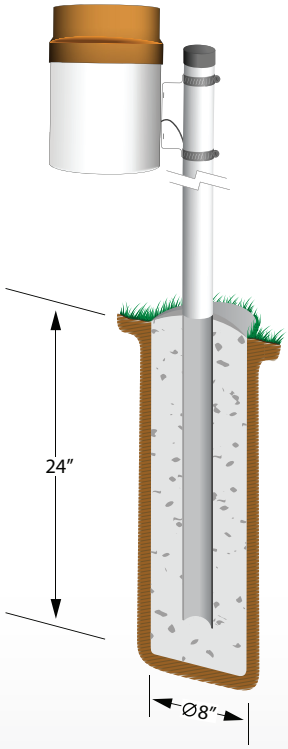
The TE525-series rain gages mount to a CM300-series Mounting Pole or a user-supplied 1.5 in. IPS pole. Several pedestal options are available to secure a CM300-series pole to the ground (see Ordering Information on page 2). Accurate measurements require the gage to be level.

Wind Screen

Campbell Scientific offers the 260-953 Wind Screen to help minimize the affect of wind on the rain measurements. This wind screen consists of 32 leaves that hang freely and swing as wind moves past them.

Snowfall Adapter

Campbell Scientific's CS705 Snowfall Conversion Adapter uses antifreeze to melt snow, allowing the TE525WS to measure the water content of snow. The CS705 cannot be directly used with either the TE525 or TE525MM. However, both the TE525 and TE525MM can be converted to a TE525WS by returning them to Campbell Scientific. For more information, refer to the CS705 brochure.



At left is a TE525 mounted onto a CM310 pole that is embedded directly in a concrete pad (-NP no pedestal base option).

Ordering Information

Tipping Bucket Raingages

Enter the cable length, in feet, after the -L. Recommended length is 25 ft, but many customers will order a 50 ft cable to place the gage away from the tower or tripod. Must choose a cable termination option.

TE525WS-L Tipping bucket with 8 inch diameter orifice and 0.01 in. tips.

TE525-L Tipping bucket with 6 inch diameter orifice and 0.01 in. tips.

TE525MM-L Tipping bucket with 24.5 cm diameter orifice and 0.1 mm tips.

Cable Termination Options (choose one)

-PT Cable terminates in stripped and tinned leads for direct connection to a datalogger's terminals.

-PW Cable terminates in a connector for attachment to a prewired enclosure.

-CWS Cable terminates in a connector for attachment to a CWS900-series interface. Connection to a CWS900-series interface allows this sensor to be used in a wireless sensor network.

-C Cable terminates in a connector for attachment to a CS110 Electric Field Meter or ET107 weather station.

-RQ Cable terminates in a connector for attachment to a RAWS-P Permanent Remote Automated Weather Station. This option is not offered for the TE525MM.

Mounting Poles

CM300 23 inch Mounting Pole with Cap

CM305 47 inch Mounting Pole with Cap

CM310 56 inch Mounting Pole with Cap

Pedestal Options for Mounting Poles (choose one)

-NP No Pedestal Base

-PJ CM340 Pedestal J-Bolt Kit

-PS CM350 Pedestal Short Legs (23 in. legs)

-PL CM355 Pedestal Long Legs (39 in. legs)

Common Accessories

CS705 Snowfall adapter for the TE525WS

10869 Four one-gallon containers of 50:50 PG:E Antifreeze; only U. S. ground shipments

CM270 CM270 Mounting Kit

260-953 Novalynx Alter-type Rain Gage Wind Screen

Specifications

	TE525	TE525WS	TE525MM
Sensor Type	tipping bucket/potted magnetic momentary contact reed switch		
Switch Ratings	30 Vdc at 2 A; 115 Vac at 1 A; closure time: 135 ms; bounce settling time: 0.75 ms		
Bucket Material	white powder coated spun aluminum		
Funnel Collector Material	gold anodized spun aluminum		
Screen Material	gold anodized spun aluminum		
Locking Snap Ring Material	stainless steel		
Operating Temperature	0° to +50°C (32° to 125°F)		
Resolution	1 tip		
Volume per Tip	4.73 ml/tip (0.16 fl. oz/tip)	8.24 ml/tip (0.28 fl. oz/tip)	4.73 ml/tip (0.16 fl. oz/tip)
Rainfall per Tip	0.01 in. (0.254 mm)		0.1 mm (0.004 in)
Accuracy	1.0% up to 2 in/hour (50 mm/hr)		
Knife Edge Funnel Collector Diameter	15.4 cm (6.1 in)	20.3 cm (8 in)	24.5 cm (9.7 in)
Height	24.1 cm (9.5 in)	26.7 cm (10.5 in)	29.2 cm (11.5 in)
Tipping Bucket Weight	0.9 kg (2 lb)	1 kg (2.2 lb)	1.1 kg (2.4 lb)
Cable	2-conductor shielded cable		
Cable Weight	0.1 kg (0.2 lb) per 10 ft length		
Warranty	three year		

APPENDIX B. CORRECTIVE ACTION REPORT

CORRECTIVE ACTION REPORT

PROJECT NAME _____

Identification of a Problem or Deficiency:

Created By: _____
Assigned To: _____
Date: _____
Verified Satisfactory: _____

Summary:

Corrective Action Taken and Results:

From: _____

Corrective Action Description:

Approved by: _____

APPENDIX C. STANDARD OPERATING PROCEDURES FOR PM_{10}, VOC, AND METEOROLOGICAL MONITORING

Calibration and Audit Standards Certification

SOP 69

July 28, 2022

Trinity Consultants

Approval of Standard Operating Procedures

Title: Calibration and Audit Standards Certification

SOP: 69

Approved By:

_____ Date:

Trinity Consultants

Project Director, Casey Lenhart

_____ Date:

Trinity Consultants

Quality Assurance Director, Linda Conger

Table of Contents

1.0 General Information

In accordance with EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, dated January 2017, reference materials are the standards against which many of the quality control checks are performed. Reference material can be gaseous standards as well as other devices. If these standards are not checked and verified as to their certified values, then the quality of data becomes suspect.

2.0 Calibration and Audit Standards Certification

Reference materials need to be certified and recertified at acceptable frequencies to maintain the integrity of the reference material. All calibration and audit standards will be certified annually or more frequent, as necessary, against National Institute of Standards and Technology (NIST), A2LA, or NVLAP certified or traceable standards. Standards used to audit or calibrate instruments are send for recertification. Upon return of the instruments, they will be acceptance tested to ensure the certification criteria held. Copies of all standards certifications will be kept in hard copy and in electronic form. A standards re-certification schedule will be kept and updated regularly. This schedule will include the audit/calibration standards make, model, serial number, certification date, and date when certifications are due. Calibration and audit standards will be clearly marked with who performed the certification, date of the certification, date due for certification, and standards ID number. Calibration or audit standards found to be out of certification will be tagged and segregated and not used until it is recertified. Calibrations will be performed using only calibration standards; audits will be performed using only audit standards. A list of PM calibration and audit standards and the schedule of recertification is presented in Table 1. Only NIST-traceable standards are used for PM monitoring programs.

TABLE 1 CALIBRATION AND AUDIT STANDARDS

Standard	Model	Serial Number	Date Due	Recertification Vendor
Sreamline Pro For FSA	Streamline Pro	S040205	**5/5/23**	Intermountain Laboratories Chinook Engineering 555 Absaraka (Marty Kjorstad) Sheridan, Wyoming 82801 (307) 672-7790 (800) 828-1407
Streamline Pro (MSI Standard)	Streamline Pro	M051202	**7/8/2022** Out for recertification	Intermountain Laboratories Chinook Engineering 555 Absaraka (Marty Kjorstad) Sheridan, Wyoming 82801 (307) 672-7790 (800) 828-1407
Streamline PRO For UTES	Streamline Pro	M080406	**3/24/23**	Intermountain Laboratories Chinook Engineering 555 Absaraka (Marty Kjorstad) Sheridan, Wyoming 82801 (307) 672-7790 (800) 828-1407
Streamline PRO	Streamline PRO	S210701	**7/29/22**	Marty Kjorstad
Streamline PRO For WDEQ	Streamline Pro	M100604	**5/5/23**	Intermountain Laboratories Chinook Engineering 555 Absaraka (Marty Kjorstad) Sheridan, Wyoming 82801 (307) 672-7790 (800) 828-1407
BGI Delta Cal	Delta Cal	000843	**4/1/23**	Mesa Labs (formerly BGI) *Include PO & RMA* 10 Park Place *w/instrument shipped* Butler, NJ 07405 ATTN: BGI SERVICE 303-987-8000 ext. 34
BGI Delta Cal	Delta Cal	1264 (Kennecott)	**8/18/22**	Mesa Labs (formerly BGI) *Include PO & RMA* 10 Park Place *w/instrument shipped* Butler, NJ 07405 973-492-8400 ATTN: BGI SERVICE 303-987-8000 ext. 34
BGI **AUDIT** DeltaCal	DeltaCal	138171	**8/18/2022**	Mesa Labs (formerly BGI) *Include PO & RMA* 10 Park Place *w/instrument shipped* Butler, NJ 07405 973-492-8400 ATTN: BGI SERVICE 303-987-8000 ext. 34
BGI DeltaCal	DeltaCal	167541 @ (AZR / Trinity - Brad Berglund)	**1/26/2023**	Mesa Labs (formerly BGI) *Include PO & RMA* 10 Park Place *w/instrument shipped* Butler, NJ 07405 973-492-8400 ATTN: BGI SERVICE 303-987-8000 ext. 34
BGI Delta CAL	Delta CAL	179773 was @ Guam	**9/29/22**	Intermountain Laboratories Chinook Engineering 555 Absaraka (Marty Kjorstad) Sheridan, Wyoming 82801 (307) 672-7790 (800) 828-1407
HiVol ORIFICE	Tisch	0379	**12/27/22**	Tisch Environmental, Inc. 145 South Miami Ave. Village of Cleves, OH 45002
HiVol ORIFICE	Tisch	2023	**12/27/22**	Tisch Environmental, Inc. 145 South Miami Ave. Village of Cleves, OH 45002
BIOS Dry Cal BIOS Dry Cal	Definer 220 High Flow Definer 220 Low Flow	140792 140619	**9/15/22** **8/26/22**	MESA LABS 10 Park Place Butler, NJ 07405 For RMA E Mail Denise McHale at mchd@biosint.com
BIOS Dry CAL BIOS Dry CAL	DEFENDER 530+ High Flow DEFENDER 530+ Low Flow	163582 168909	**8/26/2022** **8/26/2022**	MESA LABS 10 Park Place Butler, NJ 07405 For RMA E Mail Denise McHale at mchd@biosint.com

TABLE 1 CONTINUED CALIBRATION AND AUDIT STANDARDS

Standard	Model	Serial Number	Date Due	Recertification Vendor
REO TEMP Digital Thermometer	TM99-A	082818048 AUDIT KIT	10/12/22	Meteorological Solutions Laboratory
REO TEMP Digital Thermometer	TM99-A	082818046 KIT 4	10/12/22	Meteorological Solutions Laboratory
Brooklyn Digital Thermometer	6661S With utility probe	C404690 CAL KIT	2/14/23	Compared with MSI Reference Standard Thermometer. Brooklyn CT071007015-TM9
REOTEMP Digital Thermometer	TM99-A	072409005 KIT 3	2/14/23	Meteorological Solutions Laboratory
COOPER Digital Thermometer	TM99-A NEW	120219001 KIT 5	2/14/23	Compared with MSI Reference Standard Thermometer. Brooklyn CT071007015-TM9
Cole Parmer (MSI REFERENCE)	90080-12	122171278	6/10/23	SIMCO Electronics 65 Wadsworth Park Drive #101, Draper 84020 801-576-0790 tammy.barfield@simco.com
Suunto Digital Barometer	Escape	62900526 (AUD)	7/13/22	Meteorological Solutions Laboratory Compared with MSI Reference Standard Streamline Pro s/n: M051202
Suunto Digital Barometer	Escape	73403020 (CAL)	7/13/22	Meteorological Solutions Laboratory Compared with MSI Reference Standard Streamline Pro s/n: M051202
Vaisala	PTB110	G0770063 (AUD)	7/11/23	Meteorological Solutions Laboratory Compared with MSI Reference Standard Streamline Pro s/n: M051202
Vaisala	PTB110	G0770046 (CAL)	7/11/23	Meteorological Solutions Laboratory Compared with MSI Reference Standard Streamline Pro s/n: M051202
Vaisala	PTB110	C4240093 (KIT 4)	7/11/23	Meteorological Solutions Laboratory Compared with MSI Reference Standard Streamline Pro s/n: M051202
Vaisala SETRA (MSI LAB)	PTB 110 @ Indorama?? TREY ??	L0310539 (KIT 3) 3323980 (LAB)	7/11/23 7/12/22	Meteorological Solutions Laboratory Compared with MSI Reference Standard Streamline Pro s/n: M051202
Vaisala	PTB110	S3450170 (KIT 5)	3/24/23	Meteorological Solutions Laboratory Compared with MSI Reference Standard Streamline Pro s/n: M051202
Vaisala	PTB110	S2120892 (AUD-2)	3/24/23	Meteorological Solutions Laboratory Compared with MSI Reference Standard Streamline Pro s/n: M051202
Cooper Digital	TM99A	101314015 (AUD-2)	3/22/23	Meteorological Solutions Laboratory Compared with MSI Reference Standard Streamline Pro s/n: M051202

In-House Calibration of Test Equipment

SOP 106

December 7, 2020

Trinity Consultants

Approval of Standard Operating Procedures

Title: In-House Calibration of Test Equipment

SOP: 106

Approved By:

_____ Date:

Trinity Consultants

Project Director, Casey Lenhart

_____ Date:

Trinity Consultants

Quality Assurance Director, Linda Conger

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Trinity Consultants

1.0 General Information

In accordance with EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, dated January 2017, reference materials are the standards against which many of the quality control checks are performed. Reference material can be gaseous standards as well as other devices. If these standards are not checked and verified as to their certified values, then the quality of data becomes suspect. All calibration and/or audit standards will be sent to a certifying authority with standards traceable to National Institute of Standards and Technology (NIST). Calibration standards will only be used for calibration verification activities; audit standards will only be used for audit activities.

2.0 In-House Equipment Verification

Measuring devices such as thermometers and relative humidity devices will be verified annually, and calibration verifications performed at prescribed intervals and whenever the accuracy of the device appears to be suspect. Calibration verifications shall be performed against and traceable to certified calibration/verification standards having known valid relationships to NIST traceable calibration equipment. Monitoring equipment which is new, and which has not been certified against a known reference standard or is out of calibration shall be segregated and not used until a calibration verification has been established. Standards which are new and where a certification was not performed previously, not traceable to NIST or is expired, shall be segregated and not used until a certification traceable to NIST has been established. Audit and calibration standard verifications will be conducted against NIST traceable standards.

A record of the annual calibration or audit verification will be established and will include documentation of all necessary information.

A mass flow controller (MFC) is calibrated by connecting a NIST traceable standard to the output of the MFC. Ten to twenty separate points are compared between the MFC and reference standard readings over the MFC's entire flow range. If the MFC points are greater than 2% off from the reference standard reading, the points are set to match the reference standard and an "after adjustment" full calibration is performed to ensure the points are correct.

Equipment Inventory Procedure

SOP 107

December 7, 2020

Trinity Consultants

Approval of Standard Operating Procedures

Title: Equipment Inventory Procedure

SOP: 107

Approved By:

_____ Date:

Trinity Consultants

Project Director, Casey Lenhart

_____ Date:

Trinity Consultants

Quality Assurance Director, Linda Conger

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Trinity Consultants

1.0 Inventory Procedure

Once procured equipment arrives at Trinity's instrument laboratory, the packing slip is removed from the box and compared against the purchase requisition form. By project, an equipment inventory form is filled out electronically which documents the components ordered, date received, and equipment serial number by Trinity instrumentation specialists. Inventory is tracked by Trinity's senior instrument technician. Copies of the project equipment inventory forms are available in the requisite project directory. An example of this form is presented below. Packing slips are placed in the project file.

Example Equipment Inventory Form

EQUIPMENT INVENTORY SHEET

Trinity Consultants

Description	Serial #	Make	Date Ordered	Date Received	Item Location	Log In/ Date	Log Out/Date

Computer Program Validation

SOP 117

October 26, 2020

Trinity Consultants

Approval of Standard Operating Procedures

Title: Computer Program Validation

SOP: 117

Approved By:

_____ Date:

Trinity Consultants

Project Director, Casey Lenhart

_____ Date:

Trinity Consultants

Quality Assurance Director, Linda Conger

_____ Date:

Trinity Consultants

Data Manager, Wyndam Lewis

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1.0 Introduction

The procedures defined in this document are used in the validation of computer software written by Trinity programmers.

2.0 Software Verification

Software verification shall be performed during software development and after software changes to ensure that the software adequately and correctly performs all intended functions. The verification tests shall demonstrate the capability of the computer program to produce valid results for test problems encompassing the range of permitted usage defined by the program documentation. Acceptable test problem solutions are:

A. Hand calculations;

B. Calculations using comparable proven programs; or

C. Empirical data and information from technical literature.

Depending on the complexity of the computer program being tested, testing can range from a single test of the completed computer program to a series of tests performed at various stages of computer program development to verify correct translation between stages and proper working order of individual modules, followed by an overall computer program test.

3.0 Test Procedures

Test procedures or plans shall specify the following, if applicable:

A. Required tests and test sequence;

B. Required ranges of input parameters;

C. Identification of the stages at which testing is required;

D. Criteria for establishing test cases;

E. Requirements for testing logic branches;

F. Requirements for hardware integration;

G. Anticipated output values;

H. Acceptance criteria; and

I. Reports and records.

All test results developed over the testing of the software will be thoroughly documented. A Quality Assurance Form (Figure 1) will be filled out when a calculation is being verified. Test problems shall be run whenever the computer program or software updates are installed on a different computer or when significant hardware changes are made. Verification test results shall be evaluated by a responsible authority to assure that test requirements have been established.

4.0 Test Records

Verification test records shall identify the following:

A. Computer program tested;

B. Computer hardware used;

C. Date of test;

D. Test problems;

E. Tester or data recorder;

F. Results and acceptability;

G. Action taken in connection with any deviations noted; and

H. Person evaluating test results.

5.0 Software Validation

Software validation is performed at the end of the implementation phase to ensure that the code satisfies the requirements. Testing shall be the primary method of software validation. The validation of modifications shall be subject to selective regression testing to detect errors introduced during the modification of systems or system components, to verify that the modifications have not caused unintended adverse effects, or to verify that a modified system(s) or system component(s) still meets specified requirements. The selective regression testing analyzes the impact of the new code on the software's existing code.

6.0 Software Configuration Control

A configuration baseline shall be defined at the completion of each phase of the software development. Approved changes created subsequent to a baseline shall be added to the baseline. A baseline shall define the most recent approved software configuration. A labeling system shall be implemented that:

A. Uniquely identified each configuration item;

B. Identifies changes to configuration items; and

C. Provides the ability to uniquely identify each configuration of the revised software available for use.

Changes to software shall be formally documented. This documentation shall contain a description of the change, the reason for the change, and the identification of what was affected by the change. Software verification activities shall be performed for the change, as necessary to ensure the change is appropriately reflected in the software documentation and to ensure traceability. Software validation shall be performed for the change. Before updates or modifying software, ensure that the system is backed-up, and allow for a trial period utilizing all program features used in the past. Review results of system with update to ensure seamless and or improved performance without adverse effects on results (calculations compute correctly, no detrimental effects on other parts of the software program relied on).

7.0 User Documentation

User documentation at a minimum should include:

A. User instructions that contain an introduction, a description of the user=s interaction with the software, and a description of the necessary training to use the software;

B. Input and output specifications;

C. Input and output formats;

D. A description of system limitations;

E. A description of anticipated errors and how the user can respond; and

F. Information for obtaining user and maintenance support.

8.0 Problem Reporting and Corrective Action

A formal procedure of software problem and corrective action shall be established for software errors and failures. This reporting system shall assure that problems are promptly reported. Corrective action shall assure that problems are identified, evaluated, documented, and corrected. Problems should be assessed to determine the impact on past and present applications. Preventive actions and corrective results should be provided to the necessary division within Trinity.

Figure 1. Quality Assurance Form

Reviewer:	
Project:	
Date:	
Time:	
Findings/Calculations:	

Met One E-Sampler Operation

SOP 222

June 10, 2022

Trinity Consultants

Approval of Standard Operating Procedures

Title: Met One E-Sampler Operation

SOP: 222

Approved By:

_____ Date:

Trinity Consultants

Project Director, Casey Lenhart

_____ Date:

Trinity Consultants

Quality Assurance Director, Linda Conger

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Trinity Consultants

1.0 Introduction

This Standard Operating Procedure (SOP) describes procedures used by Trinity to operate, maintain, and calibrate the Met One E-Sampler to measure particulate in ambient air. The Met One Instruments, Inc. model E-Sampler is a type of nephelometer which automatically measures and records real-time airborne PM_{10}, $PM_{2.5}$, or TSP particulate concentration levels using the principle of forward laser light scatter. In addition, the E-Sampler has a built-in 47 mm filter sampler which can optionally be used to collect the particulate for subsequent gravimetric mass or laboratory evaluation. The E-Sampler combines the excellent real-time response of a nephelometer with the accuracy and traceability of a low flow manual gravimetric sampler.

2.0 Principle of Operation

Sample air is drawn into the E-Sampler and through the laser optical module, where the particulate in the sample air stream scatters the laser light through reflective and refractive properties. This scattered light is collected onto a photodiode detector at a near-forward angle, and the resulting electronic signal is processed to determine a continuous, real-time measurement of airborne particulate mass concentrations.

After the sample air stream has been measured by the E-Sampler and exits the optical engine, it passes through the built-in 47 mm filter sampler system. This system allows the particulate to optionally be collected on a filter disc as a second method to obtain airborne particulate mass data, or for laboratory analysis of the particulate.

The 47 mm filter system can also be used to determine a gravimetric K-factor (slope multiplier) to correct the E-Sampler real-time signal to match the local particulate type. In this case, a filter disc is weighed on a microbalance before and after being run in the E-Sampler for a period of time. The resulting mass of the dust on the filter is correlated with the concentrations that the E-Sampler recorded over the same time, and a correction factor is calculated. The E-Sampler can be used with no correction factor in applications where relative particulate trending is appropriate.

NOTE: This system contains a diode laser operating at 5 mW power and 670 nm wavelength. This is visible to the naked eye and can cause damage to the eye if directly exposed.

3.0 Acceptance Test

Check the sensor carefully for any signs of shipping damage. If damage occurred during transport, immediately file a claim with the carrier. Contact manufacturer or vendor to facilitate repair or replacement. Check functionality of E-sampler. An example acceptance testing form to be completed is presented as Figure 1.

FIGURE 1 EXAMPLE ACCEPTANCE TESTING FORM

ACCEPTANCE TEST FORM

Trinity Consultants

Make _____
Model _____
Serial Number _____

Date	Action/Result

4.0 Installation

The E-Sampler can be mounted to a pole, mast, or wall using the included mounting bracket. The bracket must be screwed or bolted to the pole or wall with appropriate hardware. The enclosed bolts may not be appropriate for the desired mounting. The slot on the back of the E-Sampler slips over the tab on the mounting bracket. The tab on the bottom of the E-Sampler should also be bolted to the mounting surface to ensure that the unit cannot be knocked off of its mounting.

If mounting the unit to a wall, take care to ensure that there is adequate clear space around the inlet to allow unrestricted airflow into the instrument. Wall mounting is often not considered ideal and not recommended due to the airflow and particulate obstruction of the wall itself. Mount the instrument with no large obstructions nearby whenever possible.

The Met One EX-905 aluminum tripod is the recommended mounting for the E-Sampler for most outdoor applications. Deploy the tripod as follows:

1. Remove the three stainless steel detent pins from the tripod base by pulling the rings. Unfold the three tripod legs and reinsert the three pins so that each pin secures a leg in the open position. Make sure the erected tripod is rigid and stable.

2. Lift the E-Sampler assembly and slide the slot on the back of the E-BAM over the tab on the top of the tripod. Insert the supplied ¼-20 bolt through the tab on the bottom of the E-Sampler and through the hole in the body of the tripod. Secure it with the supplied washers and nut. This prevents the E-Sampler from falling or shifting on the tripod.

3. Site the tripod on a surface that is as level as possible. The tripod feet may be secured to the ground or mounting surface with bolts, screws, or tent pegs if necessary. Secure the tripod in windy conditions.

Set up the rest of the E-Sampler hardware items and accessories as described below:

1. Installing the inlet heater assembly: The E-Sampler may be shipped with the inlet heater assembly disconnected for easier packing. Connect the heater power harness to the mating connector coming out of the top of the E-Sampler. Slip the inlet tube and heater assembly onto the top of the E-Sampler. Make sure that the inner aluminum inlet tube is seated fully into the receiver inside the top of the E-Sampler. You may need to loosen the waterproof fitting at the top of the heater assembly to seat the inlet tube correctly. Align the three holes in the base of the plastic heater body with the threaded holes in the flange on top of the E-Sampler and fasten with three 6-32 socket head screws and lock washers as provided. Tighten the waterproof fitting on top of the heater assembly securely to prevent leaks.

2. **Install PM_{10}, $PM_{2.5}$ or PM_1 cyclones and TSP inlets:** For TSP (Total Suspended Particulate) monitoring, the included weatherproof TSP inlet is simply installed directly onto the top of the E-Sampler inlet tube to keep water, insects, and debris out of the instrument. For PM_{10} or $PM_{2.5}$ monitoring, the optional sharp-cut cyclone of the desired cut-point must be installed onto the inlet tube, under the TSP inlet. Lubricate the o-rings if necessary. Never operate the E-Sampler outdoors without at least the TSP inlet in place, as the resulting water/debris damage is not covered under warranty.

3. **Internal Battery:** If the unit is to be used with the optional internal 12V battery, connect the included two-wire fused battery harness between the battery and the 2-pin connector J4 on the 81220-connector board inside the battery tray of the E-Sampler (red positive, black negative). Route the fish tape strap around the back of the battery and slide the battery into the tray. Do not ship or transport the unit with the battery installed.

4. **AC Power Supply:** If the E-Sampler is to be operated on AC line voltage, install the included power supply. Bolt it to one of the legs of the tripod with the included U-bolts. Plug the power supply output cable into the DC power input on the bottom of the E-Sampler. When the power supply is plugged into AC power, the E-Sampler will turn itself on automatically. This power supply is also used to charge the optional internal 12-volt battery.

5. **47 mm filter holder:** A 47 mm filter cassette assembly must be always installed in the filter sampler position, in order to seal the flow system. Pull down the spring-loaded filter clamp lever on the front of the unit to insert or remove the filter holder. Note: The filter cassette does not need to have a filter disc installed in it for real-time nephelometer operation, unless collection of the sample dust is desired. The backing screen disc may be left in the cassette. If a filter disc is installed, it must be removed after an appropriate amount of time. If forgotten, it will continue to collect dust until the flow system cannot continue to function.

5.0 Instrument Siting

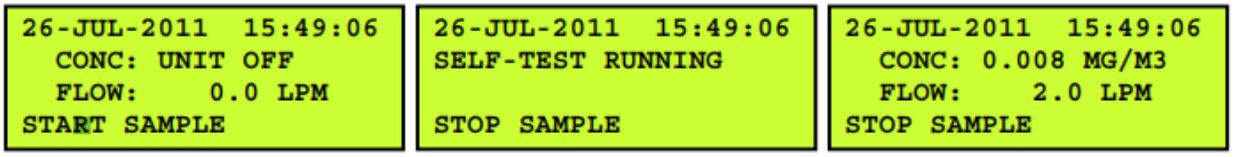
The instrument should be sited in accordance with the United States Environmental Protection Agency (U.S. EPA) Title 40, Code of Federal Regulations Part 58 Appendix E "Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring" and Quality Assurance Guidance Document 2.12 "Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods" Section 5.1.2.

6.0 Starting Operation

As soon as power is applied to the E-Sampler, the unit will boot up and display the **ABOUT** screen showing the firmware revision for a few seconds. The **ABOUT** screen can also be viewed through the menu system.

The E-Sampler will then default to the **OPERATE** screen as shown below. The **START SAMPLE** key (left menu key) must be pressed to start the unit. A confirmation screen will appear; press the YES key. The E-Sampler will begin by running a **SELF-TEST** process for about three minutes, where the optical zero and span functions will be checked.

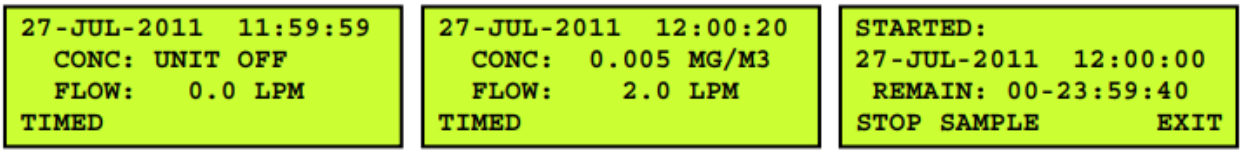
After the **SELF-Test** process, the E-Sampler is running and ready to use, if set to **CONTINUOUS** sampling mode. The date, time, real-time concentration, flow rate, and sampling condition are displayed.



Pressing the ▼ down arrow displays the other current sensor readings for AT, BP, RH, WS, WD, and battery voltage. Pressing the ◄ left arrow key will scroll back through historical logged data. Press **ESC** at any time to immediately return to the current concentration screen from any historical data screen. The **MENU/SELECT** key may be pressed at any time to enter the main menu screen. This screen is the top of a tree style menu system. Use the arrow keys to highlight an entry and press **MENU/SELECT** again to select that entry. Pressing **ESC** will back up along the tree to the top

The E-Sampler can be set to **TIMED** operation mode whenever a scheduled sampling event with a user-defined start time and duration is needed. This mode is usually used when a 47 mm filter is installed in the E-Sampler for a fixed amount of time, such as when performing a 24-hour filter samples similar to an FRM, when running an E-Sampler along with a collocated instrument with synchronized sample periods, or when creating a K-Factor using the filter collection method. Timed mode may also be used whenever the E-Sampler is set up early with a future start time to save power.

Only one timed event can be scheduled at a time. The **SETUP > SAMPLING MODE** screen is used to enable **TIMED** operation, and to set the start time and duration of the event.



When waiting for a scheduled event, the E-Sampler main **OPERATE** screen will display **UNIT OFF** for the **CONC** value as shown above left. Once a timed event has started, the E-Sampler will show the current concentration and flow rate as shown above middle. The **TIMED** soft key can be pressed to view to the **TIMED STATUS** screen with the start time and the remaining countdown time for the event as sown above right. The **STOP SAMPLE** soft key can be used to stop the event if needed. The **EXIT** or **ESC** keys return to the main **OPERATE** screen.

7.0 Establishing a Gravimetric Correction K-Factor for the E-Sampler

The main limitation of most nephelometer instruments is that the accuracy of the mass output can be negatively affected by variations in size, color, shape, and index of refraction of the sampled particles. One of the most important uses for the 47 mm filter system is determination of a gravimetric K-factor (slope multiplier) to correct the E-Sampler signal to compensate for local particulate characteristics.

A gravimetric K-Factor MUST be generated for the E-Sampler if accurate concentration measurements and good agreements with FRM or FEM methods are expected. In some applications the appropriate K-Factor will be quite significant, such as a multiplier of 3 or 4 or even more. Once determined, the K-Factor will generally remain valid for that unit and site as long as the particulate type is consistent. The E-Sampler can be used with no correction factor (K=1) in applications where relative particulate trending is appropriate.

A filter disc is carefully weighed on a microbalance scale under laboratory conditions, then placed into the E-Sampler filter holder and run for a predetermined period of time. The filter is then reweighed in the lab, and the resulting total mass of the dust on the filter is correlated with the volume of air sampled, and compared with the concentrations that the E-Sampler recorded over the same time period, then a correction factor is calculated:

1. Download and save all E-Sampler data before changing the K-Factor setting! When the K-Factor value is changed, it will apply to any previous data already stored in memory.
2. Obtain and/or pre-weigh the 47 mm disc filters on a microbalance scale according to standard protocol. Pre-weighed filters are often obtained from an outside lab due to the expense of the scale and lab setup. See 40 CFR Part 50 for more information. Many agencies already have appropriate filters for use in reference method filter samplers on hand. Met One recommends using PTFE/Teflon filters.
3. Install and check the E-Sampler. Make sure that the sampling site is representative of the local air. Use FRM/FEM siting criteria whenever possible. Make sure that the E-Sampler passes all leak checks and flow calibrations.

4. Determine the length of the sampling period. For good gravimetric results, there should be about 0.5 mg (500 µg) of mass deposited on the filter if possible. The time it takes to accumulate this amount of dust will vary greatly depending how clean or dirty the air is. In normal ambient air at moderate concentrations, you will usually need to run the E-Sampler for about 4 or 5 days.

If the typical average daily concentration levels at the site are known, then you can calculate the required run time based on the known value. You could also make a rough estimate by running the E-Sampler for a day, then download the real-time data and calculate the average in mg/m3 over the period.

For example, the E-Sampler draws 0.12 cubic meters per hour (at 2.0 LPM). If the average daily concentration at the site is about 0.035 mg/m^3, and you want .5 mg on the filter, then:

$$0.035 \text{ mg/m}^3 * 0.12 \text{ m}^3\text{/hr} = 0.0042 \text{ mg/hr}$$

$$0.500 \text{ mg} / 0.0042 \text{ mg/hr} = 119 \text{ hours} (\approx 5 \text{ days})$$

5. Set the E-Sampler to **TIMED** sampling mode and set the event duration to the estimated amount of time determined in step 3, or a similar convenient interval. Install the 47 mm filter and run the timed sample.
6. After the sample period has ended, remove the 47 mm filter and have it re-weighed in the lab. The filter must be handled carefully, transported carefully, and equilibrated properly.
7. Download the E-Sampler light scatter data and average the E-Sampler concentration data values for the entire sample period.
8. Evaluate the E-Sampler total flow over the sample period. First, check the flow values in the downloaded data for proper 2.0 lpm regulation. The E-Sampler data does not record the sample volume, so you must calculate it. If the E-Sampler ran for five days (120 hours), then the nominal sample volume would be 2.0 lpm, times 60 min/hr, times 120 hours. This equals 14,400 liters or 14.4 cubic meters of nominal sample volume.

However, you must also compensate for the fact that the sample stops for about 2.8 minutes each time the automatic self-test ran during the timed sample. For example: If the E-Sampler was set to hourly self-test, then 2.8 minutes of each hour would not have flow going through the 47 mm filter. This amounts to 2.0 lpm times 2.8 minutes, or 5.6 liters of air per hour. So, if the sample ran for five days (120 hours), then 5.6 liters per hour times 120 hours equals 672 liters. The corrected total sample volume would then be 14,400 – 672 = 13,728 liters, or 13.728 cubic meters. Note: 1 cubic meter equals 1000 cubic liters.

9. Use the change in mass results from the gravimetric filter analysis (the difference between the clean and dirty filter weight in mg) and the total sample flow volume (m3) through the filter to calculate the concentration of particulate on the 47 mm filter in mg/m^3. The concentration is calculated as total mass divided by total sample volume.

For example, if the clean filter weighed 77.643 mg and the dirty filter weighed 78.345 mg, then the total particulate mass on the filter would be 78.345 minus 77.643, or 0.702 mg. If the total sample volume was 13.728 cubic meters, then the filter total concentration would be 0.702 mg divided by 13.728 m^3, or 0.051 mg/m^3.

10. Calculate the K-Factor as the 47 mm filter total concentration divided by the E-Sampler total light scatter concentration. For example, if the filter total concentration was 0.051 mg/m3 and the E-Sampler total concentration was 0.038 mg/m3, then the K-Factor would be 0.051 divided by 0.038 or 1.342.

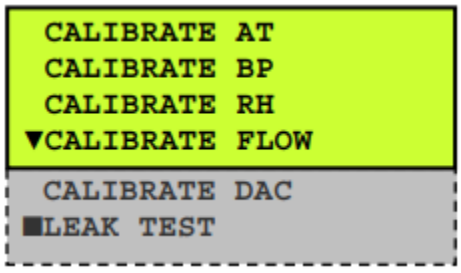
Program the calculated K-Factor into the E-Sampler **SETUP > CONCENTRATION** menu. The E-Sampler will multiply all stored and subsequent concentration measurements by the K-Factor.

8.0 Sampler Calibration

The E-Sampler has a system of calibration menus which allow the operator to audit or calibrate the airflow control system parameters for optimal performance. These parameters are often audited monthly and calibrated quarterly during continuous operation.

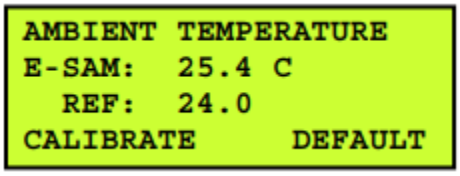
Note: The E-Sampler temperature, pressure, and leak status should always be checked before any flow calibrations are performed, since the flow calculation is dependent on these parameters.

The **CALIBRATE** menu is located in the main E-Sampler menu. Use the arrow keys to select CALIBRATE option in the main menu, then press the MENU/SELECT key to enter the menu. Use the ▲▼ keys to select the desired sub-menu and press the SELECT key again to enter. The top CALIBRATE menu is shown below:



8.1 Ambient Temperature Calibration

The **CALIBRATE AT** screen is used for field audits or calibrations of the ambient temperature measurement of the E-Sampler.



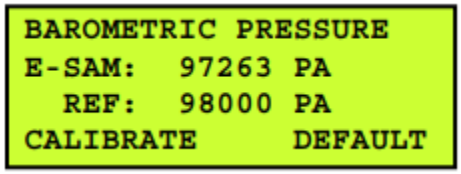
The **E-SAM** parameter is the current reading from the E-Sampler temperature sensor. The **REF** parameter is where you can enter the correct value from your traceable temperature standard, using the arrow keys. The **E-SAM** value should change to match the **REF** value when you press the **CALIBRATE** soft key.

The **DEFAULT** soft key can be pressed to clear out all previous field calibrations and restore the factory calibration for the sensor. Use this if difficulty is encountered during the calibration. Press **ESC** to escape without changes.

Note: The E-Sampler ambient temperature sensor is an unshielded thermistor bead located in the bottom of the enclosure, and as such is not particularly accurate compared to solar shielded or aspirated sensors. An accuracy of ±2 degrees C is adequate for flow control purposes.

8.2 Ambient Pressure Calibration

The **CALIBRATE BP** screen is used for field audits or calibrations of the ambient barometric pressure measurement of the E-Sampler.

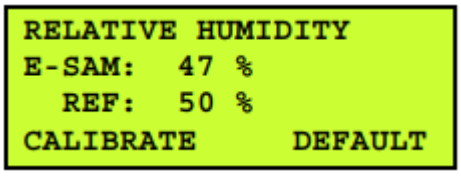


The **E-SAM** parameter is the current reading from the E-Sampler pressure sensor. The **REF** parameter is where you can enter the correct value from your traceable pressure standard, using the arrow keys. You will need to convert units if your standard outputs pressure in other units. The **E-SAM** value should change to match the **REF** value when you press the **CALIBRATE** soft key.

The **DEFAULT** soft key can be pressed to clear out all previous field calibrations and restore the factory calibration for the sensor. Use this if difficulty is encountered during the calibration. Press **ESC** to escape without changes.

8.3 External RH Sensor Calibration

The **CALIBRATE RH** screen is used for field audits or calibrations of the optional EX-593 external relative humidity measurement of the E-Sampler.



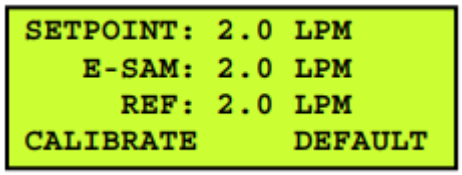
The **E-SAM** parameter is the current reading from the E-Sampler external RH sensor. The **REF** parameter is where you can enter the correct value from your traceable humidity standard, using the arrow keys. The E-SAM value should change to match the **REF** value when you press the **CALIBRATE** soft key.

The **DEFAULT** soft key can be pressed to clear out all previous field calibrations and restore the factory calibration for the sensor. Use this if difficulty is encountered during the calibration. Press **ESC** to escape without changes.

Note: This calibration screen does not apply to the internal E-Sampler filter RH sensor, which is located inside the top of the 47 mm filter cartridge receiver. The internal sensor can be audited by checking the RHi value in the main operate screen during operation. The internal RH sensor cannot be calibrated. If the E-Sampler has been operating with the inlet heater running, the inside temperature of the unit will be hotter than ambient, resulting in internal RH readings that will be lower than an ambient RH standard. If the sample RH sensor is to be audited, make sure that the inlet heater is off, and the unit has equilibrated to ambient conditions first. If the internal RH sensor fails, it will usually read an impossible value such as 125% or -25%.

8.4 Flow Sensor Calibration

The **CALIBRATE FLOW** screen is used for field audits or calibrations of the sample flow measurement of the E-Sampler. Remove the TSP inlet and any cyclones from the E-Sampler inlet tube, and then connect the top of inlet tube to the outlet of your traceable flow meter using a length of appropriate flexible tubing. The E-Sampler temperature, pressure, and leak status must be checked before performing any flow calibrations in order to prevent errors. The E-Sampler flow rate should be maintained to within ±0.1 LPM (1.9 to 2.1 LPM) for proper air volume total calculation when used with a 47 mm filter, and for proper cut-point performance of inlet cyclones.



The **SETPOINT** parameter is the target flow rate that the E-Sampler will attempt to regulate to.

The **E-SAM** parameter is the current reading from the E-Sampler flow sensor, in actual volumetric liters per minute. The E-Sampler should automatically regulate to the setpoint (2.0 LPM) when the flow calibration screen is entered. This may take a moment.

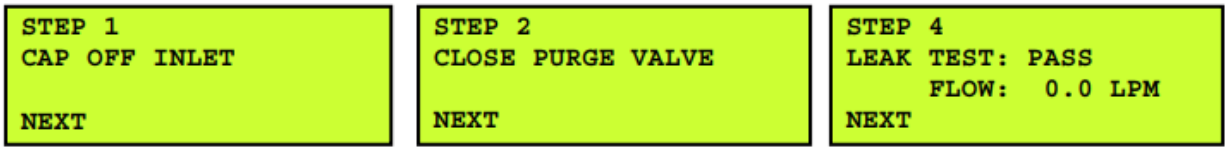
The **REF** parameter is where you can enter the correct value from your traceable flow meter, using the arrow keys. **The flow reading that you enter must be in actual conditions**. The **ESAM** value should change to match the **REF** value when you press the **CALIBRATE** soft key.

The **DEFAULT** soft key can be pressed to clear out all previous field calibrations and restore the factory calibration for the sensor. Use this if difficulty is encountered during the calibration. Press **ESC** to escape without changes.

Note: To audit the E-Sampler flow rate without changing the calibration, simply compare the **ESAM** value to your traceable standard and record the results. If the CALIBRATE soft key is not pressed, then no flow calibrations are affected.

9.0 Leak Test Calibration Checks

The **CALIBRATE > LEAK CHECK** screen is used to check for airflow system leaks which could affect the accuracy of the flow measurements or cause unwanted measurement biases.



Purge valve closed (Left) and open (Right). Located inside the battery compartment

1. Remove TSP inlet and sharp cut cyclone. Cap off the top of the E-Sampler inlet tube with a vinyl or rubber cap.
2. Open the front door and remove the battery cover plate. Locate the purge cutoff value on the left side. Rotate it to the closed position as shown above.
3. Wait for the system to zero the flow sensor reading. This step is omitted if steps 1 and 2 take longer than the zero-flow sensor process (~25 seconds). The software will automatically proceed to the step 4 when the zero-flow sensor process has completed.
4. The pump is turned on. Wait for flow reading to stabilize (about 2 minutes). The leak test will be OK if the reading is less than 0.3 liters-per-minute. The leak test will FAIL if the reading is greater than 0.3 liters-per-minute. Do not run this test for more than 5 minutes because it will reduce the lifetime of the pump motor.
5. The pump is turned off. Rotate the purge cutoff valve back to the open position as shown. Remove the vinyl cap from inlet and re-install the sharp cut cyclone

10.0 Personnel Qualifications

Installation, operation, maintenance, repair or calibration of the instrument and all support equipment will be performed by properly trained personnel. Personnel will meet all minimum requirements and qualifications commensurate with their position.

11.0 Troubleshooting Procedures

The E-Sampler contains a comprehensive system of error and alarm codes which are used to alert the operator to any problems with the unit. These error codes may be generated during normal operation or during a self-test routine. The errors appear on the E-Sampler display and are also stored in the digital alarm log as a detailed record of the time and type of the error. In addition, errors are stored in the digital data log as a code number in the data array. Table 1 describes each of the error and alarm types which can be generated by the E-Sampler, along with the conditions which cause the alarms. Many of these alarms indicate critical parameters which must be working correctly for machine operation.

TABLE 1 ERROR AND ALARM DESCRIPTIONS

Alarm/Error Message	Alarm Description
POWER OUTAGE	This alarm message indicates that the E-Sampler power has been cycled off and then back on. This can mean that there was a power failure or that someone simply unplugged the unit to turn it off. The E-Sampler alarm display will show OFF time indicating how long the power was off, and ON time indicating how long the power was on before the power failure. A second type of power alarm can be shown on the display as a COP RESET. This means "Computer Operating Properly", and will only occur when the E-Sampler firmware is flash updated by the user. This is normal and does not indicate a failure.
INTERNAL COMM DOWN!	This alarm indicates that there was an internal SPI bus failure, preventing the CPU from communicating with the I/O board for 10 seconds or more. The time and date of the error will be displayed. The E-Sampler will stop operation until internal communication is restored. If these errors occur regularly you will need to contact Met One.
LASER FAILURE	Refer to the Laser Current Logic section. Occurs when the MD engine laser current is out of range. The alarm is cleared when the laser current is within range.
ZERO CALIBRATE ERROR	Refer to the Self-Test Event logic section. Occurs during the Self-Test event when the MD engine output is out of range. The alarm is cleared when the next event does not fail.
ZERO STABILITY ERROR	Refer to the Self-Test logic section. Occurs during the Self-Test event when the MD engine output stability is out of range. The alarm is cleared when the next event does not fail.
PRESSURE FAILED	This occurs when communication is lost to the digital pressure sensor. A failed pressure sensor is forced to read 101306 Pa or 29.9 inHg.
FLOW FAILED	This alarm indicates that the flow system is more than 5% out of regulation for more than 5 minutes. The alarm display will show the actual flow rate and the time and date of the error.
BATTERY WARNING or BATTERY FAILED	The BATTERY WARNING occurs if the input voltage drops below 11.2 volts and clears when the voltage restores to above 11.7 volts. BATTERY FAILED occurs if the voltage drops below 10.5 volts and clears when the voltage restores to above 11.7 volts. The time and date of the error will be displayed, along with the actual voltage.
DETECTOR ERROR	Refer to the Self-Test Event logic. Occurs during the Self-Test event when the MD engine output is out of range. The alarm is cleared when the next event does not fail.
SOLENOID ERROR	Refer to the Self-Test Event logic section. Occurs during the Self-Test event when the MD engine output is out of range. The alarm is cleared when the next event does not fail.

Table 2 defines the possible error codes that can appear in the "alarm" column of the E-Sampler data records:

TABLE 2 ERROR CODES

Code	Error/Alarm Type
0	No alarm
1	Self-Test Failure
2	Not Used
4	Laser Current Failure
8	Pressure Sensor Failure
16	Flow Failure
32	Not Used
64	Internal Hardware (SPI bus) Failure
128	Low Battery

Note: If multiple errors or alarms occur in the same data period, then the alarm code stored in the data array will be the sum of the two individual code numbers.

Table 3 contains information on some of the more common E-Sampler problems which may be encountered, and some steps to identify and remedy the problems.

TABLE 3 COMMON E-SAMPLER PROBLEMS

Problem:	The E-Sampler won't start a measurement cycle.
Cause/Solution:	• You must press the START SAMPLE key to start continuous operation. • If the unit is set for a TIMED sample, it will not start until the scheduled time. The main display will show the TIMED soft key where you can view the schedule. • The E-Sampler may not start a measurement cycle if it detects a hardware failure, such as a pressure sensor failure or a pump failure. • The unit will not start a cycle if the input DC voltage is below the restart threshold, such as 10 volts DC. • The unit will usually display an error message on the display if it cannot start a cycle.

Problem:	Flow failures or low flow.
Cause/Solution:	• Make sure a 47 mm filter disc has not been installed and forgotten. This will eventually block the flow. • Clean the 47 mm filter cartridge backing screen, even if no filter is used. Large particles can plug this screen. • Check the PUMP and PURGE filters. These must be replaced periodically.

	• Try to DEFAULT the flow sensor calibration. If corrupted flow cal parameters are entered into the flow calibration, it may appear that the flow system is not working. • Check for insect debris or other obstructions in the small exhaust port on the bottom of the unit. It is located in the back right corner. • Verify the AT and BP sensors function. Failed sensors can affect the flow. • The sample pump itself will eventually wear out and need to be replaced. It should last at least a year under normal conditions. Check the other possibilities first.

Problem:	**Leak check failures**
Cause/Solution:	• Make sure that a 47 mm filter holder cartridge is installed correctly, even if no filter is used. It is required to seal the flow system. • Check or replace the 47 mm filter cartridge itself. Some of these can leak at the interface of the two halves of the assembly. • Make sure that the inlet is completely blocked with a rubber or vinyl cap during the leak check. Using a finger to block the inlet is not sufficient. • Make sure the inlet tube is fully seated into the top of the optical module, especially if you installed the inlet heater assembly yourself. You may need to temporarily loosen the weatherproof fitting at the top of the inlet tube to get it seated correctly. • Make sure the PUMP and PURGE filter holders are tightened fully.

Problem:	**Optical system alarms and failures**
Cause/Solution:	• The E-Sampler must be periodically returned to the factory for optical system cleaning. The period will depend on your particulate levels. • Check the PURGE filter and replace it as needed. • Make sure the manual purge valve (inside the battery compartment) is OPEN (parallel to the tubing) during normal operation. If closed, the unit will still function normally, but no purge air will circulate around the optics to keep them clean! • The laser diode has a finite lifetime which will be reduced at high temperatures. It may eventually fail and need to be replaced at the factory. • Never disassemble the MD laser optical subassembly! • Some users reported problems with the laser shutter span solenoid sticking or failing to activate in cold weather. Met One has changed to a different type of solenoid and control logic for units with R2.0.0 or later firmware (late 2012). Contact Met One if difficulty with the self-test auto span is encountered on an older unit.

Problem:	**The E-Sampler data does not match BAM or FRM data at the same site**
Cause/Solution:	• A K-Factor (multiplier) must be established for good accuracy and correlation to collocated instruments. The K-Factor will sometimes be very significant, such as a multiplier of 3 or 5. See Section 5.5. • The E-Sampler is calibrated on latex 0.6 micron micro-spheres. These provide an extremely consistent calibration, but do not generally match the characteristics of ambient particulate. • The K-Factor is only valid at the same site and for the same particulate type. If the local particulate source changes, the K-Factor may no longer be valid. • The E-Sampler TSP inlet is designed for low winds only. High winds may cause a cut-point in the TSP inlet itself. • Make sure the correct cyclone is used on the E-Sampler. The PM_{10}, $PM_{2.5}$ and PM_1 cyclones look very similar. The cyclone cut point must match the cut point used on any collocated instruments. • Clean the TSP inlet and any cyclones at least monthly. • Check the sample RH data and filter RH sensor operation. High sample RH will cause E-Sampler over-reading. The sensor itself can occasionally fail. • Check the E-Sampler for flow leaks and flow calibration problems. • Check the alarm log for optical system alarms.

12.0 Maintenance

Table 4 shows the Met One recommended period for routine maintenance items. Some of these items will need to be performed more or less often depending on the exact characteristics of your location.

TABLE 4 PERIODIC MAINTENANCE INTERVALS

Maintenance Item	Suggested Period
System leak check	**Monthly**
Flow, temperature, and pressure audits or calibration	**Monthly**
Clean sharp-cut cyclone, particle trap, and TSP inlet	**Monthly**
Check digital alarm log	**Monthly**
Clean 47 mm filter holder screen	**Monthly**
Check filter RH sensors	**6 Months**
Replace PUMP filter and PURGE filter	**12 Months**
Factory service, recalibration, and optical system cleaning	**24 Months**
Replace lithium backup battery, as needed	**5 Years**

Trinity Consultants	**STANDARD OPERATING PROCEDURE**	Title: Sub-atmospheric Pressure Canister Sampling – Time-Integrated Samples Using Simple Timer	
		Number: SOP 193	Page: 1 of 4
		Revision Number: 1	Effective Date: 6/09/2022 2/13/2005 (Rev. 0)
Approval:	Date:		Concurred By:

This procedure describes collection of time-integrated ambient air samples in evacuated stainless steel SUMMA canisters to be submitted for subsequent analysis of target compounds at a central laboratory.

Equipment

1. Chain-of-Custody documentation.
2. Stainless steel canisters prepared for sampling by an approved laboratory.
3. Flow controllers capable of maintaining a constant flow rate over a sampling period of up to 24 hours.
4. Vacuum/pressure gauge.
5. Field sampling data sheets.
6. Wrenches.
7. Sampling system with timer-controlled solenoid to automatically start and stop canister sampling.
8. Timer programming instructions will be included with the sampler.

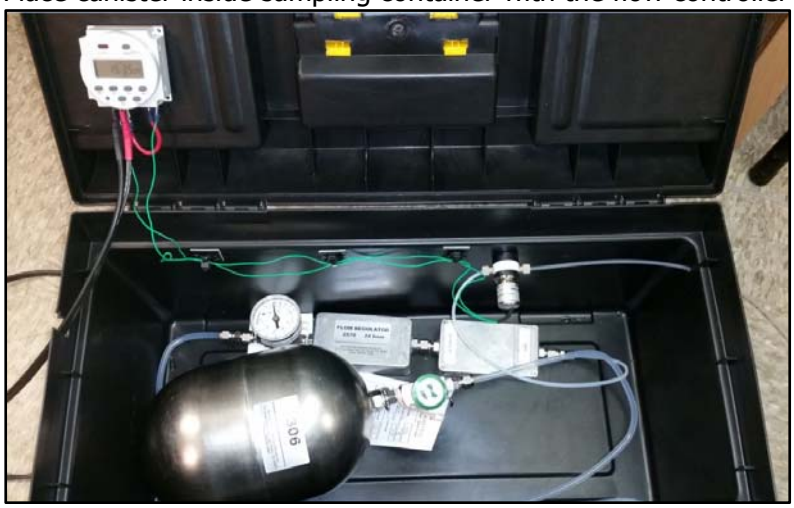
Sampling Procedure

1. Ensure the manual canister valve (shown in green) is closed. See picture below.

2. Remove the brass, screw-on cap from the upper valve of the stainless-steel canister. See picture below.

3. Connect the pressure gauge and flow controller assembly to the canister, place brass cap on inlet, open the manual canister valve (shown in green), record the start pressure, and shut the manual valve (shown in green). See picture below.

4. Place canister inside sampling container with the flow controller assembly connected.



5. Remove brass cap and install inlet tube.

6. Connect the 1/8-inch sampling tube in the sampling system to the flow controller.

7. Open the manual upper valve (shown in green) of the clean evacuated canister by turning counterclockwise.

8. Allow the timer-controlled solenoid to start the sampler at the prescribed time (midnight).

9. Record the information on the canister sampling field data sheet below. Any abnormalities surrounding the sample collection event should be recorded on the form.

10. The timer-controlled solenoid will stop the sample at the prescribed time (midnight).

11. After the sample has stopped, retrieve the canister from the system. First, close the manual canister valve (shown in green) by turning clockwise.

12. Cap the pressure gauge with the brass cap like Step 3, open the valve, record the stop pressure, close the valve (shown in green), and remove the pressure gauge.

13. Replace the brass, screw-on cap onto the manual valve (shown in green) of the stainless-steel canister.

14. Put the stainless-steel canister back into a shipping carton.

15. Put the sample collection form into the shipping carton.

16. Ship to the analytical laboratory with chain-of-custody documentation.

Example canister sampling field data sheet.

Trinity Consultants

CANISTER SAMPLING FIELD DATA SHEET

A. General Information

Site Location : _____
Site Address : _____
Sampler ID : _____
Operator : _____
Canister Leak Check Date : _____
Shipping Date : _____

B. Sampling Information

Canister Serial Number : _____
Sample Number : _____
Canister Pressure (Start) : _____
Canister Pressure (Stop) : _____
Date (Start) : _____
Date (Stop) : _____
Start Time : _____
Stop Time : _____
Elapsed Time : _____
Ambient Temp. (Start) : _____
Ambient Temp. (Stop) : _____
Flow Rate : _____

Wind Direction and Wind Speed Sensor Operation

SOP M11

October 27, 2020

Trinity Consultants

Approval of Standard Operating Procedures

Title: Wind Direction and Wind Speed Sensor Operation

SOP: M11

Approved By:

_____ Date:

Trinity Consultants

Project Director, Casey Lenhart

_____ Date:

Trinity Consultants

Quality Assurance Director, Linda Conger

Table of Contents

1.0 Introduction

Manufacturer's specifications define performance criteria of wind instruments and systems needed to meet project objectives. Performance and procurement specifications provide the basis of inspection and testing when instrumentation is received at a site. Wind speed and wind direction are typically the most important parameters measured at a meteorological monitoring site.

Although wind is a vector quantity and may be measured and processed as such, it is common to measure and/or process the scalar components of the wind vector separately; i.e., wind speed (the magnitude of the wind vector) and wind direction (the orientation of the wind vector). Wind speed determines the amount of initial dilution experienced by a plume and appears in the denominator of the steady-state Gaussian dispersion equation. In addition, wind speed is used in the calculation of plume rise associated with point source releases, to estimate aerodynamic effects in downwash calculations, and, in conjunction with other variables, in the determination of atmospheric stability. Instruments used for in situ monitoring of wind speed are of two types: those which employ mechanical sensors (e.g., cup and propeller anemometers) and those which employ non-mechanical sensors (hot wire anemometers and sonic anemometers). The non-mechanical sensors are not included in the scope of this SOP.

Wind direction is generally defined as the orientation of the wind vector in the horizontal. Wind direction for meteorological purposes is defined as the direction from which the wind is blowing and is measured in degrees clockwise from true north. Wind direction determines the transport direction of a plume or puff in air quality modeling applications. The standard deviation of the wind direction, σA, or the standard deviation of the elevation angle, E, may also be used, in conjunction with wind speed, to derive the atmospheric stability category. Wind direction may be measured directly using a wind vane or may be derived from measurements of wind speed component.

2.0 Initial Equipment Checkout

Check the sensor carefully for any signs of shipping damage. Remove the plastic nut on the propeller shaft. Install the propeller on the shaft with the serial number of the propeller facing forward (into the wind). The instrument is aligned, balanced and fully calibrated before shipment; however, it should be checked both mechanically and electrically before installation. The vane and propeller should easily rotate 360° without friction. Check vane balance by holding the instrument base so the vane surface is horizontal. It should have near neutral torque without any particular tendency to rotate.

The potentiometer requires a stable DC excitation voltage. Do not exceed 15 volts. When the potentiometer wiper is in the 5° deadband region, the output signal is "floating" and may show varying or unpredictable values. To prevent false readings, signal conditioning electronics should clamp the signal to excitation or reference level when this occurs.

Before installation, connect the instrument to an indicator as shown in the wiring diagram and check for proper wind speed and azimuth values. To check wind speed, temporarily remove the propeller and connect the shaft to an Anemometer Drive.

3.0 Installation

Proper placement of the instrument is very important. Eddies from trees, buildings, or other structures can greatly influence wind speed and wind direction observations. To get meaningful data for most applications, locate the instrument well above or upwind from obstructions. As a general rule, the air flow around a structure is disturbed to twice the height of the structure upwind, six times the height downwind, and up to twice the height of the structure above ground. For some applications it may not be practical or necessary to meet these requirements.

Grounding of the wind sensor is vitally important. Without proper grounding static electrical charge can build up during certain atmospheric conditions and discharge through the transducers. This discharge can potentially cause erroneous signals or transducer failure.

Initial installation is most easily done with two people; one to adjust the instrument position and the other to observe the indicating device. The installation example below is for a RM Young Model 05305 wind monitor.

1. Mount sensor.
   a. Place orientation ring on mounting post. Do Not tighten band clamp yet.
   b. Place Wind Monitor on mounting post. Do Not tighten band clamp yet.

2. Connect Sensor Cable.
   a. Slide junction box cover up.
   b. Route cable thru strain relief opening at bottom of junction box. Secure cable by tightening packing nut.
   c. Connect sensor cable to terminals. See wiring diagram.
   d. Slide junction box cover down.

3. Align Vane
a. Connect instrument to an indicator.
b. Choose a known wind direction reference point on the horizon.
c. Sighting down instrument centerline, point nose cone at reference point on horizon.
d. While holding vane in position, slowly turn base until indicator shows proper value.
e. Tighten mounting post band clamp.
f. Engage orientation ring indexing pin in notch at instrument base.
g. Tighten orientation ring band clamp

4.0 Calibration

Calibration procedures are in accordance with the guidelines of the EPA Quality Assurance Handbook for Air Pollution Measurement Systems: Volume IV, Version 2.0 Final (EPA, March 2008).

4.1 Calibration Equipment

The calibration technician conducting the calibration will bring the following equipment to the site:

- RM Young Model 18810 anemometer drive,
- RM Young Model 18310 Torque Disc,
- Professional classic pocket transit or precision compass with tripod,
- R.M. Young Model 18212 Vane Angle Fixture,
- R.M. Young Model 18331 Vane Torque Gauge,
- Current magnetic declination angle for site to be calibrated,
- Calibration field data sheets, and
- (optional) Theodolite and True North solar angle program for computer.

4.2 Personnel Qualifications

Installation, operation, maintenance, repair or calibration of the instrument and all support equipment will be performed by properly trained personnel. Personnel will meet all minimum requirements and qualifications commensurate with their position.

4.3 Calibration Procedures

The calibration procedures for wind speed and wind direction are presented below.

4.3.1 Wind Speed

Starting threshold is calibrated by checking sensor shaft rotational torque with a torque disc.

1. With the anemometer sensor in the horizontal position, remove the anemometer cups or propeller and install the torque disc on the anemometer shaft. Use manufacturer-provided allowable torque values or calculate the torque value that corresponds to the starting threshold of 0.5 m/s using the "k" value provided by the manufacturer and the following equation:

$$T = kU^2$$

Where:

T = torque in gm-cm
U = wind speed in m/s
k = constant (from manufacturer)

2. Install the 0.1 gm screw weight in the appropriate hole of the torque disc that corresponds to the calculated torque value and position the weight so that it is level with the anemometer shaft. Release the weight and note if the torque disk and anemometer shaft rotate freely. To measure the actual starting torque, change the position of the screw weight starting at the location closest to the shaft and move outward until the weight rotates freely from the horizontal. The weight of the screw times the distance from the shaft equals the torque in gm-cm.

3. The accuracy of wind speed measurements is tested at zero and at least two speeds within the operational range of the sensor. R.M. Young Model 18810 selectable speed anemometer drive will be used to generate stable calibration input speeds over the range of the sensor.

4. The calibration person removes the anemometer cups or propeller and joins the wind speed sensor shaft to the calibration motor with a coupling device.

5. Calculate the difference between the system and calibration wind speeds using the following equation:

Diff. = System Wind Speed - Calibration Wind Speed

The differences calculated above are compared with the US EPA PSD recommended criteria of ± 0.2 m/s.

4.3.2 Wind Direction

1. For wind direction instruments that have crossarms, prior to lowering the tower or the crossarm, determine the crossarm alignment by sighting along it using a precision compass corrected for magnetic declination. Current magnetic declination is obtained using the latitude/longitude or UTM coordinates of the site and a magnetic declination calculation computer program. Optionally, if a solar viewing is possible, a theodolite can be set up and oriented using a solar angle computer program. The calibration person views the crossarm through the theodolite to verify alignment with reference to True North.

2. Once the crossarm is lowered, the person conducting the calibration positions the wind vane exactly parallel to the crossarm and records the reading.

3. Determine accuracy and linearity by mounting a direction template or calibration fixture and fixing the vane in at least the four cardinal directions. The vane is rotated sequentially through at least the four directions clockwise and then counterclockwise and the DAS readouts are recorded. (The tip and then the tail of the vane may also be pointed at established distant sighting targets.)

4. The difference between the station and calibration wind directions is calculated using the following equation:

Diff. = System Wind Direction - Calibration Wind Direction

The differences calculated above are compared with the EPA PSD recommended criteria of ±3% for linearity and ±5% for the entire system (orientation plus linearity). If results exceed these criteria, the calibration person should recommend recalibration of the sensor or replacement of the potentiometer.

5. Determine starting threshold of the wind vane by measuring shaft rotational torque of the sensor using a torque gauge or disc. The measured torque should be less than the maximum allowable torque provided by the manufacturer corresponding to a 0.5 m/s wind speed threshold.

If the measured torque exceeds this value, the calibrator should recommend bearing and/or potentiometer replacement. If necessary, calculate the torque value that corresponds to the starting threshold of 0.5 m/s for a 10° deflection using the "k" value provided by the manufacturer and the following equation:

$$T = kU2$$

Where:

T = torque in gm-cm

U = wind speed in m/s

k = constant

The torque gauge test determines if the wind vane starting threshold is less than or equal to the required specifications. The wind vane is considered to be within the recommended criteria if the indicated torque value is less than or equal to the calculated or stated maximum starting torque value. If the wind vane fails the test, the calibrator should recommend that the bearings and/or potentiometer be replaced.

5.0 Maintenance Procedures

Given proper care, the wind sensor should provide years of service. The only components likely to need replacement due to normal wear are the precision ball bearings and the wind direction potentiometer.

Temperature Sensor Operation

SOP M13

October 27, 2020

Trinity Consultants

Approval of Standard Operating Procedures

Title: Temperature Sensor Operation

SOP: M13

Approved By:

_____ Date:

Trinity Consultants

Project Director, Casey Lenhart

_____ Date:

Trinity Consultants

Quality Assurance Director, Linda Conger

Table of Contents

1.0 Introduction

Manufacturer's specifications define performance criteria of temperature instruments and systems needed to meet project objectives. Performance and procurement specifications provide the basis of inspection and testing when instrumentation is received at a site. Temperature is typically an important parameter measured at a meteorological monitoring site.

The most common method of air temperature measurement is using devices whose resistance changed with the temperature – resistance temperature detectors (RTD) or thermistor. Thermistors and platinum wires are included in resistance bridges that allow acceptably linear and accurate voltage measurement directly by the data acquisition system.

2.0 Initial Equipment Checkout

Check the sensor carefully for any signs of shipping damage. If damage occurred during transport, immediately file a claim with the carrier. Contact manufacturer or vendor to facilitate repair or replacement.

3.0 Installation

Proper placement of the instrument is very important. For accurate measurements, the temperature probe should be installed in a protective radiation shield. Use of the probe without a radiation shield may result in large errors. Naturally ventilated or motor aspirated shields are recommended. For best performance, the probe and shield should be placed in a location with good air circulation clear of large masses (buildings, pavement, solar panels), exhaust vents, electrical machinery, motors, water fountains and sprinklers. The terminal marked "EARTH GND" should be connected to properly grounded tower, or grounding conductor, as close to the sensor as possible. Failure to do so may result in damage due to static discharge.

4.0 Personnel Qualifications

Installation, operation, maintenance, repair or calibration of the instrument and all support equipment will be performed by properly trained personnel. Personnel will meet all minimum requirements and qualifications commensurate with their position.

5.0 Calibration

Calibration procedures are in accordance with the guidelines of the EPA Quality Assurance Handbook for Air Pollution Measurement Systems: Volume IV, Version 2.0 Final (EPA, March 2008).

5.1 Calibration Equipment

The calibration technician conducting the calibration will bring the following equipment to the site:

- Mercury-in-glass thermometer or digital thermometer calibrated with a laboratory NIST-traceable thermometer.
- Thermos bottles - one with hot water, one warm water, and one ice bath or aluminum blocks at different temperatures.
- Calibration forms.

5.2 Calibration Procedures

Calibration procedures are in accordance with the guidelines of the EPA Quality Assurance Handbook for Air Pollution Measurement Systems: Volume IV, Version 2.0 Final (EPA, March 2008).

Temperature sensing systems are calibrated by collocated intercomparison with a calibrated reference standard. If immersion in water is possible, the station temperature sensing system thermistor and the calibrated thermometer are immersed in a common water bath and the readings are compared at temperatures of approximately 0°, 20°, and 40°C (or 3 points over the expected measurement range at the site) or by using aluminum blocks inserted into wide-mouth thermos bottles to provide the medium for various reference temperatures. If delta-temperature is measured, the delta-temperature is checked by simultaneous insertion of delta-temperature sensors in the same medium and comparing outputs.

Calculate the difference between the sensor and calibration temperatures using the equation:

$$\text{Diff.} = \text{System Temperature} - \text{Calibration Temperature}$$

The differences calculated above are then compared with the EPA recommended criteria of ±0.5°C and ±0.1°C for delta-temperature when the sensors are checked in the same medium.

6.0 Maintenance Procedures

Given proper care, the temperature probe is designed to offer years of service with minimal maintenance. If necessary, the probe may be periodically checked or recalibrated using normal bath calibration methods.

Precipitation Gauge Operation

SOP M14

October 27, 2020

Trinity Consultants

Approval of Standard Operating Procedures

Title: Precipitation Gauge Operation

SOP: M14

Approved By:

_____ Date:

Trinity Consultants

Project Director, Casey Lenhart

_____ Date:

Trinity Consultants

Quality Assurance Director, Linda Conger

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1.0 Introduction

Manufacturer's specifications define performance criteria of temperature instruments and systems needed to meet project objectives. Performance and procurement specifications provide the basis of inspection and testing when instrumentation is received at a site. Precipitation is typically an important parameter measured at a meteorological monitoring site.

The most common method of precipitation is a sensor that uses a collector funnel with a knife-edge that diverts the water to a tipping bucket mechanism. A magnet is attached to the tipping bucket which actuates a magnetic switch on each tip of the bucket. The water drains out of the bottom of the housing, so the sensor requires no attention or servicing. Connecting the sensor to an event counter on a data logger or display module allows for electronics record keeping of accumulated rainfall.

2.0 Initial Equipment Checkout

Check the sensor carefully for any signs of shipping damage. If damage occurred during transport, immediately file a claim with the carrier. Contact manufacturer or vendor to facilitate repair or replacement.

3.0 Installation

Proper placement of the instrument is very important. A clear and unobstructed mounting location is necessary to obtain accurate rainfall readings.

This transmitter is designed to be mounted two ways, by surface mounting or mast mounting. Surface mounting is recommended whenever possible. The transmitter housing must be mounted in a level position and in a location free from vibration. If mast mounted, make sure that the mast is properly guyed so that vibration in high winds is kept to a minimum.

After transmitter installation, remove the gold funnel and observe the black tipping bucket. It should not be held in a dead center position by the magnetic attraction of the bucket magnet and the magnetic switch. Press either end of the bucket down against the stop to be sure it is not centered. The connecting cable between transmitter and indicator can be shortened or lengthened as required.

The funnel and tipping bucket mechanism should be cleaned periodically. An accumulation of dirt, bugs, etc. on the tipping bucket will adversely affect the calibration.

4.0 Personnel Qualifications

Installation, operation, maintenance, repair or calibration of the instrument and all support equipment will be performed by properly trained personnel. Personnel will meet all minimum requirements and qualifications commensurate with their position.

5.0 Calibration

Calibration procedures are in accordance with the guidelines of the EPA Quality Assurance Handbook for Air Pollution Measurement Systems: Volume IV, Version 2.0 Final (EPA, March 2008).

5.1 Calibration Equipment

The calibration technician conducting the calibration will bring the following equipment to the site:

- Centimeter ruler for measuring gauge orifice diameter.
- Water to simulate rainfall.
- Graduated cylinder and graduated syringe or graduated burette.
- Calibration field data forms.

5.2 Calibration Procedures

The diameter of the gauge opening, or collection funnel is measured with a centimeter ruler and the area of the opening is calculated (Area = πr^2).

Precipitation gauge calibrations are performed using a graduated burette, graduated cylinder, graduated syringe or an intravenous drip bottle and tubing arrangement that allows for accurate delivery of water to the precipitation gauge at an infinitely controllable delivery rate. The delivery rate is adjusted to no more than approximately .03"/minute by monitoring the drip rate. For tipping bucket gauges, the calibration technician should deliver enough water for at least the equivalent of 10 tips per run and should calculate the average percent difference of 3 runs.

The amount of water simulating rainfall required to produce 10 tips is divided by the area of the gauge opening to calculate the amount of rainfall in inches or millimeters. This value is compared with the value reported by the site data acquisition system.

The percent difference between the station reading and the reference value is calculated using the following equation:

% diff. = (station reading - calibration reading / calibration reading) * 100

The mean of the percent differences calculated above is compared with the EPA recommended criteria of ±10% of the known calibration input.

6.0 Maintenance Procedures

Given proper care, the precipitation gauge is designed to offer years of service with minimal maintenance. As necessary, the gauge needs to be cleaned of debris.

Relative Humidity Sensor Operation

SOP M15

October 27, 2020

Trinity Consultants

Approval of Standard Operating Procedures

Title: Relative Humidity Sensor Operation

SOP: M15

Approved By:

_____ Date:

Trinity Consultants

Project Director, Casey Lenhart

_____ Date:

Trinity Consultants

Quality Assurance Director, Linda Conger

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Trinity Consultants

1.0 Introduction

Manufacturer's specifications define performance criteria of relative humidity instruments and systems needed to meet project objectives. Performance and procurement specifications provide the basis of inspection and testing when instrumentation is received at a site. Relative humidity is typically an important parameter measured at a meteorological monitoring site.

The most common method of relative humidity measurement is using devices with a capacitive RH element.

2.0 Initial Equipment Checkout

Check the sensor carefully for any signs of shipping damage. If damage occurred during transport, immediately file a claim with the carrier. Contact manufacturer or vendor to facilitate repair or replacement.

3.0 Installation

Proper placement of the instrument is very important. Sensors should be located over an open, level area at least 9 m (EPA) in diameter. The surface should be covered by short grass or the natural earth surface where grass does not grow. Sensors should be located at a distance of at least four times the height of any nearby obstruction and at least 30 m (EPA) from large, paved areas. Sensors should be protected from thermal radiation and adequately ventilated. Protect the filter at the top of the sensor from exposure to liquid water. The hydrophobic nature of the filter repels light rain but driving rain can force itself into the pore structure of the filter and take time to dry out.

Install the relative humidity sensor in a radiation shield to the tower or tripod. Attach the probe to the cable by aligning the keyed connectors, pushing the connectors together, and finger tightening the knurled ring. Long lead lengths cause errors in the measured relative humidity. The approximate error in relative humidity is 0.31% per 100 feet of cable length.

4.0 Personnel Qualifications

Installation, operation, maintenance, repair or calibration of the instrument and all support equipment will be performed by properly trained personnel. Personnel will meet all minimum requirements and qualifications commensurate with their position.

5.0 Calibration

Calibration procedures are in accordance with the guidelines of the EPA Quality Assurance Handbook for Air Pollution Measurement Systems: Volume IV, Version 2.0 Final (EPA, March 2008).

5.1 Calibration Equipment

The calibration technician conducting the calibration will bring the following equipment to the site:

- Calibrated digital RH probe or motor aspirated psychrometer.
- Booklet of psychometric tables.
- Water.
- Large plastic bucket (approx. 5-gallon size).
- Calibration forms.
- Portable barometer if using psychometric tables.

5.2 Calibration Procedures

Relative humidity sensors are calibrated using one of two methods.

1. Collocating the station RH sensor and the calibrated RH sensor inside a plastic bucket where water can be added in the bottom of the bucket to provide several different calibration points.
2. Collocating the calibrated RH sensor or motor-aspirated psychrometer adjacent to the site sensor to sense the ambient conditions. Multiple readings are taken over several hours (wet bulb, dry bulb) and converted into RH using the manufacturer's tables.

Calculate the difference between the station and calibration relative humidity's using the equation:

$$\% \text{ Diff.} = \text{Station \% RH - Calibration \% RH/ Calibration \% RH}$$

The mean of the percent differences calculated above is then compared with the EPA recommended criteria of ±7 percent relative humidity.

6.0 Maintenance Procedures

Given proper care, the relative humidity probe is designed to offer years of service with minimal maintenance but dust, debris, and salts on the filter cap will degrade sensor performance. Check the metal mesh filter on the end of the sensor for debris. If dirt or salt is engrained into the filter, it should be cleaned with distilled water or replaced. For particularly stubborn contamination, swish the entire probe tip in isopropyl alcohol (rubbing alcohol) and rinse off with distilled water. Make sure the filter is connected firmly with your fingers — do not over tighten.

Check the radiation shield monthly to make sure it is free from dust and debris. To clean the shield, remove the sensor from the shield. Dismount the shield. Brush all loose dirt off. If more effort is needed, use warm, soapy water and a soft cloth or brush to thoroughly clean the shield. Allow the shield to dry before remounting.

Replace filters that cannot be successfully cleaned. To replace the filter, unscrew the filter from the probe and pull it straight away, being careful not to bend or damage the sensors. Before putting on the replacement filter, check the alignment of the sensors with the probe, and if necessary, carefully correct the alignment before installing the filter.

A coating of salt (mostly NaCl) may build up on the radiation shield, sensor, filter and even the sensor element. A buildup of salt on the filter or sensors will delay or destroy the response to atmospheric humidity.

Long-term exposure of the relative humidity sensor to certain chemicals and gases may affect the characteristics of the sensor and shorten its life. The resistance of the sensor depends strongly on the temperature and humidity conditions and the length of the pollutant influence.

APPENDIX D. LABORATORY QUALITY ASSURANCE PLANS AND ANALYTICAL PROCEDURES

Quality Assurance Project Plan for Laboratory and Data Management Support of the Determination of Fine Particulate Matter as $PM_{2.5}$, PM_{10} and Coarse Particulate Matter as $PM_{10-2.5}$ in the Atmosphere

IML Air Science, a division of Pace Analytical Services, LLC
Sheridan, Wyoming

Revision 15.0
May 2021

IML Air Science
A Division of Pace Analytical Services, LLC
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A Project Management

A1 Title and Approval Sheet

Title: *IML Air Science Quality Assurance Project Plan for $PM_{2.5}$, PM_{10}, and $PM_{10-2.5}$*

Organization: *IML Air Science Gravimetric Laboratory*

Revision Number: *15* **Revision Date:** *05/2021*

Approval:

IML Air Science

Tim Mendenhall, Air Science Manager — Date 5/17/2021

Mary Hininger, Gravimetric Lab Supervisor — Date 05/17/21

Margaret Elliott, Quality Officer — Date 5/17/2021

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A3 Distribution List

U.S. Environmental Protection Agency (EPA), Region 8

- Greg Noah, (EPA OAQPS Field QA Coordinator), U.S. EPA, U.S. EPA-OAQPS RTP, 109 T.W. Alexander Drive, NC 27709, (919-541-2771), Noah.Greg@epa.gov

IML Air Science (a division of Pace Analytical Services, LLC)

- Tim Mendenhall, Air Science Manager, IML Air Science, 555 Absaraka Street, Sheridan, WY 82801, (307) 461-4949, Tim.Mendenhall@pacelabs.com

A4 Project/Task Organization

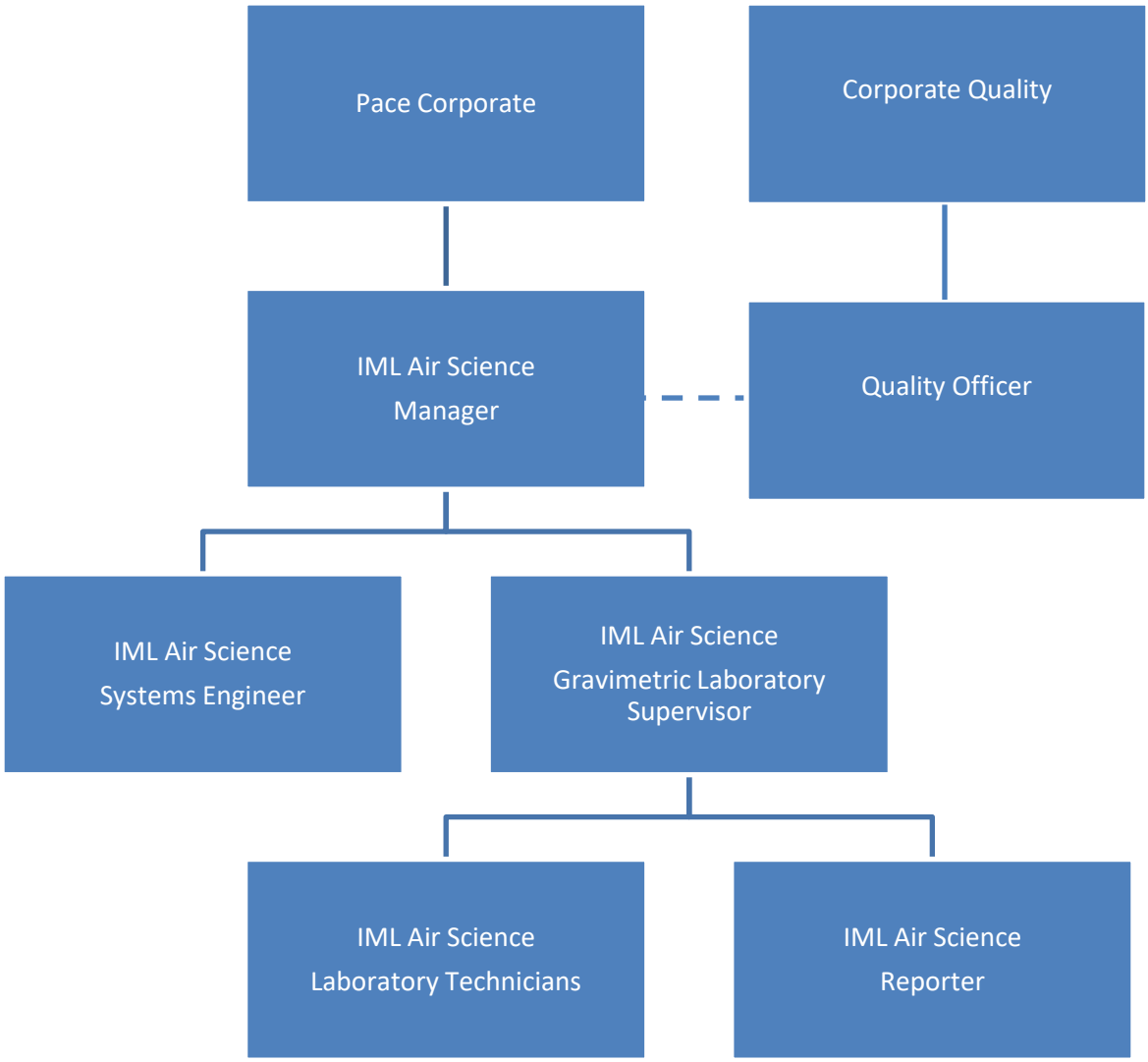
A4.1 Roles and Responsibilities

A4.1.1 IML Air Science

The roles and responsibilities of IML's Gravimetric Laboratory and Data Management System in the determination of fine particulate matter as $PM_{2.5}$, PM_{10}, and coarse particulate matter as $PM_{10-2.5}$ in the atmosphere are described below:

- IML Air Science Manager: Provides technical and managerial assistance and decision making for the lab. Maintains the official, approved Quality Assurance Project Plan (QAPP) and assists in financial matters and QAPP review. Provides managerial support to QA staff.
- Systems Engineer: Maintains the Data Management System (DMS). Responsible for IT.
- Quality Officer: Responsible for the coordination and implementation of IML Air Science Quality Management Plan (QMP). The QO trains and authorizes work on quality procedures, controls training records, organizes audits, and controls corrective actions. The QO is authorized and responsible for stopping work where quality may be compromised. The QO reports to upper levels of management where policy decisions are made. The QO is independent from data generation functions.
- Gravimetric Laboratory Supervisor: Responsible for scheduling, laboratory sample throughput, for data reporting, and for report review. Assists with laboratory operation and sample analysis. Responsible for technical, methodology, safety, quality and software training. Oversees quality assurance and quality control within the Gravimetric Laboratory. Works with QA staff to ensure understanding of quality practices through the day-to-day operation and to coordinate training with QO.
- Reporter: Generates reports, assists with laboratory operation, and performs sample analysis.
- Laboratory Technicians: Responsible for laboratory operation and sample analysis.

Figure A4-1 Project Organization and Responsibilities



A5 Problem Definition/Background

The goal of this ambient air monitoring program is to determine the concentration, in units of micrograms per cubic meter ($\mu g/m^3$), of particulate matter for $PM_{2.5}$, $PM_{10-2.5}$, and PM_{10}. The method is a manual one in which a sample is collected on a filter by passing a low volumetric flow rate of ambient air through a filter for a 24-hour period. The filter is 46.2mm in diameter and constructed of polytetrafluoroethylene (PTFE) membrane. The filter is analyzed gravimetrically, using a microbalance with sensitivity of at least 1µg, before and after sample collection to determine the net mass gain.

The aerodynamic diameter of 2.5 micrometers (µm) and less ($PM_{2.5}$) defines fine particulate. This determination follows 40 CFR 50, Appendix L, "Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere."

The mass concentration of particulate matter (PM_{10}) in low volume, ambient air over a 24-hour period defines PM_{10}. This determination follows 40 CFR 50, Appendix L, excepting that PM_{10} concentrations are corrected to standard conditions, as per memorandum "Clarification on Use of $PM_{2.5}$ Field and Laboratory Requirements for Low Volume PM_{10} Monitoring to Support PM_{10} NAAQS," dated 03/03/16 from Mike Papp, QA Team Lead, Ambient Air Monitoring Group (C304-06).

The mass concentration of particulate matter ($PM_{10-2.5}$) in ambient air over a 24-hour period defines coarse. This determination follows 40 CFR 50, Appendix O, "Reference Method for the Determination of Coarse Particulate Matter as $PM_{10-2.5}$ in the Atmosphere." The Reference Method requires $PM_{10-2.5}$ concentrations to be measured either directly from the instrument or as the arithmetic difference between separate and concurrent, collocated measurements of PM_{10} and $PM_{2.5}$.

This document covers activities that support these methods. These activities are contracted for SLAMS, SPM, and NCore networks; various state, local, and federal networks; as well as special projects (FEM testing) and privately operated networks.

A6 Project/Task Description

The IML Air Science gravimetric laboratory has been designed for filter weighing as described in 40 CFR 50 Appendix L and Appendix O for the determination of mass concentration of fine particulate matter having an aerodynamic diameter of less than or equal to a nominal 2.5 micrometers ($PM_{2.5}$), coarse particulate matter ($PM_{10-2.5}$), and particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers (PM_{10}) for low volume samplers. Each gravimetric laboratory consists of an equilibration laboratory for weighing and an ante room for pre or post weighing activities.

A6.1 Laboratory Measurements

Laboratory environmental conditions (temperature in ˚C and relative humidity in %) are measured and controlled by a data logger. Control limits for temperature and relative humidity are located in Table B5-1.

Records are kept of the 5-minute and hourly average, maximum, minimum, and standard deviation values of temperature and relative humidity. The 24-hour average of the one-second values for temperature and relative humidity data are displayed on the interface software system operating on a workstation computer in the laboratory.

A6.2 Laboratory Schedule

The operation schedule of the micro-gravimetric laboratory is typically as put forth in the table below. Holidays and other unforeseen issues may require temporary deviations from the schedule in Table A6-1. The table is designed to provide uniform equilibration times of 24 to 48 hours for all exposed sample filters. Weekly laboratory cleaning and maintenance is also integrated into the schedule.

Table A6-1 Laboratory Schedule

Monday	Tuesday	Wednesday	Thursday	Friday
Weigh clean filters for the week	Set out exposed filters that have been received to equilibrate	Set out exposed filters that have been received to equilibrate	Set out exposed filters that have been received to equilibrate	Weigh exposed equilibrated filters
Send clean filters to locations	Weigh exposed equilibrated filters	Weigh exposed equilibrated filters	Weigh exposed equilibrated filters	Clean lab

Monday	Tuesday	Wednesday	Thursday	Friday
Set out exposed filters that have been received to equilibrate			*Filters received after 12:00 noon are placed in fridge until Monday	Set out clean filters for the following week to equilibrate
				All filters received are placed in fridge until Monday

A6.3 Data Management Activities

Management of data is handled by IML Air Science's Data Management System, hereafter DMS.

Laboratory data is recorded with an information system designed specifically for use in a gravimetric laboratory. Information recorded in this system includes analytical results, QA/QC results, and laboratory temperature and relative humidity.

Sample collection data are imported with custom database programs. Sample collection data imported into the DMS or sample data recorded on the anti-static bag label and manually entered in the DMS is used to calculate standard volume. Standard volume is calculated using Equation A6-1. Laboratory and sample collection data are related by sample identification numbers.

Equation A6-1 Standard Volume

$$V_{std} = V_a \times \frac{P_a \times T_{std}}{P_{std} \times (T_a + 273)}$$

Where:

V_a – local volume (m^3)

V_{std} – standard volume (m^3)

P_a – local pressure (mmHg)

P_{std} – standard pressure (760mmHg)

T_a – local temperature (°C)

T_{std} – standard temperature (298K)

Note: Local pressure is truncated to two decimal places on BGI PQ200 (Mesa Labs) sampler downloaded data to match display values that are recorded on bag labels.

A7 Quality Objectives and Criteria

The quality of the data must be evaluated and controlled to ensure that it is maintained within the established acceptance criteria. Acceptance criteria are available in Table B5-1.

As described in 40 CFR 50 Appendix L, the lower concentration limit of the mass concentration measurement range is estimated to be approximately 2 $\mu g/m^3$, based on noted mass changes in field blanks in conjunction with the 24 m^3 nominal total air sample volume specified for the 24-hour sample. Additionally, the upper concentration limit cannot be specified precisely because it is a complex function of the ambient particle size distribution and type, humidity, the individual filter used, the capacity of the sampler flow rate control system, and perhaps other factors. Nevertheless, all samplers are estimated to be capable of measuring 24-hour $PM_{2.5}$ mass concentrations of at least 200 $\mu g/m^3$ while maintaining the operating flow rate within the specified limits. However, lower and upper concentration limits are not determined by the Gravimetric Laboratory but determined by the responsible primary quality assurance organization.

Measurement quality objectives (MQO) are designed to evaluate and control various phases of the measurement process to ensure that total measurement uncertainty is within the range required for the program. MQOs can be defined in terms of the following data quality indicators.

A7.1 Accuracy

Assessing accuracy is a process of determining how a measurement compares to the actual value. In the laboratory, measurements that are assessed for accuracy include mass, temperature, and relative humidity.

A7.2 Laboratory

Determination of the accuracy of measurements made in the laboratory is accomplished by comparison with measurements made with certified, NIST traceable reference standards.

At least quarterly, the microbalance working standards are certified with primary mass reference standards. Additionally, the microbalance is certified independently at least annually. The acceptance criteria for these certifications are presented in Section B5. The standards used in certification of the microbalance are independently certified to NIST.

The temperature and relative humidity probes in the laboratory are verified against NIST traceable standards at least monthly. Acceptance criteria are presented in Section B5.

These standards are recertified with NIST traceable standards annually or replaced with new traceable standards.

A7.3 Sample Collection and Handling

The reference method specifies procedures for assessing accuracy of the flow rate and other measurements made by samplers through verifications and audits. The performance and reporting of these procedures are the responsibility of network owners and operators.

A7.4 Precision

Precision is a measurement of mutual agreement among individual measurements of the same property usually under prescribed similar conditions, expressed generally in terms of the standard deviation. The reference method specifies procedures for evaluation of precision.

A7.4.1 Laboratory

Precision is assessed in the laboratory with two quality control procedures. The mass of a working standard is determined at least every tenth analysis. The difference between the determined mass and the certified mass of the standard is a measure of precision. In addition, replicate analyses are performed on some of the samples during each analytical session as another demonstration of measurement precision. Acceptance criteria are presented in Section B5.

A7.4.2 Complete Method

The precision of sample collection, handling, analysis, and data management is evaluated by making collocated measurements and comparing the results. $PM_{2.5}$ and PM_{10} networks include sites where two or more samplers are installed. Periodically, samples will be collected concurrently by two samplers. The handling and analyses of these samples are identical. Precision is assessed by comparison of the results of collocated measurements.

A7.5 Completeness

The completeness of a data set is the number of valid samples compared to the theoretical number of possible samples for a given time period. Completeness is reported as data recovery rate, the ratio of valid samples to possible samples and expressed as a percentage.

A8 Special Training/Certification

Adequate experience and training are integral to any monitoring program that strives for reliable and comparable data. Personnel assigned to the gravimetric laboratory ambient air monitoring program will meet the educational, work experience, responsibility, and training requirements for their positions. Personnel keep current on training by periodic review of the Gravimetric Laboratory QAPP, IML Air Science QMP, SOPs, and regulatory and guidance documents. SOPs are updated or written to reflect changes in, or new regulations, equipment, or procedures as determined by the Management Review process.

Periodic review of training qualifications by supervisors or managers determines if staff have received the latest training for their position. Records on personnel qualifications and training will be maintained in personnel files and will be accessible for review during audit activities.

Training needs will be identified for new and current employees by the supervisor, manager or Quality Officer. As training needs are identified, a trainer will be assigned to train the trainee. Training records will be completed by the trainer in a timely fashion. Training will take place using current, approved versions of SOPs or other appropriate material. SOPs are found in Appendix C: and are used for methodology and technical training. Personnel responsible for various aspects of training are listed in Section A4.1, which includes individuals' roles and responsibilities. Generalized training information can be found in the IML Air Science QMP.

A9 Documents and Records

A9.1 Activities Documentation

The $PM_{2.5}$ and $PM_{10-2.5}$ reference methods require that a large number of activities and measurements be recorded. Computers, databases, and instruments with data logging capability are utilized to record the majority of the information. Electronic data collection, use, and archiving are discussed in detail in Section B10. Other activities, required by the method, may be recorded on a paper record. These are discussed in the following sections.

Additionally, the Control of Records SOP and Document Control SOP are located in the IML Air Science QMP, which describes how documents, such as this QAPP, are available in the DMS.

A9.1.1 QA/QC

A number of bound, sequentially numbered notebooks are maintained to record QA/QC activities that are not in the DMS. The books are divided into sections, each documenting specific activities.

A9.1.2 Laboratory Notebooks

Table A9-1 Laboratory Notebook Documentation

Event	Information Recorded
Lab humidity exceeds limits %	Date, time, possible cause, corrective action
Lab temperature exceeds limits	Date, time, possible cause, corrective action
Working standards re-verified	Date, time, tech., reason for re-verification
Temperature and RH measurement system certified	Date, time, measured values, standards measurements, any changes made (calibration)
Temperature and RH system program updated	Date, time, nature of changes
Microbalance serviced	Date, time, tech., nature of service
Microbalance certified	Date, time, tech., results
Microbalance calibrated	Date, time, tech., reason for calibration
Weekly and Quarterly cleaning	Date, time, tech.
Cassette cleaning	Date, time, tech.

The Filter Lot Login Notebook is used to record receipt of clean filters, both from filter manufacturers or suppliers and clients that receive filters through the national contract. Information recorded includes the network for which the filters are to be used, date of receipt, lot number, filter numbers, and number of boxes of filters received.

The Filter Stability Evaluation (Lot Blank Notebook) is used to record all lot blank analyses. Information recorded includes lot number, date of receipt, and the resulting equilibration time required for the lot as determined by the lot blank analyses. Tables provide spaces for recording all lot blank analytical results and corresponding quality control activities. Summary tables are included for 24-hour results and final results if equilibration time is found to be greater than 24 hours.

The Working Standard Mass Verification Notebook is used to record all information associated with the verification of working standards. The form used for this procedure is tabular and provides columns for working standard identification, date of activity, analyst identification, certified mass of primary standard, measured mass of primary standard, and the difference of the two standard measurements. It is also used to record the working standard measured mass and resulting verified mass.

The Microbalance External Calibration Notebook is used to record the calibration of the microbalance. The form located in Appendix D: Forms is used to record the calibration of the microbalance and the results of mass determination of the two primary standards used in the method.

Repeatability is performed annually for each microbalance and recorded in the bound notebook for Annual Repeatability Test and in the Lab Balance Notebook. The standard deviation of difference is listed in the SOP. The SOP for Performing the Annual Repeatability Test is located in Appendix C:.

A9.1.3 Exposed Filter Archive

Exposed filters are archived according to network name and post-exposure analysis date or by month sampled. Specific exposed filters can be found in the archive by using the DMS and labeled archive containers. When exposed filters are removed from the archive for further analysis, a pack slip or chain of custody is prepared listing the exposed filters and where the exposed filters will be sent. Each time exposed filters are moved to a new location within the lab or sent somewhere else, tracking is updated in the DMS.

A9.1.4 Chain of Custody

Monitoring networks supported by IML Air Science utilize chain of custody forms to document shipment of exposed filters to the laboratory. The form may be supplied by the network or by the laboratory. An example of a chain of custody form supplied by the laboratory is located in Appendix D:.

A9.2 Reports

The range of services provided in support of ambient particulate networks can be placed in two basic categories. Some networks contract only the analytical portion of the method. These networks therefore receive gravimetric reports, as described in Section A9.2.1. Other networks contract the analytical and data management tasks. For these networks, all data comes together at the laboratory, including sample collection data, shipping and handling information, and laboratory data. Once these data are collected, all reporting requirements can be met using the laboratory's data management tools. Reports available to these networks are summarized in Section A9.2.2.

Report packages are custom designed to meet the specific needs of each network and are therefore unique. However, many individual reports in a package are similar. These reports are described in the following tables.

A9.2.1 Gravimetric Reports

Networks that contract analytical support only receive some or all of the reports summarized in Table A9-2.

Data validation is limited by the information provided by the client. According to 40 CFR 58.15, final responsibilities for data review and validation lie with each agency submitting data to AQS.

Table A9-2 Gravimetric Reports

Report	Parameters	Frequency
Analytical results	Filter ID, tare/gross date, maximum shipping temperature, tare/gross/net mass	Biweekly or Monthly
Field Blank report	Filter ID, sample date, tare/gross/net mass	Biweekly or Monthly
Lab Blank report	Lab blank filter ID, date, tare/gross/net mass	Biweekly or Monthly
Equilibration Times	Filter ID, tare equilibration, gross equilibration	Biweekly or Monthly
Working Standard report	Working standard response control chart and/or summary statistics	Monthly or quarterly
Replicate report	Filter ID, analysis type, date, original/ replicate/net	Biweekly or Monthly
Lab Conditions report	Temperature and RH control chart and/or summary statistics	Biweekly or Monthly

A9.2.2 Network Reports

Networks that contract data management in addition to gravimetric analyses have a variety of reports available to describe results and QA/QC activities. These are summarized in Table A9-3.

Data validation is limited by the information provided by the client. According to 40 CFR 58.15, final responsibilities for data review and validation lie with each agency submitting data to AQS.

Table A9-3 Network Reports

Report	Parameters	Frequency
Sampler summary	Sample date, filter ID, concentration, period, volume, average flow rate, tare/gross/net mass, invalid samples, data flags (qualifiers) (for PM_{10}, concentration and volume are reported in LTP and STP)	Monthly or quarterly
Precision report	Sample date, primary and secondary concentration, % dif., chart, precision statistics	Monthly or quarterly
Field Blank report	Site ID, sampler ID, sample date, filter ID, tare/gross/net mass	Monthly or quarterly

Report	Parameters	Frequency
Lab Blank report	Lab blank filter ID, tare/gross/net mass, gross date	Monthly or quarterly
Working Standard report	Working standard response control chart and/or summary statistics	Quarterly
Lab Conditions report	Temperature and RH control chart and/or summary statistics	Quarterly
Network summary	Sampler average and 98th percentile concentrations, data recovery rate	Quarterly and annual
AQS report	Network ID, site ID, concentration, data qualifiers	Monthly or quarterly

A9.2.3 Report Revisions

The first page in the report, may include a Title Page, which uniquely identifies the custom designed report package. If included, the Title Page would contain information that identifies the specific report. That information includes network identification, date created, and whether the report is the original report or a replacement. If the report is a replacement, it is labeled as such and the report that has been replaced is identified. If the report does not contain a Title Page, the revision would be part of the file name, generally at the end.

A9.3 Documentation Archive

All documentation used and generated in the performance of low volume $PM_{2.5}$, $PM_{10-2.5}$, and PM_{10} exposed filters follows Appendix L with the exception of PM_{10} concentration, which is reported in standard conditions. Records are archived in a secure location for a minimum of five years. Chain of custody documents and reports are archived by network.

B Data Generation and Acquisition

B1 Sampling Process Design

Sampling process design is network specific and is addressed in individual network QAPP's.

B2 Sampling Methods

Sampling methods are network specific and are addressed in individual network QAPP's.

B3 Sample Handling and Custody

B3.1 Assignment and Preparation of Pre-Exposure, Tared Filters

When a batch of pre-exposure filters is due to be sent to a network for exposure, a batch is created in the DMS, using the date the shipment will be tare weighed and shipped (several days in advance to allow time to prepare). Typically, a tare batch (shipment) is created for every Monday for each lab by alternating client networks every other week. The Tare Batch Shipment is printed from the database. The batch is cross checked for each network with a client shipping schedule to verify the quantity of filters to be sent to each network. Refer to Appendix D: for Network Tare Shipping Schedule form. After checking the schedules for agreement, pre-exposure filters are assigned in the DMS. The specified number of pre-exposure filters to be sent to each network is assigned by selecting Assign Filters in the DMS. Lab blanks and extra pre-exposure filters are assigned at the same time for each network.

Labels, with the unique bar-coded numbers corresponding to the pre-assigned, stamped number of a PTFE filter, are printed for both the pre-exposure filter and lab blank storage petri dishes. Lab blanks have an additional label printed with a unique lab blank number. The protective, anti-static shipping bag labels are printed after inspection is complete and cassette numbers have been assigned and inputted into the DMS. The anti-static bag label includes a 30-day expiration date after which the pre-exposure filter should not be used, but rather returned.

The pre-exposure filters are prepared for analysis following the procedures in Section B4. Once properly equilibrated, the tare analyses are performed. The pre-exposure filters are then loaded into uniquely labeled filter cassettes. The cassette identification number is pre-printed on the bag label. The pre-exposure filters, in cassettes, are placed into labeled, protective, anti-static bags, which are then sealed. These procedures all take place in the laboratory. Each pre-exposure filter's corresponding labeled petri storage dish is set aside in protective storage in the anteroom.

B3.2 Packaging and Shipment of Pre-Exposure Tared Filters

Pre-exposure filters, loaded in cassettes and placed in protective bags, are packaged and shipped. Pre-exposure filters are typically shipped on a biweekly schedule (every other Monday) to network operators with cooling agent and a min/max thermometer (if requested) to measure temperature on the return trip. The cooling agent need not be frozen for the shipment of pre-exposure filters, unless accompanied by a post exposed filter

for archiving by the network operator or unless requested by the client. Pre-exposed filters are then shipped to the site using a commercial parcel service. A Tare COC accompanies each shipment, identifying the quantity of pre-exposure filters, identification numbers of the pre-exposure filters, and the date shipped. The Tare COC is signed and dated by the lab technician prior to shipment. The SOP for Shipping Filters to Locations is located in Appendix C:.

B3.3 Receipt and Custody of Pre-Exposure Filters

Network operators receive the pre-exposure filters. It's recommended that the network operator record the receipt, confirm that there has been no damage in shipping, and verifies the quantity of pre-exposure filters and the pre-exposure filter numbers. The network operator places the pre-exposure filters into storage. Sequential use of the pre-exposure filters is recommended to avoid exceeding the 30-day tare expiration date.

B3.4 Sample Collection

Samples are collected on pre-exposure filters prepared by IML Air Science, according to procedures in the client network's QAPP.

B3.5 Exposed Filter Collection

Network operators collect the exposed filters from the sampler, in accordance with their network's QAPP. Exposed filters and data custody methods are defined in the client's network QAPP.

B3.6 Shipment of Exposed Filters

Exposed filters are typically sent to the laboratory on a biweekly schedule. It is recommended that every other Monday, exposed filters be returned to IML Air Science. These shipments typically alternate Mondays with the shipment of pre-exposure filters from IML Air Science to the network operator. Prior to the return shipment of exposed filters, it is recommended that the exposed filter information be recorded on the anti-static bag label along with removal date and time in the spaces provided.

Bag label information is entered into the DMS and compared to the pre-exposure filter data. Exposed filters not removed from the sampler within 177 hours after the collection period has ended will be flagged. Filters are placed inside a large, waterproof plastic bag and packed inside a cooler with frozen icepacks as a cooling agent. If a min/max thermometer

is used, the network operator ensures the thermometer is cold and has been reset, prior to placing in cooler. Exposed filters are typically returned using overnight or 2nd day parcel service to IML Air Science.

B3.7 Temperature Monitoring Upon Receipt

Upon opening the cooler at the laboratory, the maximum temperature during transit is determined by the laboratory technician, with an infrared thermometer gun. If a min/max thermometer accompanies exposed filters, the min, the max, and arrival temperatures are observed and recorded on the COC. For more detail, refer to the SOP for Receipt and Login of Exposed Particulate Filter Samples located in Appendix C:.

An infrared thermometer gun may be substituted for the min/max thermometer with client's approval. If it is apparent that the min/max thermometer was not reset prior to return shipment (e.g. icepacks are frozen solid), the arrival temperature is determined with the infrared thermometer gun and recorded as the transport temperature and the client is notified.

B3.8 Receipt of Exposed Filters

The laboratory technician removes the exposed filters from the cooler and completes the sample receipt form in the DMS and then scans the anti-static bag bar code to record receipt of exposed filters. If a chain of custody document accompanies the shipment, it is signed, dated.

Then exposed filters are prepared for post-exposure analysis or placed in cold storage; the laboratory operation schedule in Section A6, Table A6-1, determines if the exposed filters are equilibrated immediately or held in cold storage. Exposed filters received on Friday, or any day before a holiday, or during quarterly cleaning, are placed in cold storage at ≤4˚C. For more detail, refer to the SOP for Receipt and Login of Exposed Particulate Filter Samples located in Appendix C:.

B3.9 Archiving of Exposed Filters

Following post-exposure analysis, exposed filters are placed in an archiving container in cold storage (≤4˚C). Exposed filters are archived for a minimum of one year, at which time clients are notified and consulted for disposition of their exposed filters. Exposed filters may be retained longer by clients. For more detail, refer to SOP for Archiving Filter Samples located in Appendix C:.

B3.10 Cold Storage of Exposed Filters

Cold storage is used to store exposed filters due to the possibility of mass loss from volatilization. A temperature of ≤4˚C is maintained to lessen the effects of volatilization.

B3.11 Filter Contamination Prevention in the Laboratory

Several measures are taken to prevent contamination of filters while they are in the laboratory, including:

- High-Efficiency Particulate Air (HEPA) filter operates continuously
- Laboratory is pressurized to ensure no inflow of uncontrolled air when door is opened
- Daily, weekly, and quarterly cleaning of the laboratory and tool cleaning procedures
- Sticky floor mat at the entrance of a laboratory
- Laboratory coat
- Laboratory traffic minimized
- Filters handled with stainless steel forceps only
- Filter exposure to uncontrolled environment minimized

B4 Analytical Methods

The concentration of fine ($PM_{2.5}$) and coarse ($PM_{10-2.5}$) and particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers (PM_{10}) is determined by analyzing a sample filter gravimetrically before and after sample collection to determine net mass gain. The result of net mass gain divided by the actual volume of air sampled is the concentration for $PM_{2.5}$ and $PM_{10-2.5}$. For the concentration of PM_{10}, the net mass gain is divided by the volume of air sampled, corrected to standard conditions. Each of the methods described in this QAPP, $PM_{2.5,}$ $PM_{10-2.5}$, and PM_{10}, are low volume methods.

Appendix L to part 50 of 40 CFR provides for the measurement of the mass concentration of fine particulate matter having an aerodynamic diameter less than or equal to a nominal 2.5 micrometers ($PM_{2.5}$) in ambient air over a 24-hour period for purposes of determining whether the primary and secondary national ambient air quality standards for fine particulate matter specified in §50.7 and §50.13 of this part are met. The measurement process is considered to be nondestructive, and the sample obtained can be subjected to subsequent physical or chemical analyses.

The air sampler and other aspects of this reference method are specified either explicitly in this appendix or generally with reference to other applicable regulations or quality assurance guidance.

Each filter is weighed (after moisture and temperature conditioning-equilibration) before and after sample collection to determine the net gain due to collected $PM_{2.5}$. The total volume of air sampled is determined by the sampler from the measured flow rate at actual ambient temperature and pressure and the sampling time. The mass concentration of $PM_{2.5}$ in the ambient air is computed as the total mass of collected particles in the $PM_{2.5}$ size range divided by the actual volume of air sampled and is expressed in micrograms per cubic meter of air ($\mu g/m^3$).

Appendix O to Part 50 of 40 CFR provides for the measurement of the mass concentration of coarse particulate matter ($PM_{10-2.5}$) in ambient air over a 24-hour period. In conjunction with additional analysis, this method may be used to develop speciated data.

Each PM_{10c} and $PM_{2.5}$ sample collection filter is weighed (after moisture and temperature conditioning-equilibration) before and after sample collection to determine the net weight (mass) gain due to collected PM_{10c} or $PM_{2.5}$. The total volume of air sampled by each sampler is determined by the sampler from the measured flow rate at local ambient temperature and pressure and the sampling time. The mass concentrations of both PM_{10c}

and $PM_{2.5}$ in the ambient air are computed as the total mass of collected particles in the PM_{10} or $PM_{2.5}$ size range, as appropriate, divided by the total volume of air sampled by the respective samplers, and expressed in micrograms per cubic meter ($\mu g/m^3$) at local temperature and pressure conditions. The mass concentration of $PM_{10-2.5}$ is determined as the PM_{10c} concentration value less the corresponding, concurrently measured $PM_{2.5}$ concentration value.

Most requirements for $PM_{10-2.5}$ reference methods are similar or identical to the requirements for $PM_{2.5}$ reference methods as set forth in appendix L to this part. To ensure uniformity, applicable appendix L requirements are incorporated herein by reference in the sections where indicated rather than repeated in this appendix.

Appendix O describes the field sampling and laboratory requirements for both the PM_{10} and $PM_{2.5}$ component of the measurement for PM_{coarse} and reported in local conditions as described in 40 CFR Part 50 Appendix L ($PM_{2.5}$). In section A5, monitoring organizations can use low-volume PM instruments for PM_{10} monitoring. However, PM_{10} data collection for NAAQS purposes must be reported in standard temperature and pressure (STP), Appendix K (PM_{10}).

All field and laboratory requirements in the PM_{10} low volume validation template refer to the $PM_{2.5}$ method either in the regulation (40 CFR Part 50 Appendix L) or the $PM_{2.5}$ Guidance Document (Method 2.12).

Procedures and facilities used in the micro-gravimetric analyses meet the requirements of 40 CFR 50, Appendix L, "Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere" and 40 CFR, Appendix O, "Reference Method for the Determination of Coarse Particulate Matter as $PM_{10-2.5}$ in the Atmosphere." SOPs for the Gravimetric Lab are listed in Table B4-1. Exposed filter archiving is described in section B3.9.

Table B4-1 Gravimetric Laboratory SOPs

SOP Number	Title	Revision Date	Regulatory Citation
GR-501-0.0	SOP for Calibration of Microbalance	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards

SOP Number	Title	Revision Date	Regulatory Citation
GR-302-0.0	SOP for Performing Verification of the Archive Cooler and Sample Receiving Refrigerator Temperature	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-303-0.0	SOP for Performing Annual Repeatability Test	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-108-0.0	SOP for Receipt of New Micro-Gravimetric Filters	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-200-0.0	SOP for Performing a Filter Stability Evaluation (Lot Blank)	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-109-0.0	SOP for Preparing Filters for Pre-Exposure Analysis	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-600-0.0	SOP for Cleaning and Maintenance of Gravimetric Laboratory	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-110-0.0	SOP for Performing the Pre-Exposure Analysis	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards

SOP Number	Title	Revision Date	Regulatory Citation
GR-111-0.0	SOP for Shipping Filters to Locations	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-112-0.0	SOP for Receipt and Login of Exposed Particulate Filter Samples	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-113-0.0	SOP for Performing the Post-Exposure Analysis	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-114-0.0	SOP for Archiving Filter Samples	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-304-0.0	SOP for the Verification of Working Mass Reference Standards	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-301-0.0	SOP for Performing Monthly Verifications of the Laboratory Temperature and Relative Humidity	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-201-0.0	SOP for Performing Stability Test on New Lots of Anti-Static Bags	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards

SOP Number	Title	Revision Date	Regulatory Citation
GR-107-0.0	SOP for Transfer of Filters for Analysis	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-900-0.0	SOP for Validation of Software Development or Modification	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards
GR-901-0.0	SOP for Report Review	May 2021	Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards

B4.1 Clean Filters

Shipments of clean 46.2mm Teflon (PTFE) filters are logged in upon arrival at the laboratory. The network operator is notified of receipt of the shipment. For more detail, refer to the SOP for Receipt of New Micro-Gravimetric Filters located in Appendix C:. If the clean filters are from a new lot, a filter stability evaluation will be performed to determine the equilibration time required for pre-exposure analysis. For more detail, refer to the SOP for Performing A Filter Stability Evaluation located in Appendix C:. The clean filters are then placed in storage within the laboratory.

B4.2 Preparation of Pre-Exposure Filters

The procedure for preparation of filters for pre-exposure analysis is located in Appendix C:. This procedure includes inspection of clean filters and equilibration of clean filters. Equilibration of clean filters typically begins on Friday afternoon, after lab cleaning has been completed. Equilibration of clean filters continues until Monday morning. The Receipt and Preparation flow diagram is located in Figure B4-1.

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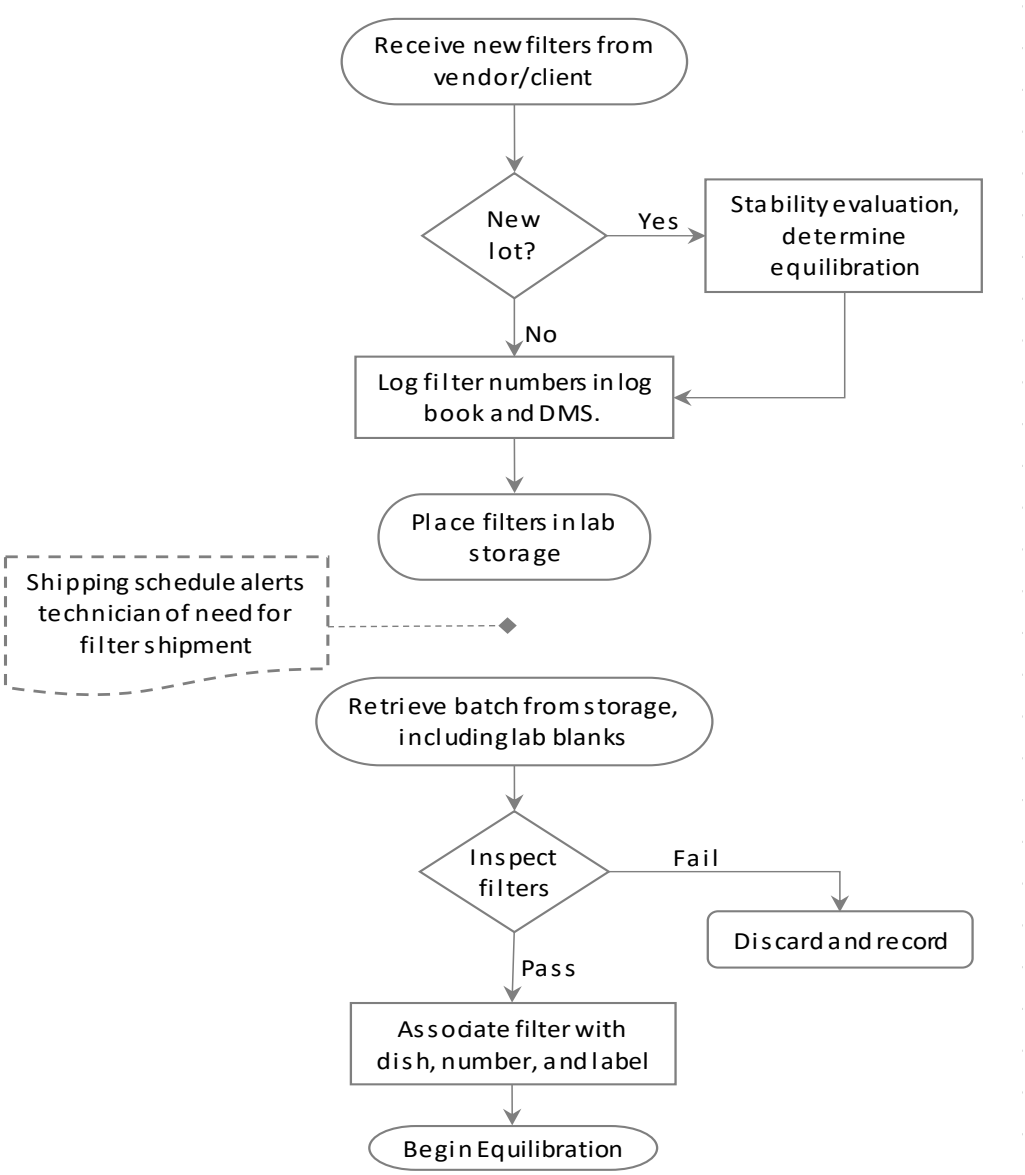
Figure B4-1 Receipt and Preparation of new filters Flow Diagram



B4.3 Analysis of Pre-Exposure Filters

After equilibration requirements have been met, pre-exposure filter analysis occurs, typically on Monday. Pre-exposure filters are then loaded into cassettes and packaged and shipped to locations. A flow diagram for pre-exposure of clean filters is located in Figure B4-2. Table B5-1 lists quality control criteria.

B4.4 Receipt of Exposed Filters

A flow diagram for receipt of exposed filters is located in Figure B4-3 which identifies equipment or instrumentation needed. Table B5-1 lists quality control criteria.

B4.5 Post-Analysis of Exposed Filters

A flow diagram for exposed filter analysis is located in Figure B4-4 which identifies equipment or instrumentation needed. Table B5-1 lists quality control criteria.

Figure B4-2 Pre-Exposure Filter Analysis Flow Diagram

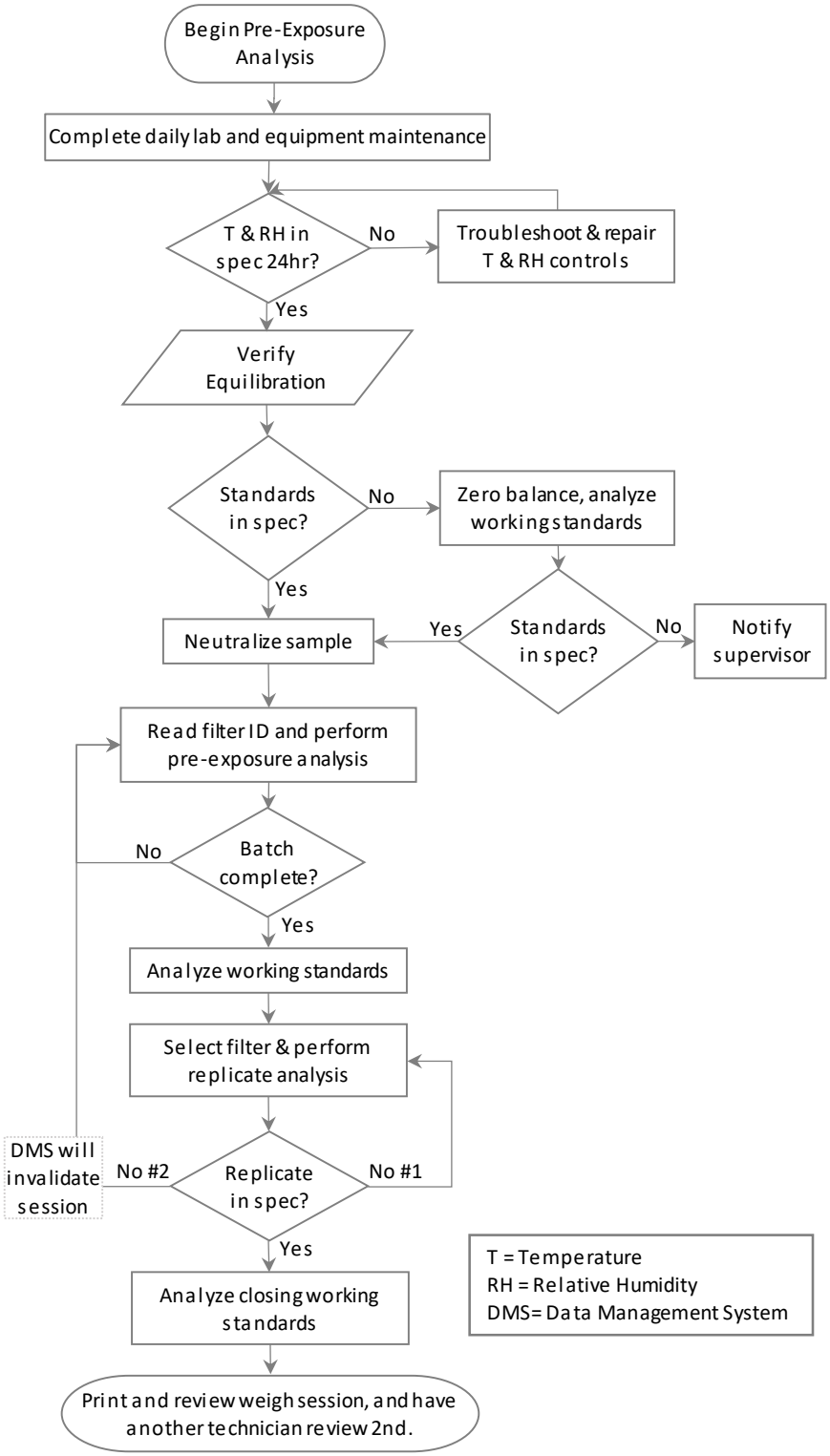


Figure B4-3 Post-Exposure Filter Receipt and Login Flow Diagram

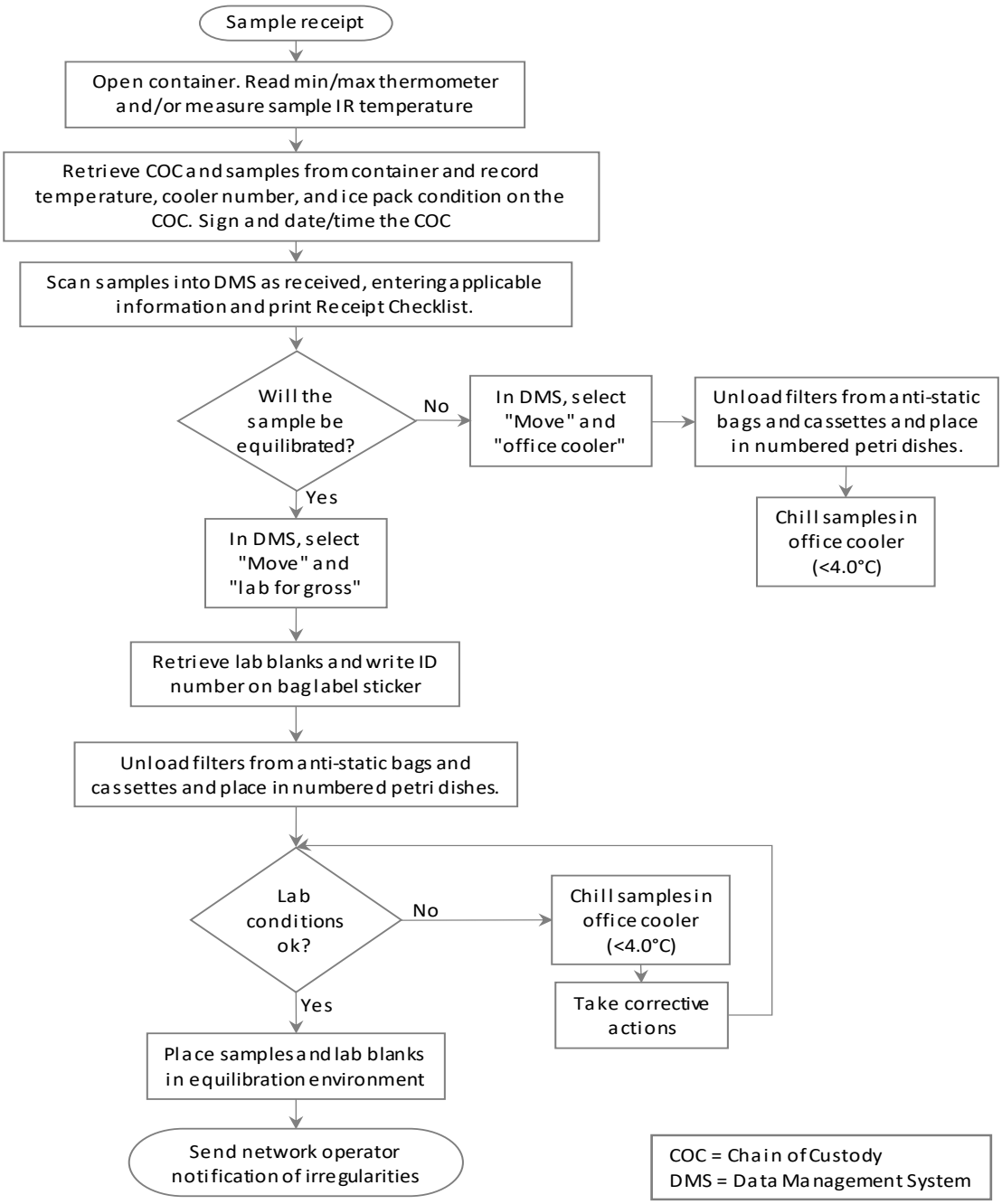
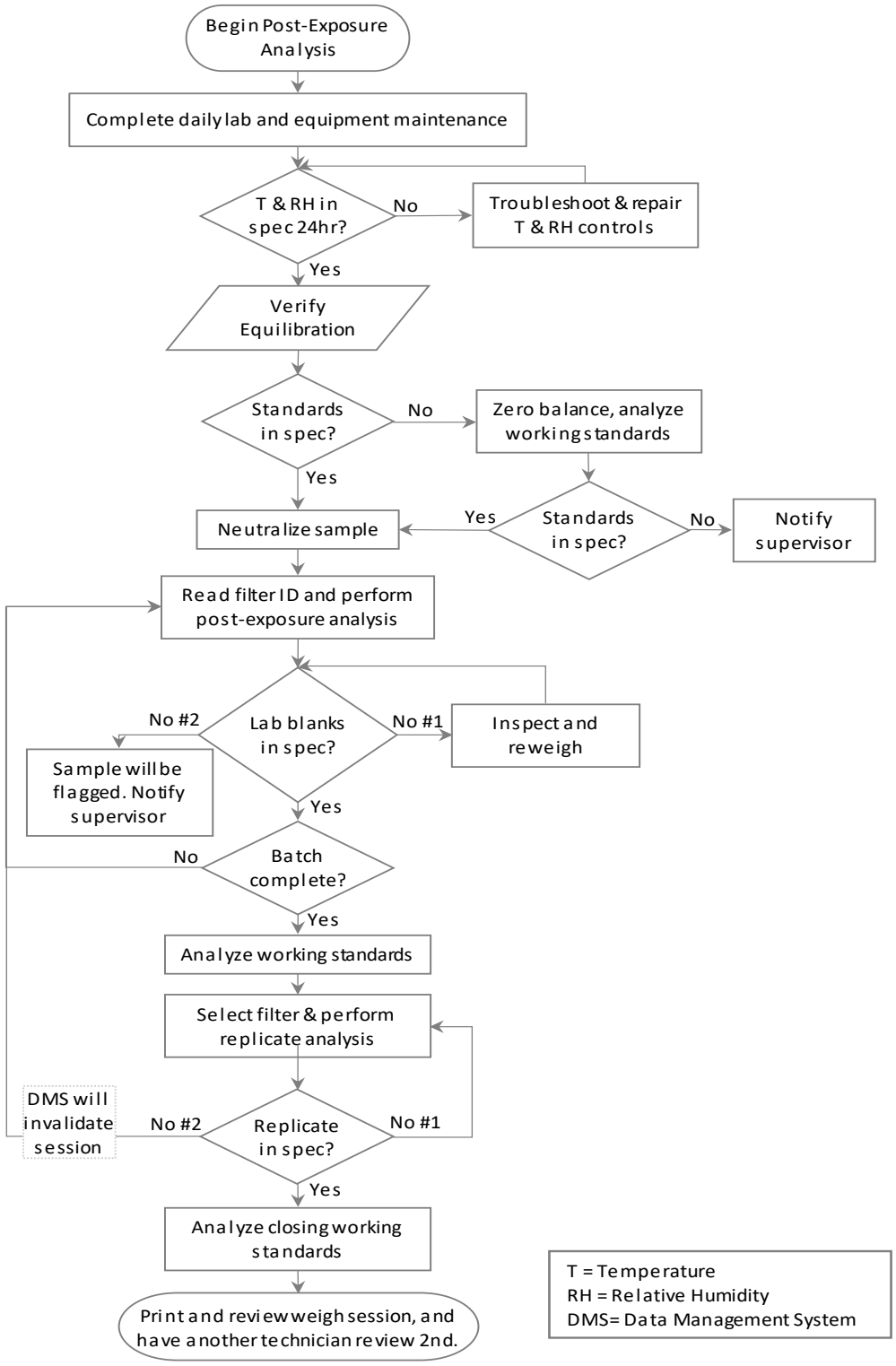


Figure B4-4 Post-Exposure Filter Analysis Flow Diagram



B5 Quality Control

B5.1 Quality Control Elements

The following table summarizes the quality control limits, quality control elements, and corrective actions used by the laboratory. Further detail is presented in subsequent sections. All excursions from control limits and all corrective actions are recorded in a laboratory notebook.

Table B5-1 Quality Control Matrix

QC Element	Frequency	Control Limits	Corrective Action
Filter integrity check	Each	Defects, see Section B5.2	Discard filter, return lot if failure rate is excessive
Sample equilibration time	Each	≥24 hours	Continue equilibration
Room relative humidity	Continuous	24-hr mean 30.0% to 40.0%, <±5.1% in 24 hours	Re-equilibrate
Room temperature	Continuous	24-hr mean 20.0˚C to 23.0˚C, <2.1˚C in 24 hours	Re-equilibrate
Relative humidity verification	Monthly	<±2.1%	Troubleshoot/ recalibrate
Temperature verification	Monthly	<±2.1˚C	Troubleshoot/ recalibrate
NIST traceable verification -Working mass standards	3 Months	<±2.1µg	Replace working standard/ calibrate microbalance
Analyze working mass standards	Every 10 analyses	<±3.1µg	Verify working standards
Independent calibration verification	Annual	±3µg	Service microbalance
Replace primary mass standards with new or recertified standards	Annual	±25µg tolerance or better	Return to manufacturer
Microbalance zero verification	Every 10 analyses	±2µg	Re-zero microbalance

QC Element	Frequency	Control Limits	Corrective Action
Lab Blanks	10% per client, or 1 per client	<±15.1µg	Investigate contamination source(s)
Replicate analysis	At least 1 per session or 10%	<±15.1µg	Re-analyze entire batch/session
Field Blank	Controlled by network	<±30.1µg	Notify client, investigate possible contamination sources within lab control
Lot stability evaluation (new lot)	9 clean filters per lot	<±15.1µg change between weighings	Extend equilibration period
Infrared thermometer	Quarterly	±2°C	Replace

B5.2 Clean Filter Integrity Check

All clean filters are visually inspected for defects before the initial analysis. A light table is used as a source of backlighting to assist in the detection of defects. If any defects are found the filter is discarded. Lots that contain an excessive number of defective filters are returned to the supplier. Specific filter defects are:

- Pinhole: A small hole appearing as a distinct and obvious bright point of light when examined over a light table.
- Wrinkle: Uneven creased area.
- Separation of ring: Any separation or lack of seal between the filter and the filter border reinforcing the ring
- Chaff or flashing: Any extra material on the reinforcing polyolefin ring or on the heat seal area that would prevent an airtight seal during sampling
- Loose material: Any extra loose material or dirt particles on the filter
- Discoloration: Any obvious, visible non-uniformity in the appearance of the filter when viewed over a light table that might indicate gradations in porosity or density across the face of the filter
- Other: A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship

B5.3 Microbalance Checks

The microbalance used for gravimetric analyses of $PM_{2.5}$ and PM_{10} filters has an independent calibration verification using NIST traceable standards annually. Before and after each weighing session the microbalance is verified against Upper and Lower (nominal) working mass reference standards. Additionally, the microbalance is checked against one of the working mass reference standards and zero after every 10 analyses. Results of all working mass reference standard checks are recorded in the DMS.

The working mass reference standards are also verified against the primary mass reference standards, as described in the SOP for Verification of Working Mass Reference Standards located in Appendix C:. Primary mass reference standards are independently certified or replaced annually.

When the microbalance is checked against primary mass standards and the control limits are exceeded, the microbalance requires calibration, as described in the SOP for Calibration of Microbalance located in Appendix C:. If the instrument calibration is unsuccessful, the microbalance is repaired or replaced.

Microbalance verifications, calibrations, and repairs are recorded in the laboratory notebooks.

B5.4 Primary and Working Mass Standards

Primary standards are used to verify working standards and the balance. If the specifications for the primary or working mass are exceeded, corrective action is taken as described in Table B5-1.

B5.5 Lot Blanks

Lot blanks are used to determine the mass stability of a new clean filter lot. When a new clean filter lot is received, a Filter Stability Evaluation is performed, as described in the SOP for Filter Stability Evaluation, located in Appendix C:.

B5.6 Lab Blanks

Lab blanks are inspected at the same time clean filters are inspected and prepared for clients. Lab blanks are stored in individually marked Petri dishes in the equilibration lab until needed. When clean filters are placed in the lab for equilibration, lab blanks from the same lot are also set out for equilibration. Lab blanks are equilibrated for the same time

period as the clean or exposed filters. Lab blanks are analyzed to determine contamination in the laboratory. Lab blanks are tare weighed with the pre-exposure filters, prior to shipment. Lab blanks are then covered and stored in the laboratory, matched to the same shipment when exposed filters are returned, and equilibrated with the exposed filters to be analyzed at the same time.

If the specification for a lab blank is exceeded, corrective action as described in Table B5-1, is taken.

B5.7 Field Blanks

Field blanks are sent to the site operators in accordance with the network's QAPP. Pre-exposure filters sent to the site operator are not distinguished as field blanks; rather, the site operator will use a pre-exposure filter at random to meet the network field blank requirement. Some networks may have the field blank pre-assigned. When field blanks are returned to the laboratory they are treated as a normal exposed filter. If field blanks are identified they are marked as field blanks in the DMS.

Corrective actions for field blank results outside acceptance limits are listed in Table B5-1.

B5.8 Replicate Analysis

Replicate analyses of pre-exposure filters or exposed filters are performed at the end of each client network's weighing session. One filter is selected from the weighing session and a second analysis is performed on the filter. Clients may request additional random replicate analysis or may request an independent analyst to perform the replicate analysis. Replicate analyses are performed as a quality assurance check against the microbalance. Corrective actions for replicate analyses outside of acceptance limits are listed in Table B5-1.

B5.9 Control Charts

Control charts are used to confirm that specific quality assurance criteria are met. Control charts are incorporated into quarterly reports. They include Working Standard Checks, Laboratory Environmental Conditions (temperature and relative humidity), Replicate Analyses, Lab Blanks, and Sample Archive Temperatures. Control charts include data from all clients in the lab.

Gravimetric Laboratory five-minute averages of temperature and relative humidity conditions are collected and control charted.

In addition to the control charts, quality assurance data for each filter is provided in the Excel spreadsheet tab labeled Exposed Lab Conditions in the monthly and/or quarterly report for clients.

Clients who receive "weights only" reports receive a modified spreadsheet tab in the report. Data is not provided from the client to calculate all the QC requirements. Clients who receive a full report with concentrations will receive the following in the Excel spreadsheet tab of the monthly or quarterly report. On the Exposed Lab Conditions tab, there are multiple columns. These columns include the filter number, the site name, the lab ID, followed by columns with all related data for the tare weigh session of the pre-exposed filter. Then the exposed weigh session data information, including the cooler temperature upon receipt, is given. The pre-exposure filter tare and exposed filter gross laboratory temperature and relative humidity differences are calculated and given in the spreadsheet along with the standard deviation for each filter. To calculate the expired tare date, the tare weigh date and time are compared to sample date and time to provide the difference in days. To calculate how many days after sampling the exposed weight analysis was performed, the exposed weigh date and time and the end of the sample period are compared to provide the difference in days. The end of the sample period date and time are compared to the sample retrieval date and time to give the difference in hours. The spreadsheet provides a quick review of the filter and quality information all in one place.

B5.10 QAPP Review

This Quality Assurance Project Plan will be reviewed annually and revised when revisions are necessary or every five years. All historic copies of the QAPP will be stored in electronic format. Network operators will be notified of the new revision.

B6 Instrument/Equipment Testing, Inspection, and Maintenance

Due to the specialized nature of laboratory equipment, maintenance consists mainly of cleaning and environmental control (temperature and relative humidity) within the laboratory. The Equipment SOP, contained in the QMP, describes how to identify, locate, determine status, and confirm equipment acceptability. A spare microbalance is kept on site. A spare set of primary standards is kept on site. A spare temperature and relative humidity probe are kept on site. Filter cassettes and coolers are replaced as needed.

B6.1 Laboratory

The sensitivity of the gravimetric analyses required by the methods 40 CFR 50, Appendix L and Appendix O, make laboratory location and design critical.

The laboratory is located in an office building at 555 Absaraka, Sheridan, Wyoming. The lab is situated in a basement room with no windows. Two walls are constructed of concrete, one of which is the only external wall. The other two walls and the ceiling are of typical wood frame and drywall construction. The floor is concrete on earth, covered with rubber, hardwood, and vinyl tile. The wood portion of the floor is cut to provide isolation from vibration in the remainder of the building. The lab door construction is for external application and therefore provides a good seal when closed. At the entrance to the lab is a sticky floor mat used to control foot-borne particulate in the laboratory environment.

The room is pressurized slightly with a small fan and a port through an internal wall. Air is fed into the room through a high efficiency particulate filer. Lab air is filtered continuously with a discrete, high efficiency particulate filter. The HEPA filter is inspected quarterly and replaced at least annually.

Two concrete tables are available for installation of analytical microbalances. A ventilated closet is adjacent to the laboratory for dissipating waste heat from the room's environmental control equipment.

B6.1.1 Laboratory Anteroom

All exposed filter handling takes place in a separate controlled space outside the laboratory to minimize possible contamination from outside sources. Activities that take place in the Laboratory Anteroom include removing exposed filters from their anti-static bags and cassettes. The room is also used for the storage of cassettes and petri dishes.

B6.2 Static Control

Controlling and neutralizing electrostatic charge is critical in the performance of micro-gravimetric analyses. Static is controlled through laboratory design and use of a variety of equipment.

Earth ground is accomplished with a half-inch diameter copper rod, inserted approximately 6 feet into the ground through the floor. One end of a lab perimeter ground bus is firmly attached to this rod. The ground bus is made of 8 AWG solid copper wire. The ground bus makes earth ground readily available throughout the lab. Instruments, anti-static mats, storage shelves, working surfaces, and operator discharging conductors are connected to the ground bus with copper wire.

One anti-static mat is located on the top of the microbalance table where it keeps electrostatic charge from accumulating around the microbalance. Another anti-static mat is located on the floor below the table to discharge the microbalance operator.

Electrostatic charge in the samples is neutralized by placing them on a $Polonium^{210}$ strip for a nominal 60 seconds prior to analysis. Strips are replaced approximately 6 months from the manufacture date. Static is further controlled through the design of the microbalance. The weighing chamber of the microbalance is coated with a static neutralizing substance that discharges any residual static from the sample.

B6.3 Temperature and Humidity Control

The temperature and relative humidity in the laboratory are continuously measured and controlled. A data logger records temperature and relative humidity once per second. This information is used to operate discrete room conditioning components including heater, air conditioner, humidifier, and dehumidifier. Control requirements are listed in Table B5-1. Temperature and relative humidity averages for 5-minute and 24-hour periods are displayed on the computer screen interface with a graphical presentation also available. This display is color-coded with green for good, yellow for warning, and red for stop. In addition, the laboratory microbalance interface will prevent weighing if conditions are outside control limits or the data from the system is not current. The conditions must be back in compliance to re-start equilibration.

Laboratory environmental conditioning equipment is controlled based on thresholds in the data logger. Control thresholds are tighter than the specifications of the method to assure that short-term transients won't exceed the specified range.

The data logger has an Ethernet connection to the network. A computer automatically collects the data every 5 minutes and loads it into the DMS. See Section B10 for further information regarding the database. The data logger records one-second readings, and then stores both 5-minute and hourly average, minimum, maximum, and standard deviation temperature and relative humidity. These measurements are periodically checked against NIST traceable standards according to Section B6.7.2 and B6.7.3.

B6.4 Microbalance

A microbalance is used for gravimetric determinations of particulate matter samples. It features automatic calibration, separate control and sensor units, and anti-static weighing chamber. Instrument specifications are listed in Table B6-1.

A serial data connection between the microbalance and a computer facilitates transfer of data directly from the instrument to the DMS. See Section B10 for further information regarding database.

B6.5 Data Handling Equipment

A computer in the laboratory continuously runs gravimetric DMS software. The software records information from three sources within the laboratory. Temperature and relative humidity data are polled from a data logger, that measures and controls laboratory environmental conditions. Sample identification numbers are input with a barcode reader. Analytical results are input into the database via a direct connection from the microbalance.

B6.6 Maintenance Equipment

Daily cleaning procedures for the laboratory and equipment are located in Appendix C:. The laboratory is cleaned weekly after all analyses are complete through general damp techniques to maximize entrainment of particles. A minimum of liquid is used to lessen the effect on relative humidity.

B6.7 Reference Standards

A variety of reference standards are used to evaluate the accuracy of measurements made in the laboratory. Each of the reference standards are described in this section.

B6.7.1 Mass Reference Standards – Primary and Working Mass Standards

Two sets of standards are used in the micro-gravimetric laboratory, primary and working.

Working standards are used to verify microbalance reproducibility throughout operating sessions. ASTM Class 0, Class 1 or Class 2 or better standards are used, typically nominal masses of 200mg and 500mg. Working mass reference standards are stored in a protective container in the laboratory.

Primary mass reference standards are used to verify the working standards periodically and/or when certain control limits are exceeded. ASTM Class 0, Class, 1, Class 2 or better standards are used, typically nominal masses of 200mg and 500mg. Primary standards are stored in the laboratory clearly labeled as the Primary Standards. Primary standards are recertified by the manufacturer or replaced annually.

B6.7.2 Temperature Reference Standard

A NIST traceable temperature standard is used to verify the laboratory temperature. Instrument specifications are listed in Table B5-1. A NIST traceable thermometer is used to verify the archive coolers. The temperature standards and min-max thermometers are recertified against a NIST traceable temperature standard annually or are replaced before the certification expires. The infrared thermometer gun is recertified quarterly.

B6.7.3 Relative Humidity Reference Standard

A NIST traceable, digital relative humidity standard is used to verify the laboratory measurement system. Instrument specifications are listed in Table B6-1. The relative humidity standard is recertified annually with reference salt standards or replaced with a new standard before the certification expires.

B6.8 Protective Bags

Once tared filters are loaded into cassettes, they are placed in protective anti-static bags. The bags are sized to provide a snug fit around the cassette, so the surface of the bag is taut and will not contact the filter. The bags are constructed of plastic with an additive which provides anti-static properties. The bags used are purchased and tested using SOP.

B6.9 Sample Archive

Two systems are used for sample archive: temporary and long term.

B6.9.1 Temporary Archive

A small refrigerator is used as a temporary archive. Exposed filters are placed in the temporary archive when they arrive at a time when they cannot immediately be placed in the equilibration lab. Exposed filters that may require further inspection or analysis are also stored in the temporary archive. The temporary archive refrigerator is located in the sample receiving area and is maintained at ≤4˚C.

B6.9.2 Long term Archive

Walk-in coolers are used as a long-term archive. After exposed filters are analyzed, they are typically placed in the long-term archive for at least one year. Exposed filters are archived by network and exposed analysis month. The archive coolers are located in the same building as the laboratory and are maintained at ≤4˚C.

B6.10 Filter Cassettes

Filter cassettes are cleaned prior to each use, by hand, using lab grade soap and warm water, and then are rinsed thoroughly with de-ionized water. The cassettes are then left to air-dry in a protected area. This procedure is located in Appendix C:.

B6.11 Shipping Containers

Insulated shipping containers capable of maintaining temperatures at or below 4°C and able to withstand the rigors of shipping are needed to transport filters from the field to the laboratory. Plastic coolers or insulated boxes are typically used.

Shipping containers are inspected prior to each use. Damaged containers are discarded.

B6.12 Min/Max Thermometers

Min/Max thermometers are used to determine the maximum temperature during return shipment of exposed filters.

B6.13 Infrared Thermometer

Infrared thermometer is used to determine the maximum temperature of return shipments of exposed filters.

Table B6-1 Instrument Specifications

Instrument	Specification
Microbalance	Readability at least ±1µg Repeatability (standard deviation) of at least ±1µg
Primary Mass/Working Mass	0.025 mg tolerance (Class 2)
Primary Mass/Working Mass Verification/Calibration Standards	0.025 mg tolerance (Class 2)
Temperature Reference	Resolution of ±0.1˚C Accuracy of ±1˚C
Relative Humidity Reference	Resolution of ±1% Accuracy of the instrument is ±2% RH at mid-range, otherwise ±4% RH

B7 Instrument/Equipment Calibration and Frequency

B7.1 Microbalance

Proper use, care, and maintenance of the microbalance are essential in gathering accurate and precise gravimetric data. Routine maintenance and calibrations assure that quality data is being obtained.

The microbalance is independently certified at least annually with ASTM/ANSI certified and NIST traceable mass reference standards. The calibration is certified before and after each weighing session using two mass reference working standards that have been verified against NIST traceable primary mass reference standards of the same nominal mass. Calibrations and resolution of deficient calibrations are documented as detailed in Section A9.

B7.2 Mass Reference Standards

Two sets of mass reference standards (primary and working) are used in the micro-gravimetric laboratory. The mass reference standards are selected to bracket the maximum and minimum expected filter weights. Typically, the upper standard is 500mg and the lower standard is 200mg. ASTM Class 0, Class 1, or Class 2 or better standards are used as the primary and working standards.

Primary standards are used to verify the working mass reference standards. The primary standards are independently certified annually or replaced.

Working standards are used to verify microbalance reproducibility throughout operating sessions. At least every three months, the working standards are verified against the primary standards. The working standards are also verified against the primary standards when drifts are detected in the mass of the working standards. The SOP for Verification of Working Mass Reference Standards is located in Appendix C:. Working standard verifications are documented on a custom form, an example of which may be found in Appendix D:

B7.3 Min/Max Thermometers

Min/Max thermometers are either certified or replaced annually.

B7.4 Infrared Thermometer

Infrared thermometer is certified quarterly. Table B5-1 lists quality control and corrective actions. Calibrations and resolution of deficient calibrations are documented as detailed in Section A9.

B7.5 Temperature and Humidity Control

The laboratory temperature and relative humidity measurement and recording equipment are verified at least monthly against a NIST traceable reference temperature and relative humidity standards. Specifications are listed in Table B6-1. The laboratory temperature and relative humidity standards are recertified against NIST traceable reference standard or replaced, as needed, before the Traceable Certificate of Calibration expires. The results of these verifications are recorded in the laboratory notebook. Table B5-1 lists quality control and corrective actions. Calibrations and resolution of deficient calibrations are documented as detailed in Section A9.

B8 Inspection/Acceptance of Supplies and Consumables

Approved vendors and NIST Traceable certifications are used to control quality critical materials. Quality critical materials generally include measurement devices, instrumentation and communications equipment. Services which are quality critical generally include instrument repair and calibration service but may include a contractor.

Vendors and service providers are selected based upon historically proven quality of service or supply; new vendors are selected based on collection of sufficient evidence of ability to provide required quality of service or supply necessary. Vendors start out as approved and acceptance of a new vendor is evidenced by a Manager signing the requisition, and/or order placement.

Additional detail can be found in IML Air Science's QMP which contains the Procurement SOP.

B9 Non-direct Measurements

This section addresses data not obtained by direct measurement. Non-measurement sources may include:

- Downloaded client monitoring data
- Sampler run data imported into the DMS
- Chemical and Physical Properties data
- Manufacturer's Operating Manual information
- Geographic location data
- Historical monitoring information
- External monitoring databases
- National Weather Service data

Any use of outside data will meet quality control criteria to the extent possible, following QA procedures outlined in this document and in applicable EPA guidance documents, including historical data.

B9.1 Chemical and Physical Properties Data

Physical and chemical properties data and conversion constants are often required in the processing of raw data into reporting units. This type of information that has not already been specified in the regulations will be obtained from nationally and internationally recognized sources. Other data sources may be used with approval from the Quality Officer. The following sources may be used without prior approval.

- National Institute of Standards and Technology (NIST)
- ISO, IUPAC, ANSI, and other widely recognized national and international standards organizations
- U.S. EPA

B9.2 Manufacturers' Literature

Another important source of information is manufacturers' literature. Operations manuals and users' manuals provide useful information.

B9.3 Historical Monitoring Information

Historical data and summary information derived from data may be used in conjunction with current results to calculate and report trends. In calculating historical trends, it is important to verify that historical data are fully comparable to current monitoring data. If different methodologies were used to gather historical data, the biases and other inaccuracies must be described in reports based on that data.

B9.4 External Monitoring Databases

No data obtained from the Internet, computer bulletin boards, or databases from outside organizations shall be used in creating reportable data or published reports unless documentation of the source of the data is attached to the report.

B9.5 Supplemental Meteorological Data

Meteorological information is gathered by the National Weather Service (NWS). Parameters generally include temperature, relative humidity, barometric pressure, rainfall, wind speed, wind direction, cloud type/layers, percentage cloud cover and visibility range. These data may be used to supplement meteorological data collected.

B10 Data Management

Data is handled by a custom DMS. The DMS contains tables to store laboratory environmental data, quality control data, pre-exposure filter data, exposed filter data, sample collection data, sample handling data, and sample tracking data.

Filter ID numbers are read electronically with bar code readers. Filter weights are electronically retrieved from the microbalance and stored in the DMS. Lab conditions are automatically retrieved and stored in the DMS. This process eliminates many of the possibilities for typographical errors.

Employees are trained in the use of the DMS and are authorized for data entry. The DMS allows authorized users to access appropriate levels within the DMS. Training is described in Section A8.

New or replacement hardware and software is validated prior to use and an acceptance checklist is completed, based upon function. Testing of hardware and software is performed to ensure it conforms to the requirements of the function. Additional information can be found in the IML Air Science QMP.

B10.1 Laboratory Data

An eight-digit sample identification number is taken from each clean PTFE filter to uniquely identify each clean filter tared in the laboratory, regardless of destination. A range of numbers needed for each tare session is created by creating a tare batch for a specific shipment day and assigning in the DMS. Numbers are confirmed by placing a clean filter in a petri dish with the same unique sample ID number on the lid after clean filters have been inspected. Discarded or damaged clean filters are noted in the DMS. Once clean filters are placed in the equilibration laboratory, individual records for each filter ID are created by performing tare equilibration in the DMS. Once a tare session is complete, the clean filter becomes a pre-exposure filter. Pre-exposure filters are assigned to a shipper and to each shipping location by entering shipping service into the DMS. The filter ID number for each tared pre-exposure filter is printed on the anti-static bag label that protects the pre-exposure filter during transportation to and from the field.

Upon return to the laboratory, the filter ID number is used to place each exposed filter back into its uniquely labeled petri dish for equilibration. The filter ID number for each exposed filter is scanned into the DMS during exposed analysis sessions. Upon the close of each

exposed analysis session, the filter ID number is used to match the exposed filter (in a Petri dish) with its protective anti-static bag for archiving.

Laboratory data is managed by a DMS, designed for gravimetric laboratory applications. The DMS runs continuously on the laboratory computer. It serves three basic functions: tracking QC activities, recording analytical results, and recording laboratory environmental conditions.

Prior to weighing, the DMS prompts the user for their name and password. The DMS automatically verifies that the lab environmental conditions are and have been in specifications for the last 24 hours. At the beginning of each weigh session, and after every 10 filters, the DMS prompts the user for microbalance zeroes and working standard checks. The DMS also records replicate and lab blank analytical results as well as notifies the operator when laboratory temperature or relative humidity approach the control limits.

Equation B10-1 Net Mass Calculation

$$M_{net} = (M_g - M_t)$$

Where:

M_{net} = total mass of PM collected during the sampling period, mg

M_g =gross mass of the equilibrated filter after sample collection, mg

M_t = tare mass of the equilibrated filter before sample collection, mg

The data acquisition system in each laboratory monitors and controls environmental conditions every second. Five-minute mean values are logged from the previous 300 one-second values. The mean calculation is a block average, not a rolling average. In addition to the mean, the minimum, maximum, and standard deviation of the one-second relative humidity and temperature are also logged for that five-minute period. Environmental condition control charts display this five-minute data.

Every time a filter is weighed, the previous 86,400 seconds of relative humidity and temperature data are averaged and recorded as the equilibration conditions for that mass determination. During gross mass determination (exposed filter), equilibration conditions are automatically compared to the tare equilibration conditions (pre-exposure filter) to ensure that conditions from tare to gross meet acceptance criteria. If these acceptance criteria are not met, the DMS prevents further weighing until the aforementioned conditions are met.

The DMS calculates the difference between pre-and post-weigh sessions for relative humidity and temperature. The technician is alerted if the difference is outside acceptance criteria. The calculations are performed as cited in Equation B10-2 and Equation B10-3.

Equation B10-2 Relative Humidity Difference

$$Relative\ Humidity\ Difference = Relative\ Humidity_{Tare} - Relative\ Humidity_{gross}$$

Equation B10-3 Temperature Difference

$$Temperature\ Difference = Temperature_{Tare} - Temperature_{gross}$$

The analysis of filters is accomplished through interaction with the DMS. Filter ID numbers are input into the DMS with a barcode reader that scans the label on the lid of each Petri dish. Once the filter ID number is input and the microbalance has a stable mass measurement, the analytical result is input into the DMS via communication between the microbalance and the laboratory computer. The date and time of each analysis is recorded in the DMS database.

B10.2 Exposed Filter Collection Data

Extracting the exposed filter collection data from the sampler is typically the network operator's responsibility. Run data are downloaded from the sampler in accordance with the network's QAPP (typically once a month). IML Air Science receives data from the client electronically.

Samplers are programmed by the operators so that each data record includes sampler serial number, exposed filter ID number, as well as the measurements recorded by the sampler (volume, sample time, average temperature, average pressure, error codes, etc.). Exposed filter ID number entry into the sampler is critical for correlating laboratory and exposed filter collection data and is the responsibility of the network operators.

Exposed filter collection data are imported from a variety of particulate samplers into tables in the DMS. The DMS checks the data for reasonableness as the data are imported.

B10.3 Sample Handling Data

The DMS contains tables to store sample handling data.

B10.3.1 Transportation

Data related to the shipping of pre-exposure filters to the network operators and the return of exposed filters to the laboratory is recorded by the DMS. When pre-exposure filters are shipped to the field, data are recorded including date and method of shipment, quantity and identity of pre-exposure filters, and the receiving location. Data recorded at the time of receipt of exposed filters at the laboratory include date, whether or not a chain of custody form was used for shipment of exposed filters to the laboratory, the receipt temperature and/or the maximum temperature of filters during shipment.

B10.3.2 Equilibration

The DMS is used to record the equilibration time of all filters and the time of each analysis.

B10.3.3 Exposed Sample Login

The receipt of exposed filters is recorded in the DMS. Information recorded includes the date of receipt, network, location (site), exposed filter ID numbers, whether a chain of custody form was used, the maximum temperature recorded by the min/max thermometer or the infrared thermometer gun, and comments. Field blanks, labeled as such, are identified at the time of exposed filter login.

B10.3.4 Data Integration

Data from the laboratory and from the field are integrated by the DMS. See Section A9.2. Where appropriate, the DMS determines data validation and qualification. The data from the lab and field DMS tables are related by filter ID number.

B10.3.5 Data Archive

Tables containing laboratory data are created by the DMS. These tables containing sample collection data and laboratory data are backed up from the network server, as described in the IML Air Science QMP.

The laboratory environmental conditions summary data are downloaded from the data logger every 5 minutes and stored in the database tables.

Data are archived on electronic storage media and stored off-site in a secure location. DMS data are maintained for a minimum of five years.

C Assessment and Oversight

C1 Assessments and Response Actions

The results of quality assurance assessments indicate whether the control efforts are adequate or need to be improved. Assessment results are reported to the Gravimetric Laboratory Supervisor, the IML Air Science Manager, and the Quality Officer.

The adequacy of the quality system is assessed annually through the QAPP review conducted by the Quality Officer or other appropriate staff. Conditions requiring revision or renewal include expiration after five-calendar-year life span following approval date, major changes to the organization, revisions to 40 CFR 58 or other regulations that affect the quality system, and assessment findings requiring corrective action.

Internal audits of the micro-gravimetric laboratory and the quality system are performed annually, as described in the Audit Procedure of the IML Air Science Quality Management Plan. Response actions are addressed through the applicable Corrective Action Procedure, also located in the IML Air Science Quality Management Plan.

C2 Reports to Management

Management Review, conducted annually by the Quality Officer or other appropriate staff, assesses the adequacy of IML Air Science's Quality System. The review includes previous management reviews, Quality Policy, Quality Objectives, corrective actions, external audits, internal audits, complaints, and resource allocations.

D Data Validation and Usability

D1 Data Review, Verification, and Validation

This section summarizes how data are reviewed, verified, and validated. The criteria used for data review, verification, and validation are described in this section.

Data validation, as used in the section, is the process of checking data quality to determine problems that exist in the collection or transportation data. Exposed filter validation is the process of determining the validity of exposed filters by comparing data to criteria specified in the Reference Method. Data reduction is the process of summarizing data from a sampler, a site, or a network. Reporting is the presentation of data to the network administrator.

D1.1 Data Validation

Data validation is a process of monitoring the quality of field operations. This process is accomplished with a computer program that imports field data records into database tables. Data are checked against reasonableness criteria. These criteria are summarized in Table D1-1. If data are found outside of these criteria, the network administrator and site operator are notified so corrective action can be implemented. If the resulting investigation determines that data criteria are violated, the data are invalidated or qualified.

Table D1-1 Data Validation Criteria for Reasonableness

Parameter	Acceptable Range	
	Lower	**Upper**
Ambient Temperature (min, max, avg.)	-50˚C	50˚C
Filter Temperature (min, max, avg.)	-50˚C	50˚C
Max Temperature Difference	0˚C	15˚C
Barometric Pressure (min, max, avg.)	500 mmHg	800 mmHg
Sample Volume	0.0 m^3	30.0 m^3
Flow Rate (min, max, avg.)	0.0 L/min	20.0 L/min
% CV	0%	8%
Date (start, stop, set, actual, T_{dif})	Current date	Current date
Time (start, stop, set, actual, T_{dif})	00.00	24.00

D2 Verification and Validation Methods

D2.1 Exposed filter Validation and Qualification

Validation of particulate mass on exposed filters occurs throughout the process. Each step in this process is evaluated for compliance with requirements, from tracking of clean, pre-exposure, and exposed filters in the lab, to the field, and returning to the lab for unloading, inspection, and final weighing. If conflict arises, consult Section C1.

Documentation on the chain of custody, bag label, sample receipt form, and in the DMS, provides data for validating particulate matter on exposed filters. Exposed filter data are tracked by filter ID. Sampler data, if available, are imported and compared to data on the bag label. If the sampler data have error codes, the DMS will flag the data and flags will appear in the QC sampler table and on the report to prompt further review of the data. These flags are in-house flags and summarized in Table D2-1.

The reporter investigates whether the sample should be qualified or invalidated using the appropriate EPA qualifier code. Qualifier codes are listed on EPA's Ambient Monitoring Technology Information Center (AMTIC).

Validation of data during reporting involves the following:

- Field Level: The operator may invalidate an exposed filter upon retrieval depending on the condition of the exposed filter or sampler. For example, if an exposed filter was dropped, contaminated, or the sampler failed to run due to machine malfunction or power failure, it may be invalidated.
- Laboratory: During unloading and inspection of exposed filters, the laboratory technician may discover filter damage or contamination. The technician may add a comment on the comment line of the database Exposed Login Form.
- DMS: Using sampler data information, sampler status codes, and internal database checks, the database will automatically flag exposed filters.

Flags in Table D2-1 are used in-house and appear in the "flags" column of the client's excel spreadsheet. These flags alert the technician of any potential problems. If the sample needs to be invalidated or qualified, the technician selects the QC button and manually codes the exposed filter with the appropriate EPA code.

Data Management System:

- For AQS files, the DMS automatically places the "W" qualifier for the concentration when the in-house FE (Flow Excursion) flag appears.
- For AQS files, the DMS automatically places the "X" qualifier for the concentration when the in-house TD (Temperature Difference) flag appears.
- For AQS files, the DMS flags data with SP (Sample Period) when the sample period is <1380 or >1500 minutes. If the sample period is <1380 minutes, the DMS uses Equation D2-1 to calculate the concentration, as if the sampler had run the full 24 hours. If the value calculated is high enough to be an exceedance, such an exceedance would be valid for the purpose of comparing to the NAAQS. If considered valid, it will carry the "Y" qualifier in AQS; the "Y" qualifier only being used with $PM_{2.5}$ sample concentrations.

Equation D2-1 Particulate Matter Concentration in micrograms per cubic meter

$$concentration\ \left(\frac{\mu g}{m^3}\right) = \frac{net\ weight\ (mg)}{volume\ (m^3)} \times 1000$$

Where:

Volume = 24 m^3 local conditions
Net weight in mg

Table D2-1 IML Air Science In-house Sample Collection and Laboratory Flags

IML Flag	Name	Description	Acceptance Criteria
CI	Collection Interval	Collection Interval Exceedance	Sample was not collected from midnight to midnight
CV	Coefficient of Variation	Coefficient of Variation	Flow rate ≤2%
FB out	Field Blank	Field Blank Exceedance	Field blank associated with sample exceeds ±30µg
FE	Flow Excursion	Flow Rate Excursion	>±5% of 16.67 L/min for >5min.
FS	Flow Stop	Flow Rate Deviation	Deviation by more than 10% from set point for more than 60 seconds
HT	Hold Time	Hold Time Exceedance	<10 days from sample end date if shipped at ambient temp, or <30 days if shipped below avg. ambient temp (or 4˚C or below

IML Flag	Name	Description	Acceptance Criteria
			for avg. sampling temps <4˚C) from sample end date
LB	Lab Blank	Lab Blank Mass Change Exceedance	Associated lab blank mass change exceeds ±15µg
LC	Lab Conditions	Lab Conditions Outside of Range during 24 hours prior to analysis	20˚C<T<23˚C 30%<RH<40%
MD	Missing Data	Sample Collection Data Missing	Sample collection data missing
NM	Negative Mass	The Gross Mass minus the Tare Mass is a Negative Mass	The gross mass minus the tare mass is a negative mass
PI	Power Interruption	Power Outage of >60sec. occurred during sampling	Power outage of >60sec. occurred during sampling
SP	Sample Period	Elapsed Sample Period differed by more than ±1 hour of programmed period	Elapsed sample period <1380 minutes or >1500 minutes
SR	Sample Removal	Sample Removal Exceedance	Sample not removed from the sampler within 177 hours of end of sample period
ST	Sample Temperature	Sample Temperature Exceedance	Sample temperature exceeded 25˚C after removal from sampler
TD	Temperature Difference	Temperature of Filter Exceedance	Measured temperature of filter exceeded the measured ambient temperature by more than 5˚C for more than 30 minutes
WD	Wrong Day	Sample Period does not match the schedule	Sample period does not match the schedule
XT	Expired Tare	Sample Period followed tare analysis by more than 30 days	Sample period followed tare analysis by more than 30 days

D3 Reconciliation with User Requirements

D3.1 Data Reduction and Reporting

Data reduction involves aggregating and summarizing results so they may be reported and interpreted by the monitoring and review organizations. Data are reduced and reported such that they may be compared with the NAAQS and to evaluate trends. Common data summaries include:

- Average concentration for a given period, typically quarterly and annual
- 98^{th} percentile 24-hour value for a given reporting period
- Data completeness for samplers, sites, and networks
- Accuracy and precision statistics

Data reduction is performed by the DMS. The DMS queries data from database tables, performs necessary data validation and reduction, and produces a report as specified by the user.

Report contents are detailed in Section A9.2.

Appendices

Appendix A: References

Appendix B: Data Qualifiers

Appendix C: Standard Operating Procedures

Appendix D: Forms

Appendix E: Revisions

Appendix F: Annual Review

Appendix A: References

Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

40 CFR 50, Appendix L; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere.

40 CFR 50, Appendix O; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10\text{-}2.5}$ in the Atmosphere.

40 CFR 50 Appendix K; Interpretation of the National Ambient Air Quality Standards for Particulate Matter.

"Clarification on Use of PM2.5 Field and Laboratory Requirements for Low Volume PM10 Monitoring to Support PM10 NAAQS," dated 03/03/2016 from Mike Papp, QA Team Lead, Ambient Air Monitoring Group (C304-06).

Appendix B: Data Qualifiers

Null Codes

Letter NullCode	Description
AA	Sample Pressure Out of Limits
AB	Technician Unavailable
AC	Construction/Repairs in Area
AD	Shelter Storm Damage
AE	Shelter Temperature Out of Limits
AF	Scheduled But Not Collected
AG	Sample Time Out of Limits
AH	Sample Flow Rate Out of Limits
AI	Insufficient Data (Can't Calculate)
AJ	Filter Damage
AK	Filter Leak
AL	Voided by Operator
AM	Miscellaneous Void
AN	Machine Malfunction
AO	Bad Weather
AP	Vandalism
AQ	Collection Error
AR	Lab Error
AS	Poor Quality Assurance Results
AT	Calibration
AU	Monitoring Waived
AV	Power Failure
AW	Wildlife Damage
AX	Precision Check
AY	QC Control Points (Zero/Span)
AZ	QC Audit (Audit)
BA	Maintenance/Routine Repairs
BB	Unable to Reach Site
BC	Multi-Point Calibration
BE	Building/Site Repair
BH	Interference/Co-Elution
BI	Lost or Damaged in Transit
BJ	Operator Error
SC	Sampler Contamination
TS	Holding Time Or Transport Temperature Is Out Of Specs.

Appendix C: Standard Operating Procedures

SOP Number	Title
GR-501-0.0	SOP for Calibration of Microbalance
GR-302-0.0	SOP for Performing Verification of the Archive Cooler and Sample Receiving Refrigerator Temperature
GR-303-0.0	SOP for Performing Annual Repeatability Test
GR-108-0.0	SOP for Receipt of New Micro-Gravimetric Filters
GR-200-0.0	SOP for Performing a Filter Stability Evaluation (Lot Blank)
GR-109-0.0	SOP for Preparing Filters for Pre-Exposure Analysis
GR-600-0.0	SOP for Cleaning and Maintenance of Gravimetric Laboratory
GR-110-0.0	SOP for Performing the Pre-Exposure Analysis
GR-111-0.0	SOP for Shipping Filters to Locations
GR-112-0.0	SOP for Receipt and Login of Exposed Particulate Filter Samples
GR-113-0.0	SOP for Performing the Post-Exposure Analysis
GR-114-0.0	SOP for Archiving Filter Samples
GR-304-0.0	SOP for the Verification of Working Mass Reference Standards
GR-301-0.0	SOP for Performing Monthly Verifications of the Laboratory Temperature and Relative Humidity
GR-201-0.0	SOP for Performing Stability Test on New Lots of Anti-Static Bags
GR-107-0.0	SOP for Transfer of Filters for Analysis
GR-900-0.0	SOP for Validation of Software Development or Modification
GR-901-0.0	SOP for Report Review

SOP GR-501-0.0

STANDARD OPERATING PROCEDURE FOR CALIBRATION OF MICROBALANCE

May 2021
IML AIR SCIENCE

REVISED BY:

Mary Hannigan _____ 05/17/21
Date

APPROVED:

_____ 5/17/2021
Manager Date

_____ 5/17/2021
Quality Officer Date

Reviewed

Initials				
Date				

Table of Contents

1.0 Scope and Application

1.1 This procedure covers the calibration of the microbalance used for micro-gravimetric analyses of particulate matter (PM) samples.

2.0 Summary of Method

2.1 When the balance is checked against primary mass standards and the control limits (<±3.1 µg) are exceeded, the balance requires calibration. Calibration is performed per manufacturer's instructions in the manual.

3.0 Definitions

3.1 Microbalance - A type of analytical balance that can weigh to the nearest 0.001 mg (that is, 1 µg or one-millionth gram).

3.2 Working standard – ASTM Class 0, Class 1, or Class 2 mass standard, traceable to NIST, that is used for routine quality control checks.

3.3 Primary standard – ASTM Class 0, Class 1, or Class 2 mass standard, traceable to NIST, that has "authority" over other standards (working standards) in the laboratory.

4.0 Health and Safety Warnings

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded. Use caution when servicing or operating electrical equipment in wet conditions.

4.2 General precautions for working with electro-mechanical equipment should be taken.

5.0 Cautions

5.1 Damage to the instrument may result if caution is not taken to properly install and maintain the device. Follow the manufacturer's instructions for maintenance of all equipment and for safe, secure installation.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained, authorized and familiar with the operation of a microbalance.

6.2 Instrumentation skills are necessary for interacting with the data acquisition system.

6.3 Familiarity with electronic and mechanical test equipment is required.

7.0 Equipment

7.1 Laboratory coat

7.2 Primary and working standards which bracket the expected filter mass. An upper standard (typically 500 mg) and a lower standard (typically 200 mg)

7.3 Primary 5 gram standard for Cahn

7.4 Microbalance; minimum sensitivity to 1 µg, minimum repeatability to 1 µg

7.5 Balance table with anti-static mat

7.6 Non-serrated, non-metallic forceps

7.7 Grounded floor mat

7.8 Microbalance calibration form

7.9 Laboratory Balance Notebook

8.0 Procedure

8.1 Review the calibration procedure in the microbalance operational manual and software within the microbalance.

8.2 Ensure that daily laboratory cleaning and maintenance preparation is complete and the lab is within specifications environmentally.

8.3 Ensure the microbalance and surrounding area is clean.

8.4 Record the current date and initial the Microbalance External Calibration Form (calibration form).

8.5 Place the upper primary standard on the microbalance and record “as found” value and the serial number of the standard on the calibration form.

8.6 Place the lower primary standard on the microbalance and record “as found” value and the serial number of the standard on the calibration form.

8.7 Follow instructions for the specific microbalance calibration. See Section 9 of this SOP for Sartorius Ultra-microbalance and Section 10 of this SOP for Cahn microbalance for instructions.

8.8 After calibration is complete, record the calibration event in the Laboratory Balance Notebook and on the calibration form. Record the conventional mass value entered in the microbalance, and record the conventional mass of the primary standards on the calibration form.

8.9 Place the upper primary standard on the microbalance and record “as left” value on the calibration form.

8.10 Place the lower primary standard on the microbalance and record “as left” value on the calibration form.

8.11 If the mass of the primary standard differs from its conventional certified value by more than ±2.1 µg, re-calibrate the microbalance starting with step 8.7.

8.12 Verify the working mass reference standards following Standard Operating Procedure for the Verification of Working Mass Reference Standards.

8.13 Return standards to their proper storage within the lab.

8.14 If the microbalance does not calibrate notify supervisor to resolve the issue.

9.0 Ultra-microbalance External Calibration for Sartorius

9.1 Check for correct primary standard information stored in the menu by using the menu soft keys below the microbalance display. The current upper primary standard is used.

9.2 Press soft key directly below Cal./Adj. on display.

9.3 Using the up and down soft keys highlight “External cal./adj. with user-defined weight”

9.4 Press right arrow soft key.

9.5 Check that the correct primary standard serial number and conventional mass are displayed.

9.6 If the primary standard is correct proceed to perform external calibration.

9.7 If the current primary standard is not stored in the menu add to the menu by pressing back to get to the main display and pressing the soft key directly below the Word Menu.

9.7.1 Using the up and down soft key highlight “Calibration/adjustment Data” and press the right arrow soft key.

9.7.2 Using the up and down soft key highlight “Define external calibration weight” and press the right arrow soft key.

9.7.3 Press the soft key directly below the word wizard.

9.7.4 Press right soft key to select next available number to store external calibration weight. Use soft keys to highlight number and press soft key directly below the word Add and then press ok.

9.7.5 Enter the conventional mass value in grams for the upper primary standard by pressing right soft key. Enter value by highlighting each digit one at a time starting with comma for the period and pressing soft key directly below "Add". When all digits are entered press soft key directly below "ok"

9.7.6 Using soft keys, arrow down to highlight Calibration weight ID. Enter the serial number of the weight using the same soft keys.

9.7.7 Using soft keys, arrow down to highlight Calibration date. Enter the date of calibration from the mass weight certificate.

9.7.8 Press soft key directly below "save"

9.8 To begin external calibration press soft key directly below Cal./Adj.

9.9 Check that the correct primary standard is displayed

9.10 Begin calibration by pressing soft key directly below "Start"; screen will prompt user to place primary standard on balance weigh pan.

9.11 Open the weighing chamber and carefully place the primary standard on the balance's weigh pan and close the weighing chamber.

9.12 The balance automatically performs the calibration. Wait for the process to complete. The balance will return to the standard weighing mode when finished.

9.13 Remove the primary standard and resume verification procedure.

10.0 Microbalance External Calibration for Cahn

10.1 To perform an external calibration, use the menu soft keys located below the microbalance display.

10.2 With microbalance on and reading 0.000 mg, press and hold tare soft key for at least two seconds. Balance will "beep", and C.I. (calibration internal) and CAL will be displayed next to the F1 key.

10.3 Press F2 key until C.E. (calibration external) is displayed where C.I. was previously displayed.

10.4 Press F1 key and the calibration readout will be displayed.

10.5 Enter the conventional mass of the primary 5 g standard using the numeric keys above the display. If a mistake is made, press the CF key in the top left corner to move back to the beginning of this step.

10.6 Press F1 key to store the new conventional mass after it has been entered correctly.

10.7 Open the weighing chamber and carefully place the 5 g standard on the balance pan and close the weighing chamber.

10.8 The balance automatically performs the calibration at this point. Wait until the balance returns to the standard weighing mode, approximately one minute.

10.9 Remove the primary standard and resume calibration procedure at step 8.8

11.0 Calculations

11.1 None.

12.0 References

12.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

12.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10\text{-}2.5}$ in the Atmosphere

12.3 Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

12.4 Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

12.5 *Cahn Technical Notes: Static Control for Balances*; Jerry Weil, Cahn Instruments, Cerritos, California

13.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP – GR-302-0.0

STANDARD OPERATING PROCEDURE FOR PERFORMING VERIFICATION OF THE ARCHIVE COOLER AND SAMPLE RECEIVING REFRIGERATOR TEMPERATURE

May 2021
IML AIR SCIENCE

REVISED BY:

Mary Hannigan 05/17/21
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APPROVED:

[signature] 5/17/2021
Manager Date

[signature] 5/17/2021
Quality Officer Date

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Table of Contents

1.0 Scope and Application

1.1 This procedure is to verify the accuracy of the Archive Cooler and Sample Receiving Refrigerator temperature dataloggers using a certified temperature standard.

2.0 Summary of Method

2.1 Temperatures in the sample archive coolers and sample receiving refrigerator are monitored by temperature probes with dataloggers, which are verified quarterly.

2.2 A NIST traceable standard is used to verify the accuracy of the temperature measurements made by the dataloggers.

3.0 Definitions

3.1 Datalogger – an electronic device that records data over time either with a built in instrument or sensor or via external instruments and sensors.

4.0 Health and Safety Warnings

4.1 Personnel should limit exposure time to the low temperatures within the coolers.

5.0 Cautions

5.1 Damage to the instrument may result if caution is not taken to properly install and maintain the device. Follow the manufacturer's instructions for maintenance of all equipment.

5.2 NIST traceable thermometer contains mercury, if broken secure the area and contact the safety department for cleanup.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement instrumentation.

6.2 Instrumentation skills are necessary for interacting with the data acquisition system.

6.3 Familiarity with electronic and mechanical test equipment is required.

7.0 Equipment

7.1 Campbell Scientific 21X data logger with two CS 107 temperature probes

7.2 Campbell Scientific CR1000 data logger with a CS 107 temperature probe

7.3 NIST traceable thermometer standard, accurate to ±0.1 ˚C

7.4 Laboratory Conditions Notebook

8.0 Procedure

8.1 Verify that the certification for the NIST traceable thermometer standard used has been certified within the last year.

8.2 If a certification is found to be out of date it must be either re-certified or replaced with a certified standard prior to performing the verification.

8.3 Record the date, the technician performing the verification and the serial number of the NIST traceable standard used in the Laboratory Conditions Notebook.

8.4 The datalogger is located in Archive Cooler (L). Place the NIST traceable thermometer in the Archive Cooler to be verified and allow the thermometer to attain equilibrium, typically 30 minutes.

8.5 The Archive Coolers are required to be ≤4.1 ºC for filter storage. If the temperature is out of specification, troubleshoot and resolve following completion of this SOP.

8.6 Measure the actual temperature in the Archive Cooler with the NIST traceable thermometer and record the value in the Laboratory Conditions Notebook.

8.7 Read directly from the datalogger the current temperature reading and the datalogger battery voltage. Record the indicated temperature and battery voltage from the datalogger in the Laboratory Conditions Notebook using the following instructions.

8.7.1 Press* 6 to see display of the measurements. Press “A” to advance to location one for the temperature in Archive Cooler L and Press “A” again to advance to location two for the temperature in Archive Cooler G. Press “A” again to advance to location three for the battery voltage. Press “B” to move backwards through the locations

8.8 Also record the time, year, and serial date from the data logger using the following instruction. Press * 5 to display the current time (24hr format). Press “A” to advance to the year. Press “A” again to advance to the serial day. Press “B” to move backwards through the locations. Press * 0 when done.

8.9 Repeat steps 8.4 through 8.7 for Archive Cooler (G).

8.10 Compare the actual and the indicated values and record difference. The datalogger indicated temperature must not differ from the actual temperature by more than ±2 °C.

8.11 If the verification values exceed the control limits, notify supervisor and corrective action must be initiated. Corrective action may include, but is not limited to: replacing the sensor, datalogger or other hardware or performing maintenance to the cooler.

8.12 Place the NIST traceable thermometer in the Sample Receiving Refrigerator located in the sample receiving area and allow it to attain equilibrium, typically 30 minutes.

8.13 Read indicated temperature from the datalogger keypad display in Lab 1 and record in the Laboratory Conditions Notebook following the steps below.

8.13.1 To access the temperature, press “enter” on the keypad to activate the screen.

8.13.2 The “Campbell Scientific” main screen should be visible, if it is some other screen, press “esc” until the screen displays “Campbell Scientific” with CR1000 Datalogger and the date/time below.

8.13.3 Press “enter” again to access the menus.

8.13.4 Use the arrow keys to place the cursor by “data” then press “enter”.

8.13.5 Verify the cursor is by “real time tables” and press “enter”.

8.13.6 Use the arrow keys to place the cursor by “public” and press “enter”.

8.13.7 Use the arrow keys to scroll down to the Office Archive Temperature, which is the reading from the temperature probe in the Sample Receiving Refrigerator.

8.13.8 To back out of the menus press “esc” until the main screen is visible.

8.14 Measure the actual temperature in the Sample Receiving Refrigerator with the NIST traceable thermometer and record the value in the Laboratory Conditions Notebook.

8.15 After the verifications are completed, return the NIST traceable thermometer to the proper storage location.

9.0 Calculations

9.1 Temperature difference = datalogger temperature – NIST traceable thermometer

10.0 References

10.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

10.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10-2.5}$ in the Atmosphere

10.3 Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.4 Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

11.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP - GR-303-0.0

STANDARD OPERATING PROCEDURE FOR PERFORMING ANNUAL REPEATABILITY TEST

May 2021
IML AIR SCIENCE

REVISED BY:

Mary Heninger — 05/17/21
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APPROVED:

Manager — 5/17/2021
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Quality Officer — 5/17/2021
Date

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Table of Contents

1.0 Scope and Application

1.1 This procedure is to verify the repeatability of the microbalance annually.

2.0 Summary of Method

2.1 The repeatability test is to measure the ability of a microbalance to display the same result in repetitive weighings of the same mass under the same measurement conditions. The term “precision” is sometimes used as a synonym.

3.0 Definitions

3.1 Lab - A room, containing the microbalance, designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

3.2 Microbalance - A type of analytical balance that can weigh to the nearest 0.001mg (that is, 1µg or one-millionth gram).

3.3 Working Standard – ASTM Class 0, Class 1, or Class 2 mass standard, traceable to NIST, that is used for routine quality control checks.

3.4 Primary Standard – ASTM Class 0, Class 1, or Class 2 mass standard, traceable to NIST, that has “authority” over other standards (working standards) in the laboratory.

3.5 Test weight – a working standard, a primary standard, or filter with an identification number.

4.0 Health and Safety Concerns

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

5.0 Cautions

5.1 Damage to the instrument may result if caution is not taken to maintain the device. Follow the manufacturer's instructions for maintenance of all equipment.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement.

7.0 Equipment

7.1 Laboratory coat

7.2 Clean, environmentally controlled conditioning room (lab)

7.3 Microbalance; minimum sensitivity to ±1µg, minimum repeatability of ±1 µg

7.4 Balance table, with anti-static mat

7.5 Non-serrated, non-metallic forceps

7.6 Grounded floor mat

7.7 Annual Repeatability Test Form

7.8 Test mass Working standards which bracket the expected filter mass. An upper standard (typically 500 mg) and a lower standard (typically 200 mg)

7.9 Laboratory Balance

8.0 Procedure

8.1 Perform the daily laboratory cleaning and maintenance (Standard Operating Procedure for Cleaning and Maintenance of Micro-gravimetric Laboratory).

8.2 Make sure the microbalance is reading zero, if not press the tare button. After the test is started do not tare the microbalance.

8.3 On the form, record the microbalance make and serial number, the date, initials of the technician(s) performing the annual repeatability test and the serial number of the working standard or the filter to be used to perform the test.

8.4 While the weigh pan is empty allow the microbalance to stabilize to zero with the “mg” appearing at the end of the display. Record the reading on the form in the left column in the space provided next to O_1 w/o Test Weight.

8.5 Open the chamber door and place the test weight carefully on the weigh pan and close the chamber door.

8.6 Allow the reading to stabilize with the “mg” appearing at the end of the display.

8.7 Record the mass on the form for the test weight in the space provided in the column on the right for O_2 with Test Weight.

8.8 Remove the test weight from the weigh chamber.

8.9 Repeat procedure with the weigh pan for the microbalance empty. Allow the microbalance to stabilize. The time should be consistent between readings.

8.10 On the form record O_3 w/o Test Weight in the place provided.

8.11 Continue the test by placing the same test weight back in the weigh chamber and close door. Allow the reading to stabilize with the “mg” appearing at the end of the display. Record the mass on the form for the test weight in the space provided O_4 with Test Weight.

8.12 Repeat procedure going back and forth until O_{20} with Test Weight is complete.

8.13 Calculate the difference between “with Test Weight” and “w/o Test Weight” for each set of observations using the equation in Section 9.2 and record in the last column of each row. Calculate the standard deviation of the differences in µg using the method described in Section 9.1. Record the standard deviation in the proper location at the bottom of the form. The standard deviation must be within ±1 µg.

8.14 Repeat entire test if the standard deviation is outside of the specification.

8.15 If the test fails again, notify supervisor and take corrective action, including verifying that the balance is level, and performing anti-static treatment and cleaning. Check microbalance for other possible problems and consider having the microbalance repaired by a service (do not try to repair microbalance) or replacing microbalance.

8.16 After performing the test, document in the Laboratory Balance Notebook.

9.0 Calculations

9.1 Standard deviation is calculated in MS Excel (Excel 2010 or greater) by using the formula STDEV.S, and then converted to µg using the equation in Section 9.3.

9.2 Difference = O_x w/ Test Weight – O_x w/o Test Weight

9.3 µg = mg*1000

10.0 References

10.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as PM2.5 in the Atmosphere

10.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as PM10–2.5 in the Atmosphere

10.3 Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.4 Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001;

January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

11.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP – GR-108-0.0

STANDARD OPERATING PROCEDURE FOR RECEIPT OF NEW MICRO-GRAVIMETRIC FILTERS

May 2021
IML AIR SCIENCE

REVISED BY:

Mary Hannigan — 05/17/21
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APPROVED:

[signature] — 5/17/2021
Manager — Date

[signature] — 5/17/2021
Quality Officer — Date

Reviewed

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Table of Contents

1.0 Scope and Application

1.1 This procedure is to receive and log shipments of new polytetrafluoroethylene (PTFE Teflon) filters for analyses using Reference Method: 40 CFR 50 Appendix L and Appendix O.

1.2 The elements of this SOP are applicable to filters for low volume air samplers.

2.0 Summary of Method

2.1 Shipments of new filters that will be used to collect particulate matter samples are logged in upon arrival. Client (Network), date received, lot number, pre-assigned filter numbers and number of boxes.

3.0 Definitions

3.1 Data Management System (DMS) – Software systems to create, store and retrieve data.

3.2 Lab - A room, containing the microbalance, designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

4.0 Health and Safety Concerns

4.1 Use proper lifting techniques.

5.0 Cautions

5.1 In all steps involving filter handling avoid filter damage.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement instrumentation.

7.0 Equipment

7.1 Filter Lot Login Notebook

7.2 46.2 mm diameter filters, Teflon membrane (PTFE)

7.3 Computer with access to the DMS

7.4 Lint-free cloth

7.5 Anti-static cleaning solution

8.0 Procedure

8.1 New PTFE filters are received, from either a vendor or a client network.

8.2 If filters are from a lot that has not previously been evaluated, perform a filter lot stability evaluation to determine the equilibration time for the lot (Standard Operating Procedure for Performing a Filter Stability Evaluation (Lot Blank)). A lot stability test will be performed once per lot prior to use. Upon client request, additional lot stability tests may be performed. For filters received from the manufacturer a lot stability test will be performed on each shipment of filters.

8.3 After filters are received from a client network, notify the client of date received and filter quantity.

8.4 Record the date received, supplier, network, lot number, number of boxes and pre-assigned filter numbers in the DMS and the Filter Lot Login Notebook.

8.5 Remove cellophane wrap and wipe each box with cloth sprayed with anti-static solution.

8.6 Write on each clear plastic box the network name and number the boxes in sequential order by filter numbers (e.g. for 23 boxes - 1/23, 2/23…etc.). Write on box “break” after each sequence break of filter numbers.

8.7 Move the filters into the lab for storage, in their original container, grouped by network, lot number and filter numbers.

9.0 References

9.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as PM2.5 in the Atmosphere

9.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as PM10–2.5 in the Atmosphere

9.3 Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

9.4 Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP – GR-200-0.0

STANDARD OPERATING PROCEDURE FOR PERFORMING A FILTER STABILITY EVALUATION (LOT BLANK)

May 2021
IML AIR SCIENCE

REVISED BY:

Mary Hannigan 05/17/21
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APPROVED:

Manager 5/17/2021
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Quality Officer 5/17/2021
Date

Reviewed

Initials				
Date				

Table of Contents

1.0 Scope and Application

1.1 The elements of this SOP are applicable to the equilibration time of filters, both pre-exposure and post-exposure for Reference Method 40 CFR 50 Appendix L and Appendix O sampling methods.

1.2 The test is to determine the minimum length of time to equilibrate filters in the laboratory conditioning room before pre-weighing and post-weighing.

1.3 If filters are from a lot that has not previously been evaluated, perform a filter lot stability evaluation to determine the equilibration time for the lot using this procedure. A lot stability test will be performed once per lot prior to use. Upon client request, additional lot stability tests may be performed. For filters received from the manufacturer, a lot stability test will be performed on each shipment of filters.

2.0 Summary of Method

2.1 Due to the potential volatilization of material from the filter into the atmosphere, or absorption of material into the filter from the conditioning environment, tests must be performed to determine the amount of equilibration (conditioning) time that is needed for the filter mass to stabilize (change of less than 15 µg in 24 hours).

2.2 The test involves testing nine random filters (taken from three random boxes), acquiring an initial mass, and weighing the filters every two hours for the first six hours, and then 24-hours for five days.

3.0 Definitions

3.1 Lab - A room, containing the microbalance, designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

3.2 Lot Blank - A filter, or group of filters, used to determine filter weight stability over a period of time due to the volatilization of material from the filter or the absorption of gaseous material into the filter from the atmosphere.

3.3 Microbalance - A type of analytical balance that can weigh to the nearest 0.001mg (that is, 1 µg or one-millionth gram).

3.4 Polonium-210 (210Po) anti-static strip - A device containing a small amount of 210Po that emits α particles (He^{2+}) that neutralize the static charge on filters, making them easier to handle and their weights more accurate.

3.5 Data Management System (DMS) – Software systems to record, store and report data.

3.6 Working Standard – ASTM Class 0, Class 1, or Class 2 mass standard, traceable to NIST, that is used for routine quality control checks.

4.0 Health and Safety Warnings

4.1 General precautions for working with electro-mechanical equipment should be taken.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter, and avoid filter damage. Filters are prepared and remain in the clean, environmentally controlled room. Wear a laboratory coat when handling filters. Keep filter Petri dishes protected. Conditioning room must be kept clean.

5.2 Errors in the gravimetric analysis of samples can result from the buildup of electrostatic charges on filters during their manufacture or during sampling. This static buildup will interfere with microbalance weighing, but it can be reduced by using static charge reduction techniques.

5.3 Handle filters with stainless steel non-serrated forceps by the support ring only. Summary of methods and work detailed in SOP.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement instrumentation.

6.2 Familiarity with electronic laboratory equipment is required.

7.0 Equipment

7.1 Clean, environmentally controlled conditioning room (Lab)

7.2 Microbalance, minimum sensitivity to ±1µg, minimum repeatability of ±1µg

7.3 46.2 mm diameter filters, Teflon membrane (PTFE)

7.4 Balance table, with anti-static mat

7.5 210Po anti-static strips

7.6 Stainless steel, non-serrated forceps

7.7 Non-serrated, non-metallic forceps

7.8 Grounded floor mat

7.9 Working standards which bracket the expected filter mass. An upper standard (typically 500 mg) and a lower standard (typically 200 mg)

7.10 Individually labeled Petri dishes

7.11 Filter Lot Stability Evaluation Form

7.12 Laboratory coat

7.13 Computer with access to DMS

8.0 Procedure

8.1 Use PTFE filters approved for collecting Particulate Matter by Reference Method 40 CFR 50 Appendix L and Appendix O.

8.2 Handle filters with stainless steel, non-serrated forceps by the support ring only.

8.3 Perform the daily laboratory cleaning and maintenance (Standard Operating Procedure for Cleaning and Maintenance of Gravimetric Laboratory).

8.4 Randomly select nine filters, three filters from three separate boxes from a single lot as “lot blanks”.

8.5 Inspect each filter following procedure (Standard Operating Procedure For Preparing Filters for Pre-exposure Analysis).

8.6 Place lot blank filters in individual Petri dishes labeled LotB1, LotB2, …thru LotB9.

8.7 On the filter stability evaluation form “Filter Lot Stability Evaluation” write the filter lot number, network, and date filters were received. Record each filter number as well as the date, time and your initials on the form.

8.8 The first measurement for each filter is taken without equilibrating in the lab. Analyze each lot blank filter and record initial weight before equilibrating.

8.9 Check that the microbalance is reading zero, if needed, press the tare button.

8.10 Analyze the upper standard. Use only non-serrated, non-metallic forceps to handle the working standards. Carefully place the standard on the balance pan and close chamber.

8.11 Allow the reading to stabilize until the “mg” appears at the end of the reading; record the mass on the form. If the measured value of the working standard disagrees with the certified value by more than ±3 µg, remove the standard, zero the balance, inspect, clean and reweigh the working standard. If the second measurement still disagrees by more than ±3 µg, contact supervisor. If necessary

verify the working standards (Standard Operating Procedure for the Verification of Working Mass Reference Standards).

8.12 Repeat steps 8.9 through 8.11 with the lower standard.

8.13 Check the balance is reading zero and record on the form, if needed, press tare to zero the balance.

8.14 Neutralize electrostatic charge from filter LotB1 by placing filter on a 210Po anti-static strip for approximately 60 seconds.

8.15 Carefully place the filter in the weigh chamber and close. Allow the reading to stabilize until the “mg” appears at the end of the reading.

8.16 Record the initial mass of filter LotB1 on the form.

8.17 Repeat steps 8.13 through 8.16 for the other eight lot blanks.

8.18 Repeat steps 8.9 through 8.12 for working standards.

8.19 Repeat step 8.13 for the zero.

8.20 Place the lot blanks in a covered, well ventilated tray, in open Petri dishes in the lab.

8.21 Repeat steps 8.9 through 8.20approximately every two hours during laboratory operation (three times).

8.22 Repeat steps 8.9 through 8.20every 24 hours. This test is continued for a minimum of 5 days to ensure that the filters are stable over longer equilibration times. This often crosses the weekend creating a two day gap of measurement, but totaling 6 days of exposure in the lab. The change in mass should be less than 15 µg in 24 hours using the calculation in section 9.1.

8.23 Determine for each filter the difference in the mass between previous day and current day and record on the form. Calculate the difference between current day and the previous day as the mass is determined and record on form using the calculation in section 9.2. (e.g. difference between day 3 and day 2)

8.24 Calculate the average length of time in days for the nine lot blanks to stabilize using the calculation in section 9.3 and 9.4. Document on the Filter Stability Evaluation Form. This average is the time in days that it takes a new box of filters to obtain a constant mass in the lab from the time the box is opened.

8.25 If the average length of time in days exceeds one day, and this is the first attempt, repeat test starting with step 8.3. If this is the second attempt, notify supervisor for corrective action.

8.26 If the lot blank filters pass the test, the filters associated with the lot are ready for use. Enter the date the lot blank test was performed in the DMS.

9.0 Calculations

9.1 mass difference = final mass – initial mass

9.2 mass difference day to day = current day mass – previous day mass

9.3 #days = first day when mass difference day to day is < 15 µg

9.4 Average length of time in days =

$$\frac{\#daysfilter1+\#daysfilter2+\#daysfilter3+\#daysfilter4+\#daysfilter5+\#daysfilter6+\#daysfilter7+\#daysfilter8+\#daysfilter9}{9}$$

10.0 References

10.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as PM2.5 in the Atmosphere

10.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as PM10–2.5 in the Atmosphere

10.3 Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.4 Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

11.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP – GR-109-0.0

STANDARD OPERATING PROCEDURE FOR PREPARING FILTERS FOR PRE-EXPOSURE ANALYSIS

May 2021
IML AIR SCIENCE

REVISED BY:

Mary Hunziger — 05/17/21
Date

APPROVED:

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Quality Officer — 5/17/2021
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Table of Contents

1.0 Scope and Application

1.1 This procedure is used to inspect and prepare clean polytetrafluoroethylene (PTFE Teflon) filters for gravimetric analysis to be used for sampling.

2.0 Summary of Method

2.1 Teflon filters are used to collect particulate matter with low volume samples for Reference Method 40 CRF 50 Appendix L and O. A Tare Batch is configured by the gravimetric lab supervisor and then added to the DMS. Filters are inspected for integrity and, if accepted, placed in Petri dishes labeled with their unique identification number. Filters are assigned to client network locations and equilibrated prior to gravimetric analysis.

3.0 Definitions

3.1 Cassette - A device supplied with particulate samplers to allow a filter to be held in place in the sampler and manipulated before and after sampling without touching the filter, and to minimize damage to the filter and/or sample during such activities.

3.2 Lab - A room, containing the microbalance, designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

3.3 Laboratory blank filter - New filters that are used to determine laboratory contamination. The laboratory blank filters shall be weighed along with the pre-sampling (tare) weighing of each set of filters for each client network. These laboratory blank filters should remain in the laboratory in protective containers during the field sampling and should be reweighed as a quality control check at gross weighing.

3.4 Data Management System (DMS) – Software systems to record, store and report data.

4.0 Health and Safety Warnings

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter, and avoid filter damage. Filters are prepared and remain in the clean, environmentally controlled room. Wear a laboratory coat when

handling filters. Keep filter Petri dishes protected and in a horizontal position during transport.

5.2 Handle filters with stainless steel non-serrated forceps by the support ring only.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement instrumentation.

6.2 Familiarity with electronic laboratory equipment is required.

7.0 Equipment

7.1 Clean, environmentally controlled conditioning room (Lab)

7.2 Laboratory coat

7.3 Light table

7.4 Petri dishes

7.5 Labels with barcode and filter numbers for Petri dishes

7.6 Stainless steel, non-serrated forceps

7.7 46.2 mm diameter filters, Teflon membrane (PTFE)

7.8 Anti-static spray

7.9 Lint-free wipes

7.10 Lint-free cloth

7.11 Computer with access to DMS

7.12 Tare batch shipment worksheet and client shipment worksheet

7.13 Anti-static bags

7.14 Lighted magnifying glass

8.0 Procedure

8.1 Supervisor or Upper Management completes sections 8.1.1 through 8.1.14 for each shipment of filters.

8.1.1 The shipping schedule is a forward looking document that lists filter quantities for the upcoming shipments. This information is provided to the gravimetric technician prior to preparing the shipment of filters for the next week.

8.1.2 Each client has a shipping schedule in an excel workbook on the network with the number of samplers and sampling frequency for each sampler. The shipping schedule calculates the number of filters needed for a two week period of sampling, including field blanks and trip blanks. – The DMS also contains a shipping schedule, however this is only for reference and the excel workbook is the official copy.

8.1.3 In the DMS (Air Data Processor), create a Tare Batch by selecting appropriate lab. Press Create Batch button for the next shipping date, typically a Monday. The batch is reviewed, client by client to make sure the correct clients are checked or unchecked. Press “OK” after the correct clients are checked.

8.1.4 Each batch contains a list of the clients to receive a filter shipment. The structure is a parent/child design. The clients are “parents” which have locations (children), and the locations may have samplers (children). Clients that use Inter-Mountain Laboratories filters appear as children of the parent client Inter-Mountain Laboratories.

8.1.5 Each client in the batch is reviewed and edited to make sure the filter quantities match the shipping schedule. If the individual samplers are listed check each for correct filter quantity for the time period given on the screen and edit as needed. Lab blank quantities are updated at the client level to ten percent or greater.

8.1.6 For clients using Inter-Mountain Laboratories filters (or other shared filter supplies), lab blanks are assigned by selecting the individual clients below the Inter-Mountain Laboratories level in the list (e.g. Black Thunder).

8.1.7 Field blank quantities are edited based on client needs then the quantities for each client are saved. These can be added at the Location level or the Sampler level. Entries are saved by pressing update for locations or save for specific samplers.

8.1.8 Trip blanks quantities are edited based on client needs. The quantities for each client are added at the Location level and press update to save.

8.1.9 Add to each client the Number Lab Extras filters needed at the client level and press update to save. Each client must have extras in case a filter is damaged during pre-weighing or loading into cassettes.

8.1.10 For clients using Inter-Mountain Laboratories filters, add the extra filters at the Inter-Mountain Laboratories level in the list, then press Update to save. (IML client filters are assigned alphabetically in sequence with shared filters and extras as needed)

8.1.11 The shipping schedule for each client is reviewed again and compared to the tare batch. Make sure the total filter shipment number matches for each client.

8.1.12 After each client is edited, press Print Batch List at top of screen and review list again to assure everything is correct.

8.1.13 Typically, the Tuesday before the next ship day Assign Filters. Have LabX_mmddyy highlighted and Press Assign Filters. Wait for the first status light to turn green indicating the filter numbers are assigned for each client. Press Print Batch List and print a hardcopy with the filter numbers. Give hardcopy to technician responsible for preparing the shipment.

8.1.14 Generally, Lab 2 filters are not assigned until Thursday before the shipment date. Waiting allows for clients using Inter-Mountain Laboratories filters to request changes.

8.2 Technicians complete for each tare shipment of filters.

8.2.1 Use only PTFE filters approved for collecting Particulate Matter by Reference Method 40 CFR 50 Appendix L and Appendix O.

8.2.2 Handle filters with stainless steel, non-serrated forceps by the support ring only.

8.2.3 Confirm that the daily laboratory/equipment maintenance is complete (Standard Operating Procedure For Cleaning And Maintenance of Gravimetric Laboratory).

8.2.4 Using the laboratory DMS and Tare batch shipment worksheet, determine the quantity of filters for each client. Ensure that a proper quantity of Petri labels necessary to process the shipment to each network location has been created; accounting for any possible discarded damaged filters. Print more if needed. Petri labels contain the filter number in digits and barcode. Labels are color coded with a marker for each shipment to identify the Petri dishes for each ship date.

8.2.5 The next filter numbers going across the worksheet are identified as lab blanks, currently labeled with three million series numbers, which are cross-referenced to the actual number on each filter. Extra filter numbers appear last, on the worksheet and should be treated the same as the other filters, except are not color coded unless actually used.

8.2.6 Clean the light table with lint free cloth sprayed with anti-static solution or alcohol wipes.

8.2.7 View filters over the light table one at a time and inspect for defects (pinholes, discoloration, non-uniformity, etc.). Use the magnifying lamp to inspect filters.

8.2.8 After inspection is complete, place the filter into its Petri dish. Apply the corresponding label and cover.

8.2.9 Place filters in sequential order, in stacks, in the specified clean trays.

8.2.10 Record damaged or missing filter numbers on tare batch shipment worksheet. Discard filters that do not pass inspection.

8.2.11 In the DMS select the appropriate client and click Filter Inspection. If there are damaged filters, scan or input filter number along with the reason rejected and press “Mark Damaged”. After damaged filters are entered into DMS before saving, verify that the client has the correct filters. Check the first and last filter numbers, lab blanks, and extra filters numbers that are listed on the worksheet match the DMS making adjustments for any damaged filters. If numbers match, then press save. Continue this process for each client. The worksheet may be printed again to verify adjustments from the removal of damaged filters. For clients using Inter-Mountain Laboratories filters, the filters for all clients are inspected and the Filter Inspection in the DMS is completed at the Inter-Mountain Laboratories level in the list. Additional filters are pre-inspected at this time (typically 100) to allow for client needs.

8.2.12 Print one hard copy of the cassette tracker by selecting the desired Tare Batch and clicking Print Cass Tracker at the top of the window in the DMS. The cassette trackers for individual networks can also be printed by selecting the client and clicking Print Cass Tracker near the center of the window.

8.2.13 Retrieve enough pre-numbered cassettes for all filters per client. A good practice is to place cassettes in numerical order and assign a cassette to each filter by writing the cassette number on the cassette

tracker worksheet. Enter cassette numbers into DMS for each filter clicking Save Cassette IDs when finished.

8.2.14 Store the cassettes in a long narrow tray in the same order listed on the cassette tracker form and retain until after weighing.

8.2.15 Print bag labels for the anti-static sealable bags from the DMS for each client or at the same time using the button at the top of the screen. Use white 2" X 4" labels for lab 1 and yellow labels for lab 2. Labels contain network name, filter ID in plain text and bar code, cassette ID, tare expiration date, and spaces for the field operator to record pertinent sample information: site, sampler ID, sample date, sample run time, sample volume, ambient average temperature and pressure, sampler status, coefficient of variation (CV), date and time filter sample removed and comments.

8.2.16 Securely place labels for each filter on the anti-static bags. Check cassette numbers match the cassette tracker worksheet. Retain the cassette tracker in laboratory until filters are loaded into cassettes and anti-static bags.

8.2.17 Filters are typically set out in trays on Fridays after weekly laboratory cleaning is complete. Carefully place filters in sequential order in a covered, well ventilated tray(s) with the Petri lids underneath the Petri dishes. Set out about 60 extra shared filters from the Inter-Mountain Laboratories filters supply. Note the first extra filter number and the last extra filter number for the shared filters. These numbers will be needed later in steps for equilibration

8.2.18 After all the filters are set out, begin tare equilibration by selecting Equilibration in the left hand panel in the DMS.

8.2.19 Select Tare Equilibration at the top of the window

8.2.20 Select the lab number. Be aware that clients using shared filters, in this case, Inter-Mountain Laboratories filters, function differently. The following instructions have no shared filters in Lab 1 and shared filters in Lab 2.

8.2.21 If Lab 1 is selected verify the filter and lab blank totals are correct then, click Save in the lower right corner

8.2.22 If Lab 2 is selected verify the filter and lab blank totals are correct exposed. Then click the Magnifying Glass to add the extra filters that have been exposed, but not assigned by entering them in the boxes for the Range of Filters. First filter number is the IML extra filter.

Verify the filter and lab blank totals again are correct after adding the extras.
Click Save in the lower righthand corner.

8.2.23 Click on Tare Batch and the current date to check that both Lab 1 and Lab 2 have all three status lights green. If all three of the status lights are not green investigate and fix.

8.2.24 Allow filters to equilibrate the amount of time required for that lot of filters (minimum 24 hours), as determined by (Standard Operating Procedure for Performing a Filter Stability Evaluation (Lot Blank)). Typically, conditioning tare filters begins on Friday afternoon and continues until Monday morning. During the equilibration period, the laboratory mean temperature must remain at 20.0-23.0˚C, with a variability of not more than ±2.1˚C over 24 hours and the mean relative humidity must remain at 30.0-40.0% with a variability of not more than ±5.0% over 24 hours. If these conditions have not been met, the filters must be properly equilibrated again.

9.0 References

9.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as PM2.5 in the Atmosphere

9.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as PM10–2.5 in the Atmosphere

9.3 Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards

9.4 Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards

10.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP – GR-600-0.0

STANDARD OPERATING PROCEDURE FOR CLEANING AND MAINTENANCE OF GRAVIMETRIC LABORATORY

May 2021
IML AIR SCIENCE

REVISED BY:

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APPROVED:

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Table of Contents

1.0 Scope and Application

1.1 The objective of this procedure is to perform daily, weekly, and quarterly cleaning and maintenance of the gravimetric laboratory and other areas that may affect the gravimetric laboratory. This procedure applies to high volume and low volume methods.

2.0 Summary of Method

2.1 Laboratory maintenance is performed to ensure that the laboratory maintains appropriate environmental conditioning, reduce potential contamination and maintain equipment to prevent failure during use. Daily, weekly and quarterly laboratory maintenance is performed. Additional cleaning in other areas is crucial to maintenance of the gravimetric laboratory.

3.0 Definitions

3.1 Lab - A room containing the balance, designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

3.2 Semi-wet cleaning methods - The use of anti-static solutions and damp, lint-free cloths

3.3 HEPA- high-efficiency particulate air

3.4 Anteroom – A room with limited access and no air flow located adjacent to the lab, for the unloading of filters from cassettes or glassines and preparing for post exposure analysis.

4.0 Health and Safety Warnings

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

4.2 Step stool is used for reaching out of reach areas.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter, and avoid filter damage. Keep filters protected and in a horizontal position during transport.

5.2 Move with caution around sensitive equipment to avoid bumping or unplugging.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be familiar with best laboratory practices for cleaning.

6.2 Familiarity with electronic laboratory equipment is required.

7.0 Equipment

7.1 Anti-static cleaning solution

7.2 Lint-free cloth

7.3 Mop with dry and moist lint-free cloth

7.4 Laboratory grade alcohol wipes

7.5 Small, anti-static, soft brush

7.6 De-ionized water

7.7 Mesh bags

7.8 Tacky mats

7.9 Replacement HEPA filter and other equipment

7.10 Vacuum

7.11 Lab coat

7.12 Laboratory Notebook

8.0 Daily Procedure

8.1 Prior to sample analysis perform daily cleaning. Only if needed, carefully brush out any loose debris from the weighing chamber of the balance with anti-static soft brush.

8.2 Each analyst should visually inspect the weighing area prior to beginning any use of the balance to determine if additional cleaning is needed. Cleaning will be conducted if inspection determines a need, even if the daily cleaning has been completed earlier in the day.

8.3 Wipe off all anti-static mats on the balance table with lint-free cloth sprayed with anti-static cleaning solution.

8.4 Clean non-serrated, non-metallic forceps with alcohol wipes.

8.5 Allow forceps to completely air dry before handling mass standards.

8.6 Make sure hands are clean and dry before handling filters.

8.7 Check humidifier and dehumidifier. Fill humidifier if needed and empty dehumidifier if needed.

8.8 Check tacky mats and peel/replace as needed.

8.9 Gather all incoming cassettes during the day in mesh bags, organized by network. Add a small amount of lab grade soap to tub in utility sink and fill with water. Place mesh bags in solution and agitate well. Drain soap solution and rinse at least twice with tap water and once with de-ionized water. Hang mesh bags in clean space to air dry. Ensure each client has a supply of clean cassettes ready for usage.

9.0 Weekly Procedure

9.1 Cap any low volume filters and carefully protect any high volume filters currently exposed, prior to cleaning. Best practice is to wait until after cleaning is complete to expose filters.

9.2 Weekly cleaning is performed after all weighing for the day is completed and filters are protected. Cleaning is completed on Friday, unless holiday or other circumstances require it to be done earlier in the week.

9.3 Wipe down work surfaces, shelves, and anti-static mats in the lab and the anteroom with lint-free cloth sprayed with anti-static cleaning solution.

9.4 Wipe down equilibration trays and racks with lint-free cloth containing anti-static solution.

9.5 If needed, dust mop floor before using mop with moist, lint-free cloth to clean floor surface in lab and anteroom. Be cautious about bringing too much moisture into the lab, causing relative humidity to increase above acceptance range.

9.6 Wipe down surfaces and clean floor in the receiving area.

9.7 Vacuum carpet and other flooring outside the anteroom weekly and as needed.

9.8 Peel top layer off tacky mats, replace when tacky mats are consumed.

9.9 Check humidifier reservoir for water level and fill as needed with deionized water.

9.10 Check dehumidifier water reservoir and empty.

9.11 Record activities in the Laboratory Notebook.

10.0 Quarterly Procedure

10.1 Move any exposed filters to anteroom prior to cleaning.

10.2 Carefully wipe down walls, surfaces, lighting, ceilings, ceiling fan, shelving, desks (including inside), and all equilibration trays and racks with a lint-free cloth sprayed with anti-static cleaning solution in the lab, anteroom, and receiving area.

10.3 Wipe off all anti-static mats with lint-free cloth.

10.4 Clean all floor surfaces with a damp mop or cloth. Vacuum carpet in receiving area and beyond.

10.5 Check the high efficiency particulate filter in the laboratory pressurization system. Replace the filter quarterly or when any discoloration or damage is apparent.

10.6 Clean dehumidifier and clean filter on back of dehumidifier.

10.7 Clean air conditioner and filter.

10.8 Clean humidifier and refill.

10.9 Inspect lab to ensure grounding wires are all connected.

10.10 Ensure the humidifier water tank is installed properly and the humidifier is on. Verify that all other laboratory control equipment is on and plugged in.

10.11 Archive refrigerators, clean shelves with lint-free cloth and sweep floors,

10.12 Apply liberally sweeping compound to back hallway floor and continue to archive refrigerators and then sweep.

10.13 Record all activities in the Laboratory Notebook.

11.0 References

11.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

11.2 *40 CFR 50, Appendix O;* Reference Method for the Determination of Coarse Particulate Matter as $PM_{10\text{-}2.5}$ in the Atmosphere

11.3 *40 CFR 50, Appendix J;* Reference Method for the Determination of Particulate Matter as PM_{10} in the Atmosphere

11.4 *40 CFR 50, Appendix B;* Reference Method for the Determination of Suspended Particulate in the Atmosphere (High-Volume Method)

11.5 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program;* EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

11.6 *Quality Assurance Guidance Document 2*.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; Environmental Protection Agency, Office of Air Quality Planning and Standards.

11.7 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Specific Methods;* EPA/600/4-77-027a; Section 2.11; January 1990; U.S. Environmental Protection Agency

12.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system.

SOP - GR-110-0.0

STANDARD OPERATING PROCEDURE FOR PERFORMING THE PRE-EXPOSURE ANALYSIS

May 2021
IML AIR SCIENCE

REVISED BY:

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Date				

Table of Contents

1.0 Scope and Application

1.1 This procedure is used to determine the mass of an equilibrated filter prior to exposure.

2.0 Summary of Method Summary of Method

2.1 Teflon membrane filters are conditioned in the lab and then analyzed gravimetrically to at least the nearest 1 µg. This tare mass is subtracted from the mass of the filter following exposure to determine the net mass of collected particles.

3.0 Definitions

3.1 Lab - A confined/defined space containing the microbalance designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

3.2 Microbalance - A type of analytical balance that can weigh to at least the nearest 0.001 mg (that is,1µg or one-millionth gram).

3.3 Polonium-210 (^{210}Po) anti-static strip - A device containing a small amount of ^{210}Po that emits α particles (He2+) that neutralize the static charge on filters, making them easier to handle and their weights more accurate.

3.4 Working Standard – ASTM Class 0, Class 1, or Class 2 mass standard, traceable to NIST, that is used for routine quality control checks.

3.5 Equilibration – Allowing the filters to come to a stable temperature and humidity in the lab, prior to weighing.

3.6 Weigh Session - A grouping of filters that are conditioned and analyzed as a single weighing event and for which laboratory 24-hour temperature and humidity conditions are documented and associated with the grouping of filters. The weigh session includes the required QC (filter blanks, standards, etc.).

3.7 Data Management System (DMS) – Software systems to record, store and report data.

3.8 Laboratory blank filter - New filters, that are used to determine laboratory contamination. The laboratory blank filters shall be weighed along with the pre-sampling (tare) weighing of each set of filters for each client network. These laboratory blank filters should remain in the laboratory in protective containers during the field sampling and should be reweighed as a quality control check at gross weighing.

4.0 Health and Safety Concerns

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter, and avoid filter damage. Laboratory coats will help minimize the potential for laboratory contamination and must be taken off before leaving the lab to minimize contamination from the external environment. Keep filters and Petri dishes protected and in a horizontal position during transport.

5.2 Errors in the gravimetric analysis of samples can result from the buildup of electrostatic charges on filters during their manufacture or during sampling. This static buildup will interfere with microbalance weighing, but it can be reduced by using static charge reduction techniques.

5.3 Handle filters with stainless steel non-serrated forceps by the support ring only.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement instrumentation and general analytical laboratory protocol.

6.2 General computer knowledge for interacting with the data management system.

6.3 Familiarity with electronic laboratory equipment is required.

7.0 Equipment

7.1 Clean, environmentally controlled room, conditioning room (Lab)

7.2 Computer with access to DMS

7.3 Laboratory coat

7.4 Analytical microbalance, minimum sensitivity to ±1µg, minimum repeatable to ±1µg

7.5 Balance table, with anti-static mat

7.6 ^{210}Po anti-static strips

7.7 Stainless steel, non-serrated forceps

7.8 Non-serrated, non-metallic forceps

7.9 Grounded floor mat

7.10 NIST traceable working mass reference standards upper and lower standards (for MTL filters, typically 500 mg and 200 mg)

7.11 46.2 mm diameter filters, Teflon membrane (PTFE)

8.0 Procedure

8.1 Verify in the DMS that the filters have equilibrated for the required amount of time from the filter lot stability test (typically 24 hours) and that room temperature and relative humidity have remained within specifications (mean temperature between 20˚C and 23˚C with less than ±2˚C variability over 24 hours and mean relative humidity between 30% and 40% with less than ±5% variability over 24 hours) during the equilibration period. Temperature and relative humidity for 5 minute and 24 hour averages are displayed on the computer screen from the DMS, with a graphical presentation also available. The display is color coded with green for good, yellow for warning, and red for stop. In addition, the DMS will prevent weighing if conditions are outside control limits. The conditions must be back in compliance to re-start equilibration.

8.2 Complete the daily laboratory cleaning, equipment maintenance and preparation. (Standard Operating Procedure For Cleaning and Maintenance of Gravimetric Laboratory).

8.3 Open the Balance Control portion of the DMS and log-in. The average of the previous 24 hours' one second readings for temperature and relative humidity will be displayed. Observe the readings and confirm compliance.

8.4 Locate the equilibration tray containing the filters to be analyzed and place the tray adjacent to the microbalance.

8.5 Retrieve the working standards and place them in a convenient location near the microbalance.

8.6 Check the balance to ensure its reading zero, if not press tare to zero the balance.

8.7 Analyze the upper working mass reference standard. Use only non-serrated, non-metallic forceps to handle the mass reference working standards. Place the standard on the balance pan and close chamber. Allow the reading to stabilize until the "mg" appears at the end of the reading, press "get weight" on the interface screen and press "next" to store weight. If the measured value of the working standard disagrees with the certified value by more the ±3 µg, remove the standard, zero the balance, inspect, clean and reweigh the working standard.

If the two measurements still disagree by more than 3 µg contact supervisor. If necessary, verify the working standards. (Standard Operating Procedure For the Verification of Working Mass Reference Standards).

8.8 Repeat step 8.7 with the lower mass reference working standard.

8.9 Perform a zero analysis by closing the weigh chamber with the weigh pan empty. Allow the balance reading to stabilize until the “mg” appears on the balance display. Press “get weight” and press “next” to store the zero reading in the database.

8.10 Neutralize the electrostatic charge of each filter by placing it on a ^{210}Po anti-static strip for approximately 60 seconds. Use only stainless steel, non-serrated forceps to handle filters. Place the filter in the weighing chamber and close. Scan the Petri dish bar code. Wait until the filter weight stabilizes and the “mg” appears on the balance display. Press “get weight” button. The mass is automatically stored in the DMS by pressing “next”.

8.11 Return each filter to its uniquely identified Petri dish following analysis and close Petri dish.

8.12 Repeat steps 8.10 and 8.11 for all the client network filters to be weighed ending with the client’s lab blanks.

8.13 Following each ten analyses, analyze the lower working standard and analyze the zero as prompted by the weighing interface.

8.14 After weighing all filters for a client network and lab blanks repeat steps 8.6-8.9 with both working standards and zero.

8.15 Ten percent reweighs may be required, or an independent analyst may be required. Typically, one reweigh analysis per client is performed. Contact supervisor if unsure of reweigh requirements.

9.0 Reweigh Procedure

9.1 Randomly select one filter from the client session, or group of filters based on client request and perform a replicate analysis. Following step 8.10 above, weigh selected filter(s). If the reweigh measurement and the original measurement differ by more than ±15 µg, reweigh the filter and save. If the second reweigh measurement is less than ±15 µg continue with the session. Manually invalidate the first replicate weight. But, if the measurements still differ by more than ±15 µg from the original weight, repeat the pre-exposure analysis for all associated filters in the entire weigh session for the client. The DMS will automatically invalidate the weigh session allowing the filters to be weighed again. Inform the supervisor of the QC failure.

9.2 After the replicate analysis, again repeat steps 8.6 – 8.9 with both working mass reference standards and zero.

9.3 Print the weigh session and review the weigh session report. Review for similar weights, breaks in filter id sequence, QC checks valid, in specification, high weights and any anomaly, Initial the bottom of the first page to document the review was performed. An independent technician must review the report and initial the bottom of the first page to verify an independent review was fulfilled. Log out of the DMS.

10.0 Independent Reweigh Procedure

10.1 In place of the random replicate analysis in section 9.0 above perform the following:

10.2 If requested by a client and after the client weigh session is complete, select 10% random filter numbers from all filters in the client network weigh session. An independent analysis will be performed on those filters.

10.3 An independent analyst must open the Balance Control portion of the DMS and log in. The average of the previous 24 hours' one second readings for temperature and humidity will automatically populate the conditions tabs.

10.4 An independent analyst must perform the replicate analyses following procedure above in section 8.2 – 8.14. If the independent replicate measurement and the original measurement differ by more than ±15 µg, reweigh the filter. If the second reweigh measurement is less than ±15 µg continue with the session. Manually invalidate the first replicate weight. But, if the measurements still differ by more than ±15 µg, the original analyst must repeat the pre-exposure analysis for all associated filters in the weigh session. As stated above at the end of 9.1, the DMS will automatically invalidate the weights of the weigh session when the second re-weigh is saved.

10.5 Print the replicate weigh session and review weigh session report. Initial and date the first page at the bottom to document the review was performed. Another technician must review the report and initial and date the bottom of the first page to verify a second review was fulfilled. Log out of the DMS.

11.0 Calculations

11.1 Working standard mass difference = Observed mass – NIST certified conventional mass

11.2 Replicate mass difference = reweigh mass – original mass

12.0 References

12.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

12.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10–2.5}$ in the Atmosphere

12.3 Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

12.4 Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

12.5 *Cahn Technical Notes: Static Control for Balances;* Jerry Weil, Cahn Instruments, Cerritos, California

13.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP - GR-111-0.0

STANDARD OPERATING PROCEDURE FOR SHIPPING FILTERS TO LOCATIONS

May 2021
IML AIR SCIENCE

REVISED BY:

Mary Hunniger 05/17/21
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APPROVED:

[signature] 5/17/2021
Manager Date

[signature] 5/17/2021
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Table of Contents

1.0 Scope and Application

1.1 This procedure is used for shipping prepared, clean, pre-weighed filters to each client location.

1.2 The elements of this SOP are applicable to filters for low volume air samplers.

2.0 Summary of Method

2.1 Following the pre-exposure mass determination, filters are loaded into cassettes, FedEx or UPS labels printed, and filters prepared for shipment. Filters are protected from electrostatic charge and handling damage by shipping in cassettes loaded into anti-static bags with stiffening labels. Filters are protected from moisture inside the cooler by placing the anti-static bags containing the filters and cassettes into waterproof bags. Cooling agents (freezer packs) are provided with the shipment to facilitate chilled return shipment of exposed filters.

3.0 Definitions

3.1 Cassette - A device supplied with samplers to allow a weighed filter to be held in place in the sampler and manipulated before and after sampling without touching the filter, and to minimize damage to the filter and/or sample during such activities.

3.2 Data Management System (DMS) – Software systems to record, store and report data.

3.3 Laboratory blank filter - New filters, that are used to determine laboratory contamination. The laboratory blank filters shall be weighed along with the pre-sampling (tare) weighing of each set of filters. These laboratory blank filters should remain in the laboratory in protective containers during the field sampling and should be reweighed as a quality control check at gross weighing.

3.4 Cooler – insulated shipping container.

4.0 Health and Safety Concerns

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter, and avoid filter damage. Filters are prepared and remain in the clean, environmentally controlled room. Wear a laboratory coat when handling filters. Keep filter Petri dishes protected and in a horizontal position during transport.

5.2 Handle filters with stainless steel non-serrated forceps by the support ring only.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of gravimetric laboratory.

7.0 Equipment

7.1 Clean filter cassettes

7.2 Cassette separation tool

7.3 Petri dishes or slides

7.4 Anti-static bags

7.5 Bar-coded labels for anti-static bags

7.6 Minimum/Maximum thermometer

7.7 Waterproof bag

7.8 Cooling agent (freezer pack)

7.9 Shipping cooler

7.10 Shipping label

7.11 Outgoing chain of custody document

7.12 Computer with access to DMS

7.13 Lab coat

8.0 Procedure

8.1 Using information provided on the shipping checklist print UPS or FedEx labels for each shipping location and organize to be ready to place on coolers as filters are packaged.

8.2 After the pre-exposure mass determination is complete, place filters into individually numbered, clean filter cassettes following the cassette tracker form already filled out and assigned. Place cassettes in magazines, metal tins or add lids if requested by client.

8.3 Close the empty Petri dishes or slides and place sequentially in the storage racks for each client in the anteroom. Lab blank Petri dishes or slides are closed with

lab blank filters inside and placed in the storage unit drawer in the laboratory for each client.

8.4 Insert the cassette with the filter into its corresponding labeled anti-static bag. Confirm that the filter ID on the bag label and the cassette tracker worksheet are the same as the filter ID on the filter. Verify the cassette ID on the cassette tracker worksheet is the same as on the actual cassette. After each client is complete initial the cassette tracker worksheet.

8.5 Rubber band evenly grouped labeled anti-static bags containing cassettes with filters inside. Put rubber band stacks of filters into waterproof bag(s) before taking filters out of laboratory.

8.6 Using the Air Data Processor portion of the DMS software, select Tare Shipment for the day of the shipment. Select the client and click Generate Tare COC at the bottom of the window. Save the excel workbook. The workbook automatically opens. Print the Tare COC.

8.7 Review the Tare COC to ensure the filters IDs are correct and match the shipment. Sign and date the Tare COC.

8.8 Make a copy of the signed Tare COC to be retained.

8.9 Pack waterproof bag inside cooler with cooling agents (the cooling agent need not be frozen when sent with the unexposed filters). Place the chain of custody documents inside a separate waterproof bag with the Tare COC and any other document. Place this waterproof bag in the cooler checking the Tare COC and filters are for the same client and shipping location. Add packing material as needed to keep contents secure after second check.

8.10 If requested, insert a minimum/maximum thermometer inside the cooler after re-setting and checking that the display comes on and is operational.

8.11 Before sealing the cooler affix the shipping label to the outside of the cooler, confirm label, client filters, and Tare COC match. Have another trained person confirm label, client filters, and Tare COC all match. Each cooler is checked by two individuals and the shipping checklist is initialed by both.

8.12 Seal the cooler with packing tape and place the cooler in the designated location for pickup by the commercial parcel service.

8.13 After all coolers for clients are finished, complete the shipment in the DMS. Select Tare Shipment from the menu for current date. For each client, one at a time Assign Shipper, fill in shipping information and hit Process Shipment. Shipment status will change to green if shipment is complete for each client. If the status is orange investigate and fix.

9.0 References

9.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

9.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10-2.5}$ in the Atmosphere

9.3 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program*; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

9.4 *Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods*; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP - GR-112-0.0

STANDARD OPERATING PROCEDURE FOR RECEIPT AND LOGIN OF EXPOSED PARTICULATE FILTER SAMPLES

May 2021
IML AIR SCIENCE

REVISED BY:

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APPROVED:

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Table of Contents

1.0 Scope and Application

1.1 This procedure is used to receive coolers with exposed filters, measure and record interior cooler temperature upon arrival of filters, verify chain of custody information and sample run data, prepare exposed filters for post-exposure gravimetric analysis, equilibrate, and complete exposed filter login.

1.2 The elements of this SOP are applicable to filters for all low volume samplers.

2.0 Summary of Method

2.1 Exposed filter samples are received from a network location typically in a cooler. Filter temperature in cooler is measured immediately and filters are removed. Filters are received into the DMS by scanning the bag label bar code and then are prepared and placed into the equilibration environment. The filter IDs are checked against the Chain of Custody to make sure the filters listed on the COC match. All pertinent data is logged into the DMS.

3.0 Definitions

3.1 Cassette - A device supplied with samplers to allow a weighed filter to be held in place in the sampler and manipulated before and after sampling without touching the filter, and to minimize damage to the filter and/or sample during such activities.

3.2 Lab - A room containing the microbalance designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

3.3 Data Management System (DMS) – Software systems to record, store and report data.

3.4 Anteroom – A room with limited access and no air flow located adjacent to the lab, for unloading cassettes, storing Petri dishes, and clean cassettes.

4.0 Health and Safety Concerns

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter, and avoid filter damage. Wear laboratory coat when handling filters in lab and anteroom. Keep filter Petri dishes protected and in a horizontal position during transport.

5.2 Handle filters with stainless steel non-serrated forceps by the support ring only.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement instrumentation.

6.2 Familiarity with electronic laboratory equipment is required.

7.0 Equipment

7.1 Clean, environmentally controlled room

7.2 Laboratory coat

7.3 Auto-retracting blade utility knife

7.4 Cut resistant glove

7.5 Insulated shipping container (cooler)

7.6 Minimum/maximum thermometer

7.7 Infrared thermometer gun, verified to NIST traceable thermometer

7.8 Completed Chain of Custody documents (COC)

7.9 Cassette separation tool

7.10 Petri dishes with labels

7.11 Stainless steel, non-serrated forceps

7.12 46.2 mm diameter filters, Teflon membrane (PTFE)

7.13 Lint-free wipes

7.14 Computer with access to DMS

8.0 Procedure

8.1 Open cooler and immediately determine the receipt, maximum, and minimum temperatures, upon arrival of the filters and record as indicated by the min/max thermometer. If it's apparent that the min/max thermometer was not reset prior to shipment, use the infrared thermometer gun to determine the receipt temperature. If a thermometer has not accompanied the filter samples in shipment use the infrared thermometer gun when network clients approve substitution.

To obtain the temperature with the infrared thermometer gun, open cooler and immediately point the gun into the cooler at the filter samples with the trigger held down watching the digital read out until it stabilizes, typically a few seconds. Record the reading and apply the correction factor, from the most recent certification. If the reading is unstable check and replace the batteries in the thermometer gun. Current certification stickers are placed on the IR thermometer following each new calibration.

8.2 If included, retrieve Chain of Custody and/or sampler collection data (field sheets) for the filter samples received. Record the cooler temperature and cooler number on the Chain of Custody. Document the condition of the ice packs on the bottom of the COC. Sign the COC and record the date and time.

8.3 In the DMS under “Receive Samples” on the sample receipt form fill out all relevant information on the screen and then receive filter samples.

8.4 Remove the filter sample anti-static bags from the waterproof bag(s). Filters are received into the DMS by scanning each bar code on the filter anti-static bag labels. Finish filling out client information on the screen; save and print sample receipt form.

8.5 In the DMS, press move and select all to track filters to the lab for post-exposure analysis or other options.

8.6 Match the filter IDs on the anti-static bags against the Chain of Custody to assure the filters listed on the COC are received. Note any discrepancies on the sample receipt form.

8.7 Take filter samples to the anteroom where Petri dishes are stored.

8.8 Retrieve ten percent laboratory blanks with the same pre-exposure date as the exposed samples and open the Petri dish placing the lid underneath the dish. If filters are not going to be exposed at this time close each dish, stack, and place in Office cooler. On the first anti-static bag place a sticker at the top and write the lab blank id numbers. Put the laboratory blanks in the well-ventilated tray in front of the corresponding client Petri dishes.

8.9 Remove samples from their protective anti-static bag, noting that the cassette ID on the anti-static bag matches the cassette ID on the cassette in the bag. If the cassette in the bag does not match the cassette ID written on the bag label make a note on the sample receipt form and contact the supervisor.

8.10 Using a cassette separation tool, open the cassette and carefully remove the filter using stainless steel, non-serrated forceps, contacting the filter’s support ring only. Verify the filter ID on the bag label matches the number on the Petri dish lid and the number stamped on the filter itself. Place the filter in the Petri dish. Continue unloading each filter according to steps 8.9 and 8.10. Inspect

filters while unloading from cassettes into the Petri dishes. Record any defects on the sample receipt form.

8.11 Verify that the laboratory conditions are within specifications by viewing Lab Conditions interface on the computer screen or lab status open on the desktop of the computer in the laboratory. If the lab conditions have not been met, the DMS will alert you. If the lab conditions are out of specifications take corrective actions to ensure that they return to specifications. Corrective action may include repair or replacement of environmental measurement and control components. Store exposed filter samples at <4˚C until the laboratory conditions respond to the corrective action and return to specifications.

8.12 Place Petri dishes containing exposed samples in well-ventilated trays, with the lid underneath the Petri dish, typically in sequential order of filter numbers.

8.13 Take trays of filters into lab.

8.14 In the DMS, click equilibration button. Select exposed filters and add lab blank ID numbers by clicking on the "magnifying glass"  in the upper righthand corner of the screen. Input the lab blank ID number and save, repeating this step for each lab blank. Once the lab blanks have been selected save the equilibration batch by clicking on the save button in the lower righthand corner of the screen.

8.15 Review the COC and sample receipt form with the anti-static bag labels to make sure everything matches, and any issues are fully documented. Place in tray for final review by supervisor.

8.16 To minimize the amount of volatilization, exposed samples should not be left to equilibrate over extended periods (weekends, holidays etc.). If it is Friday or the day before a holiday, place exposed samples in cold storage at <4 ˚C. Stack closed Petri dishes in trays in sequential order before placing in the refrigerator. Lab blanks remain in closed Petri dishes in the lab.

8.17 After equilibration is initiated, log the samples into the DMS by going to exposed login for the date received; select a client and begin. The scanner will read the barcode and save the image of the bag label. Images are not saved for all clients. Log in varies by client. Fill in the information on the screen for each filter.

8.18 The supervisor will notify the network operator of any unusual situations (cooler temperature out of specification, missing samples, etc.) in a timely fashion.

9.0 References

9.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

9.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10-2.5}$ in the Atmosphere

9.3 Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

9.4 Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.0 Revision Record

Revision #	Date	Person Responsible	Description of Changes
0.0	05/14/21		• New template and new numbering system

SOP – GR-113-0.0

STANDARD OPERATING PROCEDURE FOR PERFORMING THE POST-EXPOSURE ANALYSIS

May 2021
IML AIR SCIENCE

REVISED BY:

Mary Hemingen — 05/17/21
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APPROVED:

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Manager — Date

[signature] — 5/17/2021
Quality Officer — Date

Reviewed

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Date				

Table of Contents

1.0 Scope and Application

1.1 This procedure is to determine the mass of an equilibrated filter after sampling.

2.0 Summary of Method

2.1 Teflon membrane filters containing particle samples are conditioned in a lab and then analyzed gravimetrically. This mass is used in conjunction with tare mass (pre-exposed), to determine the net mass of the collected particles.

3.0 Definitions

3.1 Lab - A room, containing the microbalance, designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

3.2 Microbalance - A type of analytical balance that can weigh to at least the nearest 0.001mg (that is,1 µg or one-millionth gram).

3.3 Polonium-210 (^{210}Po) anti-static strip - A device containing a small amount of ^{210}Po that emits α particles (He2+) that neutralize the static charge on filters, making them easier to handle and their weights more accurate.

3.4 Working Standard – ASTM Class 0, Class 1, or Class 2 mass standard, traceable to NIST, that is used for routine quality control checks.

3.5 Equilibration – Allowing the filters to come to a stable temperature and humidity in the lab, prior to weighing.

3.6 Weigh Session - A grouping of filters that are conditioned and analyzed as a single weighing event and for which laboratory 24-hour temperature and humidity conditions are documented and associated with the grouping of filters. The weigh session includes the required QC (filter blanks, standards, etc.).

3.7 Data Management System (DMS) – Software systems to record, store and report data.

3.8 Laboratory blank filter - New filters, that are used to determine laboratory contamination. The laboratory blank filters shall be weighed along with the pre-sampling (tare) weighing of each set of filters for each client network. These laboratory blank filters should remain in the laboratory in protective containers during the field sampling and should be reweighed as a quality control check at gross weighing.

4.0 Health and Safety Concerns

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded. Use caution when servicing or operating electrical equipment in wet conditions.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter and avoid filter damage. Laboratory coats will minimize the potential for laboratory contamination and must be taken off before leaving the lab to minimize contamination from the external environment. Keep filters and Petri dishes protected and in a horizontal position during transport.

5.2 Errors in the gravimetric analysis of samples can result from the buildup of electrostatic charges on filters during their manufacture or during sampling. This static buildup will interfere with microbalance weighing, but it can be reduced by using static charge reduction techniques.

5.3 Handle filters with stainless steel non-serrated forceps by the support ring only.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement instrumentation and general analytical laboratory protocol.

6.2 General computer knowledge for interacting with the data management system.

6.3 Familiarity with electronic laboratory equipment is required.

7.0 Equipment

7.1 Clean, environmentally controlled room

7.2 Laboratory coat

7.3 Analytical microbalance, minimum sensitivity to ±1µg, minimum repeatable to ±1µg

7.4 Balance table, with anti-static mat

7.5 ^{210}Po anti-static strips

7.6 Stainless steel, non-serrated forceps

7.7 Non-serrated, non-metallic forceps

7.8 Grounded floor mat

7.9 NIST traceable upper and lower working mass reference standards

7.10 Computer with access to DMS

7.11 46.2 mm diameter filters, Teflon membrane (PTFE)

8.0 Procedure

8.1 Verify in the DMS that the filters have equilibrated for the required amount of time as determined by the filter lot stability test (typically 24 hours) and that room temperature and relative humidity have remained within specifications (mean temperature between 20˚C and 23˚C with less than ±2˚C variability over 24 hours and mean relative humidity between 30% and 40% with less than ±5% variability over 24 hours) during the equilibration period. Temperature and relative humidity for 5 minute and 24 hour averages are displayed on the computer screen interface, with a graphical presentation also available. The display is color coded with green for good, yellow for warning, and red for stop. In addition, the DMS will prevent weighing if conditions are outside control limits. The conditions must be back in compliance to re-start equilibration.

8.2 Complete the daily laboratory and equipment maintenance and preparation. (Standard Operating Procedure For Cleaning and Maintenance of Gravimetric Laboratory)

8.3 Log into the Balance Control portion of the DMS. The average of the previous 24 hours' one second readings for temperature and relative humidity will be displayed. Observe the readings and confirm compliance.

8.4 Locate the equilibration tray containing the exposed filters to be analyzed and place the tray adjacent to the microbalance.

8.5 Retrieve the working standards and place them in a convenient location near the microbalance.

8.6 Check the balance to make sure it's reading zero, if not press the tare button.

8.7 Analyze the upper working mass reference standard. Use only non-serrated, non-metallic forceps to handle the mass reference working standards. Place the standard on the balance pan and close chamber. Allow the reading to stabilize until the mg appears at the end of the reading, press "get weight" on the interface screen and press "next" to store weight. If the measured value of the working standard disagrees with the certified value by more the ±3 µg, remove the standard, zero the balance, inspect, clean and reweigh the working standard. If the two measurements still disagree by more than ±3 µg, contact supervisor. If

necessary, verify the working standards (Standard Operating Procedure For the Verification of Working Mass Reference Standards).

8.8 Repeat step 8.7 with the lower working mass reference standard.

8.9 Perform a zero analysis by closing the weigh chamber with the weigh pan empty. Allow the balance reading to stabilize until the “mg” appears on the balance. Press “get weight” and then press “next” to store the zero reading in the database.

8.10 Weigh lab blanks associated with client network before the exposed filters by following step 8.11. If the lab blank exceeds control limits of ±15µg the filter will be inspected and reweighed. Stop weighing and notify supervisor. The filters associated with the lab blank will be flagged on the report. An investigation and corrective action procedures will be performed after weighing. Corrective action may include cleaning the lab.

8.11 Neutralize the electrostatic charge of each filter by placing it on a ^{210}Po anti-static strip for approximately 60 seconds. Use stainless steel, non-serrated forceps to handle filters. Place the filter in the weighing chamber and close. Scan the Petri dish bar code. Wait until the filter weight stabilizes and the “mg” appears on the balance display. Click on the “get weight” button. The mass is automatically stored in the DMS by pressing “next”.

8.12 If the filter is a field blank or trip blank and out of specification (30 µg and 15 µg respectively). Inspect, and reweigh. If still out of specification, notify supervisor.

8.13 Return each filter to its uniquely identified Petri dish following analysis and close Petri dish.

8.14 Repeat steps 8.11 and 8.13 for all the client filters to get exposed weight.

8.15 Following each ten analyses, analyze the lower working mass reference standard and analyze the zero as prompted by the weighing interface.

8.16 After weighing all filters for a client network repeat steps 8.6 - 8.9 with both working mass reference standards and zero.

8.17 Reweighs are client specific. Ten percent reweighs may be required, or an independent analyst may be required. Typically, one reweigh analysis per client is performed. Contact supervisor if unsure of reweigh requirements.

9.0 Reweigh Procedure

9.1 Randomly select one filter from the client session, or group of filters based on client request and perform a replicate analysis. Following step 8.11 above, weigh selected filter(s). If the replicate measurement and the original measurement

differ by more than ±15 µg, reweigh the filter and save. If the second reweigh measurement is less than ±15 µg continue with the session. Manually invalidate the first replicate weight. But, if the measurements still differ by more than ±15 µg, repeat the post-exposure analysis for all associated filters in the entire weigh session for the client. The DMS will automatically invalidate the weigh session allowing the filters to be weighed again. Inform the supervisor of the QC failure.

9.2 After the replicate analysis, again repeat steps 8.6 – 8.9 with both working mass reference standards and zero.

9.3 Print the weigh session and review the weigh session report. Initial the bottom of the first page document the review was performed. An independent technician must review the report and initial the bottom of the first page to verify an independent review was fulfilled. Log out of the Balance Control portion of the DMS.

10.0 Independent Reweigh Procedure

10.1 In place of the random replicate analysis in section 0 above, perform the following:

10.2 If requested by a client, and after the weighing session is complete, select 10% random filter numbers from all filters in the client network weigh session. An independent analysis will be performed on those filters.

10.3 An independent analyst must open the Balance Control portion of the DMS and log-in. The average of the previous 24 hours' one second readings for temperature and humidity will automatically populate the conditions tabs.

10.4 An independent analyst must perform the replicate analyses following procedure above in section 8.2 – 8.14 skipping step 8.9 for the lab blanks. If the independent replicate measurement and the original measurement differ by more than ±15 µg, reweigh the filter. If the second reweigh measurement is less than ±15 µg continue with the session and manually invalidate the first replicate weight. But, if the measurements still differ by more than ±15 µg, the original analyst must repeat the post-exposure analysis for all associated filters in the weigh session. As stated above at the end of section 9.1, the DMS will automatically invalidate the weights of the weigh session when the second re-weigh is saved.

10.5 Print the weigh session and review the weigh session report. Initial the first page at the bottom to document the review was performed. Another technician must review the report and initial the bottom of the first page to verify a second review was fulfilled. Log out of the balance control interface software.

11.0 Calculations

11.1 Working standard mass difference = Observed mass – NIST certified conventional mass

11.2 Net mass = exposed mass - original mass

11.3 Replicate mass difference = reweigh mass – original mass

12.0 References

12.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

12.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10\text{-}2.5}$ in the Atmosphere

12.3 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program*; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

12.4 *Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods*; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

12.5 *Cahn Technical Notes: Static Control for Balances*; Jerry Weil, Cahn Instruments, Cerritos, California

13.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP – GR-114-0.0

STANDARD OPERATING PROCEDURE FOR ARCHIVING FILTER SAMPLES

May 2021
IML AIR SCIENCE

REVISED BY:

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APPROVED:

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Manager — Date

[signature] — 5/17/2021
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Reviewed

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Date				

Table of Contents

1.0 Scope and Application

1.1 This procedure is used to archive filter samples, in cold storage, after post-exposure analysis.

2.0 Summary of Method

2.1 Following the post-exposure analysis, samples are archived in a temperature controlled (0 to 4˚C) environment. Chilled archiving is used to minimize mass loss due to volatilization. The length of storage is per client's requirements. EPA recommends storage for 5 years with a minimum of at least one-year cold storage.

3.0 Definitions

3.1 Data Management System (DMS) – Software systems to record, store and report data.

4.0 Health and Safety Concerns

4.1 Personnel should limit exposure time to the low temperatures within the coolers.

4.2 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter, and avoid filter damage. Keep filter cassettes protected and in a horizontal position during transport.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with filter handling.

6.2 Familiarity with electronic laboratory equipment is required.

7.0 Equipment

7.1 Archiving containers with network name and dates

7.2 Computer with access to DMS

7.3 Cooler capable of maintaining temperature control (0 to 4˚C)

8.0 Procedure

8.1 Following the post-exposure analysis, place filters in labeled Petri dishes capped inside labeled anti-static bags. Place the group of filters from a network together in a plastic bag and label the bag with the post-exposure analysis date or by sampling period for small networks.

8.2 Place the bag in the cold storage cooler in a network specific archiving container.

8.3 To move filters to archive in the Air Data Processor portion of the DMS for tracking, select Move Filters in the left panel.

8.4 Select the Network from the dropdown menu

8.5 Select the current location of the filters in the Move Samples from dropdown menu (in this case Lab for Gross)

8.6 Select the location to move the filters from the To: dropdown menu.

8.7 Select which filters to move (there are options for multi-selecting at the bottom of the window)

8.8 Click Move.

8.9 After one full year of archiving filters in cold storage, clients are contacted to determine disposition of filters. Filters should be returned to client or other arrangements made for long term storage. Record in DMS following similar steps to 8.3 - 8.8 when filters are moved. After removal from cold storage, the filters should be relocated to a clean, dry area, where they will be protected from light, vibrations, and dust sources.

9.0 References

9.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

9.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10\text{-}2.5}$ in the Atmosphere

9.3 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program*; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

9.4 *Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods*; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP – GR-304-0.0

STANDARD OPERATING PROCEDURE FOR THE VERIFICATION OF WORKING MASS REFERENCE STANDARDS

May 2021
IML AIR SCIENCE

REVISED BY:

Mary Hemingen — 05/17/21
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APPROVED:

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Manager — Date

[signature] — 5/17/2021
Quality Officer — Date

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1.0 Scope and Application

1.1 This procedure is used to verify the conventional value of working mass reference standards used routinely for quality control in the micro-gravimetric analyses of filter samples.

2.0 Summary of Method

2.1 Working Standards are used multiple times during each weighing session of filters. The conventional masses of the working standards are verified against traceable, conventional mass of the primary standards. Verifications are completed periodically (every 3 months) and/or when control limits (±3µg) for working standard drift are exceeded. Primary mass reference standards are independently certified or replaced annually.

3.0 Definitions

3.1 Lab - A room, containing the microbalance designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

3.2 Primary standard – ASTM Class 0, Class 1, or Class 2 mass standard, traceable to NIST, that has “authority” over other standards, (working standards), in the laboratory.

3.3 Microbalance - A type of analytical balance that can weigh to at least the nearest 0.001 mg (that is,1 µg or one-millionth gram).

3.4 Data Management System (DMS) – Software systems to record, store and report data.

3.5 Working standard – ASTM Class 0, Class 1, or Class 2 mass standard, traceable to NIST, that is used for routine quality control checks.

4.0 Health and Safety Concerns

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

5.0 Cautions

5.1 In all steps involving mass reference standard handling; be extremely careful to avoid any contamination and avoid damage.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement instrumentation.

6.2 General computer knowledge for interacting with the DMS.

6.3 Familiarity with electronic laboratory equipment is required.

7.0 Equipment

7.1 Primary and working standards which bracket the expected filter mass. An upper standard (typically 500 mg) and lower standard (typically 200 mg)

7.2 Calibration Certificates for the Primary and working standards.

7.3 Microbalance, minimum sensitivity to ±1µg, minimum repeatable to ±1µg

7.4 Balance table, with anti-static mat

7.5 Laboratory coat

7.6 Non-serrated, non-metallic forceps

7.7 Grounded floor mat

7.8 Working Mass Reference Standard Verification form

7.9 Data management system (DMS)

7.10 Laboratory balance notebook

8.0 Procedure

8.1 Before beginning verifications, perform the daily laboratory cleaning and equipment maintenance (Standard Operating Procedure For Cleaning and Maintenance of Micro-Gravimetric Laboratory).

8.2 Record the date and initial the working mass reference standard verification form.

8.3 Confirm the primary standards serial numbers and conventional masses match the calibration certification traceable forms received with the standards. Enter all four digits right of the decimal place in the mass reference standard conventional value on the form.

8.4 Confirm the working standards serial numbers and conventional masses match the calibration certification traceable forms received with the standards. Enter all

four digits right of the decimal place in the mass reference standard conventional value on the form.

8.5 Check to make sure the balance is reading zero. Allow the balance to return to zero of its own accord, if not press the tare button. Ensure the balance returns to reading zero in between each measurement.

8.6 Log into the Balance Control portion of the DMS, making sure to click on button for Verify Working Standards. Select appropriate primary and working lower standards in drop down menu.

8.7 On the Working Mass Reference Standard Verification form, fill in each box with the correct information from the standard calibration certificate.

8.8 Measure the weight of the lower working standard. When the reading is stable press “get weight” to store the reading in the DMS. Record the weight on the form “O_1” in the place provided. If the working standard is not ±3 µg of conventional mass, remove the standard from the balance and allow the balance to go to zero and weigh again. If the working standard is not ±3 µg, troubleshoot.

8.9 While the pan balance is empty, allow the balance to return to reading zero, if not press the tare button.

8.10 Measure the weight of the lower primary standard; when the reading is stable press “get weight”. Record the weight on the form “O_2”. If the primary standard is not ±3 µg of conventional mass, remove the standard from the balance and allow the balance to go to zero and weigh again. If the primary standard is not ±3 µg, then troubleshoot and if necessary, perform microbalance calibration (Standard Operating Procedure for Calibration of Microbalance).

8.11 Remove the standard in between measurements.

8.12 Measure again the lower primary standard and when the reading is stable press “get weight”. Record the weight on the form “O_3”.

8.13 Then measure the weight of the lower working standard and when the reading is stable press “get weight”. Record the weight on the form “O_4”.

8.14 Calculate the apparent mass correction and difference, by clicking on the computer control interface screen calculate button. Record reading and calculations on the form.

8.15 If the difference is within ±2 µg, press the “Save” button to store results in the DMS. If the difference is not within ±2 µg, repeat steps 8.5 – 8.15 once. If working standard(s) still do not meet requirements, troubleshoot.

8.16 Repeat steps 8.5 through 8.15 for the upper working standard verification.

8.17 Review form for both working standards to make sure the form is completely filled out and requirements are met.

8.18 Return the standards to their proper storage in the lab conditioning room.

8.19 Document activity in Laboratory Balance notebook.

9.0 Calculations

9.1 Apparent mass correction (mg)

$$Apparent\ mass\ correction = C_p + \frac{O_1 - O_2 + O_4 - O_3}{2} + N_p - N_w$$

Where:

C_p is the conventional mass of the primary standard (mg)
O_1 working standard, observation 1 (mg)
O_2 primary standard, observation 2 (mg)
O_3 primary standard, observation 3 (mg)
O_4 working standard, observation 4 (mg)
N_p is the nominal value of the primary standard (mg)
N_w is the nominal value of the working standard (mg)

9.2 Difference (µg) = (C_w – Apparent Mass Correction) x 1000

Where: C_w is the conventional mass of the working standard (mg)
Apparent Mass Correction (mg)

10.0 References

10.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as PM2.5 in the Atmosphere

10.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as PM10–2.5 in the Atmosphere

10.3 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program*; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.4 *Quality Assurance Guidance Document 2.12; Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods*; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

11.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

SOP – GR-301-0.0

STANDARD OPERATING PROCEDURE FOR PERFORMING MONTHLY VERIFICATIONS OF THE LABORATORY TEMPERATURE AND RELATIVE HUMIDITY

May 2021
IML AIR SCIENCE

REVISED BY:

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1.0 Scope and Application

1.1 This procedure is used to verify (audit) the laboratory temperature and relative humidity sensors using an independent data logger audit kit with certified temperature and relative humidity standards. This procedure applies to both high volume and low volume methods.

1.2 This procedure must be performed monthly.

1.3 Temperature and relative humidity standards require NIST re-certification annually.

2.0 Summary of Method

2.1 IML gravimetric laboratory uses an electronic Data Acquisition System (DAS) to monitor and control the temperature and relative humidity within the laboratory. NIST traceable standards are used to verify the accuracy of the laboratory temperature and relative humidity measurements made by the DAS.

3.0 Definitions

3.1 Lab - A room containing the balance, designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

3.2 NIST- Acronym for the National Institute of Standards and Technology, which is the federal technology agency that works with industry to develop and apply technology, measurements, and standards.

3.3 Reference verification data acquisition system (Verification DAS) – A separate data acquisition system that records the reference standard temperature and humidity readings for comparison to the laboratory data acquisition system’s readings.

4.0 Health and Safety Warnings

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded. Use caution when servicing or operating electrical equipment in wet conditions.

5.0 Cautions

5.1 Damage to the instrument may result if caution is not taken to properly install and maintain the device. Follow the manufacturer's instructions for maintenance of all equipment and for safe, secure installation.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be familiar with laboratory operation and conditions.

6.2 Instrumentation skills are necessary for interacting with the data acquisition system.

6.3 Familiarity with electronic and mechanical test equipment is required.

7.0 Equipment

7.1 NIST traceable relative humidity standard (hygrometer) with ±0.8 % accuracy at 10 °C to 30 °C.

7.2 NIST traceable temperature standard with ±0.1 °C accuracy at 20 °C to 30 °C.

7.3 Verification DAS

7.4 Electronic Data Acquisition System (DAS) connected to laboratory sensor for temperature and relative humidity.

7.5 Laboratory notebook

8.0 Procedure

8.1 Verify that the certification for the NIST traceable standards in use are current.

8.2 If certification is found to be out of date the standard must be either re-certified or replaced with certified standard prior to performing the verification.

8.3 Record the date, the technicians performing the verification, and the serial number of NIST standard used in the Laboratory notebook.

8.4 Connect the verification DAS to the Ethernet port in the lab

8.5 Connect the reference NIST temperature and relative humidity standard to the verification DAS.

8.6 Power on the system and place the temperature/relative humidity probe near the lab probe.

8.7 Use the keypad to select which lab will be verified and start.

8.8 The system will equilibrate for 30 minutes, and then record and compare readings from the lab DAS and verification DAS for six measurements of five minute averages.

8.9 Once the verification is complete the system will email notification of the results, along with the readings from the lab and the verification system.

8.10 Compare the actual and the indicated values and record the difference in the Laboratory notebook. The lab DAS indicated temperature must not differ from the actual temperature measured by the verification DAS temperature standard by more than ±2 °C. The Lab DAS indicated relative humidity must not differ from the verification DAS relative humidity standard by more than ±2 %.

8.11 If the verification values exceed the acceptance limits, corrective action must be initiated. Corrective action may include, but is not limited to: replacing the sensor, datalogger or other hardware or performing maintenance. Repeat the verification after corrective action is completed to provide evidence acceptance criteria is met.

8.12 Record the results in the Laboratory notebook and save results on network.

8.13 After the verifications are completed, return the verification DAS to proper storage.

9.0 References

9.1 *40 CFR 50, Appendix L;* Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

9.2 *40 CFR 50, Appendix O;* Reference Method for the Determination of Coarse Particulate Matter as $PM_{10-2.5}$ in the Atmosphere

9.3 *40 CFR, Appendix J;* Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere

9.4 *40 CFR 50, Appendix B;* Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High-Volume Method)

9.5 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program;* EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

9.6 *Quality Assurance Guidance Document 2.*12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

9.7 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Specific Methods;* EPA/600/4-77-027a; Section 2.11; January 1990; U.S. Environmental Protection Agency

10.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system • Combined SOP for Hivol and Lowvol methods • Add references

SOP – GR-201-0.0

STANDARD OPERATING PROCEDURE FOR PERFORMING STABILITY TEST ON NEW LOTS OF ANTI-STATIC BAGS

May 2021
IML AIR SCIENCE

REVISED BY:

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1.0 Scope and Application

1.1 To verify that a new lot of anti-static bags are contamination free.

1.2 This procedure must be performed on each new lot of anti-static bags. (And each box of 1000 bags)

2.0 Summary of Method

2.1 The Laboratory uses anti-static bags for storage, transport and protection of low volume filter samples in cassettes. New lots of bags are tested for possible contamination prior to use.

3.0 Definitions

3.1 Lab - A room, containing the microbalance designed to maintain the filter conditioning requirements for temperature and humidity. Filters are held in this area until they have reached a steady state of moisture.

3.2 Microbalance - A type of analytical balance that can weigh to at least the nearest 0.001mg (that is,1µg or one-millionth gram).

3.3 Polonium-210 (^{210}Po) anti-static strip - A device containing a small amount of ^{210}Po that emits α particles (He^{2+}) that neutralize the static charge on filters, making them easier to handle and their weights more accurate.

3.4 Working Standard – ASTM Class 0, Class 1, or Class 2 mass standard, traceable to NIST, that is used for routine quality control checks.

3.5 Cassette - A device supplied with $PM_{2.5}$ samplers to allow a weighed Teflon® filter to be held in place in the sampler and manipulated before and after sampling without touching the filter, and to minimize damage to the filter and/or sample during such activities.

4.0 Health and Safety Concerns

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter and avoid filter damage. Laboratory coats will minimize the potential for laboratory contamination and must be taken off before leaving the lab to minimize contamination from the external environment. Keep filters and Petri dishes protected and in a horizontal position during transport.

5.2 Errors in the gravimetric analysis of samples can result from the buildup of electrostatic charges on filters during their manufacture or during sampling. This static buildup will interfere with microbalance weighing, but it can be reduced by using static charge reduction techniques.

5.3 Handle filters with stainless steel non-serrated forceps by the support ring only.

5.4 Damage to the instrument (microbalance) may result if caution is not taken to properly operate and maintain the device. Follow the manufacturer's instructions for maintenance of all equipment and for safe operation.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement instrumentation.

6.2 General computer knowledge for interacting with the data management system.

6.3 Familiarity with electronic laboratory equipment is required.

7.0 Equipment

7.1 Laboratory coat

7.2 Analytical microbalance, minimum sensitivity to ±1µg, minimum repeatable to ±1µg

7.3 Balance table, with anti-static mat

7.4 ^{210}Po anti-static strips

7.5 Stainless steel, non-serrated forceps

7.6 Non-serrated, non-metallic forceps

7.7 Grounded floor mat

7.8 Working standards which bracket the expected filter mass. An upper standard (typically 500 mg) and lower standard (typically 200 mg)

7.9 46.2 mm diameter filters, Teflon membrane (PTFE)

7.10 New lot of Anti-static sealable bags

7.11 Anti-Static Bag Stability Test Form (test form)

8.0 Procedure

8.1 This procedure is performed in the lab.

8.2 Use only filters approved for the method of collecting particulate matter.

8.3 Confirm that the daily laboratory and equipment maintenance are complete (Standard Operating Procedure For Cleaning and Maintenance of Micro-gravimetric Laboratory).

8.4 Use filters not suitable for sampling. These filters will not affect the results of the anti-static bag stability test. The test does not require sampling on the filter, only weighing to determine weight gain or loss.

8.5 Select ten filters for the test and place them in Petri dishes labeled 1-10.

8.6 Carefully place the filters in sequential order in a covered, well ventilated tray, and leave in the environmentally controlled room to begin equilibration. Write the unique filter numbers on the test form and record the date and time. Also record the lab conditions on the form. Lab conditions are located on the computer software interface display.

8.7 Allow filters to equilibrate for the minimum requirement as determined by (Standard Operating Procedure For Performing A Filter Stability Evaluation (Lot Blank)). During the equilibration period, temperature must remain at 20-23 ˚C, with a variability of not more than ±2 ˚C over 24 hours and the relative humidity must remain at 30-40 % with a variability of not more than ±5 % over 24 hours. If these conditions have not been met, the filters must be properly equilibrated again.

8.8 Following equilibration, check the microbalance is reading zero, if needed, press the tare button.

8.9 Analyze the upper working standard. Use only non-serrated, non-metallic forceps to handle the working mass reference standard. Carefully place the standard on the balance pan and close chamber. Allow the reading to stabilize until the “mg” appears at the end of the reading, record the mass on the form. If the measured value of the working standard disagrees with the certified value by more than ±3 µg, remove the standard, zero the balance, inspect, clean and reweigh the working standard. If the two measurements still disagree by more than ±3 µg, contact supervisor. If necessary, verify the working standards (Standard Operating Procedure for the Verification of Working Mass Reference Standards).

8.10 Repeat step 8.8 and 8.9 with the lower working standard.

8.11 Check that the microbalance is reading zero when the pan is empty and the chamber door is closed, if not press the tare button on the balance display. Record zero reading on the form.

8.12 Locate the tray containing the equilibrated filters to be analyzed and place it on the anti-static mat adjacent to the balance.

8.13 Neutralize the electrostatic charge of each filter by placing it on a ^{210}Po anti-static strip for a nominal 60 seconds. Place the first filter in the weighing chamber and wait until the balance reading stabilizes and “mg” appears. Record the filter weight on the test form next to the filter number.

8.14 Return each filter to its uniquely identified Petri dish immediately following analysis.

8.15 Determine the weight of each filter, repeating steps 8.13 through 8.15. After the ten filters are weighed perform closing standards by repeating steps 8.8 – 8.11.

8.16 Load the filters into individually clean numbered cassettes. Randomly pick ten anti-static, sealable bags from the box being tested. Insert the cassette loaded with the filter into individual anti-static bag.

8.17 Leave the anti-static bags with filters in cassettes in the lab for 24 hours on a tray, for equilibration.

8.18 After the 24 hour equilibration period remove the first anti-static bag from the tray and take the filter out of the anti-static bag and cassette. Immediately determine its weight following steps 8.8 - 8.15 (skipping step 8.12)

8.19 Repeat the process of analyzing one (1) filter each working day until all ten (10) filters have been analyzed.

8.20 Determine the difference between the original weight obtained prior to loading into the cassettes and anti-static bags to those weights that were obtained after exposure to the anti-static bag. The pre- and post- weight determination must not differ by more than ±15 µg.

8.21 If the difference in the two weights exceeds the control limits, repeat the test using new bags and new filters. If the test fails a second time the box of Anti-static bags must not be used. Test each box and only use boxes of the anti-static bags that pass this test.

8.22 When the test is complete and all filters pass, the anti-static bags may be put into use.

9.0 Calculations

9.1 Net Difference = Original filter weight – measured weight after exposure to the anti-static bag

9.2 Difference = Observed mass – NIST certified mass

10.0 References

10.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

10.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10\text{-}2.5}$ in the Atmosphere

10.3 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program*; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.4 *Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods*; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.5 *Cahn Technical Notes: Static Control for Balances*; Jerry Weil, Cahn Instruments, Cerritos, California

11.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	0514/21	Document	• New template and new numbering system

SOP – GR-107-0.0

STANDARD OPERATING PROCEDURE FOR TRANSFER OF FILTERS FOR ANALYSIS

May 2021
IML AIR SCIENCE

REVISED BY:

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1.0 Scope and Application

1.1 The objective of this procedure is to transfer exposed filter samples to an off-site laboratory for additional analysis following gravimetric analysis and reporting.

1.2 This procedure applies to low volume and high-volume filter samples requiring additional analysis.

2.0 Summary of Method

2.1 Filters requiring additional analysis, such as speciation testing are prepared, transferred, and tracked during the additional analysis period.

3.0 Definitions

3.1 Data Management System (DMS) – Software systems to record, store and report data.

4.0 Health and Safety Warnings

4.1 General safety precautions related to transport of filters by vehicle or shipping service.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter and avoid filter damage or contamination.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained, familiar with the operation of environmental sampling and chain of custody documentation.

6.2 Skills necessary to perform work include, ensuring report is complete and chain of custody is correct.

7.0 Equipment

7.1 Exposed filters in pink anti-static bags or glassine envelopes

7.2 Data management system (DMS)

7.3 Secure container to protect filters

7.4 Chain of Custody forms (COC) or pack slip

8.0 Procedure

8.1 Following gravimetric analysis, filters identified by the client as needing additional analysis are returned to the closed Petri dish and anti-static bag or glassine envelope with corresponding filter number. The report is reviewed to make sure analysis and re-weighs have been performed and are correct. The report may be re-generated to determine correctness. The chain of custody is reviewed to assure the correct filters are taken and in order as listed on the chain of custody. Transfer occurs as soon as possible after reporting particulate matter concentrations is complete.

8.2 For the low volume exposed filters update tracking in the DMS by clicking on “move filters”, choose the appropriate network from the dropdown menu, select the current location from the “move samples from” dropdown menu, then select the filters to be moved, select the location to move the filters to from the “To” dropdown menu, and finally click “move” to move the filters in the DMS.

8.3 Filters are transported between locations by IML personnel or a shipping service. Filters are transported in secure containers to protect filters from damage. The original chain of custody (COC) form from the client or a revised COC is placed in the shipping container with the filters. At times a revised COC is more appropriate due to corrections to the original COC. If the original COC is not appropriate, a new COC is completed with client information, filter numbers of the samples to be analyzed, and the analysis requested. The COC form is signed and dated by the lab personnel who transferred/relinquished the filters.

8.4 A signed copy of the COC is retained and filed.

8.5 When analysis is completed, the filters are returned to the gravimetric lab with an accompanying COC signed and dated by the relinquishing and the receiving laboratory personnel. If the extraction procedure is destructive and the filter is partially destroyed, the remaining part of the filter is returned.

8.6 The filters are archived according to the Standard Operating Procedure for Archiving Filter Samples according to the low volume or high volume SOP.

8.7 For the low volume exposed filters update tracking in the DMS to document the filters were returned the gravimetric lab by following the same step 8.2.

9.0 References

9.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

9.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10\text{-}2.5}$ in the Atmosphere

9.3 *40 CFR 50, Appendix J;* Reference Method for the Determination of Particulate Matter as PM_{10} in the Atmosphere

9.4 *40 CFR 50, Appendix B;* Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High-Volume Method)

9.5 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program;* EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

9.6 *Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods;* EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

9.7 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Specific Methods;* EPA/600/4-77-027a; Section 2.11; January 1990; U.S. Environmental Protection Agency

10.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/1121	Document	• New template and new numbering system • Combined high volume and low volume SOP

SOP – GR-900-0.0

STANDARD OPERATING PROCEDURE FOR AIR DATA MANAGEMENT SYSTEM DEVELOPMENT, VALIDATION AND MODIFICATION

May 2021
IML AIR SCIENCE

REVISED BY:

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1.0 Scope and Application

1.1 Inter-Mountain Laboratory (IML) Air Science has developed a Data Management System (DMS) for tracking gravimetric filters, processing of raw data, calculating results, and dissemination of results to clients. This SOP describes the process for modifying the interface or data tables that may affect the calculation, assessment, or reporting for gravimetric results.

2.0 Summary of Method

2.1 IML Air Science employs an Agile Lean development methodology. Where possible, software unit tests are created to ensure that expected results are obtained. All mathematical calculations are automatically compared to the expected results when the test application is run. Once modification or new functionality is ready to be tested, code changes are recorded in IML's source control system with the software version number. After a new version is released, software is tested to ensure changes yield the intended results.

3.0 Definitions

3.1 Source Control –The management of changes to documents, computer programs, and other collections of information. Changes are identified as a revision. Each revision is associated with a timestamp and the person making the change. Revisions can be compared, restored, and with some types of files, merged.

3.2 Unit test – A software development process in which a testable part of an application is individually tested for proper.

4.0 Health and Safety Concerns

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacle and equipment must be properly grounded. Use caution when servicing or operating electrical equipment in wet conditions.

5.0 Personnel Qualifications

5.1 Persons performing this SOP must be familiar with Microsoft SQL Server, Microsoft .Net framework, and Microsoft Access.

6.0 Equipment

6.1 Server running Microsoft SQL Server.

6.2 Microsoft Visual Studio.

7.0 Procedure

7.1 When new functionality or modification to the DMS is identified, a request is made by creating an issue in IML's issue database. The requestor describes the issue, modification, or defect and sets the appropriate flag (immediate issue, high, medium or low).

7.2 Modifications to the DMS code or data tables are made and where appropriate unit tests are updated or created. Once modifications are ready for testing, the changes are recorded in source control and linked to the issue and revision. Then the issue status is set to "In Review" and notification is sent to the requestor to verify the changes.

7.3 Comments are added to the issue database, either to confirm that the modification gives the intended result or that further modifications are necessary. This continues until all the issues are resolved.

7.4 The issue is marked as resolved and closed.

8.0 References

8.1 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program*; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

8.2 *Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods*; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

8.3 *Good Automated Laboratory Practices: Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations with Implementation Guide*; EPA-220-B-95-006; August 1995; U.S. Environmental Protection Agency

9.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	5/2021	Document	New template and new numbering system.

SOP – GR-901-0.0

STANDARD OPERATING PROCEDURE FOR REPORT REVIEW

May 2021
IML AIR SCIENCE

REVISED BY:

Mary Hemmingsen 05/17/21
Date

APPROVED:

[signature] 5/17/2021
Manager Date

[signature] 5/17/2021
Quality Officer Date

Reviewed

Initials				
Date				

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1.0 Scope and Application

1.1 This procedure identifies the steps necessary to review air monitoring gravimetric reports. Information and steps to review filter data is outlined and checked.

2.0 Summary of Method

2.1 All gravimetric reports are thoroughly reviewed, verifying data and information for each filter before the final report is released to client.

3.0 Definitions

3.1 Data Management System (DMS) – Software systems to record, store and report data. Reports and Documents are tracked by versions for updates and changes.

3.2 Laboratory blank filter – New filters, that are used to determine laboratory contamination. The laboratory blank filters shall be weighed along with the pre-sampling (tare) weighing of each set of filters. These laboratory blank filters should remain in the laboratory in protective container during the field sampling and should be reweighed as a quality control check at gross weighing.

4.0 Health and Safety Concerns

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

5.0 Cautions

5.1 Use general precautions when operating a computer.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be familiar with the operation of the laboratory DMS.

7.0 Equipment

7.1 Computer with access to DMS

7.2 Computer with access to Electronic storage medium

7.3 Gravimetric Reporting Review Checklist form

7.4 Reports ready for review

7.5 Sample bag labels or images

7.6 Chain of Custody (COC)

7.7 Field data sheets

7.8 Sample instrument data

7.9 Sample Receipt form

8.0 Procedure

8.1 Weight Only Report Review

8.1.1 Open the report and checklist review files in the DMS. Check the client name and date range for the report to be reviewed.

8.1.2 Review bag labels, field data sheets, COC, and sample receipt form for any discrepancies or comments.

8.1.3 Review filters with comments concerning damage, holes, specks, smudges, etc. and take a photograph if necessary.

8.1.4 Review net masses and identify any high masses potentially over the standard for concentration. Check trip blanks and field blanks for acceptance criteria. If over the acceptance limit, inspect the filter if not already performed. Make sure the filter was reweighed.

8.1.5 “Weight only report” confirm number of filters listed on “Equilibration Times” tab match the number of filters received in the DMS accounting for voids and expired filters too.

8.1.6 Check the equilibration times for Tare and Gross positive values? If okay continue, if not investigate and correct.

8.1.7 Are the COC and Weigh Sessions scanned if required and stored in DMS in the appropriate location?

8.1.8 Check number of lab blanks is appropriate and net mass values within acceptance criteria.

8.1.9 Check replicates for tare and gross analysis net mass values with acceptance criteria.

8.1.10 Check Formatting – e.g. Print area is set correctly for each page or tab.

8.1.11 Data validation is completed by client as the report has only the net mass.

8.2 Concentrations Report Review (monthly and quarterly)

8.2.1 Report saved in correct location in DMS. Open the report and checklist review file and confirm the client name and date range for the report review is correct.

8.2.2 Using Air Data Processor program select menu “Export Utilities” then under Export Option select “Sample Information” pick client currently reviewing and then the date range for the report. Click on Export. An excel spreadsheet will open with bag label data and imported sampler data to review and compare.

8.2.3 Start checking data, the order of checks is not critical, but all checks must be completed.

8.2.4 Compare the bag label data with the sampler data. Sampler data all imported? Do filter numbers and dates match? Discrepancies will be highlighted in red. Each value highlighted in red will need to be investigated to determine which value is correct. Login errors are to be corrected.

8.2.5 Confirm run days are correct for each sampler. Investigate any wrong days or missing days. Find evidence the sampler ran on wrong day or missed day from bag label, field data sheets, COC and sampler data if available.

8.2.6 Review client comments on COC, sampler data and bag labels.

8.2.7 Check retrieval dates and times are correct.

8.2.8 Verify maximum arrival temperatures are correct for each shipment.

8.2.9 Field blanks and trip blanks on correct sample day and sampler.

8.2.10 Check for lab blanks within acceptance criteria and at least one lab blank for each gross weigh day.

8.2.11 Verify total number of filters and voids/expired for report to be billed. Does this count match the number on the equilibration page?

8.2.12 Average temperatures and pressures inputted for all valid samples with no sampler data.

8.2.13 Look at average temperatures and pressures are values similar to other samplers on same sample day?

8.2.14 Status codes and flag codes SP, SR, CI, XT, HT and more on the report, are samples that will be individually evaluated and potentiality invalidated with appropriate null code according to QA Handbook Volume II, Appendix D "PM2.5 Filter Based Local Conditions Validation Template" and client's direction.

8.2.15 QC Samples saved and complete. Check sample information table for yes in QCed column and Filter location.

8.2.16 Precision chart reviewed for proper scaling, range and missing values.

8.2.17 Collocated filters with difference between pair more than 5 µg/m^3 concentration investigate, inspect, review sampler data, reweigh filters. Look for evidence of why the filter concentrations do not match.

9.0 Calculations

9.1 Difference (net weight) = gross weight – tare weight

9.2 LTP Concentration (µg/m^3) = (net weight)/Va) x 1000

Where:

Net weight (net mass) in mg
Va = (m^3) local temperature and pressure conditions

9.3 STP Concentration (µg/m^3) = (net weight)/Vstd) x 1000

Where:

Net weight (net mass) in mg
Vstd = (m^3) standard temperature and pressure conditions

9.4 Standard Volume

$$V_{std} = V_a \times \frac{P_a \times T_{std}}{P_{std} \times (T_a + 273)}$$

Where:

Va – local volume (m^3)
Vstd – standard volume (m^3)
Pa – local pressure (mm Hg)
Pstd – standard pressure (760 mm Hg)
Ta – local temperature (K)
Tstd - standard temperature (298 K)

10.0 References

10.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

10.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10\text{-}2.5}$ in the Atmosphere

10.3 *Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program*; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.4 *Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods*; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

11.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21	Document	• New template and new numbering system

Appendix D: Forms

Microbalance External Calibration Form

Date	Tech	Balance SN:		Primary Standards Lower Standard	 Upper Standard	Calibration Standard
			Serial Number:			
			Certified Mass:			
			As Found:			
			As Left:			

Date	Tech	Balance SN:		Primary Standards Lower Standard	 Upper Standard	Calibration Standard
			Serial Number:			
			Certified Mass:			
			As Found:			
			As Left:			

Date	Tech	Balance SN:		Primary Standards Lower Standard	 Upper Standard	Calibration Standard
			Serial Number:			
			Certified Mass:			
			As Found:			
			As Left:			

Date	Tech	Balance SN:		Primary Standards Lower Standard	 Upper Standard	Calibration Standard
			Serial Number:			
			Certified Mass:			
			As Found:			
			As Left:			

Date	Tech	Balance SN:		Primary Standards Lower Standard	 Upper Standard	Calibration Standard
			Serial Number:			
			Certified Mass:			
			As Found:			
			As Left:			

Date	Tech	Balance SN:		Primary Standards Lower Standard	 Upper Standard	Calibration Standard
			Serial Number:			
			Certified Mass:			
			As Found:			
			As Left:			

Date	Tech	Balance SN:		Primary Standards Lower Standard	 Upper Standard	Calibration Standard
			Serial Number:			
			Certified Mass:			
			As Found:			
			As Left:			

Filter Lot Login Sheet

Network	Date Received	Lot #	Filter # 's	Network Notification Date	# of Boxes Rec'd

Pace Analytical

Sheridan, WY and Gillette, WY

- CHAIN OF CUSTODY RECORD -

All shaded fields must be completed.

This is a legal document; any misrepresentation may be construed as fraud.

#WEB

Client Name	Project Identification	Sampler (Signature/Attestation of Authenticity)	Telephone #

Report Address	Contact Name	Email	Phone

Invoice Address	Purchase Order #	Quote #

ANALYSES / PARAMETERS

REMARKS

ITEM	LAB ID (Lab Use Only)	DATE SAMPLED	TIME SAMPLED	SAMPLE IDENTIFICATION	Matrix	# of Containers									
1															
2															
3															
4															
5															
6															
7															
8															
9															
10															
11															
12															
13															
14															

LAB COMMENTS	Relinquished By (Signature/Printed)	DATE	TIME	Received By (Signature/Printed)	DATE	TIME

SHIPPING INFO

- ❑ UPS
- ❑ FedEx
- ❑ USPS
- ❑ Hand Carried
- ❑ Other_____

MATRIX CODES

Water	WT
Soil	SL
Solid	SD
Filter	FT
Other	OT

TURN AROUND TIMES

Check desired service

- ❑ Standard turnaround
- ❑ **RUSH - 5 Working Days**
- ❑ **URGENT - < 2 Working Days**

Rush & Urgent Surcharges will be applied

COMPLIANCE INFORMATION

Compliance Monitoring ? Y / N

Program (SDWA, NPDES,…)

PWSID / Permit #

Chlorinated? Y / N

Sample Disposal: Lab Client

ADDITIONAL REMARKS

PM 2.5 TARE EQUILIBRATION

For shipment date _____
Equilibration date _____
Equilibration time _____

CLIENT	# Filters	Total
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		
21		
22		
23		
24		
25		
26		
27		
28		
29		
30		
31		
32		

For shipment date _____
Equilibration date _____
Equilibration time _____

CLIENT	# Filters	Total
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		
21		
22		
23		
24		
25		
26		
27		
28		
29		
30		
31		
32		

Working Standard Mass Verification

Lab __ - Balance __

Date: _____

Analyst: _____

Balance

Balance Make:	
Balance Model:	
Balance Serial Number:	

Working Standard

Nominal Mass (N_w):	
Serial Number:	
Certified Conventional Mass (C_w):	

Primary Standard

Nominal Mass (N_p):	
Serial Number:	
Certified Conventional Mass (C_p):	

Observations

O_1 Working Standard:		mg
O_2 Primary Standard:		mg
O_3 Primary Standard:		mg
O_4 Working Standard:		mg
Apparent Mass Correction:		mg
Difference:		µg

must be within ±2µg

$$Apparent\ mass\ correction = C_p + \frac{O_1 - O_2 + O_4 - O_3}{2} + N_p - N_w$$

Difference = (C_w^* - Apparent Mass Correction) x 1000

* Working and Primary Conventional mass values use the number of decimal places displayed

Appendix E: Revisions

Revision Number	Date	Section Number	Description of Changes
15	12/2020	A3	Updated recipients as needed.
15	12/2020	A4.1	Revised roles and responsibilities.
15	12/2020	Figure A4.1-1	Revised Organization Chart.
15	12/2020	A6	Added detail to project/task description.
15	12/2020	A6.1	Removed control limits for temperature and relative humidity and referenced table containing control limits for temperature and relative humidity.
15	12/2020	Table A6-1	Added detail.
15	5/2021	A7	Added lower and upper concentration limits.
15	12/2020	A7.2	Added detail and improved readability by restructuring sentences.
15	12/2020	A.7.4	Added detail to precision.
15	12/2020	A.7.4.2	Included PM_{10} networks in discussion.
15	12/2020	A8	Revised training section to match Quality Management Plan.
15	12/2020	A9.1	Replaced hardcopy records with paper record and added reference to the Control of Records SOP and Document Control SOP and described where documents are available.
15	12/2020	A9.1.1	Replaced laboratory information system with data management system.
15	12/2020	Table A9-1	Removed lab blank out of specification notebook.
15	12/2020	A9.1.2	Revised notebook names to match notebooks. Added reference to forms appendix and SOP appendix.
15	12/2020	Table A9-2	Revised frequencies and added equilibration times and replicate report.
15	12/2020	Table A9-3	Revised to match reports and removed sample validation and qualification report.
15	12/2020	A9.2.3	Added how reports that do not contain a title page are revised.
15	12/2020	A9.3	Revised retention of records time frame.
15	12/2020	B3.1	Referenced Appendix D for various forms.

Revision Number	Date	Section Number	Description of Changes
15	12/2020	B3.2	Added detail.
15	12/2020	B3.3	Added detail.
15	12/2020	B3.4	Moved sample collection section ahead of exposed sample filter collection.
15	12/2020	B3.6	Added detail and operation of min/max thermometer.
15	12/2020	B3.7	Revised temperature monitoring description.
15	12/2020	B3.8	Revised receipt of exposed sample filters. Removed detail described in the SOP and added reference to SOP.
15	12/2020	B3.9	Revised archiving of exposed sample filters. Removed detail described in the SOP and added reference to SOP.
15	12/2020	B4	Added detail to analytical methods and removed list of references.
15	5/2021	Table B-1	Added table of Gravimetric Laboratory SOPs.
15	12/2020	B4.1	Revised clean filters. Removed detail described in the SOP and added reference to SOP.
15	12/2020	B4.2	Revised preparation of pre-exposure filters. Removed detail described in the SOP and added reference to SOP.
15	12/2020	Figure B4.2-1	Revised flow diagram.
15	12/2020	B4.4	Added reference to flow diagram for pre-exposure of clean filters which identifies equipment or instrumentation needed. Referenced quality control criteria. Referenced archiving of exposed filters.
15	12/2020	B4.5	Removed references to pre-exposure and receipt of exposed sample filters. Referenced flow diagram to identify equipment or instrumentation needed.
15	12/2020	Figure B4.3-1	Revised flow diagram.
15	12/2020	Figure B4.3-2	Revised flow diagram.
15	12/2020	Figure B4.3-3	Revised flow diagram.
15	12/2020	B5.1	Added detail.
15	12/2020	Table B5-1	Updated control limits.

Revision Number	Date	Section Number	Description of Changes
15	12/2020	B5.3	Revised microbalance checks. Removed detail and added reference to SOP.
15	12/2020	B5.4	Added primary and working mass standards.
15	12/2020	B5.5	Revised lot blanks. Removed detail and added reference to SOP.
15	12/2020	B5.6	Added detail.
15	12/2020	B5.7	Removed redundant detail and added reference to corrective action.
15	12/2020	B5.8	Added reference to corrective action.
15	12/2020	B5.9	Clarified “weights only” reports from full reports.
15	12/2020	B6.3	Removed control limits and replaced with reference to table with control limits.
15	12/2020	B6.4	Removed instrument specifications and replaced with reference to table containing specifications.
15	12/2020	B6.5	Replaced laboratory information system with data management system.
15	12/2020	B6.7	Added detail.
15	12/2020	B6.7.1	Defined mass reference standards and replaced ultra class with ASTM classes.
15	12/2020	B6.7.2	Removed mercury from NIST traceable thermometer. Removed specifications and referenced table with specifications.
15	12/2020	B6.7.3	Removed specifications and referenced table with specifications.
15	12/2020	B6.10	Added filter cassettes to instrument/equipment testing, inspection, and maintenance section.
15	12/2020	B6.11	Added shipping containers to instrument/equipment testing, inspection, and maintenance section.
15	12/2020	B6.12	Added min/max thermometers to instrument/equipment testing, inspection, and maintenance section.
15	12/2020	B6.13	Added infra-red thermometers to instrument/equipment testing, inspection, and maintenance section.
15	12/2020	Table B6-1	Added table for instrument specifications.

Revision Number	Date	Section Number	Description of Changes
15	12/2020	B7	Removed laboratory, static control, filter cassettes, and shipping containers from instrument/equipment calibration and frequency section.
15	12/2020	B7.1	Removed cleaning of the balance detail.
15	12/2020	B7.2	Removed detail and referenced SOP.
15	12/2020	B7.3	Added min/max thermometers.
15	12/2020	B7.4	Added infra-red thermometer.
15	12/2020	B7.5	Removed specifications and referenced table containing specifications.
15	12/2020	B8	Revised inspection/acceptance of supplies and consumables. Removed detail and added reference to SOP.
15	12/2020	B10.1	Removed acceptance criteria for relative humidity and temperature. Removed redundant paragraphs.
15	12/2020	Equation B10.1-1	Added equation name.
15	12/2020	C1	Added QAPP review responsibility and location of audit SOP and corrective action SOP.
15	12/2020	C2	Added staff to conductance of Management Review.
15	12/2020	D1	Added purpose of section.
15	12/2020	Equation D2.1-1	Added title of equation.
15	12/2020	Appendix A	Added one reference.
15	12/2020	Document	Consistent use of Pre-exposure filter, exposed filter, clean filter, lab or conditioning environment.
15	12/2020	Document	Spelling, grammar, and formatting.

Appendix F: Annual Review

Date	Initials	Printed Name

SOP - GR-112-1.0

STANDARD OPERATING PROCEDURE FOR RECEIPT AND LOGIN OF EXPOSED PARTICULATE FILTER SAMPLES

June 2022
IML AIR SCIENCE

REVISED BY:

Kayley Smith — 6/2/22
Date

APPROVED:

[signature] — 6/2/2022
Manager — Date

[signature] — 6/2/2022
Quality Officer — Date

Table of Contents

1.0 Scope and Application

1.1 This procedure is used to receive coolers with exposed filters, measure and record interior cooler temperature upon arrival of filters, verify chain of custody information and sample run data, prepare exposed filters for post-exposure gravimetric analysis, equilibrate, and complete exposed filter login.

1.2 The elements of this SOP are applicable to filters for all low-volume samplers.

2.0 Summary of Method

2.1 Exposed filter samples are received from a network location typically in a cooler. Filter temperature in cooler is measured immediately and filters are removed. Filters are received into the DMS by scanning the bag label bar code and then are prepared and placed into the equilibration environment. The filter IDs are checked against the Chain of Custody to make sure the filters listed on the COC match. All pertinent data is logged into the DMS.

3.0 Definitions

3.1 Cassette - A device supplied with samplers to allow a weighed filter to be held in place in the sampler and manipulated before and after sampling without touching the filter, and to minimize damage to the filter and/or sample during such activities.

3.2 Lab - A room containing the microbalance designed to maintain the filter conditioning requirements for temperature and humidity.

3.3 Data Management System (DMS) – Software systems to record, store and report data.

3.4 Anteroom – A room with limited access and no air flow located adjacent to the lab, for unloading cassettes, storing Petri dishes, and clean cassettes.

3.5 Laboratory blank filter – New filters that are used as a quality control check to determine laboratory contamination.

4.0 Health and Safety Concerns

4.1 General safety precautions related to electrical hazards must be observed at all times when working with electronic equipment. Electrical receptacles and equipment must be properly grounded.

5.0 Cautions

5.1 In all steps involving filter handling, be extremely careful to avoid any gain or loss of particulate matter and avoid filter damage. Wear laboratory coat when

handling filters in lab and anteroom. Keep filter Petri dishes protected and in a horizontal position during transport.

5.2 Handle filters with stainless steel non-serrated forceps by the support ring only.

6.0 Personnel Qualifications

6.1 Persons performing this SOP must be trained and familiar with the operation of environmental measurement instrumentation.

6.2 Familiarity with electronic laboratory equipment is required.

7.0 Equipment

7.1 Clean, environmentally controlled room

7.2 Laboratory coat

7.3 Auto-retracting blade utility knife

7.4 Cut resistant glove

7.5 Insulated shipping container (cooler)

7.6 Minimum/maximum thermometer

7.7 Infrared thermometer gun, verified to NIST traceable thermometer

7.8 Completed Chain of Custody documents (COC)

7.9 Cassette separation tool

7.10 Petri dishes with labels

7.11 Stainless steel, non-serrated forceps

7.12 46.2 mm diameter filters, Teflon membrane (PTFE)

7.13 Lint-free alcohol wipes

7.14 Computer with access to DMS

8.0 Procedure

8.1 Open cooler and immediately determine the receipt, maximum, and minimum temperatures of the filters and record as indicated by the min/max thermometer. If it's apparent that the min/max thermometer was not reset prior to shipment, use the infrared thermometer gun to determine the receipt temperature; if receipt

temperature of the min/max thermometer is above 4°C, use the infrared thermometer gun to verify current temperature. If a thermometer has not accompanied the filter samples in shipment, use the infrared thermometer gun when network clients approve substitution.

> *To obtain the temperature with the infrared thermometer gun, open cooler and immediately point the gun into the cooler at the filter samples with the trigger held down watching the digital read out until it stabilizes, typically a few seconds. Record the temperature reading to one decimal place. If the reading is unstable, check and replace the batteries in the thermometer gun. Current certification stickers are placed on the IR thermometer following each new calibration.*

8.2 If included, retrieve Chain of Custody and/or sampler collection data (field sheets) for the filter samples received. Record the cooler temperature and cooler number on the Chain of Custody. Document the condition of the ice packs on the bottom of the COC. Sign the COC and record the date and time.

8.3 Remove the filter sample anti-static bags from the waterproof bag(s).

8.4 In the DMS under “Receive Samples,” fill out all information on the screen for the relevant fields. When entering receipt temperature obtained from the infrared thermometer gun, be sure to enter the truncated, whole number for temperature reading.

8.5 Filters are received into the DMS by scanning each bar code on the filter anti-static bag labels; save and print sample receipt form.

8.6 All filter activities are tracked in the DMS. After receipt, move received filter samples to Lab for Gross, or Office Cooler as necessitated from Laboratory Schedule, by pressing “select all” and “move.”

8.7 Match the filter IDs on the anti-static bags against the Chain of Custody to assure the filters listed on the COC are received. Note any discrepancies on the sample receipt form.

8.8 Take filter samples to the anteroom where Petri dishes are stored. Retrieve Petri dishes assigned to the filter samples received. Retrieve ten percent laboratory blanks with the same pre-exposure date as the exposed samples. To minimize the amount of volatilization, exposed samples should not be left to equilibrate over extended periods (weekends, holidays etc.). If it is Friday or the day before a holiday, place exposed samples in cold storage at <4 ˚C. Stack closed Petri dishes in trays in sequential order before placing in the Office Cooler. Lab blanks remain in closed Petri dishes in the lab.

8.9 For filter samples to be exposed, arrange Petri dishes in a well-ventilated tray with the laboratory blank(s) at the beginning of the group, placing the Petri dish

lid underneath the dish with the filter ID visible, typically in sequential order of filter ID. On the first anti-static bag, place a sticker at the top and write the lab blank ID number(s).

8.10 Clean stainless steel, non-serrated forceps and cassette separation tool with lint-free alcohol wipe. Allow to air dry.

8.11 Remove samples from their protective anti-static bag, noting that the cassette ID on the anti-static bag matches the cassette ID on the cassette in the bag. If the cassette in the bag does not match the cassette ID written on the bag label, make a note on the sample receipt form and contact the supervisor.

8.12 Using a cassette separation tool, open the cassette and carefully remove the filter using stainless steel, non-serrated forceps, contacting the filter's support ring only. Verify the filter ID on the bag label matches the number on the Petri dish lid and the number stamped on the filter itself. Place the filter in the Petri dish. Check anti-static bags for any particulate matter and carefully add any material onto the filter. Note sample condition on receipt form. Continue unloading each filter according to steps 8.11 and 8.12. Inspect filters while unloading from cassettes into the Petri dishes. Record any defects on the sample receipt form.

8.13 Verify that the laboratory conditions are within specifications by viewing Lab Conditions interface on the computer screen or lab status open on the desktop of the computer in the laboratory. If the lab conditions have not been met, the DMS will alert you. If the lab conditions are out of specifications, take corrective actions to ensure that they return to specifications. Corrective action may include repair or replacement of environmental measurement and control components. Store exposed filter samples at <4˚C until the laboratory conditions respond to the corrective action and return to specifications.

8.14 Take trays of filters into lab.

8.15 In the DMS, click equilibration button. Select exposed filters and add lab blank ID numbers by clicking on the "magnifying glass"  in the upper righthand corner of the screen. Input the lab blank ID number and save, repeating this step for each lab blank. Once the lab blanks have been selected, save the equilibration batch by clicking on the save button in the lower righthand corner of the screen.

8.16 Review the COC and sample receipt form with the anti-static bag labels to make sure everything matches, and any issues are fully documented. Place in tray for final review by supervisor.

8.17 After equilibration is initiated, log the samples into the DMS by going to exposed login for the date received; select a client and begin. The scanner will

read the barcode and save the image of the bag label. Images are not saved for all clients. Log in varies by client. Fill in the information on the screen for each filter.

8.18 The supervisor will notify the network operator of any unusual situations (cooler temperature out of specification, missing samples, etc.) in a timely fashion.

9.0 References

9.1 *40 CFR 50, Appendix L*; Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere

9.2 *40 CFR 50, Appendix O*; Reference Method for the Determination of Coarse Particulate Matter as $PM_{10\text{-}2.5}$ in the Atmosphere

9.3 Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II. Ambient Air Quality Monitoring Program; EPA-454/B-17-001; January 2017; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

9.4 Quality Assurance Guidance Document 2.12; Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods; EPA-454/B-16-001; January 2016; U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards.

10.0 Revision Record

Revision #	Date	Section	Description of Changes
0.0	05/14/21		New template and new numbering system
1.0	06/2022	8.1	Removed use of correction factor for IR gun; updated temperature receipt and input requirements
1.0	06/2022	8.0	Rearranged items in procedure
1.0	06/2022	3.0	Added/updated definition for “Laboratory blank filter” and updated “Lab” definition

Quality Manual

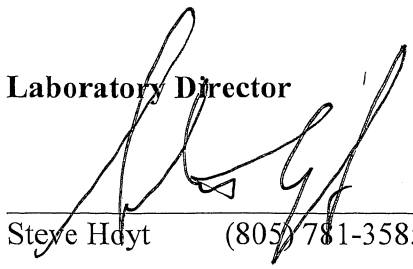
ENVIRONMENTAL
Analytical Service, Inc.

The Air Measurement Specialists

173 Cross Street
San Luis Obispo, California 93401
Phone: (805) 781-3585
www.easlab.com

Quality Manual
Environmental Analytical Service
Approvals Page

Laboratory Director



Steve Hoyt (805) 781-3585

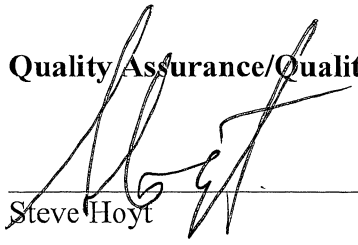
1/5/22
Date

Technical Director

Steve Hoyt

1/5/22
Date

Quality Assurance/Quality Manager



Steve Hoyt

1/5/22
Date

Laboratory Manager

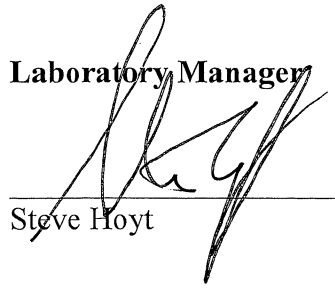


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Table of Contents

1.0 INTRODUCTION

The Quality Manual for Environmental Analytical Service, Inc. (EAS) is the document that describes the quality system and the process by which EAS conducts its activities in order to provide our clients with data of known and documented quality. The quality system documents the process by which appropriate analytical methods are selected, their capability is evaluated, and their performance is documented. The EAS Quality Manual meets the requirements for NELAC Chapter 5, 2009 and is in the process of being updated to 2016.

1.1 Scope of Testing

Environmental Analytical Service, Inc. is a Special Analytical Services Laboratory providing air and gas testing services to consulting engineers, industrial clients, commercial laboratories, and regulatory agencies. EAS specializes in the analysis of ambient air, indoor air, source test samples, gas samples, and product testing for organic compounds and inorganic gases. EAS provides analytical testing services for environmental samples, product testing, agricultural testing, and for research projects.

Environmental Testing

The Environmental testing methods and QC are based on Environmental Protection Agency (EPA), American Society for Testing of Materials (ASTM), and California Air Resources Board (CARB) methods. The Quality Systems used are based on NELAC Chapter 5 standards. The Laboratory Manager oversees the analytical testing, the Quality Manager monitors the data quality, and the Laboratory Director reviews the analytical report.

Product Testing

The product testing methods used by EAS are based on ASTM methods and other related methods. The Project Manager supervises the analytical testing, the Quality Manager monitors the data quality, the Laboratory Director reviews the analytical report, and the final report is prepared under the direction of the Technical Director.

Agricultural Testing

Agricultural testing methods are based on EPA and ASTM test methods. The Quality System is based on EPA GLP systems. The Project Manager supervises the analytical testing, the Quality Manager monitors the data quality, and the Technical Director reviews the analytical report.

Research Projects

Under the direction of Dr. Steven Hoyt, the Technical Director, EAS provides analytical

services, consulting, and report preparation for research projects in the environmental, product testing and agricultural areas. For research projects, a Study Director is assigned to the project. The Study Director and the Technical Director design the project, and the Study Director and their staff execute the project. The Quality Manager monitors the data quality, and the Study Director and the Technical Director prepare the final report.

2.0 ORGANIZATIONAL ROLES AND RESPONSIBILITIES

Environmental Analytical Service, Inc. (EAS) is a private air-testing laboratory, and has one facility that is located in San Luis Obispo, California. EAS is a Type C Corporation, registered in California and is an entity that is legally responsible. There is a Board of Directors that is responsible for the overall direction of the laboratory. EAS is not part of any other organization.

2.1 Management System

The management at EAS has developed a management system that is designed to cover the administrative, quality, and technical operations of the laboratory. EAS has defined and documented its policies, systems, programs, and procedures necessary to assure the quality of its operations.

The Laboratory Mission Statement and the Quality Policy Statement developed by the Board of Directors is described in Section 3.1. The implementation of the Quality Policy Statement into procedures is the responsibility of the top management, which includes the Administrative Director, the Laboratory Director, and the Technical Director. The implementation of the tasks and procedures is the responsibility of the Quality Manager, Project Director, Laboratory Manager, individual technicians and support personnel.

2.2 Laboratory Organization

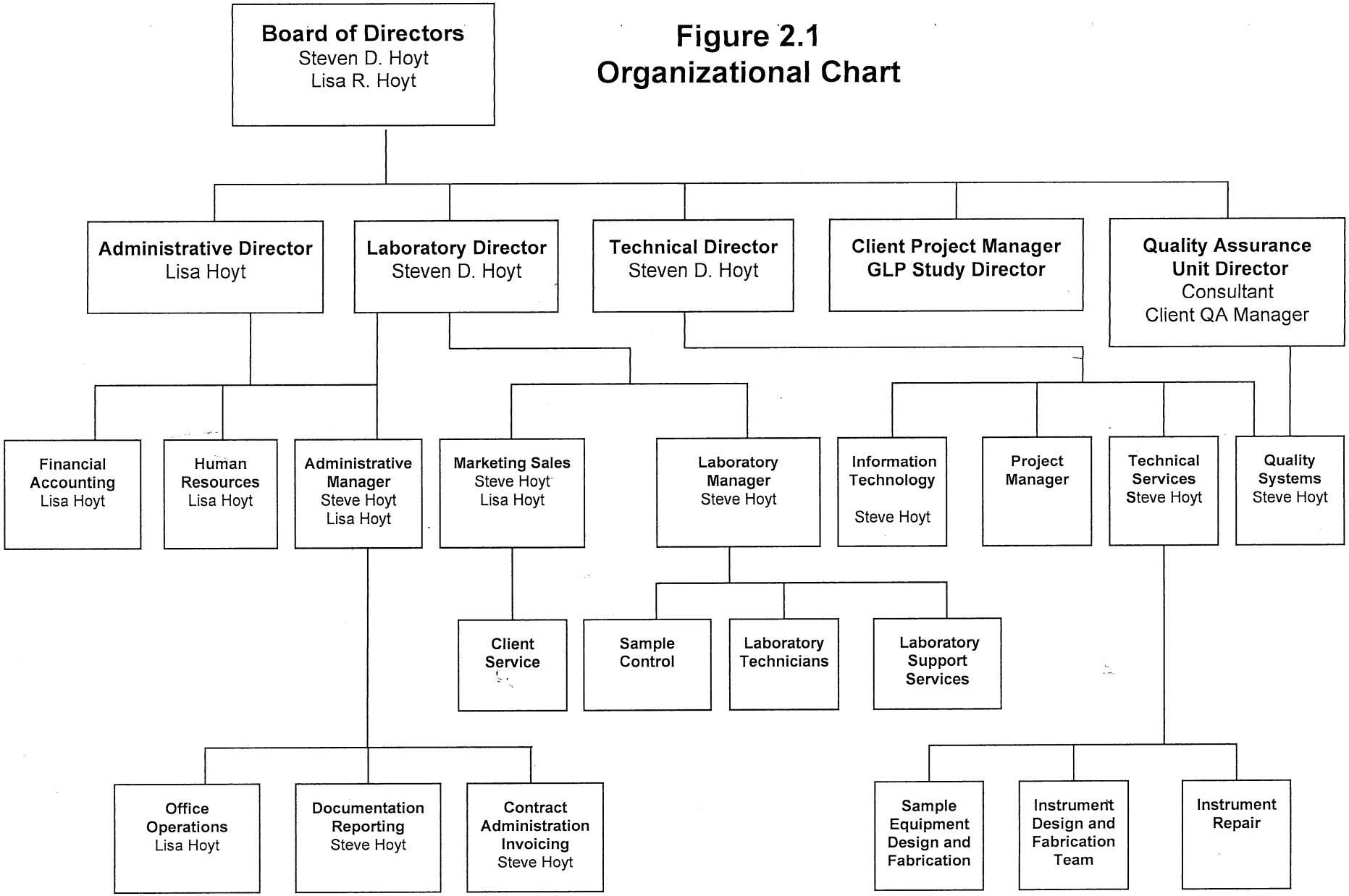
Environmental Analytical Service is divided up into Management Systems or Units, which are listed in Table 2.1. The organizational chart graphically showing the management structure and reporting structure for EAS is shown in Figure 2.1. EAS is an independent corporation with a board of directors, which sets the corporate policy. EAS is a laboratory that provides analytical services to other companies (or individuals), and the companies often have a project manager or study director that specifies and verifies the project requirements, which is independent of the EAS management. The companies also have a Quality Assurance Director for the project who is independent from the other management. The organizational chart shows how the Client Project Manager or Study Director and Quality Assurance Unit interact with the EAS management.

Below the Board of Directors is the upper management of EAS. This consists of the Administrative Director, Laboratory Director, and Technical Director. Because of the size of EAS the Laboratory Director Technical Director, and Quality Manager are the same person. They are defined as three separate positions to clearly define their duties and responsibilities. The upper management is responsible for implementing the policy established by the Board of Directors, and the policies dictated by the clients. The upper management has defined a management system based on operational units that are headed by a unit manager. The units are defined in a manner that provides independence to the key QC units; so the person analyzing samples is different than the QC person.

Table 2.1
Management Systems or Areas

Position	Operational Unit	Reports To
Board Member	Board of Directors (BOD)	
Upper Management		
Administrative Director	Administration	BOD
Laboratory Director	Administration	BOD
Technical Director	Administration	BOD
Client Project Manager GLP Study Director	Client	Client
Client Quality Assurance Director	Client	Client
Management		
Financial Manager	Financial	Administrative Director
Human Resources Manager	Human Resources	Administrative Director
Administrative Manager	Administrative	Administrative Director
IT Manager	Information Technology	Technical Director
Quality Manager	Quality	Technical Director
Technical Services Manager	Technical Services	Technical Director
Project Manager	Project	Technical Director
Marketing Manager	Marketing and Sales	Laboratory Director
Laboratory Manager	Laboratory	Laboratory Director
Personnel		
Client Services	Marketing and Sales	Marketing Manager
Sample Control	Laboratory	Laboratory Manager
Laboratory Technician II	Laboratory	Laboratory Manager
Laboratory Technician I	Laboratory	Laboratory Manager
Lab Support Personnel	Laboratory	Laboratory Manager

Figure 2.1
Organizational Chart



2.3 Job Descriptions and Quality Responsibility

Each employee at EAS has a documented job description in the Administrative Manual, Section AM2 on Management Systems. Organizational Chart (Table 2.1) also outlines the quality system reporting relationship of each position in the organization.

Impartiality

The Laboratory Director is impartial for the following reasons: 1) The Laboratory Director is fully committed to quality data and understands that the very existence of the laboratory is based on data quality, 2) The Laboratory Director does not analyze samples except in special circumstances, and all sample data and QC data is reviewed and evaluated by the Laboratory Manager, 3) All reports and QC are also reviewed by the Laboratory Director. The American Association for Laboratory Accreditation (A2LA) states in their book "General Requirements for Accreditation of Laboratories" that (for some laboratories, probably based on size) the Quality Manager may also be the same person as the Laboratory Director. This is also described in NELAC Chapter 5.

The Quality Manager can evaluate data outside of management influence because the Quality Manager does not run samples. The Laboratory Manager or Principal Analyst for the instrument is responsible for the operation, calibration, and determination that the instrument meets the QC Criteria. They sign a Daily Analytical Batch (DAB) form indicating whether the QC criteria for the method has been met. Discussions about data quality are made jointly between the Quality Manager and the Laboratory Supervisor and any decision has to be unanimously.

2.3.1 Board of Directors

The Board of Directors establishes the corporate policies, goals and objectives for the business and management systems necessary to run the business.

2.3.2 Client Project Manager – GLP Study Director

The Client Project Manager or GLP Study Director manages the project or study for the client and provides the EAS Technical Director with a Quality Assurance Project Plan (QAPP) or Master Schedule defining the project. After accepting a project, the EAS Laboratory Director and Technical Director will follow the QAPP or Master Schedule and take direction from the Client Project Manager or GLP Study Director. They have overall responsibility for the technical conduct, protocol and protocol changes, documentation, and reporting of results.

2.3.3 Administrative Director

The Administrative Director is responsible for the implementation of board policies for the financial, human resources, and administrative operations of EAS.

2.3.4 Laboratory Director

The Laboratory Director is responsible for the implementation of policies and procedures in support of the Board of Directors. The Laboratory Director controls the laboratory performance to make sure it achieves the corporate objectives. The Laboratory Director determines what analytical tests will be conducted by EAS, and, in conjunction with the Laboratory Manager, the volume of samples that can be analyzed based on the laboratory capacity. The Laboratory Director also maintains a cooperative and safe working environment that contributes to the overall laboratory quality.

2.3.5 Technical Director

The Technical Director is responsible for technical operations of the laboratory. They have an advanced degree in chemistry or a related field, and experience with analytical methods. The Technical Director provides technical support for projects and sample analysis. They are responsible for the initial design, validation, and quality control criteria for each of the analytical methods. The Technical Director provides technical support on instrument design, maintenance, repair, and troubleshooting.

The Technical Director works with the Client Project Manager or Study Director to make sure that the analytical methods used by EAS will meet the objectives of the project, and will recommend analytical solutions for the project requirements. The Technical Director also works with the Quality Assurance Unit Director to define the quality objectives of the project. The Technical Director communicates the project quality objectives to the EAS Quality Manager, Project Manager, and Laboratory Manager

The Technical Director conducts training programs for the laboratory technicians on the theory and operations of equipment and methods. He supervises the preparation of standards, laboratory audit samples, QC samples and any NIST traceable equipment. In the absence of the Technical Director, the Lab Manager will act as the Technical Director.

2.3.6 Quality Assurance Unit Director

The Quality Assurance Unit Director is a person that is independent of the personnel engaged in the direction and conduct of the project or study. This person is not an EAS employee. They are an independent consultant provided by the Study Director (for GLP projects), a person from the clients quality unit, or an independent data reviewer hired by the project manager. This person assures that the facilities, equipment, methods, QC controls, and records comply with the QAPP or Master Schedule. Any problems, which affect the project integrity, are communicated to the EAS management through the EAS Technical Director.

2.3.7 Quality Manager

The EAS Quality Manager oversees the development the EAS Quality System and its implementation. The Quality Manager has documented training in statistics and over 10 years experience with QA/QC procedures, and has read and is knowledgeable of the NELAC Quality Systems, and EPA GLP procedures. The Quality Manager is responsible for preparing and updating the Laboratory Quality Manual and has full knowledge of the analytical test methods and Standard Operating Procedures. The Quality Manager is also responsible for the oversight and review of QC data and implementing corrective actions when criteria are not met. The Quality Manager notifies the Board of Directors of any quality system deficiencies. He also oversees and maintains records of proficiency tests for laboratory technicians. In the absence of the Quality Manager, the Laboratory Manager can provide this information.

2.3.8 Administrative Manager

The Administrative Manager is responsible for office operations, contract administration, purchasing, invoicing, document management, and report management. The Administrative Manager has an AA degree in business, and experience with contracts and document control. They work with the Administrative Director on human resources, employee and training files, and bookkeeping. They are responsible for the administrative end of the Health and Safety Program.

2.3.9 Technical Services Manager

The Technical Services Manager has experience in scientific instrumentation fabrication and electronics. They are responsible for instrument repair, instrument design and fabrication, configuration of instrumentation for methods, and sample equipment fabrication. The Technical Services Manager works under the Technical Director who provides the technical expertise for method development, and instrumentation design.

2.3.10 Information Technology Manager

The Information Technology Manager has computer experience and is responsible for the EAS Computer System. They design and maintain the computer network, information servers, Laboratory Information System, computer workstations, and backup server.

2.3.11 Laboratory Manager/Supervisor

The Laboratory Manager/Supervisor schedules sample analysis in the laboratory, and ensures that all samples are analyzed according to the Standard Operating Procedures, and that they meet the method QC Criteria. They communicate to the Laboratory Director the laboratory sample load and maintain sample load consistent with the Quality Program.

The Laboratory Manager/Supervisor reviews the Daily Analytical Batches (DAB) from each instrument, verifies that each DAB meets the QC criteria established for the project, and prepares or reviews the analytical reports. The Laboratory Manager/Supervisor meets with the Laboratory Director, Quality Manager, Project Managers, and Laboratory Technicians to make sure all project requirements are met and sample analysis is on schedule. They are responsible for the technical end of the Health and Safety Program.

2.3.12 Project Manager

The Project Manager is responsible for design, management, technical communication and completion of assigned projects. Project Managers are assigned based on their education, experience, and the complexity of the project. Projects can range from a few samples that only require an Analytical Report to the testing of products that require the preparation of a Project Plan and Schedule. The Project Manager makes sure that lab personnel are aware of any special requirements for a project such as special target lists, QC, reports or electronic deliverables. The Project Manager is responsible for contacting the clients concerning technical questions. The Project Manager is responsible for generating, reviewing, and writing QC comments for the final report.

2.3.13 Laboratory Technician I and II

The Laboratory Technician's responsibilities include the routine daily startup and shutdown for the instruments, and the analysis of the Daily Analytical Batch (DAB) QC samples which include standards, quality control samples, and blanks. The Laboratory Technician records all samples analyzed and instrument conditions in the DAB worksheet. The technician performs initial checks on the validity of the data, and records any deviations on the DAB form.

2.3.14 Sample Control

Sample Control is responsible for the reception and logging in of samples into the Laboratory Information Management System (LIMS) system. Sample Control assigns each batch of samples a unique Sample Delivery Group (SDG) number and creates an SDG file for each batch. They also fill out the Quotation sheets for billing and generate the Laboratory Worksheets for review by the Project Manager. Sample Control faxes or emails the client a copy of the Sample Receipt Notification that includes the signed Chain of Custody. Sample Control verifies the integrity of the samples when they are received and maintains custody over the samples.

2.3.15 Laboratory Support Personnel

The Laboratory Support Personnel have a scientific background and also work as Laboratory Technicians. The function of this position is to provide support services to the laboratory personnel. They clean canisters, clean and adjust flow controllers, prepare sorbent media for shipment, clean and prepare solutions for impingers. They enter

cleaning batch information into LIMS, prepare packing slips, and box sample equipment for shipment to the client.

2.3.16 Client Services

The Client Services person is responsible for keeping client information updated in the LIMS system, taking calls from clients for sample equipment orders on existing projects, providing pricing information, and sending materials to clients. They will contact clients with updated information on the report delivery schedule, requests for information, and other non-technical project information.

3.0 QUALITY SYSTEMS

The Laboratory Quality System includes all quality assurance (QA) policies and quality control (QC) procedures. The Quality Manual documents these policies and procedures for Environmental Analytical Service. For Environmental Projects that have a Quality Assurance Project Plan (QAPP) and for EPA GLP Projects, there will be separate documentation that defines the quality assurance necessary for that project. There will also be a separate QC person, who is not an employee that will review the data and reports submitted by EAS. A written status report for the project, noting any problems with the project data or QC and what corrective action was taken, will be provided to the Project Manager or Study Director as well as the EAS Technical Director.

3.1 Quality Policy Statement

The quality policy at Environmental Analytical Service, Inc. is based on the company mission statement. The Board of Directors and the upper management wrote the Mission Statement and the Quality Policy Statement. Prominent in the Mission Statement are the words quality and reliable showing the emphasis of upper management on quality.

EAS Mission Statement

Environmental Analytical Service, Inc. is an environmentally conscious company offering quality analysis of air using state of the art procedures. The well-trained staff employed at EAS is dedicated to providing the customer with prompt and reliable analysis.

Quality Policy Statement:

The Board of Directors and the Laboratory Director understand the extreme importance of laboratory data quality and ethical reporting of results to the client. The financial future of the laboratory depends on this. This is communicated to each employee as they are hired and is reiterated on an ongoing basis. The purpose of the management systems is to provide the documentation to the laboratory personnel in a way that they can carry out their duties in accordance with the stated methods and customers requirements.

The Board of Directors and the Laboratory Director are committed to the international standard as expressed in ISO 17025 and are continuously striving to improve the effectiveness of the management systems and the data quality.

The standard of service is that the analytical testing conducted by Environmental Analytical Service, Inc. will always be done in a technically competent manner to provide the client with accurate results that meet the quality objectives of the project.

The training program at EAS ensures that all of the lab personnel are familiar with the Quality Manual and the Standard Operating Procedures for the tests that they conduct.

3.2 Quality Manual

The EAS Quality Manual was prepared by the EAS Quality Manager and Technical Director with input from the operational unit managers, and was written to meet the requirements for NELAC Chapter 5, 2003/2009, and EPA 40 CFR part 160 for GLP. The Quality Manual is the foundation of the EAS quality system and is given a high priority by the upper management.

The Quality Manual is reviewed yearly and changes are incorporated into the manual as needed to establish new policies or to make for a better understanding of existing policies.

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4.0 DOCUMENT MANAGEMENT

This section defines the documents, manuals, procedures, and standard operating procedures used at EAS. All of the documents appear on the Master Document List.

4.1 Documents

The term document applies to a variety of materials, both internal and external, that are used in the laboratory operations. Documents include Administrative Procedures, Laboratory Procedures, manuals, books, articles, and methods.

A. Controlled Document
These are documents that are an integral part of the lab operations and report generation. They are controlled.

B. Reference Document
These are external documents such as agency methods, instrument manuals, etc. that are a manual of a PDF of a manual. They are not document controlled.

4.1.1 Manuals

The manuals are a collection of documents and procedures that define the operations in a particular area.

4.1.2 Administrative Procedures

Administrative Procedures are procedures that have no impact on the analytical results. Some examples are answering the phone or filling out time cards are procedures that to not part of the analytical report generating process and are not document controlled.

4.1.3 Procedure

These are documents that describe procedures such a loading software on a computer, building flow controllers, etc. They are not document controlled.

4.1.4 Standard Operating Procedures

The Standard Operation Procedures (SOP) used at Environmental Analytical Service are written by the Technical Director and document controlled. These procedures describe the sample analysis, calibration, report generation, and other function that directly relate to the results reported to the client in the Analytical Report. The format for the Standard Operating Procedure is described in NELAC Chapter 4.2.8.5. This procedure contains the 23 sections recommended in NELAC.

4.2 Document Organization

The following sections define the types of documents used by EAS as part of its operations. The documents are stored on the EAS Computer System, and there are also printed versions available. The organization of documents is shown in Table 4.2 which shows all of the documents and their location.

Table 4.2a
MASTER DOCUMENT LIST

					DISTRIBUTION/LOCATION			
Number	**Description**	**Year Last Date of Review or Revision**	**Current Version**	**Doc. Type**	**Archive Server**	**EAS Server**	**Hard Copy**	**Client**
	ADMINISTRATION MANUAL							
AM2	Management Systems	2020		AP	YES	NO	NO	NO
AM3	Human Resources	2020		AP	YES	NO	NO	NO
AM4	Office Procedures	2020		AP	YES	NO	NO	NO
AM5	Document Management	2020		AP	YES	NO	NO	NO
AM6	Report Management	2020		AP	YES	NO	NO	NO
AM7	Contracts	2020		AP	YES	NO	NO	NO
AM8	Shipping and Receiving	2020		AP	YES	NO	NO	NO
	HEALTH & SAFETY MANUAL							
HS	Health and Safety	2020		M	YES	NO	NO	NO
	INFORMATION TECHNOLOGY							
IT1	Computer System	2020		P	YES	NO	NO	NO
IT2	Documents and Reports	2020		P	YES	NO	NO	NO
IT3	EDD	2020		P	YES	NO	NO	NO
IT4	Backup	2020		P	YES	NO	NO	NO
IT5	LIMS	2020		P	YES	NO	NO	NO
IT6	Workstations	2020		P	YES	NO	NO	NO
IT7	Software	2020		P	YES	NO	NO	NO
	LABORATORY PROCEDURES							
	LABORATORY SUPPORT							
LS1.01	Preparation of Standard Operating Procedures	2020	07	SOP	YES	YES	YES	YES
LS1.02	Employee Training & Method Capability Certification	2020	08	SOP	YES	YES	YES	YES
LS1.03	Glassware Cleaning	2020	03	SOP	YES	YES	YES	YES
LS1.04	Receiving and Storing Chemical Inventory	2020	05	SOP	YES	YES	YES	YES
LS1.05	Recording Refrigerator and Freezer Temperatures	2020	06	SOP	YES	YES	YES	YES
LS1.06	Preparation of NIST Traceable Standards	2020	03	SOP	YES	YES	YES	YES

Number	Description	Year Last Date of Review or Revision	Current Version	Doc. Type	Archive Server	EAS Server	Hard Copy	Client
	SAMPLE ANALYSIS PROCEDURES							
LP2.01	Setting Up a Method for HP Chemstation for GC and GC/MS	2020	05	SOP	YES	YES	YES	YES
LP2.02	Sample Analysis-Daily Analytical Batch	2020	09	SOP	YES	YES	YES	YES
LP2.03	Analytical File Naming and Storage	2020	04	SOP	YES	YES	YES	YES
LP2.04	Preparing and Initial Calibration for GC/MS	2020	07	SOP	YES	YES	YES	YES
LP2.05	Manual Integration	2020	05	SOP	YES	YES	YES	YES
LP2.06	Tuning the GCMS for Passing BFB	2020	06	SOP	YES	YES	YES	YES
LP2.08	TIC Compound Identification	2020	05	SOP	YES	YES	YES	YES
	REPORTING PROCEDURES							
LP3.02	Preparing Daily Analytical Batch for Processing	2020	12	SOP	YES	YES	YES	YES
LP3.02	Assembly of Analytical Report	2020	11	SOP	YES	YES	YES	YES
LP3.03	Review of SDG Folder and Sample Scheduling	2020	08	SOP	YES	YES	YES	YES
LP3.04	Processing Client Reports	2020	19	SOP	YES	YES	YES	YES
LP3.05	Electronic Data Deliverable (EDD) Procedure	2020	12	SOP	YES	YES	YES	YES
LP3.06	EPA TO-14 Hydrocarbon Processing GC/FID	2020	09	SOP	YES	YES	YES	YES
LP3.07	The Excel Analytical Report Template	2020	06	SOP	YES	YES	YES	YES
LP3.08	Typing Labels on Chromatogram	2020	02	SOP	YES	YES	YES	YES
	LABORATORY SOPs							
MSA								
MSA.TO 15.01	EPA TO-15 Volatile Organics by GC/MS Full Scan	2020	16	SOP	YES	YES	YES	YES
MSA.TO 17.01	EPA TO17 Volatile Organics by GC/MS Full Scan	2020	7	SOP	YES	YES	YES	YES
MSB								
MSB.TO 15.01	EPA TO14-15 Volatile Organic Compounds by	2020	04	SOP	YES	YES	YES	YES

Number	Description	Year Last Date of Review or Revision	Current Version	Doc. Type	Archive Server	EAS Server	Hard Copy	Client
	GCMS SIM							
MSG								
MSG.TO 13.03	EPA TO-04, TO-10 AND TO-13 Semi Volatile Organic Compounds by GC/MS Full Scan	2020	08	SOP	YES	YES	YES	YES
MSG.TO 4.04	EPA TO-04A Pesticides by GC/MS SIM	2020	09	SOP	YES	YES	YES	YES
GCK								
GCK.TO 15.01	EPA TO-15M Hydrocarbon Speciation GC/FID	2020	04	SOP	YES	YES	YES	YES
GCL								
GCL.3416.01	ASTM 3416 Methane and CO by GC/FID	2020	06	SOP	YES	YES	YES	YES
GCL.EAS06.01	EAS Method for Nitrous Oxide in Air	2020	02	SOP	YES	YES	YES	YES
GCO								
GCO.1946.01	ASTM 1945/1946 and EPA 3C, Permanent Gases	2020	10	SOP	YES	YES	YES	YES
GCP								
GCP.EPA16.01	EPA 16/15 Modified Reduced Sulfur by GC/FPD	2020	11	SOP	YES	YES	YES	YES
HPT								
HPT.TO 08.01	EPA TO-8 Phenols ad Cresols	2020	09	SOP	YES	YES	YES	YES
HPT.TO 11.01	EPA TO-11A/CARB Aldehydes and Ketones	2020	13	SOP	YES	YES	YES	YES
	SAMPLE MEDIA PREPARATION							
SP4.01	Canister Evacuation and Cleaning	2020	12	SOP	YES	YES	YES	YES
SP4.02	Preparing Thermal Desorption Tube Media	2020	09	SOP	YES	YES	YES	YES
SP4.04	Testing and Leak Checking of Flow Orifices	2020	08	SOP	YES	YES	YES	YES
SP4.05	Flow Orifice and Gauge Cleaning	2020	10	SOP	YES	YES	YES	YES

Number	Description	Year Last Date of Review or Revision	Current Version	Doc. Type	Archive Server	EAS Server	Hard Copy	Client
SP4.06	Flow Controller Setting	2020	05	SOP	YES	YES	YES	YES
SP4.07	Preparing SVOC Media	2020	07	SOP	YES	YES	YES	YES
	EQUIPMENT MAINTENANCE							
SP5.01	Instrument Maintenance	2020	07	SOP	YES	YES	YES	YES
SP5.02	Rough Vacuum Pump Maintenance and Repair	2020	06	SOP	YES	YES	YES	YES
	SAMPLE RECEIVING							
SP6.01	Sample Receipt and Handling	2020	10	SOP	YES	YES	YES	YES
SP6.02	Canister Pressure Recording and Pressurization	2020	12	SOP	YES	YES	YES	YES
SP6.03	Logging Samples in LIMS System	2020	11	SOP	YES	YES	YES	YES
SP6.04	Generating Shipping Requests, Packing Slips and Shipments	2020	12	SOP	YES	YES	YES	YES
SP6.05	Subcontracting Procedure	2020	06	SOP	YES	YES	YES	YES
	QA PROCEDURES							
SP7.01	Internal Audits and Audit Samples	2020	07	SOP	YES	YES	YES	YES
SP7.03	NIST Traceable Laboratory Calibration Procedure	2020	05	SOP	YES	YES	YES	YES
SP7.04	Determining the Method Detection Limit	2020	06	SOP	YES	YES	YES	YES
SP7.05	Calculating Laboratory Control Limits	2020	09	SOP	YES	YES	YES	YES
	METHODS PDF							
	PDF of Agency Methods			RD	YES	YES	NO	NO
	QUALITY MANUAL							
QA	Quality Manual	2020	35	CD	YES	YES	YES	YES
	TECHNICAL SERVICES PROCEDURES							
TS1	Instrument Manuals	2020		M	YES	YES	NO	NO
	HP5890	2020		M	YES	YES	NO	NO
	HP5890 Series II	2020		M	YES	YES	NO	NO
	HP5970 MSD	2020		M	YES	YES	NO	NO

Number	Description	Year Last Date of Review or Revision	Current Version	Doc. Type	Archive Server	EAS Server	Hard Copy	Client
	HP Chemstation	2020		M	YES	YES	NO	NO
	Shimadzu HPLC	2020		M	YES	YES	NO	NO
TS2	Support Equipment Manuals	2020		M	YES	YES	NO	NO
	ADDITIONAL DOCUMENTS							
	NELAC 2016 Technical Requirements	2016	2016	RD	YES	YES	YES	NO
	EPA Definition of Procedure for Determination of the Method Detection Limit	2016	2016	RD	YES	YES	YES	NO
	Interpretation of Mass Spectra	1980	1980	RD	YES	YES	YES	NO
	HP MSD Maintenance	2015	2015	RD	YES	YES	YES	NO
	Method TO-15A	2019	2019	RD	YES	YES	YES	NO

Document Type

CD Controlled Document
P Procedure - Not Controlled
RD Reference Document - External Not Controlled
SOP Standard Operating Procure - Controlled
AP Administrative Procedure - Not Controlled
M Manual - Not Controlled

4.3 Generating New Procedures

Procedures are written by either the Lab Director or Technical Director. If the procedure is a Laboratory Procedure and is document controlled it is given to the Document Control Manager, who reviews and corrects the formatting of the document and assigns it a number and tracks it.

Approving and Reviewing Procedures

The completed Document Controlled Procedure goes back to the Technical Director who reviews, approves and signs the document. The signed document is returned to Document Control, who makes a Secured PDF copy of the signed document and enters it into the Document Control Log. The secured copy can be printed, but cannot be modified.

Revision of Procedures

All Document Controlled Procedures are reviewed every two years by the Technical Director or the Quality Manager to make sure they reflect the current procedure used.

Distribution of Procedures

The Document Administrator tracks the distribution of the controlled documents. In most cases, an electronic copy in PDF form that cannot be edited is placed on the EAS Server. The EAS Document Directory is a defined location on the EASSERVER that is available to the personnel that need to use the document. The prior copy is removed from the EAS Document Director.

Archiving Procedures

After a procedure is signed and copied, the original Microsoft Word procedure, and the PDF procedure are filed in the Document directory on the ARCHIVE computer. The Document Archive is located on a computer that is accessible only to the Document Administrator, the Technical Director and the Quality Manager.

4.4 Control of Documents and Procedures

The Laboratory Director sets the policy for document control for the laboratory. The implementation of this policy is assigned to the Document Administrator who maintains document control of all current and past EAS Documents and Procedures that require document control..

4.5 Computer Storage of Documents

The EAS computer storage system facilitates both of these requirements. To facilitate these requirements, protected versions of the original documents which are saved and archived on the ARCHIVE computer. The locations of other documents is shown in Table 4.5. The table also shows what the security is on each of the locations, and who has access to that location.

Table 4.5
Computer Document Storage Locations

Computer Location	Document	Security	Access
ARCHIVE	All Documents – Word All Documents – PDF All Procedures - Word All Procedures - PDF	Access to Computer Restricted with User Name and Password	Document Administrator Technical Director
EASSERVER	PDF of Lab SOP	Document Protected to Track Changes. User Name and Password	Document Administrator Lab Director Technical Director Laboratory Technicians
Workstations	No Documents	Password	Laboratory Technicians

5.0 RECORD MANAGEMENT

Records can be divided up into Administrative Records and Technical Records. Administrative records include accounting information, purchase orders, invoices, personal records, correspondence, and insurance. Technical Records include internal audit reports, calibration standard preparation, analytical reports, raw analytical data, project reports, and calibration reports.

Records are archived on the ARCHIVE Computer. Records are maintained for a minimum of five years.

5.1 Record Management and Storage

The records stored by EAS are listed below. There is redundancy built into the data storage because the raw data that makes up an analytical report is stored on the EASSERVER and a complete copy of the analytical report and its raw data is stored in another location on the EASSERVER. The Server is built on a redundant RAID drive system, and has an external hard drive backup, as well as an off-site backup.

5.1.1 Audit Reports

Audit Reports are stored in PDF format on the computer.

5.1.2 Calibration Data

Calibration data is stored electronically on the computer in the Excel Analytical Report for each sample, and electronically in the raw HP Chemstation data files. The data is stored for a period of at least five years.

5.1.3 Laboratory Information System (LIMS)

Sample information and sample receipt information is stored in the EAS LIMS system. The LIMS system is stored on LIMS and is backed up. The LIMS system contains all the pertinent information about each sample, including the tests requested, sample receipt information, sample login information, billing information, and call logs (communications) with the client about the samples.

5.1.4 Analytical Reports and Supporting Data

Analytical Reports and supporting laboratory data are stored electronically in a Sample Delivery Group (SDG) file, which is stored on the EASSERVER hard drive for a period of at least five years. The different types of information that make up the SDG file and how it is stored is described below:

1. SDG Files: Each sample delivery group (SDG) is assigned a unique number and file folder. All information related to the SDG is placed in this file folder. This includes shipping labels, fax notices, quotation, analytical reports, all of the Daily Analytical Batch data related to the SDG group, notes, phone logs, and review forms. The SDG files are placed in filing cabinets in the Administrative Assistant's office in order of SDG number. The files are stored in a file drawer and, after 6 months, are copied into a PDF file and archived on the EAS Server under the SDG number. The files are located by looking up the information in the EAS LIMS system and obtaining the SDG number for the file of interest. The Administrative Assistant then retrieves the file. The electronic files are stored for a period of five years.

2. Instrument Raw Data Files: Each instrument has a computer data collection system, which collects and stores the raw data. The raw data for each sample and the blanks, QC data, calibration data, and methods are stored on the EASSERVER, filed by instrument number and date.

3. Excel Analytical Report Templates (EART): The instrument quantitation data is put into an Excel Workbook. The workbook contains calibration data, calculation of results, dilution factors, and the final Analytical Report. The EART also has the information to generate the electronic data deliverable (EDD) files. The Excel files are stored on the EASSERVER in the Report Files.

4. Sample Delivery Group (SDG) Data: Sample delivery groups are assigned a unique SDG number and laboratory number by sample control. This information is stored in a bound notebook. Data about the samples is entered in the EAS LIMS system. The LIMS system is stored on the EASSERVER and is backed up on a regular basis.

5. Instrument Run Logs: Each analytical instrument has a run log which the laboratory technician records the DAB information each day. This includes instrument conditions, analytical method, calibration curve, standards, blanks, QC, and samples analyzed in that batch. The run logs are part of the DAB database program. A copy of the DAB form for each day is filed with the raw data in the SDG folder.

6. Standard Log Book(s): All information about the preparation of standards at EAS is stored in a bound notebook. These notebooks are stored in the laboratory. The data from the logbooks is entered into the computer and a standard report sheet is generated. This report sheet contains all of the information in the logbook and provides a secondary backup of the Standard Log Book(s).

7. Reagent Preparation Log Book(s): All information about the preparation of reagents used at EAS is stored in a bound notebook. These notebooks are stored in the laboratory.

8. Canister Cleaning and Certification Log: Canisters are cleaned in batches and are batch certified unless otherwise requested. A log is maintained in LIMS showing which canisters are cleaned in each batch, how they were cleaned, a reference to the

canister used to certify the batch, and whether the batch passed or failed the certification.

9. Chemical Inventory: As chemicals are received, they are assigned a unique number and entered in the log book.

10. Maintenance Logs: Maintenance information on the instruments is stored in the EAS LIMS System. When repair work is done on the instruments, the information is entered in the computer, where it is saved.

11. The chain of custody records for each sample are converted to a PDF file and stored with the SDG records on the Archive Computer.

5.2 Computer Storage of Records

For protection the records are archived on the ARCHIVE Computer that has only limited access as defined in Table 4.2. In addition to the archived copy, there is a copy of the record stored on the EASSERVER, so that laboratory personnel can refer to past reports, data, worksheets, or calibrations. Table 5.1 shows the location and the security on each of the computer locations, and who has access to that location.

Table 5.1
Computer Report Storage Locations

Computer Location	Report	Security	Access
ARCHIVE	All Reports – Word All Reports – Excel All Reports – PDF	Access to Computer Restricted with Password	Document Administrator Technical Director
LIMS	LIMS	Access to Computer Restricted with User Password	IT Manager Document Administrator Lab Technicians
EASSERVER	All Reports – Word All Reports – Excel Raw Data All Reports – PDF Protected Controlled Documents Uncontrolled Materials of Interest	Document Protected Password Controlled	Document Administrator Lab Technicians
Workstations	Raw Data Methods	Working Templates and Files	Lab Technicians

6.0 AUDITS AND MANAGEMENT REVIEW

There are two types of laboratory audits, an Internal Audit and an External Audit. The Quality Assurance Officer at EAS conducts Internal Audits approximately every year. External Audits are conducted either by clients or laboratory accreditation groups. External audits are conducted on a frequency of every two years by the clients or the group responsible for accreditation.

6.1 Internal Audits

The Internal Audits are conducted as part of the EAS Quality System and are performed about every year by the Quality Assurance Officer (QAO). The Internal Audit is designed to be an internal assessment tool to evaluate whether the Quality System is being implemented at the operational level of the laboratory. The Internal Audits include a technical audit to verify compliance with method-specific requirements and operations and a systems audit to verify the laboratory's compliance with the quality system.

For the Internal Audit, the Quality Assurance Officer will follow the checklist that covers both the technical audit and the systems audit. The audit will be done in conjunction with the Laboratory Manager/Supervisor to make sure that all items are independently audited.

6.2 External Audit

The External Audit involves a series of meetings and reports, and is initiated when the letter arrives stating that an audit will take place and documentation is requested.

Pre-Audit Meeting

Before each audit, the Laboratory Director, Technical Director, Laboratory Manger, and the Administrative Manager will meet to review what the audit will cover, the date the audit is scheduled to take place, and the materials that have been requested for the audit. The meeting will also go over whether there are documents that are currently in for revision that need to be revised before the audit.

Audit

When the audit takes place, the Auditor will meet with at least the Laboratory Director, the Quality Manager, and the Laboratory Manager depending on the nature of the audit. The auditor will complete the audit forms and, at the end of the audit, go over the forms with the laboratory staff.

Post Audit Meeting

Following the audit, the Auditor will send a list of deficiencies to the laboratory for correction before final approval. The Laboratory Director, Technical Director,

Laboratory Manger, and the Administrative Manager will meet to review the audit findings and deficiencies

Corrective Action Plan

The Laboratory Director and Quality Assurance Officer formulate a Corrective Action Plan (CAP), with input from key laboratory personnel from the Post Audit Meeting.

1. The plan is formulated by making a list of each deficiency item or problem on the audit report.

2. The deficiencies are individually discussed in a meeting with key laboratory personnel to determine the cause of the deficiency.

3. At the same meeting, plans are discussed for correcting the deficiencies. The notes of this meeting are used by the Quality Assurance Officer to develop a written Corrective Action Plan.

4. The implementation of each item on the Corrective Action Plan is assigned to the appropriate laboratory personnel. A follow-up date is determined based on the estimated amount of time for full implementation and utilization of the procedure. The Quality Assurance Officer will work with the laboratory personnel to provide assistance or clarification on the implementation during this period. A time limit of 30 days will be used for implementation of routine corrective action items. If an item is expected to take longer then 30 days, a specific time limit will be set at the meeting and indicated in the Corrective Action Plan.

5. The laboratory personnel assigned to implement the procedure will collect data or information showing that the corrective action has solved the problem. A time period of 60 days is set for the demonstration of the implementation. If there are indications that the corrective action has not solved the problem, the Quality Assurance Officer will have a meeting with the Laboratory Director and the laboratory personnel to revise the implementation plan.

6.3 Performance Audit

The Performance audit includes a technical audit to make sure that the methods are being followed, and that the appropriate calibration procedures are being followed. This could include taking an analytical report and checking the calculations back to the primary calibration source.

The performance audit would also look at those items that would contribute to the data quality

Internal Quality Control

The internal quality control procedures include daily blanks, continuing calibration, laboratory control spikes, etc.

Replicate Testing or Duplicates

Duplicate testing is part of each Daily Analytical Batch (DAB). The normal QC protocol calls for a duplicate of either the laboratory control spike, a matrix spike, or a sample. The results of this test are put into the QC Report for the DAB, which calculates the percent RPD for the pair and compares it to the control limits.

In addition to the DAB duplicate analysis, many samples are routinely duplicated to either verify unexpected results or to verify field duplicate results that are out of the field duplicate control limits. These values are also used to verify the duplication of the analytical method. Sample duplicates are also analyzed to evaluate matrix effects on high level samples.

Proficiency Testing Samples (Performance Evaluation)

The results for the blind audit are reviewed in a meeting with the Quality Manager, the Laboratory Director, Laboratory Manager/Supervisor. A written set of findings is prepared, and implemented..

6.4 Systems Audit

The systems audit system is used to verify the laboratory's compliance with the quality system. The EAS internal audit forms have sections covering the systems audit components and the evaluation of the compliance with the quality system and Quality Manual.

6.5 Management Review

The Laboratory Director, who represents the Board of Directors, will have a meeting each year in March with the Technical Director, Quality Assurance Officer, Laboratory Director to review the management system and testing and calibration activities to ensure their suitability and effectiveness.

- Suitability of policies and procedures.
- Reports from management and recommendations for improvements
- The review will include the results of the yearly Internal Audit.
- Review the findings of External Audits.
- Proficiency samples that have been analyzed and any inter-laboratory comparisons.
- Changes in volume and type of work
- Staffing for the summer season.

- Client feedback
- Complaints.
- Corrective and preventative actions.

7.0 PERSONNEL, TRAINING, DATA INTEGRITY

Through the hiring and evaluation process, EAS has taken steps to ensure the competence of all who operate specific equipment, perform environmental tests, evaluate results, and sign test reports. Personnel performing analytical tests are qualified on the basis of their education, training, and experience. The Laboratory Director and Technical Director, Dr. Steve Hoyt oversees the training of all laboratory personnel. Dr. Hoyt has a Masters degree in Analytical Chemistry and a PhD in Environmental Science and Engineering. He taught chemistry classes and environmental engineering classes at the university level for over five years, before founding EAS, and also worked as the Technical Director of a major environmental laboratory which offered testing in the areas of water, soil, and air analysis.

7.1 Job Descriptions

Job Descriptions

Environmental Analytical Service is a specialty air-testing laboratory with a staff of approximately six people that provide laboratory services in the specific area of air testing. The job descriptions at EAS are defined below. Since EAS is considered a small laboratory, some employees occupy more than one position.

Administrative Director

The Administrative Director is responsible for the overall management of the office end of the laboratory operations. The Administrative Director oversees the Human Resources department, the Office Operations, Documentation, financial planning, Accounting and Marketing.

Laboratory Director

The Laboratory Director is responsible for the laboratory operations and supervises the Laboratory Manger, Technical Director, Quality Manager, Client Services, Information Technology, and Marketing. The Laboratory Director plans the methods that are used and assists in the development of the QC criteria for the methods. He also meets with the different departments to coordinate their activities.

Technical Director

The Technical Director is responsible for method development, project planning, instrument design and fabrication and reporting new methodology to the Laboratory Director. He plans and oversees the standard preparation process, and calibration of equipment. He is also responsible for repairing the instruments, and scheduling the routine maintenance.

Quality Manager

The Quality Manager oversees the EAS Quality Program. The Quality Manager is responsible for writing and updating the Quality Manual and setting the QC criteria in conjunction with the Laboratory Director and Laboratory Manager. He needs a degree in Chemistry and experience in Quality Control.

Administrative Manager

The Administrative Manager supervises the office operations, which include the Administrative Manual that contains all of the office systems and their procedures. She is in charge of the EAS filing system and document control, and oversees the client reports and invoicing. The Administrative Manager needs experience in office management.

Laboratory Manager

The Laboratory Manger is the head of the Laboratory Team and Support Team, and works with the Laboratory Director to schedules sample analysis and evaluate the sample load in the laboratory. The Laboratory Manager manages the Support Team that is responsible for the canister cleaning, media preparation and certification.

Project Manger

The Project Manager is an internal EAS person that manages the flow of project through EAS. The Project Manager needs a degree in Chemistry.

Client Services

The Client Service person takes calls from clients and enters sample information in to the computer system. The Client Service's person needs to have taken a class in Chemistry or have a science background.

Information Technology Manager

The Information Technology Manager maintains the EAS computer system and needs experience with computers and computer networks.

Technical Services Manager

The Technical Services Manager supervises the construction repair and testing of instruments. They need knowledge and experience in fabrication and electronics.

Laboratory Technicians

The Laboratory Technicians work under the Laboratory Manager on the Laboratory Team. The Lab Technicians are responsible for analyzing samples and preparing reports. They need to have taken classes in Chemistry of have a science background.

7.2 Training

Training and Evaluation Procedures

There are four types of training programs provided by EAS to help improve the performance and allow for advancement of employees. The training programs are listed below.

1. In-house Technical Training: Dr. Steve Hoyt, the Technical Director for EAS, conducts this program.

2. In-house Business Training: This program is conducted by Lisa Hoyt, who has a Masters Degree in Education Administration..

3. Seminars conducted locally by national seminar groups: These programs usually focus of developing supervisory and organizational skills.

Training Documentation

All training received by staff is documented in the personnel file maintained on each employee.

7.3 Data Integrity

Environmental Analytical Service has a policy established by the Board of Directors to provide its customers with data of the highest quality. This is the basis for the data integrity system. A large component of data integrity is the communication of the EAS policies on ethical and legal responsibilities of each employee. The four required elements of data integrity are:

1. Data Integrity Training

2. Signed "Ethical and Professional Policies" Documentation for each Employee yearly. See Section 7.4 for form and training

3. Periodic Monitoring of Data Integrity

4. Data Integrity Procedure Documentation

Data Integrity Training

Data Integrity Training is provided before a technician can analyze samples.

Signed Documentation for each Employee

Every year all employees at EAS are given a yearly employee evaluation. Part of this evaluation is to review the EAS Data Integrity policies, sign the "Ethical Professional Policies" form. This form is signed and placed in his/her employee file.

Periodic Monitoring of Data Integrity

The Laboratory Director is ultimately responsible to the Board of Directors for monitoring the EAS Data Integrity program and making sure it is implemented by all employees. This is done by daily interaction between technician and the Technical Director and through the data review process.

Data Integrity Procedure Documentation

Data Integrity is documented in the yearly employee review forms, where each employee signs an "Ethical Professional Policies" form.

7.4 Ethics

Training Staff for Ethical Responsibilities

In order to insure that the data generated by Environmental Analytical Service is of the highest quality, a training program has been established to detect and prevent improper, unethical, or illegal actions. During an employee's probationary period, their trainer is responsible for introducing all laboratory procedures reviews and explains the EAS Ethical and Legal Policy and how it applies to daily laboratory procedures.

Improper Actions

Improper actions are defined as deviations from good analytical practices in the laboratory whether intentional or unintentional. All employees at EAS have a responsibility to follow established method procedures as defined in the Standard Operating Procedures for that method and established EAS QC Criteria as defined in the EAS Quality Manual. If you deviate or are asked to deviate from established methods except in the case of special client specific analytical methods, or EAS QC Criteria then you need to get approval on the Daily Analytical Batch (DAB) form

Unethical Actions

Unethical Actions are defined as deliberate falsification of analytical or quality control data (QC) to misrepresent the quality of data.

Examples of Improper Practices

- Improper manual integration of peak areas other then to correct for poor baseline because of sample moisture, peak shifting, peak doubling, or peak tailing to misrepresent calibration or method QC requirements.

- Intentional misrepresentation of the date or time of analysis in order to meet holding times: This usually involves resetting the computer clock. Since the computers at EAS are older and operate on Windows 95 the daylight savings time does not set correctly. Employees should check the instrument clocks periodically to make sure they are set correctly.

- Falsification of results to meet method requirements.

- Reporting results without analysis.

- Selective exclusion of data to meet QC criteria: Dropping calibration points without justification.

- Use of Matrix Interference notation for exceeding acceptance limits in a clean matrix.

- Omitting sample preparation steps for QC. This would be calling a laboratory control spike a matrix spike.

- Improper checking for passing the GC/MS tune, even though the tune no longer has any scientific purpose or value. A tune is not required on selective ion monitoring.

Ethical Professional Policies
Environmental Analytical Service

Overview

Environmental Analytical Service (EAS) expects it employees to act as ethical professionals in the laboratory. At EAS the Technical Manager and Quality Manager are always available in the laboratory and will observe and instruct employees on the proper and ethical manner in which to perform their analytical duties. The EAS ethical program focuses on professionals working together to providing scientifically valid data for the client and providing QC information that accurately reflects the uncertainties of the measurements.

Improper Actions

Improper actions are defined as deviations from good analytical practices in the laboratory whether intentional or unintentional. All employees at EAS have a responsibility to follow established method procedures as defined in the Standard Operating Procedures for that method and established EAS QC Criteria as defined in the EAS Quality Manual. If you deviate or are asked to deviate from established methods except in the case of special client specific analytical methods, or EAS QC Criteria then you need to get approval on the Daily Analytical Batch (DAB) form

Unethical Actions

Unethical Actions are defined as deliberate falsification of analytical or quality control data (QC) to misrepresent the quality of data. Two critical actions are listed below.

_____Improper manual integration of peak areas other then to correct for poor baseline because of sample moisture, peak shifting, peak doubling, or peak tailing to misrepresent calibration or method QC requirements.

_____Intentional misrepresentation of the date or time of analysis in order to meet holding times.

I have read and understand the above information. I have initialed that I have read and been trained on LP3.02 on Manual Integration, and that I will not reset the clocks on the computers to meet holding times.

Employee: _____ Date: _____

Confidentiality Procedures

The laboratory has each employee sign a form that he/she understands that any information that is supplied to the laboratory by the client and the analytical data is the property of the client who arranged the testing is confidential and cannot be disclosed to anyone.

Before any information is given out to someone other then the client that contracted the work, the client is called to verify that they want the data released. An example is a consultant or government agency calling to request copies of analytical reports for a project done for a company.

8.0 LABORATORY CAPACITY REVIEW

At Environmental Analytical Service, the process of accepting samples and entering into contracts starts with the Laboratory Director and the Client Service staff. When clients request analytical services the Client Service staff initiates the request with a Call Log in the LIMS system. If the project is more then a few samples or is technically complicated, the Lab Director contacts the client to go over the details of the project. The Lab Director will go to the lab and go over the canister inventor and other projects with the Lab Technicians and Sample Control.

For all projects, a Project Sheet is prepared in the LIMS system, where the tests, cost for the tests, number of samples, turn around time, QC criteria, and any other special instructions are recorded. For written contracts, all information is included, and deviations from the written instructions are recorded. All discussions with the client about the work and any changes are documented in the Call Log in the LIMS system.

9.0 PHYSICAL FACILITIES AND EQUIPMENT

9.1 Physical Facilities

Environmental Analytical Service, Inc. is located in a modern, 3000 sq foot facility that is dedicated to ambient air and gas analysis. The equipment at EAS is dedicated to air and gas analysis.

Instrumentation Room

EAS has a 1500 sq foot instrumentation room that has three GC/MS systems with full data processing and NIST mass spectral libraries, gas chromatographic systems equipped with FID, and ECD detectors. One gas chromatograph (GC) with FID detector is dedicated to methane, while another is dedicated to non-methane hydrocarbons.

Sample Control/Sampling Equipment

The Sample Control area houses the 300+ SUMMA canister and 100+ flow regulator inventory, as well as other sampling equipment. Samples are shipped and received in this area. The area contains a refrigerator for holding samples that require refrigeration. All canisters are tracked by the EAS computer system, and all samples are logged into the LIMS system as they are received. This room also houses the canister cleaning system and the pressurization system for pressurizing canisters.

Chemistry Laboratory

The Chemistry Laboratory is located in an isolated area not connected directly to the instrumentation room. The Chemistry Laboratory is equipped with hoods and an extraction area for handling semi-volatile sample extractions. The Chemistry Laboratory houses two GC/MS systems for semi-volatile organic compounds. The room also houses three GC systems for fixed gases, sulfur compounds, and a GC with an FID for liquid injections. There is also a HPLC system used for TO-11 aldehydes, a spectrophotometer, and analytical balance.

9.2 Equipment

Environmental Analytical Service has a complete complement of equipment for the analysis of air and gas samples. EAS is equipped with all sampling, measurement, and test equipment required for the correct measurement of parameters required in the methods performed by EAS. A list of equipment is given in Table 9.1.

9.2.1 Support Equipment

Support equipment includes all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include balances, ovens, refrigerators, freezers, flow meters, pressure gauges, and temperature measuring devices.

9.2.2 Analytical Equipment

When a test method is developed at EAS, an instrument is designated for that method. The Standard Operating Procedures (SOP's) are written for the method and instrument, which describes the procedure, QC criteria, maintenance, and troubleshooting. The equipment used to perform analytical testing is operated by trained and authorized personnel. The calibration of this equipment is described in Section 13, and the quality control procedures to verify performance of the instrumentation is described in Section 12.

Table 9.1
EAS Equipment List

Windows Computer Server System
EAS LIMS System
Data Backup System
HP 5890A GC, HP 5970 MSD and MS/DOS Chemstation (4)
HP 5890 GC with TCD
HP 5890A GC with FID/ECD
HP 5890A GC with dual FID (2each)
HP 5890 II GC with FPD
HP 3365 Chemstation GC Data System
Shimadzu HPLC System, with Variable Wavelength Detector
Top Loading Balance (o.01)
EAS Cryogenic Concentrator Systems (5 each)
Canister Pressurization System
EAS Canister Cleaner
NIST Traceable-referenced standards
Stainless steel canisters (300)
Integrated ambient air samplers (100)
Soil Gas Samplers (50)
Fume hood for extractions
Digital Document Center
Facsimile machine
Soxhlet Extractors (8 station)
Oven
Semi Micro Analytical Balance

9.2.3 Equipment Maintenance

Instrumentation is an important part of the EAS analytical program. To obtain the best performance from instrumentation it must be serviced, calibrated, and often repaired. EAS has the facilities and the trained personnel to repair and calibrate the laboratory instrumentation and computers.

The EAS LIMS system has a section for instrument repair and maintenance. The Technician communicates the problem to the Technical Director who enters significant problems, maintenance, or repairs into the LIMS maintenance log. When the repair is completed, the repair information is entered into the computer and the record marked as completed.

9.2.4 Preventative Maintenance

EAS has a preventative maintenance program to keep the instruments in top working condition. Most of the instrumentation is complicated and requires cleaning or repairing at least yearly. At the time repairs are made, much of the routine maintenance is also done.

Table 9.2
Preventative Maintenance Activities

Instrument	Maintenance Parameters	Frequency
Gas chromatograph (GC)	Liner Insert, column, glass wool plug, detector, thermal traps Replace septa Gas drying and purifying cartridges	As needed basis; determined by analyst in order to meet method QA/QC requirements As needed When indicated to be necessary Every 6 months
Gas chromatograph /mass spectrometer (GC/MS)	Same as GC plus the following: -Vacuum pump oil -Trap Beads -Ion source and analyzer cleaning -Cryogenic Traps	 About once a year when oil is dirty As needed As needed As needed
High Pressure Liquid Chromatograph	-Replace Pump Seals -Clean detector -Replace Column	As needed As needed As needed
Canister Cleaner	-Replace Pump Oil - Replace Trap Beads	About very 12 months As needed

10. MEASUREMENT TRACEABILITY

The goal for the measurement and traceability program at EAS is to be able to take a final report containing either an amount or concentration of a compound and reference it back to an NIST traceable standard.

For an analytical testing laboratory like EAS, the standards are divided into types: Standards for calibrating Support Equipment as described in Section 10.1 and chemical standards for calibrating the analytical instrumentation. This section describes the traceability, storage, and labeling of reference standards.

10.1 Reference Standards for Support Equipment

EAS has Reference Standards for calibrating Support Equipment and Reference Chemical Standards for calibrating Analytical Instrumentation. This section discusses the traceability of these standards.

Primary Calibration Standards for Support Equipment

The primary calibration standards are calibrated yearly by an outside source that can provide an NIST traceable certificate of calibration. The primary standards are used to calibrate secondary standards that are used more frequently in the laboratory.

Table 10.1
Primary Calibration Standards

Description	Standard	Calibration Frequency	Calibration Source
Digital Flow Check	Flow	Yearly	Primary Bubble Meter
Analytical Balance	Weight	Yearly	Wine Country Balance
NIST Pressure Gauge	Pressure	2 Years	EAS - Vapor Pressure of Gas at STP

Secondary Calibration Standards for Support Equipment

The secondary calibration standards are standards that are used more frequently in the daily laboratory operations and are calibrated against the primary standard.

Table 10.2
Secondary Calibration Standards

Description	Standard	Calibration Frequency	Primary Standard
Instrument Flow Controllers	Flow	Weekly	Digital Flow Check
Analytical Balance	Weight	Yearly	Wine Country Balance
Standard Loops for Concentrators	Volume	Evidence of Deterioration	Manufacturer
Glass Syringes	Volume	Evidence of Deterioration	For Standards the weight of the liquid is recorded not volume.

Gas Sample Volume Measurement

Gas volume measurements are important for air and gas analysis. The gas volume measurements commonly used by EAS are listed below.

1. Large volume gas sample measurements (50 to 1000 ml): These sample volumes are measured using a mass flow controller and a timer. The individual mass flow controllers are calibrated against an NIST traceable flow meter.

3. Low volume gas sample measurements (0.05 to 20 ml): These sample volumes are measured using a calibrated loop and a gas-sampling valve. The loops are purchased pre-calibrated from the manufacturer.

4. Gas standard volume measurements (0.1 to 20 ml): The standard volumes are measured using a calibrated loop and a gas-sampling valve. The loops are purchased pre-calibrated from the manufacturer.

10.2 Primary Standards for Instrument Calibration

Environmental Analytical Service specializes in the measurement of ambient air and gas samples. The instrumentation used for these measurements is primarily gas chromatography (GC) or GC/MS. The primary standards used are gas standards prepared at a known concentration in cylinders.

EAS purchases some gas standard mixes from commercial vendors that sell this type of standard. The concentrations of gases in these cylinders are reported in a volume (mole) ratio that is temperature and pressure independent, and are usually in the 0.1 to 1000 ppmv range.

EAS prepares most gas standards in different size cylinders from pure chemicals or gases. The procedure for preparing gas standards is described in detail in a standard operating

procedure (SOP) on gas standard preparation. The NIST traceability of the standards prepared by EAS is accomplished by weighing the liquid standard on a balance that is NIST traceable, using the volume of the cylinder from the manufacturer, and measuring the pressure of the cylinder using an NIST traceable pressure gauge.

For liquid injections, NIST traceable liquid standards, in the units of ug of compound/ml of liquid, are used. Commercially available certified (NIST traceable) standards are used for tests, where they are available. For special target compounds, EAS can prepare liquid standards. These mixes are prepared by weight, using an NIST traceable balance.

The instrumentation at EAS is calibrated using standards that are NIST traceable. Table 10.3 shows the standards used for the calibration of different compounds and the calibration frequency. Different gas concentrations for calibration are prepared using the EAS gas dilution system based on fixed volume loops.

Table 10.3
Traceability of Instrument Calibration Standards

Description	Standard	Calibration Frequency	NIST Traceability
VOC Standards, TO-14 Compounds	Gas Cylinder AL 150	5 years	Traceable by Weight and Volume
VOC Standards, TO-15 Compounds	Gas Cylinder AL150	5 years	Traceable by Weight and Volume
Hydrocarbon Standards	Gas Cylinder AL150	5 years	Traceable by Weight and Volume
Permanent Gases	Gas Cylinder	5 years	Traceable by Weight and Volume
Semi-Volatile Pesticides	Liquid Standard	2 years	Commercial Standard Supplier
Aldehydes and Ketones	Liquid Derivative Standard	1 year	Commercial Standard Supplier

The calibration frequency for the high pressure gas cylinders can be extended by verifying cylinder against secondary standard. This can add an additional 2 years to the standard.

10.3 Labeling of Standards and Reagents

Logbooks are maintained for the preparation of standards and reagents. These are separate bound books, and contain detailed entries of how each standard and reagent are prepared. There is a labeling system to track each standard and reagent, based on a unique number.

10.3.1 Labeling Standards

Standards are labeled using the characters ST followed by a five-digit number. The new standard identification number is assigned the next number in the series. See the example below:

ST60001 (where the previous assigned number was ST60000)

This number appears in the standard logbook with a description of how the standard is made. Commercial standards are also logged in the book with the same type of standard number. Along with the number the following information is included: the date the standard was prepared, how it was prepared, the expiration date, who prepared the standard, and any other information needed to verify the NIST traceability of the standard.

A label is prepared for each standard with the standard number, a name for the standard, the expiration date, and nominal concentration of the compounds in the standard.

10.3.2 Labeling Reagents

Reagents are labeled using the Character R followed by a five-digit number. The new reagent identification number is assigned the next number in the series. See the example below:

R60001

This number appears in the reagent logbook with a description of how the reagent is made. Commercial reagents are also logged in the book with the same type of reagent number. Along with the number the following information is included: the date the reagent was prepared, how it was prepared, the expiration date, and who prepared the reagent.

A label is prepared for each standard with the reagent number, a name for the reagent, and the expiration date.

11.0 TEST METHOD EVALUATION

This section describes the process of evaluating a test method for use in the laboratory.

11.1 Test Method Evaluation

The development of a new test method for compounds starts with a literature search to determine if there is an existing method or procedure that has already been used for that compound, or if the compound is similar to other compounds that are in an established method. The Technical Director compiles the formation from the literature search into a draft method. If the test method is totally new or a major modification of an existing method, it requires an Initial Method Validation. If the method follows an existing method or is a minor modification of an existing method, it will require a Demonstration of Capability.

11.2 Initial Method Validation

Test methods are validated when they are first established and evaluated on an ongoing basis by a Demonstration of Capability. The initial method validation process involves the collection of data to establish how the method behaves in the following categories.

Description	QM Section
Limit of Detection	12.2
Initial Calibration	12.3.1
Calibration Check Sample	12.3.2
Continuing Calibration Verification	12.3.3
Method Blanks	12.4
Laboratory Control Spike	12.5
Duplicates	12.7
Surrogates and Internal Standards	12.8

Once the Initial Method Validation has been completed, a set of quality control criteria (QC Criteria) is established that is used for the future Demonstration of Capability and the Daily Batch Validation process.

11.3 Demonstration of Capability

The Demonstration of Capability (DOC) is used to confirm that personnel can properly perform all methods before analyzing client samples. The DOC is used for the initial setup of an established method, where complete Method Validation is not needed. The procedure is described in Appendix C of the NELAC Chapter 5. The DOC is done anytime there is a change in instrument type, personnel, or test method. Tests that require a group of analysts (work cell) have the group tested together. When new personnel are hired, they are under direct, close supervision until they complete their

DOC. The DOC is documented on the Demonstration of Capability form that is signed by the Technical Director (also the Quality Manager) and the Laboratory Manager/Supervisor and appears in their personnel file.

The DOC steps require that the method be set up and calibrated according to the method procedures and that a clean method blank is obtained.

The following steps are performed for each of the test methods.

1. A sample containing all of the target compounds for the method is run replicate times over a 2 to 6 month time period. (an analysts initial DOC will be done over a 2 month period).
2. Four of the runs are selected for comparison and put in their file.
3. The results are compared against either established method criteria or the mean recovery, and standard deviation is calculated to establish a measure of precision and accuracy. See Section 14 on Quality Control for more detailed information.
4. If parameters fail, the problem causing the failure is corrected, and the above steps are repeated.
5. Once the initial DOC for the method has been completed under the supervision of the Technical Director, and the QC criteria has been established, the DOC form for each analyst is stored in their personnel file.

11.4 Daily Batch Validation

Each daily analytical batch associated with samples includes standards, control spikes, and blanks to verify that the instruments used to analyze samples meet the established QC criteria of the method or the Project Specific QC criteria. If the Daily Batch Validation fails, the appropriate corrective action is taken. Section 17 describes the corrective action.

11.5 Standard Operating Procedures

Once the Demonstration of Capability or the Initial Method Evaluation has been completed, a Standard Operating Procedure (SOP) is prepared for that method.

11.6 Proficiency Testing

The final part of the method evaluation is analyzing a Proficiency Test (PT) sample usually obtained from an independent commercial source. The PT sample checks the accuracy of the method by comparing the results among a group of laboratories. While PT standards are not currently required, the results are used to validate the methods internally.

12.0 QUALITY ELEMENTS FOR AIR TESTING

12.1 Quality Elements

In Section 11 the initial method development process was discussed. This process involved using several measurements that determine data quality and usability. These quality elements are determined either through the Initial Method Validation or through established Quality Parameters that are contained in a specific method. From this information, Quality Control Criteria is established for the method along with the frequency with which it needs to be checked. The QC Criteria then becomes part of the method, and are listed for each of the EAS methods in Section 13 on Analytical Methods.

This QC Criteria is used to do a daily validation of the method for each Daily Analytical Batch (DAB). The daily DAB validation provides data that is used to establish that the method is "In Control" for that day and that group of samples.

The elements of the QC program are defined in NELAC Chapter 5 in Appendix D. Since EAS is an Air Testing laboratory the QC program centers on those elements described in Appendix D.5 on Air Testing. The elements of the program are defined below.

12.2 Limit of Detection

12.2.1 Method Detection Limit

The method detection limit (MDL) is the lowest concentration of an analyte that can be measured and reported with a 99% confidence that the analyte concentration is greater then the base line noise of the method. The determination of the MDL is described in 40 CFR 136 Appendix B, and in more detail in the EAS SOP SP7.04 Determining the Method Detection Limit.

The general procedure is to run seven replicate samples and calculate the standard deviation of the concentration of all runs. The MDL is then determined using the following formula.

$$MDL = STDEV(Conc) \times 3.14$$

In addition to the MDL, which is a statistical value related to the detectability of a compound, there are other parameters often requested by clients related to the MDL and the reporting of data.

MDL Method Detection Limit: Lowest concentration of an analyte that can be measured and reported with a 99% confidence that the analyte concentration is greater then the base line noise of the method. The MDL is a statistical quantity developed by J.D. Weinforder and involves making replicate measurements of the noise level, and then calculating the 99% confidence limit. At EAS this involves making seven replicate

measurements at about five times the estimated MDL. NELAC uses LOD instead of MDL.

LOD Limit of Detection: This is the measure of the lowest quantity that can be measured and reported as defined in NELAC Chapter 5. The limit of detection is determined by running a QC spike at 1-2 times the claimed LOQ. NELAC does not require a LOD or MDL for results that are reported within the range of the initial calibration curve.

12.2.2 Limit of Quantitation

The limit of quantification is defined by NELAP, Chapter 5 as the lowest concentration that can be (LOQ) verified to meet the QC requirements of the method. This can be defined as the lowest standard in the calibration curve that passes the criteria or can be determined by running a low level standard and verifying that it meets the QC requirements.

Other measurements relating to the lowest amounts to be reported that are not based on a statistical measurement, but are often requested, are listed below.

RL Reporting Limit: The California ELAP defines the reporting limit as a client specified number based on project limits or other factors. For ambient air samples the RL is set above the global background for F12, F11, and chloromethane.

PQL Practical Quantitition Limit: This is generally 3-5 times the MDL and is often used to take into account matrix or blanking problems. At EAS, we do not use the PQL unless required to do so by the client, since it has no statistical significance.

LOQ Limit of Quantitation: The LOQ is the lowest standard on the calibration curve. This is checked when the low standard on the initial calibration curve is run. The LOQ must be greater than the LOD by a factor of at least 2 or 3, depending on the project.

CRQL Contract Required Quantitation Limit: The CRQL is a project specific requirement for some projects (such as EPA contract work). This is the lowest limit that can be quantitated, so it is similar to the LOQ. Projects that have a CRQL will usually have a CRQL Standard requirement. This requirement is that a standard be made up at the CRQL limit concentration for the project and be analyzed each day before sample analysis is started.

12.3 Calibration Procedures

Laboratory instruments are calibrated using NIST traceable standards as described in Section 10 on traceability. Instruments are calibrated using the procedures described in the Standard Operating Procedures (SOP) for that particular method. The specifics of calibration are described in the SOP.

12.3.1 Initial Calibration Curve

Calibration curves are produced when two or more standards are analyzed on a particular instrument and the relative response is calculated. Traditionally, for a linear curve, the data are calculated using a least-squares best fit. For many instruments, the resulting line is forced through a zero intercept on the postulate that the calibration blank is defined as both the absence of target analyte and the zero signal point. The correlation coefficient, R, is also calculated.

For the GC, HPLC, and GC/MS, EAS uses the relative response factors (RRF) of the instrument at different concentrations, since the QC criteria for calibrations used for environmental work are generally expressed as a relative response factor, also known as the RRF.

Relative Response Factor (RRF)

$$RRF = \frac{(A_x C_{is})}{(A_{is} C_x)}$$

A_x = Area of characteristic ion of compound measured

A_{is} = Area of characteristic ion of Internal Standard

C_x = Concentration of compound

C_{is} = Concentration of Internal Standard

The average RRF is calculated over the calibration range. The % RSD of the RRF's must be less than required by the method. The GC/MS methods generally specify a RSD of 30% for most stable compounds and 40% for some compounds that are easily adsorbed on surfaces. The acceptable percent RSD is specified in the QC criteria for the method.

Run when a system is initially set up, calibration curves are repeated whenever a major change is made to the instrument or if the continuing calibration verification (CCV) standard does not pass.

12.3.2 Calibration Check Sample (CCS)

The Calibration Check Sample (CCS) is a standard that is analyzed to verify the accuracy of the method. In most cases the CCS is from a secondary source when available.

12.3.3 Continuing Calibration Verification (CCV)

The Continuing Calibration Verification (CCV) standard is analyzed with every daily analytical batch to demonstrate that the system response has not drifted significantly since the current initial calibration. The instrument calculates a response factor (RF) and compares it to the average RF for the initial calibration. A percent difference (%D) is calculated for each compound. Section 13 shows the CCV criteria for each method, which is usually one-half the high point on the calibration curve. Some methods have target compounds that are not very stable or chromatograph poorly, for these compounds a larger %D deviation of the RF from the initial calibration may be acceptable.

12.3.4 Contract Required Quantitation Limit (CRQL)

The Contract Required Quantitation Limit (CRQL) is a project specific requirement for some projects, such as EPA contract work. Projects that have a CRQL will usually have a CRQL Standard requirement. This requirement is that a standard be made up at the CRQL limit concentration for the project and be analyzed each day before sample analysis is started. The recovery for the CRQL Standard is calculated and compared to the project limit.

12.4 Method Blanks

Method blanks consist of zero air (or clean sorbent) carried through the analytical system like a sample. The method blank serves to measure contamination associated with laboratory storage, preparation, or instrumentation. For most tests, one method blank is analyzed in every analytical batch of samples.

For Air Samples, NELAC, Chapter 5 for Air Testing states that if the Method Blank is greater than the LOQ and contributes greater than 10% of the total concentration of a sample compound (adjusted for sample volume), it must be investigated and the results qualified with a “B” flag. If the method blank value contributes less than 10% of the sample, it does not need to be qualified. While the goal is to have no detectable contaminants, each method blank is critically evaluated as to the nature of the interference and the effect on each sample in the batch. Steps are then taken to minimize or eliminate the problem (NELAC Chapter 5, Appendix D).

The goal at EAS is to have method blanks that have no target compounds detected. Some sample batches have both high level samples and low level samples and sometimes the high level samples contaminate the instrumentation so that small amounts of the contaminant are detected in the low level samples. EAS applies the NELAC procedure of critically evaluating each method blank and its effect on the sample batch to decide on the best solution.

12.5 Laboratory Control Spike

An important quality control parameter for analytical methods is the laboratory control spike (LCS). The LCS is also called the laboratory control sample or laboratory blank spike. The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. The ideal LCS will have a matrix similar to the samples. The LCS can be combined with a laboratory control spike duplicate to get the percent recovery and RPD for a measure of precision (See section 12.7 on Duplicates for calculation of percent RPD). These are run at a frequency of 1 per Daily Analytical Batch (or 20 samples).

Due to the nature of the air testing methods, the LCS is a gas cylinder standard that is separate from the actual calibration standard. The assigned value of the LCS is determined by running the LCS against the current curve over a couple of day period and using averaging the values. As the curve drifts current runs are put into the average. If the calibration standard must be used, it will be specified in the case narrative. Spike recoveries are calculated as follows:

$$\%Recovery = 100\ (A)/T$$

where A = Concentration of Laboratory Control Spike
T = Theoretical value of Laboratory Control Spike

The control limits for the LCS can be set in two ways. LCS limits are often specified by the method or project specific QC requirements. The LCS limits can also be set based on historical data on the recoveries of LCS's over time. The historical data can be used to set an upper control limit (UCL) and a lower control limit (LCL). The UCL and the LCL can be used to establish a set of QC criteria for each particular instrument. The calculation of control limits and control charts is explained in detail in the SOP SP7.05 on Calculation of Control Limits. Since most of the tests done by EAS are based on methods that have established QC limits those are used for the LCS.

NELAC Chapter 5 (D.1.1.2.1) has stated that for those test methods that have extremely long lists of analytes, a representative number may be chosen. The following criteria is used to determine the minimum number.

Target Compounds	Minimum Spike	Comments
1 to 10	Spike all normal target compounds	
11 to 20	10 or 80%	
20 or More	16	

NELAC Chapter 5 (D.1.1.2.1) has stated that for large numbers of analytes in the LCS it becomes statistically likely that a few will be outside control limits without indicating that the system is out of control.

The number of allowable marginal exceedance (ME) is based in the number of compounds in the LCS. These should be random, otherwise it may indicate a systematic problem. If there are more marginal exceedances, they should be reported in the case narrative for the client.

Analytes in LCS	Number of Allowed ME
11	1
11 to 30	3
31 to 50	4
51 to 70	6

12.6 Matrix Spike

Matrix effects are somewhat more difficult to characterize. The primary guard against method deficiencies in this area is the analysis of matrix spikes. The spike results provide an assessment of the efficiency of extraction for a particular matrix and can alert the analyst to a system problem. Running the spikes in duplicate also provides verification of system reproducibility on a given matrix. Matrix spikes are analyzed if requested by the client.

12.7 Duplicate Samples

Duplicates are a second analysis of a spike or sample. A duplicate sample analysis is a good way to determine the precision of the method for a particular type of sample. The duplicate spike, either a Laboratory Control Spike Duplicate (LCSD) or a Matrix Spike Duplicate (MSD), allows simultaneous estimation of precision and accuracy on a given sample matrix. A minimum of one duplicate is run per analytical batch.

The relative percent difference (RPD) between duplicate spikes measures the precision of a given analysis and is calculated as follows:

$$\% \text{ RPD} = \frac{S1 - S2}{Sav \times 100}$$

where S1 and S2 = observed concentrations of analyte in the spike and its duplicate
Sav = average of observed analyte concentrations in spike and its duplicate

12.8 Surrogates and Internal Standards

Surrogates are measured amounts of compounds, which are not expected to be found in the sample and are added before sample concentration or extraction. Surrogate recovery is a measure of the concentrator or extraction performance. For organic compound analysis, surrogates provide a guard against matrix bias. Where appropriate and available, these compounds are added to the sample. Since they are chemically similar to

compounds of interest, but are not themselves environmental contaminants, their recovery is considered a good indicator of system performance.

Internal standards are measured amounts of compounds (not expected to be found in the samples), which are added after sample preparation or extraction. They are used in an internal standard calibration method to correct for instrumental bias. Internal standard calibration is used for all of the GC/MS methods, but is not used for the GC methods unless specified in the project.

12.9 Completeness

The main factors within the laboratory's control of data completeness are holding time compliance and batch data validation. Since batch analysis does not start until the method validation is complete and approved, method validation is not generally a factor in data completeness.

Holding Time: This is one of the most important factors in data completeness. EAS works closely with the client to schedule samples to ensure that the EPA or State specified holding times are met.

Data Validation: The technician validates daily batches during the batch analysis. All QC criteria are run and met before sample analysis is started, and individual samples are checked as they are analyzed. If problems occur, samples are reanalyzed to prevent expiration of holding times.

12.10 Control of Data

The combination of all of the QC parameters for a method is referred to as the QC Criteria. This is a list of QC items that are checked to verify the Daily Analytical Batch validation. The QC criteria used by EAS is shown in Section 13.0, under the analytical method.

12.11 Project Specific QC Requirements

In addition to the EAS QC Criteria, there are project or client specific QC criteria, which are different criteria applied to specific projects. The project or client specific QC criteria are entered in the LIMS.

13.0 ANALYTICAL METHODS

The analytical methods used by Environmental Analytical Service, Inc. (EAS) follow established agency methods for testing air, gas, or source samples. For Air Testing, most of the methods come from one of the sources listed below:

Method Source	Method Designation	Method Type
EPA	Toxic Organic (TO)	Ambient Air Indoor Air
EPA	Indoor (IP)	Indoor Air
EPA	EPA (Number)	Source Test Method
ASTM	ASTM	Product Source
NIOSH	NIOSH	Indoor Air Ambient Air
Local, State, and Air District Methods	CARB, SCAQMD, BAAQMD, etc.	

Most of the Local, State, and Air District Methods are similar to the EPA methods but can have different Compound Lists, QC Requirements, and Sample Collection Requirements. EAS does these methods and treats them as Project Specific methods with special compound lists and QC requirements. The EPA IP methods for indoor air testing are very similar to the TO methods, so the TO methods are often requested in place of them.

Modification to Methods

In order to provide our clients with the latest technological improvements and the most extensive target compound lists, EAS (like most air laboratories) uses modified methods. Most laboratories use modified EPA TO Compendium methods because the published methods were originally prepared as a guidance document. These published methods often lack specifics on target compounds or have unrealistic QC requirements established for a limited list of compounds or estimated from other methods. For example, ASTM methods (for compounds such as methane) were originally written for petroleum testing but have been modified for use in ambient air analysis. All methods that have been modified are designated “modified” on the analytical reports.

Methods used for source testing follow established and well-documented methods and can be run with no modification for compliance tests ***if requested in advance***.

Laboratory Test Methods

Environmental Analytical Service, Inc. (EAS) specializes in the analysis of ambient air, indoor air, source test samples, and gas samples, testing for organic compounds. The

instrumentation for this testing is gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS), and high-pressure liquid chromatography (HPLC).

Environmental Testing

The Environmental testing methods and QC requirements used by EAS are based on the established methods of the Environmental Protection Agency (EPA), American Society for Testing of Materials (ASTM), NIOSH, OSHA, and California Air Resources Board (CARB).

NELAC Test Methods

Environmental Analytical Service has its National Environmental Laboratory Accreditation with the Florida Department of Health Services.

The following list of test methods are part of the NELAC Accreditation.

Method	Reference
EPA TO-15	13.7
EPA TO-11	13.4
EPA TO-4A	13.2

13.1 TO-3/15 Detailed Hydrocarbon Analysis (DHA) PAMS Compounds

Method TO-3/15 DHA uses cryogenic trapping and a gas chromatograph with a flame ionization detector (FID) to measure hydrocarbons collected in Summa Canisters. The EAS concentrator used for the method complies with EPA TO-3, EPA TO-14A (simple referred to as TO-14) and EPA TO-15 depending on the water management system used. For the EPA TO-15 FID a three bed VOC sorbent trap is used for water management as described in the TO-15 Method. To differentiate between TO-15 by GC/MS the designation TO-15 FID is used. EAS performs a modified version of the method that follows the calibration protocol contained in the EPA Guidance Document "Technical Assistance Documents for Sampling and Analysis of Ozone Precursors". The method is used to determine individual hydrocarbons, including the 55 Photochemical Assessment Monitoring Stations (PAMS) compounds in air and gas samples. The FID is calibrated using hexane and the response of individual hydrocarbons is calculated against these compounds in ppbC according to the procedure described in the guidance document. This method can be used for PIANO analysis of air and gas samples.

Table 13.1c
Summary of QC Criteria for TO-3/15 Modified for DHA and PAMS Hydrocarbon Analysis

Parameter	EAS TO-3/15 DHA Modified	TO-3 PAMS Method
Initial Calibration	Four points relative response factors run on Hexane. See Table 13.1d	4 points with a linear regression Weekly
Continuing Calibration Verification (CCV)	Hexane Daily (24 hours) See Table 13.1d	Prior to Sample Analysis and every 4-6 hours PAMS: Calibrate for hexane propane
Method Blank	Target analytes less than RL	Not Specified
Laboratory Control Spike	With daily batch See Table 13.1d	Not Specified
Duplicate-Either Lab Control Dup Sample Dup	With daily batch See Table 13.1d	Not Specified
Canister Holding Times	30 days	Not Specified
Canister Certification	Certification <0.2 ppbv by full scan GC/MS	

Laboratory Control Spike is made up of a short list of hydrocarbon compounds. They appear in Table 13.1d in bold.

Table 13.1d
Method TO-3 DHA/15 Compound List

The following compounds can be reported as part of the TO-3/15 DHA analysis. All of the compounds are calibrated against hexane according to the EPA guidance document. Compounds in bold are in the LCS sample. The method detection limit (MDL) and reporting limit (RL) are determined in ppbC for hexane and adjusted for the other compounds based on the number of carbon atoms.

Analyte			Criteria			
	MDL ppbV	RL ppbV	ICAL %RSD	CCV %R	LCS %R	Duplicate %RPD
Ethene	0.40	1.20				<25
Acetylene	0.40	1.20				<25
Ethane	0.40	1.20				<25
Propene	0.27	0.80				<25
Propane	0.27	0.80				<25
i-Butane	0.20	0.60				<25
Methanol	0.80	2.40				<25
1-Butene	0.20	0.60				<25
n-Butane	0.20	0.60				<25
t-2-Butene	0.20	0.60				<25
c-2-Butene	0.20	0.60				<25
Ethanol	0.40	1.20				<25
i-Pentane	0.16	0.48				<25
1-Pentene	0.16	0.48				<25
Isopropanol	0.27	0.80				<25
n-Pentane	0.16	0.48				<25
Isoprene	0.16	0.48				<25
t-2-Pentene	0.16	0.48				<25
c-2-Pentene	0.16	0.48				<25
2,2-Dimethylbutane	0.13	0.40				<25
Cyclopentene	0.16	0.48				<25
2,3-Dimethylbutane	0.13	0.40				<25
2-Methylpentane	0.13	0.40				<25
3-Methylpentane	0.13	0.40				<25
n-Hexane	0.13	0.40	<30	<30		<25
Methylcyclopentane	0.13	0.40				<25
2,4-Dimethylpentane	0.11	0.34				<25
Benzene	0.13	0.40			**70-130**	<25
Cyclohexane	0.13	0.40				<25
2-Methylhexane	0.11	0.34				<25
2,3-Dimethylpentane	0.11	0.34				<25

Analyte	MDL ppbV	RL ppbV	Criteria			
			ICAL %RSD	CCV %R	LCS %R	Duplicate %RPD
3-Methylhexane	0.11	0.34				<25
2,2,4-Trimethylpentane	0.10	0.30			**70-130**	<25
n-Heptane	0.11	0.34				<25
Methylcyclohexane	0.11	0.34				<25
2,5-Dimethylhexane	0.10	0.30				<25
2,4-Dimethylhexane	0.10	0.30				<25
2,3,4-Trimethylpentane	0.10	0.30				<25
Toluene	0.11	0.34			**70-130**	<25
2,3-Dimethylhexane	0.10	0.30				<25
2-Methylheptane	0.10	0.30				<25
3-Methylheptane	0.10	0.30				<25
n-Octane	0.10	0.30				<25
Ethylbenzene	0.10	0.30				<25
m,p-xylene	0.10	0.30			**70-130**	<25
Styrene	0.10	0.30				<25
o-xylene	0.10	0.30			**70-130**	<25
n-Nonane	0.09	0.27				<25
i-Propylbenzene	0.09	0.27				<25
n-propylbenzene	0.09	0.27				<25
a-Pinene	0.08	0.24				<25
3-Ethyltoluene	0.09	0.27				<25
4-Ethyltoluene	0.09	0.27				<25
1,3,5-Trimethylbenzene	0.09	0.27			**70-130**	<25
2-Ethyltoluene	0.09	0.27				<25
b-Pinene	0.08	0.24				<25
1,2,4-Trimethylbenzene	0.09	0.27				<25
n-Decane	0.08	0.24				<25
1,2,3-Trimethylbenzene	0.09	0.27				<25
d-Limonene	0.08	0.24				<25
1,3-Diethylbenzene	0.08	0.24				<25
1,4-Diethylbenzene	0.08	0.24				<25
n-Butylbenzene	0.08	0.24				<25
Undecane	0.07	0.22				<25
Decane	0.07	0.20				<25

13.2 TO-4A Pesticides and PCB's

Method TO-4A is used for the analysis of pesticides and PCB's in ambient and indoor air. These methods can be used for organochlorine and organophosphate pesticides. Method TO-4A uses a high volume air sampler that samples 250 L/min of air. The filter and PUF plug are extracted for 18 hours in a Soxhlet extractor using hexane/diethyl ether (as specified in the method), and are concentrated to their final volume. Method TO-4A allows several types of detectors but recommends GC/MS-SIM EAS uses the GC/MS SIM detector. For high level samples a GC/MS full scan method is sued.

The EAS modifications to the method include QC criteria and target list. The TO-4A does not specify many elements of QC. The QC criteria also reflect a GC/MS SIM analysis.

Table 13.2a
Summary of QC Criteria for TO-4A

Parameter	EAS TO-4A Modified	TO-4A Method
DFTPP Tune	Daily (24 hours) Not Required for SIM	Not Specified
Tuning Criteria	Not Required for SIM	Not Specified
Initial Calibration	5 points minimum See Table 13.2b One compound may exceed QC limits A PCB curve is generated when PCB's are detected and identified	
Continuing Calibration Verification (CCV)	Daily (24 hours) See Table 13.2b	
Internal Standard (IS) RT	See Table 13.2c	
Surrogate	See Table 13.2d	p-terphenyl-d14 is "commonly used" 60-120% recovery
Method Blank	No target analytes above RL Every 20 samples	<10 ng/sample for individual components. One per extraction batch or every 10 samples. Method specifies both a field and process blank.
Laboratory Control Spike	With Daily Batch See Table 13.2b	
Duplicate Laboratory Control Dup Sample Dup	Duplicate with each 20 samples See Table 13.2b	Recoveries of 65 to 125%
Extraction	Soxhlet for 18 hours in 10% diethyl ether in hexane	TO-10A specifies 5% diethyl ether in hexane. TO-4 specifies 10% diethyl ether in hexane. TO-10A specifies a minimum 16 hr extraction.
Holding Times and Storage	Store at <4C during shipment and at laboratory Extract within 7 days of sampling. Analyze within 40 days of extraction	Store at <4C during shipment and after receipt at laboratory Extract within 7 days of sampling. Analyze within 40 days of extraction

Table 13.2b
Method TO-4A Compound List

Analyte	MDL ug	LOQ ug	Criteria		
			ICAL CCV %D	LCS %R	Duplicate %RPD
a-BHC	0.1	0.5	<30	65-125	<30
g-BHC	0.1	0.5	<30	65-125	<30
b-BHC	0.1	0.5	<30	65-125	<30
d-BHC	0.1	0.5	<30	65-125	<30
Heptachlor	0.1	0.5	<30	65-125	<30
Aldrin	0.1	0.5	<30		<30
Heptachlor Epoxide	0.1	0.5	<30		<30
Endosulfan I	0.1	0.5	<30	65-125	<30
p,p'-DDE	0.1	0.5	<30	65-125	<30
Dieldrin	0.1	0.5	<30		<30
Endrin	0.1	0.5	<30		<30
Endosulfan II	0.1	0.5	<30	65-125	<30
4,4'-DDD	0.1	0.5	<30	65-125	<30
Endrin Aldehyde	0.1	0.5	<30		<30
Endosulfan Sulfate	0.1	0.5	<30		<30
4,4'-DDT	0.1	0.5	<30		<30
Endrin Ketone	0.1	0.5	<30		<30
Methoxychlor	0.1	1	<30		<30
Determined from EIC					
Technical-Chlordane	1	10			
Toxaphene	1	10			
Aroclor 1221	1	10			
Aroclor 1232	1	10			
Aroclor 1242	1	10			
Aroclor 1248	1	10			
Aroclor 1254	1	10			
Aroclor 1260	1	10			

Table 13.2c
Internal Standards

Compound	Target Recovery %
Acenaphthene-d10	50- 200
Phenanthrene-d10	50- 200

Table 13.2d
Surrogate Standards

Compound	Recovery %
p-Terphenyl-d14	50- 150

13.3 TO-8 Phenols and Cresols

Method TO-8 is used for the analysis of phenols in air samples that are collected in midget impingers. These samples are analyzed by reverse phase high-pressure liquid chromatography (HPLC) using UV detection.

The EAS modification to this method includes the calibration procedure and QC criteria.

Table 13.3a
Summary of QC Criteria for TO-8

Parameter	EAS TO-8 Modified	TO-8 Method
Initial Calibration	3 points minimum 0.12ug/ml – 1.2ug/ml See Table 13.3b	Method specifies standards run in triplicate; 5 points minimum. 0.999 correlation coefficient
Continuing Calibration Verification (CCV)	Daily (24 hours) Mid range standard Same Criteria as initial calibration	Daily (24 hours) Mid range standard <10% D
Method Blank	No target analytes above LOQ	Specifies use of a field blank, see comments. One field blank per 10 samples.
Laboratory Control Spike	See Table 13.3b	Not specified.
Duplicate Lab Control Dup Sample Dup	1 duplicate with each 20 samples See Table 13.3.b	Specifies use of a sample duplicate, see comments. One sample duplicate per 10 samples. <20% D
Holding Times and Storage	14 days from sampling date Store at 4C	Time between refrigeration and analysis not to exceed 48 hours

13.4 TO-11A Aldehydes and Ketones

The EAS method for aldehydes and ketones is a combination of the EPA TO-11A and CARB 1004 and is used for the analysis of aldehydes and ketones that have been collected in DNPH sorbent cartridges in ambient air and automobile exhaust samples. The DNPH solution forms a hydrazone derivative with the target compounds. The cartridges are extracted with acetonitrile and are analyzed by reverse phase high-pressure liquid chromatography (HPLC) using UV detection. EAS has modified the method and QC requirement to be able to accommodate both methods and target lists. The calibration and QC are based on the CARB 1004 target list, which represents the commonly found carbonyl compounds in both air and exhaust samples, but the initial calibration includes all the target compounds from both methods. The cartridges are available in a diffusion sampler package.

The EAS modification to this method includes the calibration procedure and QC criteria. The initial calibration for the combined method is done by analyzing duplicate, 5 calibration points for the CARB 1004 standard and duplicate, 5 calibration points for a second source TO-11A standard. With this procedure an initial calibration is generated with 4 replicate runs on most of the compounds.

Table 13.4a
Summary of QC Criteria for TO-11A and CARB 1004

Parameter	EAS TO-11A Modified CARB 1004 Modified	TO-11A Method
Initial Calibration	5 points minimum TO-11A and CARB 1004 Duplicate Runs See Table 13.4b Curve prepared as needed	Method specifies standards run in triplicate. Method criteria 0.999 correlation coefficient. Minimum every 6 months
Calibration Check Sample (CCS)	With Initial Calibration TO-11A Standard See Table 13.4b	
Continuing Calibration Verification (CCV)	Daily (24 hours) CARB 1004 Standard Mid range standard See Table 13.4c	<15% D for calibration verifications
Method Blank	Less than LOQ	Average Blank Subtraction
Laboratory Control Spike	1 per Daily Batch See Table 13.4c	Not Specified
Duplicate Lab Control Dup Sample Dup	Duplicate with each 20 samples See Table 13.4c	50% of sampling events should have a collocated sample. <20% D
Holding Times	Refrigerated Samples 30 days	Extract 14 days from Sample Date
Ending Table	Check 11A & CARB Method	Technically since there is no IS we need an ending standard

Table 13.4b TO/11ACARB 1004 Modified Calibration, MDL, RL for Combined List

The EPA TO-11A and CARB 1004 Standards are both used for Initial Calibration

Analyte	MDL ug	LOQ ug	Criteria		
			ICAL %D		
Formaldehyde	0.05	0.24	<20		
Acetaldehyde	0.08	0.24	<20		
Acrolein	0.20	0.24	<20		
Acetone	0.20	0.24	<20		
Propionaldehyde	0.08	0.24	<20		
Crotonaldehyde	0.08	0.24	<20		
Methacrolein	0.10	0.24	<20		
Butyraldehyde	0.10	0.24	<20		
2-Butanone	0.10	0.24	<20		
Butyraldehyde	0.10	0.24	<20		
Isovaleraldehyde	0.10	0.24	<20		
Valeraldehyde	0.10	0.24	<20		
o-Tolualdehyde	0.10	0.24	<20		
m-Tolualdehyde	0.10	0.24	<20		
p-Tolualdehyde	0.10	0.24	<20		
Hexaldehyde	0.20	0.24	<20		
2,5-Dimethylbenzaldehyde	0.20	0.24	<30		

Table 13.4c CARB 1004 List

Standard used for CCV, LCS, Duplicate TO-11A and CARB 1004

The CARB 1004 Standard is used for the CCV, LCS, LCD

Analyte	MDL ug	LOQ ug	Criteria		
			CCV %D	LCS %R	Duplicate %RPD
Formaldehyde	0.05	0.24	<20	80-120	<20
Acetaldehyde	0.08	0.24	<20	80-120	<20
Acrolein	0.20	0.24	<20	80-120	<20
Acetone	0.20	0.24	<20	80-120	<20
Propionaldehyde	0.08	0.24	<20	80-120	<20
Crotonaldehyde	0.08	0.24	<20	80-120	<20
Methacrolein	0.10	0.24	<20	80-120	<20
Butyraldehyde	0.10	0.24	<20	80-120	<20
2-Butanone	0.10	0.24	<20	80-120	<20
Benzaldehyde	0.10	0.24	<20	80-120	<20
Valeraldehyde	0.10	0.24	<20	80-120	<20
m-Tolualdehyde	0.10	0.24	<20	80-120	<20
Hexaldehyde	0.20	0.24	<20	80-120	<20

Table 13.4d TO-11A

The EPA TO-11A Standard used for CSS

Analyte	MDL ug	LOQ ug	Criteria		
			CCS %D		
Formaldehyde	0.05	0.24	<20		
Acetaldehyde	0.08	0.24	<20		
Acrolein	0.20	0.24	<20		
Acetone	0.20	0.24	<20		
Propionaldehyde	0.08	0.24	<20		
Crotonaldehyde	0.08	0.24	<20		
Butyraldehyde	0.10	0.24	<20		
Benzaldehyde	0.10	0.24	<20		
Isovaleraldehyde	0.10	0.24	<20		
Valeraldehyde	0.10	0.24	<20		
o-Tolualdehyde	0.10	0.24	<20		
m-Tolualdehyde	0.10	0.24	<20		
p-Tolualdehyde	0.10	0.24	<20		
Hexaldehyde	0.20	0.24	<20		
2,5-Dimethylbenzaldehyde	0.20	0.24	<30		

13.5 TO-12 NMOC

TO-12 is run as part of EPA TO-3/15 FID. See this method for procedure and QC criteria.

13.6 TO-13A PAH Compounds

Method TO-13A is used for the analysis of samples collected in either high or low volume PUF/XAD cartridges with a filter. The filter and cartridge are extracted with pentane/ethyl ether in a soxhlet extractor as described in the method. The solvent is concentrated and analyzed by GC/MS in either the full scan or SIM mode. The target list for the method is a select group of polynuclear aromatic hydrocarbons (PAH). The method is also used for a modified 8270.

The EAS modifications to the method include the target list, calibration, and QC criteria.

Summary of Extraction Method for TO-13A

Samples are extracted using hexane/diethyl ether in a soxhlet extractor as described in the method and are concentrated to a 1 ml volume.

Parameter	EAS Method	TO-13A
Extraction	Soxhlet for 18 hours in 10% diethyl ether in hexane. EAS uses a combination PUF and XAD sampling media, so dichloromethane is not suitable for extraction.	Soxhlet for 18 hours in 10% diethyl ether in hexane. Extraction solvent for XAD is listed as dichloromethane. PUF extraction solvent is listed is hexane.
Sample Medium	Whatman Quartz Filter and PUF Cartridge, High Volume	Whatman Quartz Filter and PUF Cartridge, High Volume
Holding Times	Store at 4C and extract in 7 days. Analyze within 40 days of extraction	Store at 4C and extract in 7 days. Analyze within 40 days of extraction

Table 13.6a
Summary of QC Criteria for TO-13A

Parameter	EAS TO-13A Modified	TO-13A Method
Extraction	18 hours with 10% ethyl ether/hexane mix.	18 hours with 10% ethyl ether/hexane mix.
DFTPP Tune	Every 24 hours	Every 12 hours
Tuning Criteria with DFTPP	TO-13 50 ng DFTPP TO-13A criteria	TO-13 50 ng DFTPP TO-13A criteria
Initial Calibration	5 Points Minimum 1 ug/ml to 50 ug/ml See Table 13.6d One cmd may exceed	5 Points Minimum 0.1 – 2.5 ug/ml <30% RSD for compounds listed in the method.
Calibration Check Sample (CCS)	Not Run	After Initial Calibration
Continuing Calibration Verification (CCV)	24 hours – Midpoint on Curve See Table 13.6d	12 hours – Midpoint on Curve Same criteria as initial calibration
Internal Standard (IS)	See Table 13.6b	1,4-Dichlorobenzene-d4 is not listed as an internal standard in the TO-13A method, but does appear in EPA 8270. Response 50-200%
Surrogate (lab)	All samples prior to extraction See Table 13..6c	Fluorene-d10 Pyrene-d10 20 uL of a 50 ug/mL solution 60-120% recovery
Method Blank	1 per batch of 20 Less than LOQ	With each batch of up to 20 samples. Less than MDL
Laboratory Control Spike	1 per batch of 20 samples See Table 13.6c	With each batch of up to 20 samples 60-120% recovery
Duplicate Lab Control Dup Sample Dup	1 per batch of 20 samples See Table 13.6d	Method does not specify an LCD

Table 13.6b
Internal Standards

Compound	Recovery %
1,4-Dichlorobenzene-d4	50- 200
Naphthalene-d8	50- 200
Acenaphthene-d10	50- 200
Phenanthrene-d10	50- 200
Chrysene-d12	50- 200
Perylene-d12	50- 200

Table 13.6c
Surrogate Standards

Compound	Recovery %
Nitrobenzene-d5	50- 200
2-Fluorobiphenyl	50- 200
p-Terphenyl-d14	50- 200

Table 13.6d
Method TO-13A PAH Compound List

Analyte	Full Scan LOQ ug	SIM LOQ ug	Criteria	
			ICAL CCV %D	Duplicate %D
Naphthalene	1.0	0.1	<30	<30
Acenaphthene	1.0	0.1	<30	<30
Fluorene	1.0	0.1	<30	<30
Phenanthrene	1.0	0.1	<30	<30
Anthracene	1.0	0.1	<30	<30
Fluoranthene	1.0	0.1	<30	<30
1-Methylnapthalene	1.0	0.1	<30	<30
2-Methylnapthalene	1.0	0.1	<30	<30
Pyrene	1.0	0.1	<30	<30
Chrysene	1.0	0.1	<30	<30
Benz[a]anthracene	1.0	0.1	<30	<30
Benzo(b)fluoranthene	1.0	0.1	<30	<30
Benzo(k)fluoranthene	1.0	0.1	<30	<30
Benzo(a)pyrene	1.0	0.1	<30	<30
Indeno(1,2,3-cd)pyrene	1.0	0.1	<30	<30
Dibenz(a,h)anthracene	1.0	0.1	<30	<30
Benzo(g,h,i)perylene	1.0	0.1	<30	<30

Table 13.6e
Laboratory Control Standards

Analyte	LCS %R
1,4-Dichlorobenzene	50-200
N-nitrosodi-n-propylamine	50-200
1,2,4-Trichlorobenzene	50-200
Acenaphthene	50-200
Pyrene	50-200

These are a modification of the EPA Method 8270 limits.

13.7 TO-15 and TO-14A Full Scan GC/MS Volatile Organic Compounds

Method TO-15 and TO-14A are used for measuring volatile organic compounds collected in SUMMA canisters by Full Scan GC/MS. The difference between TO-14 and TO-15 is the method of water management. The TO-14 method uses a Nafion dryer to remove water, and TO-15 uses a three bed sorbent trap to remove water. The designation of TO-15 is applied to most samples because it is the most current method. The sample size can be between 0.025ml and 1000ml depending on the levels of the VOC's in the samle. The sample is thermally desorbed and cryofocused on the GC column. The samples are analyzed by GC/MS full scan or by selected ion monitoring (SIM). Method TO-15 does not specify a target list of compounds.

The EAS modifications to the method include the target list, limit of quantitation, and QC criteria. EAS also runs TO-15 with no modification for a special target group. EAS has standards and can analyze over 125 volatile organic compounds.

Note: The calibration criteria, LCS criteria, and LCD criteria used in writing EPA TO-15 was based on a 16 compound target that covers most compounds detected in air and gas samples. A full LCS is run at the clients request. The EAS (and other laboratories) modifications to these parameters are based on a substantially larger list that contains oxygenate and SVOC compounds which can have RSD values greater then 30%. These criteria are listed by compound in Table 13.7b

TO-15 Sample Collection Criteria

Parameter	EAS TO-15 Modified	TO-15 & TO-14A Method
Canister Holding Times Tedlar Bag	30 days 72 hours	30 days from sampling date
Canister Certification	Certification <RL by full scan GC/MS for target compounds	Certification <0.2 ppbv
Passive Flow Regulator	Certification <RL with the Canister for target compounds	

Canisters and flow regulators are batch certified unless individual certification is requested. It is recommended that canisters and flow regulators be individually certified for low level analysis, or soil gas analysis.

Table 13.7a
Summary of QC Criteria for TO-15 Modified

The following Table lists the QC Criteria for the EAS TO-15 Method. Besides the Normal or Standard TO-15 63 target list, EAS also offers Special Lists such as the Extended List (similar to 8260B list) and the Indoor Air list. There are many compounds on these lists that are not standard TO-15 compounds. An initial calibration curve is prepared for these compounds but the continuing calibration check is preformed on the normal TO-15 list and curve.

Parameter	EAS TO-15 Modified	TO-15 Method
BFB Tune	Daily (24 hour) 12 hours if Required	Daily (24 hour)
Tuning Criteria with BFB	TO-15 Tune Criteria	TO-15 Tune Criteria
Initial Calibration Verification (ICV)	Five points minimum for normal TO-15 compounds See Table 13.7b 90% compounds meet criteria	5 points minimum RSD < 30% TO-14 List 2 Compounds can exceed criteria by 10%
Calibration Check Sample (CCS) with each curve	Yearly Same Percent RSD as Initial Calibration	After Initial Calibration Same Percent RSD as Initial Calibration
Continuing Calibration Verification (CCV)	Daily (24 hours) See Table 13.7b 90% compounds meet criteria	Daily (24 hours) 10 ppbv Std Same Percent RSD as Initial Calibration
Internal Standard (IS)	Pentafluorobenzene 1,4-Difluorobenzene RT < 0.5 min daily std. Response 50% to 200%	Pentafluorobenzene Chlorobenzene-d5 1,4-Difluorobenzene RT < 0.33 min daily std. Response 60% to 140%
Surrogate	Toluene-d8 70-130% recovery	Toluene-d8 70-130% recovery
Method Blank	<RL	<RL
Laboratory Control Spike	1 per Daily Analytical Batch 70-130% for LCS list See Table 13.7b	1 per Daily Batch 70-130% for LCS list
Duplicate Lab Control Dup Sample Dup	Duplicate with each 20 samples <30% for LCS spike list See Table 13.7b 90% meet criteria	1 Duplicate with each 20 samples <25% for LCS spike list
Ending Standard	None unless specified	Not required since surrogate are used

Table 13.7b
Method TO-15 Compounds Calibration and QC

The TO-15 Compound List includes 63 volatile organic compounds. See Table 13.7d for the TO-15 Low Level MDL and RL values. The following MDL and RL are based on a 1000 ml load volume.

Compound	Initial Cal %D	CCV %D	LCS %R	Precision %D
Dichlorodifluoromethane	<30	<30	70-130	<25
Chloromethane	<30	<30	70-130	<25
Freon 114	<30	<30	70-130	<25
Vinyl chloride	<30	<30	**70-130**	<25
1,3-Butadiene	<30	<30	70-130	<25
Bromomethane	<30	<30	70-130	<25
Chloroethane	<30	<30	70-130	<25
Ethanol	<50	<50	60-140	<25
Trichlorofluoromethane	<30	<30	70-130	<25
Acetone	<40	<40	60-140	<25
2-propanol	<50	<50	60-140	<25
1,1-Dichloroethene	<30	<30	**70-130**	<25
Freon 113	<30	<30	70-130	<25
Dichloromethane	<30	<30	**70-130**	<25
Carbon disulfide	<30	<30	70-130	<25
trans-1,2-Dichloroethene	<30	<30	70-130	<25
Methyl tert butyl ether	<30	<30	70-130	<25
1,1-Dichloroethane	<30	<30	**70-130**	<25
Vinyl acetate	<40	<40	70-130	<25
2-Butanone	<40	<40	60-140	<25
Ethyl acetate	<40	<40	60-140	<25
Bromochloromethane	<30	<30	60-140	<25
Tetrahydrofuran	<40	<40	60-140	<25
cis-1,2-Dichloroethene	<30	<30	70-130	<25
Chloroform	<30	<30	**70-130**	<25
1,1,1-Trichloroethane	<30	<30	**70-130**	<25
1,2-Dichloroethane	<30	<30	70-130	<25
Cyclohexane	<30	<30	70-130	<25
Benzene	<30	<30	**70-130**	<25

Compound	Initial Cal %D	CCV %D	LCS %R	Precision %D
Carbon tetrachloride	<30	<30	**70-130**	<25
n-Heptane	<30	<30	70-130	<25
1,2-Dichloropropane	<30	<30	70-130	<25
1,4 Dioxane	<40	<30	60-140	<25
Trichloroethene	<30	<40	**70-130**	<25
Bromodichloromethane	<30	<30	70-130	<25
Methyl methacrylate	<40	<30	70-130	<25
4-Methyl-2-pentanone	<40	<40	60-140	<25
cis-1,3-Dichloropropene	<30	<40	70-130	<25
Toluene	<30	<30	**70-130**	<25
trans-1,3-Dichloropropene	<30	<30	70-130	<25
1,1,2-Trichloroethane	<30	<40	70-130	<25
2-Hexanone	<40	<30	60-140	<25
Dibromochloromethane	<30	<30	70-130	<25
1,2-Dibromoethane	<30	<30	70-130	<25
Tetrachloroethene	<30	<30	**70-130**	<25
Chlorobenzene	<30	<30	70-130	<25
Ethylbenzene	<30	<30	**70-130**	<25
m,p-Xylenes	<30	<30	**70-130**	<25
Styrene	<30	<30	70-130	<25
Bromoform	<30	<30	60-140	<25
o-Xylene	<30	<30	**70-130**	<25
1,1,2,2-Tetrachloroethane	<30	<30	70-130	<25
4-Ethyltoluene	<30	<30	60-140	<25
1,3,5-Trimethylbenzene	<30	<30	**70-130**	<25
1,2,4-Trimethylbenzene	<30	<30	70-130	<25
1,3-Dichlorobenzene	<40	<40	70-130	<25
Benzyl chloride	<40	<40	50-150	<25
1,4-Dichlorobenzene	<40	<40	70-130	<25
1,2-Dichlorobenzene	<40	<40	70-130	<25
1,2,4-Trichlorobenzene	<50	<50	50-150	<25
Naphthalene	<50	<50	50-150	<25
Hexachlorobutadiene	<50	<50	50-150	<25
1,1-Difluoroethane	<40	<40	60-140	<25

Table 13.7c
Method TO-15 Compounds with MDL and RL

The TO-15 Compound List includes 63 volatile organic compounds. The following MDL and RL are based on a 1000 ml load volume. The MDL for Dichlorofluoromethand, chloromethane, and trichlorofluoromethane are set about their global background level.

CAS#	Compound	MDL PPBV	RL PPBV	MDL UG/M3	RL UG/M3
75-71-8	Dichlorodifluoromethane	0.65	0.75	0.25	1.24
74-87-3	Chloromethane	0.65	0.75	0.10	0.52
76-14-2	Freon 114	0.05	0.25	0.35	1.76
75-01-4	Vinyl chloride	0.05	0.25	0.13	0.64
106-99-0	1,3-Butadiene	0.05	0.25	0.11	0.56
74-83-9	Bromomethane	0.10	0.25	0.19	0.98
75-00-3	Chloroethane	0.05	0.25	0.13	0.66
64-17-5	Ethanol	0.25	0.75	0.47	1.41
75-69-4	Trichlorofluoromethane	0.35	0.40	0.28	1.35
67-64-1	Acetone	0.25	0.63	0.59	1.46
67-63-0	2-propanol	0.57	0.63	0.61	1.41
75-35-4	1,1-Dichloroethene	0.05	0.25	0.20	0.98
76-13-1	Freon 113	0.05	0.24	0.38	1.83
75-09-2	Dichloromethane	0.10	0.24	0.35	0.84
75-15-0	Carbon disulfide	0.35	0.63	0.78	1.44
156-60-5	trans-1,2-Dichloroethene	0.05	0.18	0.20	0.72
1634-04-4	Methyl tert butyl ether	0.05	0.18	0.18	0.66
75-34-3	1,1-Dichloroethane	0.05	0.25	0.20	1.01
108-05-4	Vinyl acetate	0.05	0.22	0.18	0.77
78-93-3	2-Butanone	0.20	0.50	0.59	1.50
141-78-6	Ethyl acetate	0.10	0.22	0.36	0.70
74-97-5	Bromochloromethane	0.05	0.13	0.26	0.70
109-99-9	Tetrahydrofuran	0.10	0.25	0.29	0.74
156-59-2	cis-1,2-Dichloroethene	0.10	0.27	0.40	1.06
67-66-3	Chloroform	0.05	0.25	0.24	1.22
71-55-6	1,1,1-Trichloroethane	0.05	0.22	0.27	1.21
107-06-2	1,2-Dichloroethane	0.05	0.23	0.20	0.92
110-82-7	Cyclohexane	0.05	0.19	0.17	0.66
71-43-2	Benzene	0.10	0.25	0.16	0.81
56-23-5	Carbon tetrachloride	0.05	0.24	0.31	1.49
142-82-5	n-Heptane	0.25	0.62	1.02	2.48
78-87-5	1,2-Dichloropropane	0.05	0.24	0.23	1.11
123-91-1	1,4 Dioxane	0.41	0.50	0.72	1.47

CAS#	Compound	MDL PPBV	RL PPBV	MDL UG/M3	RL UG/M3
79-01-6	Trichloroethene	0.03	0.23	0.16	1.25
75-27-4	Bromodichloromethane	0.05	0.10	0.33	0.67
80-62-6	Methyl methacrylate	0.20	0.68	0.82	2.77
108-10-1	4-Methyl-2-pentanone	0.20	0.76	0.82	3.10
10061-01-5	cis-1,3-Dichloropropene	0.05	0.26	0.23	1.18
108-88-3	Toluene	0.10	0.25	0.38	0.98
10061-02-6	trans-1,3-Dichloropropene	0.05	0.26	0.23	1.18
79-00-5	1,1,2-Trichloroethane	0.05	0.26	0.27	1.40
591-78-6	2-Hexanone	0.25	0.63	1.02	2.90
124-48-1	Dibromochloromethane	0.05	0.10	0.43	0.85
106-93-4	1,2-Dibromoethane	0.05	0.12	0.38	0.93
127-18-4	Tetrachloroethene	0.03	0.12	0.20	0.82
108-90-7	Chlorobenzene	0.05	0.23	0.23	1.05
100-41-4	Ethylbenzene	0.11	0.26	0.46	1.15
1330-20-7	m,p-Xylenes	0.11	0.26	0.46	1.15
100-42-5	Styrene	0.10	0.26	0.44	1.10
75-25-2	Bromoform	0.05	0.07	0.52	0.69
95-47-6	o-Xylene	0.10	0.26	0.45	1.12
79-34-5	1,1,2,2-Tetrachloroethane	0.05	0.12	0.34	0.85
622-96-8	4-Ethyltoluene	0.17	0.41	0.81	2.04
108-67-8	1,3,5-Trimethylbenzene	0.10	0.26	0.51	1.27
95-63-6	1,2,4-Trimethylbenzene	0.10	0.25	0.50	1.25
541-73-1	1,3-Dichlorobenzene	0.10	0.14	0.60	1.11
100-44-7	Benzyl chloride	0.10	0.51	0.52	3.14
106-46-7	1,4-Dichlorobenzene	0.10	0.17	0.60	1.04
95-50-1	1,2-Dichlorobenzene	0.10	0.16	0.60	0.97
120-82-1	1,2,4-Trichlorobenzene	0.25	0.34	1.85	2.55
91-20-3	Naphthalene	0.05	0.08	0.26	0.42
87-68-3	Hexachlorobutadiene	0.25	0.27	2.67	2.83
75-37-6	1,1-Difluoroethane	0.75	1.50	2.03	4.05

Additional Target Compounds

EAS can analyze over 125 volatile organic compounds by TO-15. There is an indoor air pollutant list, a modified 8270 list, siloxane list, and a hydrocarbon list. These lists are run using the QC criteria listed above. A 5 point initial calibration is prepared for all target compounds, the above continuing calibration list is used, and the above LCS and LCD list and criteria is used unless specified in advance.

TO-15 Modified Siloxane

Siloxanes are measured in ambient air and source samples using the normal TO-15 Modified method described above. A separate processing method with a 5 point initial calibration is used for a secondary processing of the main TO-15 analysis. The QC including the LCS and LCD from the main TO-15 analysis (Table 13.7b) is reported unless otherwise specified. Additional siloxanes can be added at TIC compounds

Table 13.7c
Method TO-15 Siloxanes Target Compounds

CAS#	Compound	MDL PPBV	RL PPBV	ICAL CCV %	LCS %	DUP RPD%
75-76-3	Tetramethylsilane	20.0	40.0	<40	NA	NA
3277-26-7	Tetramethyldisiloxane	5.0	10.0	<40	NA	NA
107-46-0	Hexamethyldisiloxane	5.0	10.0	<40	NA	NA
541-05-9	Hexamethylcyclotrisiloxane	5.0	10.0	<40	NA	NA
107-51-7	Octamethyltrisiloxane	5.0	10.5	<40	NA	NA
556-67-2	Octamethylcyclotetrasiloxane	5.0	10.0	<40	NA	NA
141-62-8	Decamethyltetrasiloxane	5.0	10.0	<40	NA	NA
541-02-6	Decamethylcyclopentasiloxane	25.0	50.0	<40	NA	NA

Table 13.7c
Method TO-15 Siloxane by TIC

CAS#	Compound	MW		Ion	Ion	Ion
1066-40-6	Trimethylsilanol	98.5	TIC	75	47	
1825-63-4	Propoxytrimethylsilane	132.1	TIC	117	75	
1438-82-0	Pentamethyldisiloxane	148.1	TIC	113	147	
1825-62-3	Ethoxytrimethylsilane	118.1	TIC	103	75	
1825-65-6	Butoxytrimethylsilane	146.1	TIC	111	75	
540-97-6	Dodecamethylcyclohexasiloxane	348.1	TIC	73	147	207
141-63-9	Dodecamethylpentasiloxane	281	TIC	281	147	

The dodecamethylsiloxanes can also be done by TIC.

TO-15 Modified TPH and TVOC

The total petroleum hydrocarbons (TPH) and Total Volatile Organic Compounds (TVOC) can be determined from the TO-15 GC/MS analysis by integrating the total ion chromatogram from the indicated range, subtract the internal standard areas, and then calculate the TPH against the Toluene-d8 internal standard. The TPH result is normally reported as gasoline. The results for TPH and TVOC includes BTEX and other target compounds.

Since the calculation is done against the internal standard, there is no initial calibration curve or continuing calibration other then the normal TO-15 calibration and QC (Table 13.7b).

Analyte	Quant Ion	MW	ICAL	CCV
TPH gas C5 to C12 Hydrocarbons Before i-pentane to end	TIC	95	NA	NA
TVOC C3 to C12 Volatile Organic Compounds	TIC	100	NA	NA
Internal Standards				
Toluene-d8	TIC		NA	NA

EAS TO-15 Modified Air Phase Petroleum Hydrocarbons

The EAS APH Method is an internally developed processing method that is based on our TO-15 Method and the Massachusetts APH method. The Massachusetts APH method is very specific and has very detailed procedures, QC requirements, and has a special hydrocarbon list that is used for calibration and calculation of results. The EAS APH processing method uses a different hydrocarbon calibration list and follows the EAS TO-15 Method, so while the APH range results will not be significantly different, it does not qualify for the Massachusetts APH method if that method is specifically required.

Any target APH compounds like BTEX or naphthalene are measured and reported on the normal TO-15 analysis. These compounds as well as other non-APH compounds like acetone, ethyl acetone are subtracted for the APH calculation.

A separate processing method is used for a secondary processing of the main TO-15 analysis. The LCS and LCD from the main TO-15 analysis (Table 13.7b) is reported unless otherwise specified.

Table 13.7d
Method TO-15 Air Phase Hydrocarbons
Calibration and QC

Analyte	**Quant IS**		**ICV**	**CCV**
C5 to C12 Aliphatic Hydrocarbons Sum of C5-C8 AH and C9-C12 AH	NA		NA	NA
C5 to C8 Aliphatic Hydrocarbons Before i-pentane to before nonane	1,4-DFB		NA	NA
C9 to C12 Aliphatic Hydrocarbons Before nonane to end	CB-d5		NA	NA
C9 to C10-Aromatic After o-xylene to before naphthalene	CB-d5		NA	NA
Calibration Compounds	**Quant Ion**	**Calibration**		
Hexane	TIC	C5-C8 AH	<40%	60-140%
2,2,4-Trimethylpentane	TIC	C5-C8 AH	<30%	70-130%
n-Heptane	TIC	C5-C8 AH	<30%	70-130%
Octane	TIC	C5-C8 AH	<30%	70-130%
Nonane	TIC	C9-C12 AH	<30%	70-130%
Decane	TIC	C9-C12 AH	<30%	70-130%
Isopropylbenzene	120	C9-C10 A	<30%	70-130%
1,3,5-Trimethylbenzene	120	C9-C10 A	<30%	70-130%
1,2,4-Trimethylbenzene	120	C9-C10 A	<30%	70-130%
isopropyltoluene	134	C9-C10 A	<30%	70-130%
n-Butylbenzene	134	C9-C10 A	<30%	70-130%
Internal Standards				
1,4-Difluorobenzene (1,4-DFB)	114			
Chlorobenzene-d5 (CB-d5)	117			

13.8 TO-15/TO-14A GC/MS Selected Ion Monitoring (SIM)

This method is part of the TO-15/TO-14 method except the mass spectrometer is operated in the selected ion-monitoring mode (SIM), which gives a lower detection limit for a select group of compounds. The improvement in sensitivity over full scan is because instead of measuring 200 plus ions in a scan, SIM measures about 20 ions per scan. SIM is best used for a short list of compounds that need low MDL's.

The EAS modifications to the method include the target list and the QC criteria. Table 13.8b shows the compounds that are routinely run by TO-15 SIM; but, to get the best sensitivity, a subset of this list should be requested.

Table 13.8a
Summary of QC Criteria for EPA TO-15/ TO-14A SIM

Parameter	EAS TO-15/TO-14A SIM Modified
BFB Tune	Not Applicable to SIM Run if Specified in Project QC Criteria
Tuning Criteria with BFB	TO-15 Tune Criteria
Initial Calibration	See Table 13.8b 1 to 10 compounds one out 1 to 20 compounds two out 2 to 30 compounds three out
Calibration Check Sample (CCS)	Every year Same Percent RSD as Initial Calibration Use Full Scan Standard
Continuing Calibration Verification (CCV)	See Table 13.8b 1 to 10 compounds one out 1 to 20 compounds two out 2 to 30 compounds three out Only Target Compounds
Internal Standard (IS)	Response 50% to 200%
Surrogate	Toluene-d8 65 - 135% recovery
Method Blank	Dry Nitrogen <RL
Laboratory Control Spike	1 per Daily Batch Short List of Compounds (16) Bold Compounds Table 13.8b Full List if Required by Project 90% of compounds meet criteria
Duplicate Lab Control Dup Sample Dup	Duplicate with each 20 samples See Table 13.8b 90% of compounds meet criteria
Ending Standard	Not required since Internal Standards are used

Table 13.8b
Method TO-15 SIM/TO-14A SIM QC Criteria

Component	MDL ppbv	Initial Calibration %D	CCV %D	LCS %R	Precision %D
1,1-Difluoroethane	0.010	<30	<30	70-130	<30
1,3-Butadiene	0.010	<30	<30	70-130	<30
Vinyl chloride	**0.003**	**<30**	**<30**	**70-130**	<30
Bromomethane	**0.005**	**<30**	**<30**	**70-130**	<30
1,1-Dichloroethene	**0.003**	**<30**	**<30**	**70-130**	<30
Dichloromethane	**0.005**	**<30**	**<30**	**70-130**	<30
t-1,2-Dichloroethene	0.003	<30	<30	70-130	<30
Methyl tert butyl ether	0.005	<30	<30	70-130	<30
1,1-Dichloroethane	**0.003**	**<30**	**<30**	**70-130**	<30
c-1,2-Dichloroethene	0.003	<30	<30	70-130	<30
Chloroform	**0.003**	**<30**	**<30**	**70-130**	<30
1,2-Dichloroethane	**0.003**	**<30**	**<30**	**70-130**	<30
1,1,1-Trichloroethane	**0.003**	**<30**	**<30**	**70-130**	<30
Benzene	**0.050**	**<30**	**<30**	**70-130**	<30
Carbon Tetrachloride	0.003	<30	<30	70-130	<30
1,2-Dichloropropane	0.005	<30	<30	70-130	<30
Trichloroethene	**0.003**	**<30**	**<30**	**70-130**	<30
1,1,2-Trichloroethane	0.003	<30	<30	70-130	<30
Toluene	**0.003**	**<30**	**<30**	**70-130**	<30
1,2-Dibromoethane	0.002	<30	<30	70-130	<30
Tetrachloroethene	**0.003**	**<30**	**<30**	**70-130**	<30
Chlorobenzene	0.005	<30	<30	70-130	<30
Ethylbenzene	**0.003**	**<30**	**<30**	**70-130**	<30
m &p-Xylenes	**0.003**	**<30**	**<30**	**70-130**	<30
Styrene	0.005	<30	<30	70-130	<30
o-Xylene	**0.003**	**<30**	**<30**	**70-130**	<30
1,1,2,2- Tetrachloroethane	0.003	<40	<40	70-130	<30
1,3,5-Trimethylbenzene	0.005	<40	<40	60-140	<30
1,2,4-Trimethylbenzene	**0.005**	**<40**	**<40**	**60-140**	<30
1,3-Dichlorobenzene	0.005	<40	<40	70-130	<30
1,4-Dichlorobenzene	0.005	<40	<40	70-130	<30
1,2-Dichlorobenzene	0.005	<40	<40	70-130	<30
Naphthalene	0.005	<50	<50	50-150	<30

13.9 EPA TO-17 Volatile Organic Compounds

Method TO-17 is used to analyze samples for volatile organic compounds collected on multi-bed sorbent tubes, which are thermally desorbed and cryo-focused on the capillary column and then analyzed by GC/MS. The range of compounds analyzed by the method depends on the selection of the sorbent cartridge. EAS follows the method recommendation that the calibration and QC criteria for Method TO-17 follow the TO-15 method.

The modifications done by EAS include the target list. The method recommends using the Method TO-15 QC criteria. EAS uses the modified TO-15 QC criteria listed in Table 13.9c.

Table 13.9a
TO-17 Sorbent Cartridge Selection Guide

Tube Name	Compounds	Packing	Desorption Temperature
Tenax TA	BTEX Diesel Range Organic	Tenax TA	300C
Carbotrap 300	General VOC	Carbopak C Carbopak B Carboseive SIII	325C
VOC	General VOC	Tenax TA Carboxen 1000 Carboseive SIII	325C

TO-17 tubes can also be sampled passively using special adapters. The tubes are desorbed and analyzed in the same manner as the normal TO-17, and the TO-17 QC criteria is used.

Table 13.9b
TO-17 Recommended Sampling Times

Final Volume	Flow Rate	Time
500 ml	100 ml/min	5 min
480 ml	1 ml/min	8 hours
720 ml	0.5 ml/min	24 hours

13.10 ASTM D1946, ASTM D1945 Permanent Gases

ASTM D1946, D1945, and EPA 3C are used for the analysis of permanent gases (also called fixed gases) and light hydrocarbons at the percentage level in all types of samples. The samples can be collected in canisters, Tedlar bags, or other approved containers. Samples are analyzed by gas chromatography with a thermal conductivity detector (TCD). The methods for ASTM D1945, ASTM D1946, and EPA 3C are the same, but there are different QC criteria and compounds specified for each method. These methods were originally designed for high-level source analysis and have been modified for environmental analysis. The results for ASTM D1946 and D1945 are normalized to 100% when all compounds are analyzed.

The modifications done by EAS include the calibration and QC criteria.

Table 13.10a
Summary of QC Criteria for ASTM D1946/D1946 Modified

Parameter	EAS D1945 Modified	ASTM D1945
Initial Calibration	Results quantitated from Daily Calibration	
Calibration Check Sample (CCS)	Pure air has nitrogen at 78.1% and Oxygen at 21.9% (includes argon)	
Daily Calibration	One or more daily calibration standard(s) are run for quantitation. The primary standard is run to bracket: Hydrogen 0-5% Oxygen 1-30% Nitrogen 5-80% Methane 1-75% Carbon Dioxide 0-20% Helium 0-20%	Reference standard within ½ concentration of and 10 mol % of target compounds. <1% for duplicate runs of CCV
Method Blank	Oxygen <0.3% Nitrogen < 1% Others <0.01%	Not Specified
Laboratory Control Spike	1 per Daily Batch See Table 13.10b	Not Specified
Duplicate Lab Control Dup Sample Dup	Duplicate with each 20 samples See Table 13.10b	Not Specified
Sum of Components	85% - 110% (results normalized to 100% for full list)	
Canister Holding Times	30 days	Not Specified

Table 13. 10b
Method ASTM D1946/D1945 Compound List

Analyte	RL %	RL ppmV	Criteria		Duplicate %RPD
			ICAL %D	LCS %R	
Oxygen	0.15	500	NA	80-120	<20
Nitrogen	0.30	1000	NA	80-120	<20
Methane	0.01	100	NA	80-120	<20
Carbon Monoxide	0.01	100	NA	80-120	<20
Carbon Dioxide	0.01	100	NA	80-120	<20
Special Request			NA		
Hydrogen	0.15	500	NA	80-120	<20
Helium	0.06	600	NA	80-120	<20
Ethene	0.005	50	NA	80-120	<20
Ethane	0.005	50	NA	80-120	<20
Ethylene	0.005	50	NA	80-120	<20
Propene	0.005	50	NA	80-120	<20
Propane	0.005	50	NA	80-120	<20

ASTM D1945 Permanent Gases

ASTM D1945 is similar to ASTM D1946 for permanent gases and light hydrocarbons, but has additional hydrocarbons on the compound list. EAS normally analyzes these additional hydrocarbons by Method 18 Modified for source samples, and TO-3 modified for ambient air samples. These methods use GC/FID and are more suited to reporting more detailed hydrocarbon analysis.

13.11 ASTM D3416 Low Level Methane and CO

ASTM D3416 measures methane and carbon monoxide in ambient air with a detection limit of about 0.1 to 0.2 ppmv. The air sample is loaded on a loop and injected onto a molecular sieve 5A column, which separates the methane and carbon monoxide. If only methane is measured, the sample goes from the column directly to an FID detector that measures the methane. If carbon monoxide is measured, the output from the column goes into a reduction catalyst that converts the carbon monoxide into methane. ASTM no longer supports the method, but the procedure used is accurately described in the method.

The modifications used by EAS include the calibration and QC criteria.

Table 13. 11a
Summary of QC Criteria for ASTM D3416

Parameter	EAS D3416 Modified	ASTM D314
Initial Calibration	3 point initial calibration See Table 13.11b	
Calibration Check Sample (CCS)	Each daily analytical batch includes the analysis of ambient air as a secondary check for methane (1.85 ppmV).	
Continuing Calibration Verification (CCV)	Daily (24 hours) See Table 13.11b	
Method Blank	<RL	Not Specified
Laboratory Control Spike	1 per Daily Batch See Table 13.11b	Not Specified
Duplicate Lab Control Dup Sample	Duplicate with each 20 samples See Table 13.11b	Not Specified
Canister Holding Time Tedlar Bag	30 days 72 hours	Not Specified
Canister Certification	Upon Request	

Table 13. 11b
Method ASTM D3416 Compound List

Analyte	MDL ppmV	RL ppmV	Criteria		Duplicate %RPD
			ICAL/ CCV %D	LCS %	
Methane	0.1	0.3	<20	80-120	<20
Carbon Monoxide	0.1	0.3	<20	80-120	<20

13.12 EPA Method 15/16 Sulfur Compounds

EPA Method 16, CARB 16, and EPA Method 15 are all methods for the analysis of reduced sulfur gases and Total Reduced Sulfur (TRS) in source samples. These methods have been modified for the analysis of sulfur gases collected on-site and shipped to EAS in special canisters or in Tedlar bags. EPA and CARB Method 16 have superseded Method 15 for fixed laboratory analysis of TRS. The method uses a flame photometric detector (FPD). A 0.1 ml to 10 ml sample is analyzed using a fused silica capillary column for the identification and quantification of the TRS compounds and 18 other sulfur compounds. Other sulfur compounds not in the standard like sulfur dioxide can be calculated against hydrogen sulfide since the detector response is based on the sulfur content of the compound.

The modifications to the method are the sample introduction system, the sample collection procedure, the target list, standards, the calibration procedure, and the QC criteria.

Table 13.12a
Summary of QC Criteria for EPA Method 15/16

Parameter	EAS EPA 15/16 Modified	EPA 15/16
Initial Calibration	3-point minimum initial calibration compound list. Since the method is used in a fixed lab the initial calibration is used until the CCV no longer passes. See Table 13.12b	3-point minimum initial calibration for H2S, COS, CS2 Standards run in triplicate, agree within 5% of their mean.
Calibration Check Sample (CCS)	No Secondary Source	Not specified
Continuing Calibration Verification (CCV)	Daily (24 hours) Continuing Calibration Verification standard is run in triplicate. See Table 13.12b	Daily (24 hours) Standard run in triplicate, agree within 5% of their mean.
Method Blank	<RL	Not Specified
Laboratory QC	Duplicate Standard recovery is calculated from Init Cal.	Not Specified
Duplicate CCV Duplicate	With each 20 samples See Table 13.12b	Not Specified
Holding Times Silico Canister	Tedlar Bag: 72 hours 7 days	Not Specified

Table 13.12b
EPA Method 15/16 Compound List

Analyte	MDL ppmV	LOQ ppmV	Criteria		Duplicate %RPD
			ICAL/ CCV %RSD		
Hydrogen Sulfide	0.14	0.25	<15		<15
Methyl Mercaptan	0.14	0.25	<15		<15
Ethyl Mercaptan	0.14	0.25	<15		<15
Dimethyl Sulfide	0.14	0.25	<15		<15
Carbon Disulfide	0.14	0.25	<15		<15
i-Propyl Mercaptan	0.14	0.25	<15		<15
Ethyl Methyl Sulfide	0.14	0.25	<15		<15
n-Propyl Mercaptan	0.14	0.25	<15		<15
Thiophene	0.14	0.25	<15		<15
Isobutyl Mercaptan	0.14	0.25	<15		<15
Diethyl Sulfide	0.14	0.25	<15		<15
t-Butyl Mercaptan	0.14	0.25	<20		<20
n-Butyl Mercaptan	0.14	0.25	<20		<20
Dimethyl Disulfide	0.14	0.25	<20		<20
3-Methylthiophene	0.14	0.25	<20		<20
Tetrahydrothiophene	0.14	0.25	<20		<20
2,5-Dimethylthiophene	0.14	0.25	<20		<20
Diethyl Disulfide	0.14	0.25	<20		<20
2-Ethylthiophene	0.14	0.25	<20		<20

The MDL is an estimate since the reporting limit (RL or LOQ) is used as the lower limit in the Method.

13.13 EPA Method 18

EPA Method 18 is a source test method for the analysis of volatile organic compounds collected in Tedlar bags. The method has a very specific protocol for calibration and QC that must be followed. Before using the method a screening sample is collected and used to identify compounds in the source and to prepare a standard containing these compounds. EAS analyzes samples with the unmodified Method 18 and with the modifications listed below.

The modifications made by EAS to the method include collecting samples in Canisters, calibration procedure, target list, and QC criteria. The unmodified method is not used for source testing.

Table 13.3a
Summary of QC Criteria for EPA Method 18

Parameter	EAS EPA 18 Modified	EPA 18 Method
Initial Calibration	3 points minimum. See Table 13.13b	3 points minimum for calibration standards prepared in Tedlar bags. Analyzed in triplicate until <5% RSD
Continuing Calibration Verification (CCV)	Daily (24 hours) See Table 13.13b	Daily (24 hours) < 5% D. A screening sample is run to check for components and concentrations.
Method Blank	<RL	
Laboratory Control Spike	With Daily Batch See Table 13.13b	Not specified
Duplicate Sample Dup	Duplicate with each 20 samples See Table 13.13b	Method specifies a recovery study that may include matrix spikes.
Holding Time - Canister Tedlar Bag	 30 days 72 hours	30 days
Canister Certification		Not Specified

For the Modified EPA Method 18, the TO-3 compound list, MDL, and RL values are used. The modified method is used for non-compliance source testing where the ambient air method TO-3 is not applicable. There is no specific target list or MDL for Method 18. This information is determined during the pretest study, when samples are collected and analyzed to determine the compounds present and their levels. Table 13.13b shows the BTEX compound list with the LCS compounds.

14.0 PREVENTATIVE ACTION

EAS has a pro-active program to identify ways to verify data and improve methods rather then wait for problems or complaints. The Technical Director is constantly monitoring laboratory generated data.

1. If there is an anomaly in any client samples in a group these samples will be reanalyzed before the data is sent out to verify the results.
2. If there is a question about matrix interferences, a sample is analyzed on a different instrument to verify results. For example, a GC/FID sample might be analyzed on GC/MS to verify identities or concentrations.
3. Special spikes or standards are prepared to assist in peak identification or to check results.
4. An aliquot of a sample will be spiked to check peak identification or recovery (like a matrix spike).

15.0 PROCEDURES FOR HANDLING SAMPLES

Environmental Analytical Service, Inc. does not do any sampling. EAS will occasionally assist client with their sampling efforts, in which case the client will have a sampling plan. Because of the complexity of the air sampling process the Technical Director will often work with clients in designing their sampling plans. The goal of all sampling is to make sure that representative samples are collected that meet the project objectives of the client.

15.1 Sample Receipt

EAS receives primarily air and gas samples in the types of containers listed in Table 15.1. This table also gives the holding time and preservation recommended by the method.

The clients ship samples to Environmental Analytical Service. Samples that do not require refrigeration are sent in standard cardboard boxes, and samples that require refrigeration as a preservative are sent in ice chests. The temperature of the ice chest is recorded when it is received.

Sample Control opens the boxes as they arrive and immediately labels each sample with a sample delivery group number and a sequential laboratory number from a hardbound logbook and by the LIMS system. The samples are then logged into the EAS LIMS system.

Table 15.1

Container	Holding Time	Preservative	Storage Location
SUMMA Canister	30 days (1)	None	Laboratory Shelf
Tedlar Bag	3 days	None	Laboratory Shelf
VOC Sorbent Tubes	30 days	Refrigerate	Sample Control Refrigerator
SVOC Sorbents and Filter	7 days	4 C	Sample Control Refrigerator
SVOC Extracts	40 days	4 C	Sample Control Refrigerator
DNPH Cartridges	14 Days	4 C	Sample Control Refrigerator
Impinger Solutions	14 days	4 C	Sample Control Refrigerator

(1) For compliance samples, special projects or studies the holding time can be up to 90 days for stable VOC compounds.

15.2 Sample Identification and Tracking

Sample Control is the department that documents the receipt of incoming samples and initiates all paperwork for that project. EAS has a computerized Laboratory Information Management System (LIMS) and a Sample Logbook to track incoming samples and outgoing sampling media.

Priority is given to the processing of samples at or near the method holding time. In addition, samples that require an expedited turn around for results are logged in before routine analyses.

Semi-volatile resins, condensates, and washes waiting for processing are held in a refrigerator used only for that purpose. An exception to this is when the samples are received in sealed coolers, in which case they are not unsealed until they are processed. Samples with short holding times do not usually come to the laboratory this way, and those that do are only from certain clients, so EAS asks to be advised in advance to open these and process them first.

Documentation received with the sample is reviewed for accuracy and completeness of information regarding the client, the sampler, the description of the samples, the date sampled, and any special requests. If the samples come to the laboratory with a chain of custody, as is recommended, the chain of custody document is signed by the Sample Control Officer after it has been determined that all samples listed on that chain of custody have arrived. Notes regarding the condition of the samples, such as seals being intact or bottles received broken, are made by the Sample Control Officer on the chain-of-custody document. Inconsistencies require an immediate call to the client.

The samples are then logged into the LIMS system and the Sample Logbook. Each sample is given a unique log number with a description. Information entered into LIMS include the date and time received by the laboratory, the client's name, the sampler's name, the description of the sample, the date and time sampled, the sample destination, each analysis requested, the initials of the person logging in the sample, each type of container the sample is in, and any additional notes regarding that sample, such as a turnaround time. "RUSH" samples are highlighted and given directly to the analysts.

Once the sample is logged in, the sample containers are clearly marked with the correlating sample number. The sample log numbers are also recorded on the chain of custody for future reference.

A Project sheet is generated by Sample Control, and is used for several purposes. The Accounting Department uses it for invoicing, and the client immediately receives copy which serves as confirmation that his/her request has been properly understood. Any paperwork regarding the samples is attached to the Project sheet, including the original Chain of Custody, the completed original worksheets, the entire computer generated results, and a copy of the invoice.

Laboratory Worksheets are then prepared by Sample Control. The worksheet includes information such as the client's name, the sampler's name, the sample number, the date and time collected, the date and time received, the turnaround time required, and the sample location. If a sample is a RUSH, the turnaround time is highlighted to make sure laboratory personnel notice it. The analysis requested, the holding time for that analysis,

the EPA Method number, and the detection limit are also listed as the work is completed, both on the work order and by direct entry into the LIMS system.

15.3 Sample Storage

Following login, but prior to analysis, the Sample Control Officer determines a proper location for the storage of the sample. Once the sample is stored, the worksheets are distributed to the proper departments and the key personnel are notified of any unusual or important information such as rushes, unusual matrices, unusual analyses, samples approaching their holding times, or especially hazardous samples.

Samples collected in SUMMA Canisters are stored until the analytical reports have been reviewed. Tedlar bag samples are saved for at least 10 days. SVOC extracts are stored for at least 60 days. Sorbent tubes are not stored since they cannot be reanalyzed once they are desorbed.

15.4 Sample Disposal

EAS primarily analyzes ambient air samples collected in SUMMA Canisters. When the reports are reviewed and completed, the canisters are re-evacuated by pumping any remaining sample out of the canister using a vacuum pump. Samples collected in Tedlar bags are vented in the hood.

Any liquid or product samples are disposed of by an appropriate method after the completion of the project.

15.5 Sample Records

All records associated with the shipping, receipt, or login of the samples are stored in the Sample Delivery Group (SDG) file with all of the analytical data. This file is converted into an electronic PDF by scanning all of the materials and storing them on the EAS Server.

16.0 REPORTING RESULTS

16.1 Analytical Reports

The Analytical Reports are prepared in a standardized multi-sheet Excel Workbook called the Excel Analytical Report Templates. The GC data is manually entered into the Excel Analytical Report Template (EART) from the electronic integrator output, and the GC/MS data is electronically linked to the EART by the HP Chemstation software.

The technician processes the Daily Analytical Batch (DAB) . The area counts from the chromatograms are transferred either automatically by the computer (GC/MS) or manually by the technician (GC, HPLC) into a computer Excel workbook for the calculation of results and report generation. Each sample analyzed is part of a DAB, which includes calibration standards, blanks, and QC samples.

Once the final results for all of the samples and QC samples have been calculated for a particular batch, the technician, who prepares the QC batch reports, and validates that batch of data, reviews the DAB. Any data points that do not meet the QC batch requirements are flagged with the standard EPA data flag letters. The QC batch report is included with all sample reports from that batch.

The results of the DAB folders are combined into the Project SDG folder by the Project Manager and are reviewed in context of the analytical tests requested, the batch QC, and the method validation data. Any additional changes, flags, or qualifiers are added at this stage. After data review, the Project Manager will assemble the final report in the SDG folder and sign the cover sheet for the analytical results.

The data is reviewed before it is sent to the client. The review is documented on the DAB Review form and the SDG Review form. Each person preparing the report or reviewing data signs one of these forms, which becomes a permanent part of the final SDG file.

Technician Review: Once the final results for all of the samples and QC samples has been calculated for a batch, the batch data is reviewed by the technician, who then prepares the QC batch report and validates that batch of data.

Project Manager: The results of the DAB folders are combined into the Project SDG folder by the Project Manager and are reviewed in context of the analytical tests requested, the batch QC, and the method validation data.

Laboratory Director: After the final report is assembled and the invoice generated, the final package is given a final review by the Laboratory Director.

16.2 Data Deliverable Packages

Some projects require a data deliverable package often called a Level 4 package. The level 4 data package headers are printed from LIMS.

Level 4 Data Deliverable Package
Cover Letter and Case Narrative
Method Blank Report
Laboratory Control Spike/Duplicate Report (and/or project specific QC reports)
Analytical Reports
TIC and Library Search (if needed)
Chromatograms, Ion Spectra, and Raw Data
Standard Certificates
Correspondence

16.3 Approved Signatures

All reports produced by Environmental Analytical Service are reviewed and signed by the Laboratory Director on the Cover Letter and the SDG Review Form. An electronic signature of the Lab Director appears on the Laboratory Case Narrative page for Lab Certification.

16.4 Amendments to Reports

Amendments to a report are sent with a cover letter describing what changes have been made to the report and any changed sheets that need to be put in the old report. If there are extensive changes, or the client requests it, a complete report will be sent. EAS used the following categories for amendments.

Revised Report: This is a report that is changed because the client made an error on the Chain of Custody, or requested the wrong target list or test.

Corrected Report: This is a report that is changed because the laboratory made an error in the headers or target list.

Supplemental Report: This is additional testing on the original samples that is requested by the client.

16.5 Electronic Reports and Deliverables

Environmental Analytical Service generates a PDF file of all analytical reports sent. This PDF file is stored on the EAS Server in the SDG folder with all of the raw data. The PDF file report is a copy of the analytical report, so it meets all of the requirements of the standard. This report can be e-mailed to the client or posted to their FTP site.

17.0 CORRECTIVE ACTION

17.1 Corrective Action

Quality control (QC) failures logically fall into two categories: single QC outliers, indicating batch failure, and systematic problems, which reveal method failure. EAS uses a two-tiered corrective action program to deal with these two levels.

Analytical control limits are maintained for laboratory control standards (LCSs, also known as Quality Control Check Samples QCCSs), method blanks, spike recoveries, duplicate spike relative percent differences (RPDs), and surrogate recoveries. In addition, many analytical SOPs specify quality control criteria for calibrations, sensitivity checks, and other method-specific quality checks that are performed routinely.

If one of the above checks does not meet acceptance limits, the analyst initiates corrective action. Such action can take several forms, but can always be accomplished with little interruption in analysis. Re-calibration, for example, might resolve a failure to meet calibration criteria; or review of the sample data might indicate that only the method blank was contaminated in the laboratory, so the blank's failure to meet acceptance criteria would not compromise the quality of the data. Table 17.1a lists possible corrective action steps to take in response to QC failures for all methods used by EAS. Every corrective action requires appropriate flagging of the data. A list of data flags used by EAS is enclosed with each analytical report.

Systematic failures of a method, issues of method compliance, consistent contamination that the analyst cannot resolve, or QC failures that impact data already reported are examples of more serious quality issue. For these problems, the Technical Director/Quality Manager is always notified.

Table 17.1a
Laboratory Corrective Action Summary

QC Parameter Out of Control	Corrective Action
Holding Times	1. Extract sample immediately 2. Call Project Manager to assess impact to data usability 3. Depending on use of data run out of holding time or resample
Tuning Criteria (GC/MS only)	1. Retune 2. Clean Source
Initial Calibration	1. Evaluate System 2. Evaluate use of data and target compounds and run samples if data quality is not effected 3. Rerun Initial Calibration
Continuing Calibration Verification (CCV)	1. Evaluate System 2. Rerun standard 3. Check concentrator 4. Check Tune (for GC/MS) 5. New Initial Calibration
Internal Standard	1. Rerun Sample in holding time 2. Cut load volume for matrix effects. 3. Check concentrator and correct if necessary 4. If rerun is out of holding time report both results
Surrogate	1. Rerun Sample 2. Cut load volume for matrix effects. 3. Check concentrator and correct if necessary 4. If rerun is out of holding time report both results
Method Blank	1. Analyze another Blank 2. If blank levels are dropping significantly continue running blanks 3. If two reruns of blanks are the same, clean concentrator with heat gun 4. Runs samples in holding times and flag blanks
Laboratory Control Spike	1. Reanalyze LCS 2. Recalibrate LCS
Matrix Spike	1. Reanalyze MS 2. Recalibrate MS
Duplicate	1. Reanalyze LCD, sample, MSD 2. Repair Concentrator
Canister Certification	1. Reclean canisters 2. Use canisters for project with higher MDL 3. Use canisters for project that does not contain compound that failed.

17.2 Corrective Action Form

The EAS LIMS system has sections to record any deviations from QC that occurs during the sample receipt or reporting of data. These sections print out on the Case Narrative form in the Analytical Report

Because of the size of the laboratory, the Corrective Action Form (CAF) is part of the Daily Analytical Batch (DAB) form and any nonconforming work or departures from policies and procedures in the quality system at EAS are recorded on this form. The Daily Analytical Batch (DAB) is used for the laboratory technicians to record and check for any QC deviations. The Technical Director consults with the analysts to decide what to record and what action to take.

17.3 Departures from Documented Policy

Occasionally it is necessary to deviate from either the Standard Operating Procedures or QC requirements. This occurs when:

1. Samples are received for special projects
2. Samples have a difficult matrix that cause QC limits to be exceeded
3. Samples have to be analyzed and reported in a short period of time

Each of these circumstances is handled differently according to the procedures described below. Ultimately it is the Technical Director that makes final approval of deviations from documented procedures.

Samples are received for special projects: For these projects, the Technical Director will write out any modifications to procedures that will be used for that project. These modifications will be communicated to the Laboratory Manager/Supervisor, who will go over the modifications with the laboratory staff before the start of the project.

Samples have a difficult matrix that cause QC limits to be exceeded: When laboratory technicians encounter any samples or situations that cause variations from established QC limits, they are required to get approval by the Technical Director before analyzing samples.

Samples have to be analyzed and reported in a short period of time: The Laboratory Manager/Supervisor closely monitors Rush samples and any deviations require the approval of the Technical Director as described above.

18.0 CONTROL OF NON-CONFORMING WORK

Non-conforming work is any aspect of the environmental testing or the results of the testing that does not conform to the EAS procedures or requirements of the client. When the Laboratory Director identifies non-conforming work, the following procedure is used.

1. The initial report of the non-conforming work will be made on the corrective action form/form. This form will document the non-conforming work and insure that all responsible parties will evaluate the non-conformance by signing off on the form and any resulting corrective action.

2. The Laboratory Director (also the Technical Director) will meet and determine the significance of the non-conformance.

4. If the data quality is impacted, the client is contacted to participate in the decision about what to do. The contact with the client is documented in the Contact Log..

5. The responsibility for resuming work resides with the Technical Director.

19.0 CLIENT SERVICES

Environmental Analytical Service has client service representatives who are responsible for maintaining contact with the clients and managing their projects. Because of the size of the laboratory, this person is often the Technical Director. When a client calls about possible analytical work, the client service representative starts a Client Contact Log with a summary of the conversation. If the client is requesting canisters for an existing project they will look up the Project Quotation and generate a Shipping Request for the sample media. For a project that does not have a contract, the Client Service person will fill out a Project Worksheet and generate a quotation to send to the Client.

When a project becomes active the Client Service person will open the Project Worksheet and mark the project Active. They will check with the Laboratory Manager/Supervisor to make sure that the media can be prepared and ready by the ship date. A Shipping Request will be generated for the media shipment.

When samples are received, the Client Services Team will receive them and log them in based on the information the client provides on the Chain of Custody and the information gathered for the project. A Sample Delivery Group (SDG) number is assigned to the samples and a folder started. If there are any questions during login, the client service representative will contact the client for clarification. After the folder is completed it will be sent to the Laboratory Manager/Supervisor for review. After review, a sample receipt notification sheet is e-mailed to the client, listing all of his tests and the turn around time for the tests to be completed.

20.0 SUBCONTRACTING

Environmental Analytical Service rarely subcontracts analytical services. Generally the client will deal directly with the subcontract lab. Any subcontracting must meet the following requirements:

1. The client is always notified that samples are going to be subcontracted, to what laboratory, and what the certifications and qualifications are for that laboratory.

2. The subcontracting laboratory is required to provide EAS all of the QC information required for the report requested.

3. In the EAS LIMS system there is a section in the SDG form for entering subcontracting the subcontracting laboratory, their certifications and qualification. that will appear on the Case Narrative in the Analytical Report.

The subcontractors are all listed in the Administrative Rolodex which is located in the LIMS system and has their contact information, what services they provide and if the are a Qualified Vendor or just a Vendor. EAS uses Qualified Vendors for subcontracting. A Qualified Vendor will have NELAC or California ELAP certificate on file at EAS, and proof of insurance.

21.0 PURCHASING SERVICES AND SUPPLIES

EAS purchases all supplies and standards (see section on calibration standards) from reliable commercial sources that normally provide these services to analytical laboratories. All purchases are entered into the Purchase Order module of the EAS LIMS system. This can be searched later, if necessary, for reorders.

The vendors are all listed in the Administrative Rolodex which is located in the LIMS system and has their contact information, what products they supply and if the are a Qualified Vendor or just a Vendor. EAS used Qualified Vendors for the purchase of lab supplies related to the quality system and laboratory operations. A Qualified Vendor is a nationally recognized supplier of laboratory supplies or a vendor that has been investigated by the Technical Director who approves their services. A Vendor is someone that supplies items not related to lab quality such as paper towels, light bulbs, printer repair, postage.

22.0 OUTSIDE SUPPORT

Environmental Analytical Service does all of its own maintenance and instrument service. The following outside resources are used to help evaluate problems when the Technical Director is not available. The support vendors are all listed in the Administrative Rolodex which is located in the LIMS system and has their contact information, what services they supply and if the are a Qualified Service Vendor.

Table 22.1
Outside Support and Suppliers

Component	Service	Outside Support
Computers and Computer Network	Service and Parts	• Coastal Computers
Agilent Gas Chromatographs and Mass Spectrometers	Parts and Repairs	• Agilent Technologies • EBAY
EAS Concentrators	Parts Flow Meters Electronics Valves Fittings	• McMaster Carr • Cole-Parmer • Newark • Ventura Valve and Fitting
Chromatographic Supplies	Chromatographic Supplier	• Supelco • Restek
Analytical Balance	Calibration	• Wine Country Balance
Instrument Fittings	Purchase	• CCFST
Solvents and Lab Supplies	Purchase	• VWR • Fisher
Calibration Gases	Purchase	• Scott Specialty • Scott Marrin
Instrument Gases	Purchase	• AirGas

23.0 COMPLAINTS

Either Customer Service or the Laboratory Director receives complaints. The complaint is initially recorded in a CALL LOG in the EAS LIMS system. The Laboratory Director reviews all complaints and contacts the customer to provide a resolution to their complaint.

State of Florida
Department of Health, Bureau of Public Health Laboratories
This is to certify that

E871125

ENVIRONMENTAL ANALYTICAL SERVICE
173 CROSS STREET
SAN LUIS OBISPO, CA 93401

has complied with Florida Administrative Code 64E-1,
for the examination of environmental samples in the following categories

AIR AND EMISSIONS - EXTRACTABLE ORGANICS, AIR AND EMISSIONS - PESTICIDES-HERBICIDES-PCB'S, AIR AND EMISSIONS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Public Health Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

Date Issued: July 01, 2022 Expiration Date: June 30, 2023

Susanne Crowe, MHA
Interim Chief Bureau of Public Health Laboratories
DH Form 1697, 7/04
NON-TRANSFERABLE E871125-08-07/01/2022
Supersedes all previously issued certificates

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ENV-MAN-SHRT-0001

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Michelle LaGory (990324)	Manager - Quality	20 Apr 2020, 12:51:35 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Emily Agola (006966)	Manager - Quality	20 Apr 2020, 12:45:36 PM	Approved
Dennis Leeke (007079)	Regional Director - Operations	20 Apr 2020, 12:54:52 PM	Approved
Kevin Chartier (990308)	General Manager 1	20 Apr 2020, 01:46:45 PM	Approved
Thomas Patten (990330)	Manager	20 Apr 2020, 04:51:17 PM	Approved
Daelene Truax (990340)	Administrative Assistant	21 Apr 2020, 10:34:08 AM	Approved
Eric Brandjord (990304)	Manager	21 Apr 2020, 04:54:34 PM	Approved
Mary Slipp (990337)	Supervisor	22 Apr 2020, 12:23:44 PM	Approved
Dave White (004945)	Regional Manager - Systems	22 Apr 2020, 02:48:51 PM	Approved
Amy Eady (990593)	Supervisor	27 Apr 2020, 10:56:26 AM	Approved
Wade Nieuwsma (990328)	Supervisor	27 Apr 2020, 04:26:32 PM	Approved
Karen Secor (990336)	Supervisor	29 Apr 2020, 11:45:10 AM	Approved

TITLE PAGE

LABORATORY QUALITY MANUAL

Prepared for:

Pace Analytical Services, LLC – Sheridan, WY Laboratory
1673 Terra Avenue
Sheridan, WY 82801
Phone: 307-672-8945

Pace Analytical Services, LLC – Gillette, WY Service Center
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Gillette, WY 82801
Phone: 307-682-8945

Manual Approval Signatories

Approval of this manual by managerial personnel is recorded on the Signature Manifest located before the Title Page of this manual.

The individuals listed below represent the management team that was in place on the effective date of this version of the manual for the following location:

Pace Analytical Services, LLC
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Sheridan, WY 82801
Phone: 307-672-8945

Each of the following individuals is a signatory for the manual for the location listed above. The application of their signature to the manual signifies their commitment to communicate, implement, and uphold the requirements, policies and procedures specified in this manual and their commitment to continuously improve the effectiveness of the quality management system based on customer feedback and internal assessment.

Name[1]	Title	Address[2]	Phone[2]
Dennis Leeke	Regional Director-Operations	1700 Elm Street SE, Minneapolis, MN 55414	612-656-2279
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Michelle LaGory	Manager - Quality		
Thomas Patten	Manager – Lab Operations		
Eric Brandjord	Manager - Client Services	555 Absaraka St. Sheridan WY 82801	307-674-7506
Daelene Truax	Health & Safety, however named.		
Wade Nieuwsma	Supervisor - Radiochemistry [3]		
Amy Eady	Supervisor- Wet Chem[3]		
Karen Secor	Supervisor - Mining[3]		
Mary Slipp	Supervisor-Trace Metals [3]		

[1] Members of the local management team are subject to change during the life-cycle of this document version.
[2] Include if different from the physical address and phone number of the facility.
[3]This individual serves as an Acting Technical Manager for TNI for one or more fields of accreditation.

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Michelle LaGory	Manager - Quality	1673 Terra Avenue Sheridan WY 82801	307-672-8945
Eric Brandjord	Manager - Client Services	555 Absaraka Street Sheridan, WY 82801	307-674-7506
Daelene Truax	Health & Safety, however named.	1673 Terra Avenue Sheridan WY 82801	307-672-8945

[1] Members of the local management team are subject to change during the life-cycle of this document version.
[2] Include if different from the physical address and phone number of the facility.
[3] This individual serves as an Acting Technical Manager for TNI for one or more fields of accreditation.

Table of Contents

1.0 PURPOSE AND SCOPE

1.1 Purpose

This quality manual (manual) outlines the quality management system and management structure of the laboratories and service centers affiliated with Pace Analytical Services, LLC (PAS). A laboratory is defined by PAS as any PAS facility, however named, that provides testing, sampling, or field measurement services. When the term 'laboratory" is used in this manual, the term refers to all locations listed on the Title Page of this manual and in Section 4.1.3 unless otherwise specified.

The PAS quality management system is also referred to as the quality program throughout this document. In this context, the phrase "quality management system" and "quality program" are synonymous.

The quality management system is the collection of policies and processes established by PAS management to consistently meet customer requirements and expectations, and to achieve the goals to provide PAS customers with high quality, cost-effective, analytical measurements and services.

The quality management system is also intended to establish conformance[1] and compliance with the current versions of the following international and national quality system standards:

- ISO/IEC 17025: *General requirements for the competence of testing and calibration laboratories*
- NELAC/TNI Standard Volume 1: *Management and Technical Requirements for Laboratories Performing Environmental Analysis*

[1]The statement of conformity to these Standards pertains only to testing and sampling activities carried out by the laboratory at its physical address, in temporary or mobile facilities, in-network, or by laboratory personnel at a customer's facility.

In addition to the international and national standards, the quality management system is designed to achieve regulatory compliance with the various federal and state programs for which the laboratory provides compliance testing and/or holds certification or accreditation. When federal or state requirements do not apply to all PAS locations, the requirements for compliance are provided in addendum to this manual or in other documents that supplement the manual. Customer-specific project and program requirements are not included in the manual in order to maintain client confidentiality.

- A list of accreditation and certifications held by each laboratory associated with this manual is provided in Appendix A.
- A list of analytical testing capabilities offered by each laboratory associated with this manual is provided in Appendix B.

1.2 Scope and Application

This manual applies to each of the PAS locations listed on the Title Page and in Section 4.1.3.

The manual was prepared from a quality manual template (template) created by PAS corporate quality personnel. The template outlines the minimum requirements PAS management considers necessary for every PAS laboratory, regardless of scope of services or number of personnel, to establish in order to maintain a quality management system that achieves the objectives of PAS's Quality Policy (See 4.2.2). In this regard, the template is the mechanism used by the corporate officers (a.k.a. 'top management') to communicate their expectations and commitment for the PAS quality program to all PAS personnel.

The laboratory also has the responsibility to comply with federal and state regulatory and program requirements for which it provides analytical services and holds certification or accreditation. When those requirements are more stringent than the template, the requirements for compliance are provided in addendum to this manual or in other documents that supplement the manual. This document structure maintains consistency in the presentation of the quality management system across the network while providing the laboratory a mechanism to describe and achieve compliance requirements on a program basis.

1.2.1 Quality Manual Template

The quality manual template is developed by the Corporate Quality Director with contribution and input from corporate quality personnel and the corporate officers. Approval of the template by the corporate officers (aka "top management") confirms their commitment to develop and maintain a quality management system appropriate for the analytical services offered by the organization and to communicate their expectations of the quality program to all personnel.

The template and instructions for use of the template are released by corporate quality personnel to quality assurance manager(s) responsible for each laboratory (Local QA). Local QA uses the template to prepare the laboratory's manual by following the instructions provided. Since the template provides the minimum requirements by which all PAS locations must abide, the laboratory may not alter the font, structure or content of the template except where specified by instruction to do so. As previously stated, program specific requirements are provided in addendum or in documents that supplement this manual.

The template is reviewed by corporate quality personnel every two years and updated if needed. More frequent review and revision may be necessary to manage change, to maintain conformance and compliance to relevant standards, or to meet customer expectations.

See standard operating procedure (SOP) ENV-SOP-CORQ-00015 *Document Management and Control* for more information.

1.2.2 Laboratory Quality Manual

The manual is approved and released to personnel under the authority of local management. The manual is reviewed annually and location specific information is updated, if needed. More frequent review and revision may be necessary when there are significant changes to the organizational structure, capabilities, and resources of the laboratory. Review and revision of the manual is overseen by local QA. If review indicates changes to the main body of the manual are necessary to maintain conformance and compliance to relevant standards, or to meet customer expectations, local QA will notify corporate quality personnel to initiate review and/or revision of the template.

See SOP ENV-SOP-CORQ-00015 *Document Management and Control* for more information.

1.2.3 References to Supporting Documents

The template and the manual includes references to other laboratory documents that support the quality management system such as policies and standard operating procedures (SOPs). These references include the document's document control number and may include the document title.

This information is subject to change. For example, an SOP may be converted to a policy or the document's title may change. For these types of administrative changes, the manual and template are updated to reflect the editorial change during the document's next scheduled review/revision cycle or the next time a new version of the document is released, whichever is sooner.

Local QA maintains a current list of controlled documents used at each PAS location to support the quality management system. This list, known as the Master List, lists each document used by document control number, title, version, effective date, and reference to any document(s) that the current version supersedes. When there is a difference between the template and/or manual and the Master List, the document information in the Master List takes precedence. The current Master List is readily available to personnel for their use and cross-reference. Parties external to the laboratory should contact the laboratory for the most current version.

2.0 REFERENCES

References used to prepare this manual include:

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, most current version.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.
- "Methods for Chemical Analysis of Water and Wastes", EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.
- U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis, current version.
- U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis, current version.
- "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF.
- "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.
- "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society of Testing and Materials.
- "NIOSH Manual of Analytical Methods", U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health, most current version.
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (Sep 1986).
- Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.
- Methods for Non-Conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C.
- Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February, 1992.
- Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July, 1990.

- Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, most current version.
- National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.
- ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories- most current version.

  The following are implemented by normative reference to ISO/IEC 17025:

  - ISO/IEC Guide 99, *International vocabulary of metrology –Basic and general concepts and associated terms*
  - ISO/IEC 17000, *Conformity assessment – Vocabulary and general principles*
- Department of Defense Quality Systems Manual (QSM), most current version.
- TNI (The NELAC Institute) Standard- most current version applicable to each lab.
- UCMR Laboratory Approval Requirements and Information Document, most current version.
- US EPA Drinking Water Manual, most current version.

3.0 TERMS AND DEFINITIONS

Refer to Appendix C for terms, acronyms, and definitions used in this manual and in other documents used by the laboratory to support the quality management system.

4.0 MANAGEMENT REQUIREMENTS

4.1 Organization

4.1.1 Legal Identity

Pace Analytical Services, LLC is authorized under the State of Minnesota to do business as a limited liability company.

4.1.1.1 Change of Ownership

If there is a change of ownership, if a location goes out of business, or if the entire organization ceases to exist, Pace Analytical Services, LLC ensures that regulatory authorities are notified of the change within the time-frame required by each state agency for which the location is certified or accredited.

Requirements for records and other business information are addressed in the ownership transfer agreement or in accordance with appropriate regulatory requirements, whichever takes precedence.

4.1.2 Compliance Responsibility

Laboratory management has the responsibility and authority to establish and implement procedures and to maintain sufficient resources necessary to assure its activities are carried out in such a way to meet the compliance requirements of the quality management system.

4.1.3 Scope of the Quality Management System

The quality management system applies to work carried out at each location covered by this manual including permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

The permanent and mobile facilities to which this manual applies includes:

Name	Pace Analytical Services, LLC
Address:	1673 Terra Avenue
City, State, Zip	Sheridan, WY 82801
Phone Number	307-672-8945
Service Type:	Laboratory
Name	Pace Analytical Services, LLC
Address:	4506 Wigwam Drive, Suite D
City, State, Zip	Gillette, WY 82718
Phone Number	307-682-8945
Service Type:	Laboratory

4.1.4 Organization History and Information

Founded in 1978, Pace Analytical Services, LLC (PAS) is a privately held scientific services firm operating one of the largest full service contract laboratory and service center networks in the United States. The company's network offer inorganic, organic and radiochemistry testing capabilities; specializing in the analysis of trace level contamination in air, drinking water, groundwater, wastewater, soil, biota, and waste.

With over 90 laboratories and services centers in the contiguous US and in Puerto Rico, the network provides project support for thousands of industry, consulting, engineering and government professionals.

Pace delivers the highest standard of testing and scientific services in the market. We offer the most advanced solutions in the industry, backed by truly transparent data, a highly trained team, and the service and support that comes from four decades of experience.

4.1.4.1 Organization Structure

Each location maintains a local management structure under the oversight and guidance of corporate personnel. Local management is responsible for making day-to-day decisions regarding the operations of the facility, implementing the quality management system, upholding the requirements of the quality program, and for supervision of personnel.

Local management is provided by a General Manager (GM), Quality Manager (QM), Manager - Client Services (MNGR-CS), Information Technology (IT) Manager, Department Managers (DM) and/or Department Supervisors (DS), however named.

Some locations may also have any one of the following management positions: Regional Quality Manager, Manager - Operations (MNGR - OPS), Technical Specialist (TS), or Technical Manager (TM), however named. When the location

does not have a TS or TM, technical management is provided jointly by the GM, QM, DM, and DS.

The GM, however named, reports to a Regional Operations Manager (RGM), who is responsible for the management of multiple laboratories and service centers within a geographical region, and who reports directly to the Chief Operating Officer (COO). The QMs have indirect reporting relationship to the Corporate Director of Quality.

Refer to the organization charts provided in Appendix D to view the management structure, reporting relationships, and the interrelationships between positions.

4.1.5 Management Requirements

4.1.5.1 Personnel

The laboratory is staffed with administrative and technical personnel who perform and verify work under the supervision of managerial personnel.

- Technical personnel include analysts and technicians that generate or contribute to the generation of analytical data and managerial personnel that oversee day to day supervision of laboratory operations. Including the reporting of analytical data and results, monitoring QA/QC performance, and monitoring the validity of analysis to maintain data integrity and reliability.
- Administrative personnel support the day-to-day activities of the laboratory.
- IT personnel maintain the information technology systems and software used at the laboratory.
- Client services personnel include project managers and support staff that manage projects.
- Managerial personnel make day-to-day and longer term decisions regarding the operations of the facility, supervise personnel, implement the quality management system and uphold the requirements of the quality program.

All personnel regardless of responsibilities are expected to carry out their duties in accordance with the policies and processes outlined in this manual and in accordance with standard operating procedures (SOPs) and other quality system documents. The laboratory's policies and procedures are designed for impartiality and integrity. When these procedures are fully implemented, personnel remain free from undue pressure and other influences that adversely impact the quality of their work or data.

4.1.5.1.1 Key Personnel

Key personnel include the management positions that have the authority and responsibility to plan, direct, and control, activities of the division (corporate) or the laboratory.

The following tables list key personnel positions by PAS job title and the position's primary deputy:

Key Personnel: Corporate

Key Personnel	Primary Deputy
Chief Executive Officer	Chief Operating Officer
Chief Operating Officer	Chief Executive Officer
Chief Compliance Officer	Quality Director
Corporate Quality Director	Chief Compliance Officer
Health and Safety Director	Chief Compliance Officer
IT Director	LIMS Administrator, however named.

Key Personnel: Laboratory

Key Personnel	Primary Deputy
Regional Director - Operations	Chief Operating Officer or as designated.
General Manager	Regional Director - Operations
Manager - Quality	Corporate Quality Manager or as designated.
Manager - Client Services	General Manager
Local IT	Corporate IT Director or as designated.
Department Manager	General Manager
Quality Manager[1]	Corporate Quality Manager
Technical Specialist[1]/Manager[1] Acting Technical Manager TNI	Manager - Quality
Manager - Operations[1]	General Manager

[1] *Position may not be staffed at each location.*

Some state certification programs require the agency to be notified when there has been a change in key personnel. Program-specific requirements and time-frames for notification by agency, are tracked and upheld by local QA, when these requirements apply.

4.1.5.2 Roles and Responsibilities

The qualifications, duties, and responsibilities for each position are detailed in job descriptions maintained by PAS's corporate Human Resource's Department (HR).

The following summaries briefly identify the responsibility of key personnel positions in relation to the quality management system.

Chief Executive Officer (CEO): The CEO has overall responsibility for performance of the organization and endorses the quality program. Working with corporate and laboratory management, the CEO provides the leadership and resources necessary for PAS locations to achieve the goals and objectives of the quality management system and quality policy statement.

Chief Operating Officer (COO): The COO oversees all aspects of operations management including, strategic planning, budget, capital expenditure, and management of senior management personnel. In this capacity, the COO provides leadership and resources necessary to help top management at each PAS location achieve the goals and objectives of the quality management system and quality policy statement.

Chief Compliance Officer (CCO): The CCO oversees the quality assurance and environmental health and safety programs (HSE) for each business unit. The CCO is responsible for planning and policy development for these groups to ensure regulatory compliance and to manage risk. The position provides leadership and

guidance necessary for all PAS locations to achieve the goals and objectives of the quality and HSE programs.

The CCO also serves as the Ethics Officer (ECO). The ECO develops the Ethics and Data Integrity Policy and Training Program, and provides oversight for reporting and investigation of ethical misconduct to maintain employee confidentiality during the process. The ECO provides guidance and instruction for follow-up actions necessary to remedy the situation and deter future recurrence.

Corporate Director of Quality: The Corporate Director of Quality is responsible for developing and maintaining the PAS quality program under guidance and assistance from the CEO, COO, and CCO. This position helps develop corporate quality policy and procedure and analyzes metric data and other performance indicators to assess and communicate the effectiveness of the quality program to top management. The position provides leadership and guidance for implementation of the quality program across all PAS locations.

Corporate Director of Information Technology: The Corporate Director of IT oversees the systems and processes of information technology used to support the quality program. These systems include Laboratory Information Management Systems (LIMS); data acquisition, reduction, and reporting software; virus-protection, communication tools, and ensuring the integrity and security of electronic data.

Regional Director - Operations (RGM): The RGM has full responsibility for administrative and operations management and performance of a group of PAS laboratories and service centers. Working with the COO and local laboratory management, the RGM provides leadership, guidance and resources, including allocation of personnel, necessary to achieve the goals of PAS quality program.

General Manager (GM): The GM is responsible for the overall performance and administrative and operations management of a PAS location and associated service center(s). This position is responsible to provide leadership and resources, including allocation and supervision of personnel, necessary for the location to implement and achieve the goals of the PAS quality program. In this capacity, the position assures laboratory personnel are trained on and understand the structure and components of the quality program defined in this manual as well as the policies and procedures in place to implement the quality management system.

The GM of NELAC/TNI Accredited laboratories are also responsible for the designation of technical personnel to serve as acting technical managers for TNI for the fields of accreditation held by the laboratory (See Section 4.1.5.2.2) and for notifying the accreditation body (AB) of any extended absence or reassignment of these designations.

Quality Manager (QM): The QM oversees and monitors implementation of the quality management system and communicates deviations to laboratory management. The QM is independent of the operation activities for which they provide oversight and has the authority to carry out the roles and responsibilities of their position without outside influence.

Additionally, in accordance with the TNI Standard, the QM:

- serves as the focal for QA/QC and oversees review of QC data for trend analysis;
- evaluates data objectively and perform assessments without outside influence;
- has document training and experience in QA/QC procedures and the laboratory's quality system;
- has a general knowledge of the analytical methods offered by the laboratory;
- coordinates and conducts internal systems and technical audits;
- notifies laboratory management of deficiencies in the quality system;
- monitors corrective actions;
- provides supports to technical personnel and may serve as the primary deputy for the acting TNI Technical Manager(s).

Manager - Client Services (MNGR-CS): The MNGR-CS oversees project management personnel. This position is responsible for training and management of client facing staff that serve as the liaison between PAS and the customer to ensure that projects are successfully managed to meet the expectations and needs of PAS customers. This position is also responsible for sharing positive and negative customer feedback with laboratory management so that this information may be used to improve the quality program.

Local IT Manager, however named: Local IT managers are responsible for maintaining the IT systems used to support the quality program. These systems include Laboratory Information Management Systems (LIMS); data acquisition, reduction, and reporting software; virus-protection, communication tools, and ensuring the integrity and security of electronic data.

Department Manager (DM): The DM is responsible for administrative and operations management and implementation of the quality management system in the work area he/she oversees. These responsibilities include but are not limited to: training and supervision of personnel, monitoring work activity to maintain compliance with this manual, SOPs, policies and other instructional documents that support the quality management system; method development, validation and the establishment and implementation of SOPs to assure regulatory compliance and suitability for intended purpose; monitoring QA/QC performance, proper handling and reporting of nonconforming work, purchasing of supplies and equipment adequate for use, maintaining instrumentation and equipment in proper working order and calibration, and general maintenance of administrative and technical processes and procedures established by the laboratory.

Quality Manager 2 (QM2): The QM2 provides support to the quality manager and assists the quality manager with implementation of the quality management system for one or more site locations.

Technical Specialist (TS): The TS provides technical oversight and guidance to laboratory personnel. Responsibilities may include but are not limited to: research and development, method development and validation, development of standard

operating procedures, proposal and contract review. The TS may also be responsible for QA/QC trend analysis, technical training, and technology improvement.

Manager - Operations (MNGR-OPS): The MNGR-OPS is responsible for management of production and/or other duties assigned by the GM or RGM.

4.1.5.2.1 Acting Technical Manager (TNI Accreditation):

For PAS locations that are NELAC/TNI accredited:

The TNI Standard specifies requirements for the qualification and duties of technical personnel with managerial responsibility. These requirements are associated in the Standard to the designation 'technical manager(s), however named'. These responsibilities may be assigned to multiple individuals and are not associated with any specific job title.

For PAS, these TNI requirements for personnel that provide technical oversight correlate with PAS's job descriptions for Department Manager or Supervisor. However, the duties may be assigned to any PAS employee that meets the TNI specified qualifications.

Personnel assigned this designation retain their PAS assigned job title. The job title may be appended with *"acting as technical manager for TNI"* and the technology or field of accreditation for which the employee is approved, if necessary.

When TNI Accreditation Bodies (AB) refer to these employees as 'technical manager' or 'technical director' on the official certificate or the scope of accreditation, this reference is referring to their approval to carry out duties of the 'technical manager, however named' as specified in the TNI Standard.

In accordance with the TNI Standard, the acting Technical Manager(s) for TNI are responsible for monitoring the performance of QC/QA in the work areas they oversee.

If the absence of any employee that is approved as acting technical manager for TNI exceeds 15 calendar days, the duties and responsibilities specified in the TNI Standard are reassigned to another employee that meets the qualifications for the technology or field of accreditation or they are assigned to the position's deputy, the quality manager.

4.1.5.3 Conflict of Interest

A conflict of interest is a situation where a person has competing interests. Laboratory management looks for potential conflict of interest and undue pressures that might arise in work activities and then includes countermeasures in policies and procedures to mitigate or eliminate the conflict.

See policy COR-POL-0004 *Ethics Policy* for more information.

4.1.5.4 Confidentiality

Laboratory management is committed to preserving the confidentiality of PAS customers and confidentiality of business information.

Procedures used by the laboratory to maintain confidentiality include:

- A Confidentiality Agreement which all employees are required to sign at the time of employment and abide by the conditions of throughout employment;
- Record retention and disposal procedures that assure confidentiality is maintained;
- Physical access controls and encryption of electronic data; and
- Protocol for handling Confidential Business Information (CBI).

Client information obtained or created during work activities is considered confidential and is protected from intentional release to any person or entity other than the client or the client's authorized representative information provided to PAS, except when the laboratory is required by law to release confidential information to another party, such as a regulatory agency or for litigation purposes. In which case, the laboratory will notify the client of the release of information and the information provided.

The terms of client confidentiality are included in PAS Standard Terms and Conditions (T&C). With the acceptance of PAS Terms and Conditions and/or the implicit contract for analytical services that occurs when the client sends samples to the laboratory for testing, the client authorizes PAS to release confidential information when required.

See policy COR-POL-0004 *Ethics Policy* for more information.

4.1.5.5 Communication

Communication is defined as the imparting or exchanging of news and information. Effective (good) communication occurs when the person(s) you are exchanging information with actively gets the point and understands it.

4.1.5.5.1 Workplace Communication

Good communication in the workplace is necessary to assure work is done correctly, efficiently, and in accordance with client expectations.

Instructions for how to carry out work activities are communicated to personnel via written policy, standard operating procedures, and standard work instructions.

Information about laboratory performance (positive and negative) and ideas for improvement are communicated using various communication channels such as face to face meetings, video conferencing, conference calls, email, memoranda, written reports, and posters.

4.1.5.5.2 External Communication

Communication with external parties such as customers, vendors, business partners, and regulatory agencies takes place every day.

Laboratory management ensure personnel learn to communicate in professional and respectful ways in order to build strong relationships, and learn to communicate effectively to avoid misunderstanding.

4.2 Quality Management System

4.2.1 Quality Management System Objectives

The objectives of the laboratory's quality management system are to provide clients with consistent, exemplary professional service, and objective work product that is of known and documented quality that meets their requirements for data usability and regulatory compliance.

Objective work product is analytical services, data, test results, and information that is not influenced by personal feeling or opinions. The quality of being objective is also known as 'impartiality'.

4.2.1.1 Impartiality

The laboratory achieves and maintains impartiality by implementing and adhering to the policies and processes of the quality management system, which are based on industry accepted standards and methodologies.

The laboratory's procedures for handling nonconforming work (See 4.9), corrective and preventive actions (See 4.11) and management review (See 4.15) are the primary mechanisms used to identify risk to impartiality and to prompt actions necessary to eliminate or reduce the threat when risk to impartiality is suspected or confirmed.

4.2.1.2 Risk and Opportunity Assessment

Risks are variables that make achieving the goals and objectives of the quality management system uncertain. An opportunity is something that has potential positive consequences for the laboratory.

Laboratory personnel manage risks and opportunities on a daily basis by carrying out the processes that make up the quality management system. Some of the ways in which the quality management system is designed to identify, minimize, or eliminate risk on a daily basis include but are not limited to:

- Capability and capacity reviews of each analytical service request to assure the laboratory can meet the customer's requirements;
- Maintenance of accreditation and certification for test methods in multiple states and programs to cover a broad range of jurisdiction for regulatory compliance;
- SOPs and other controlled instructional documents are provided to personnel to eliminate variability in process. These documents include actions to counter

risk factors inherent in the process and are reviewed on a regular basis for on-going suitability and relevancy;

- Participation in proficiency testing programs and auditing activities to verify on-going competency and comparability in performance;
- Provision of on-the-job training and established protocol for quality control (QC) corrective action for nonconforming events;
- An established program for ethics, and data integrity;
- Tiered data review process;
- Culture of continuous improvement;
- Monitoring activities to assess daily and long term performance; and
- Annual critical review of the effectiveness of the quality management system.

PAS also promotes a continuous improvement culture based on the principles of lean manufacturing. These principles include 3P (Process, Productivity, Performance) and Kaizen. 3P is a platform used by PAS to share best practices and standardization across the network to achieve operational excellence. Kaizen is a team based process used to implement tools and philosophies of lean to reduce waste and achieve flow with the purpose of improving both external and internal customer satisfaction. PAS's lean programs and activities help to mitigate risk because they generate a collective understanding of vulnerabilities and utilize group-effort to develop and implement solutions at all levels.

Risk and opportunities may also be formally identified using specific risk and opportunity assessment methods such as SWOT Analysis (Strength, Weakness, Opportunity, Threats) and 3-Stage Impact/Probability Grids.

4.2.1.3 Communication of the Quality Management System

This manual is the primary mechanism used by laboratory management to communicate the quality management system to laboratory personnel.

To assure personnel understand and implement the quality program outlined in the manual:

- All laboratory personnel are required to sign a Read and Acknowledgement Statement to confirm the employee has: 1) been informed of the manual by laboratory management, 2) has access to the manual, 3) has read the manual 4) understands the content of the manual, and 5) agrees to abide by the requirements, policies and procedures therein.
- Personnel are informed that the manual provides the "what" of the quality management system. The "how to" implementation of the quality management system is provided in policy, SOPs, standard work instructions, and other controlled instructional documents.

4.2.2 Quality Policy Statement

The quality policy of the laboratory is to provide customers with data of known and documented quality fit for their intended purpose. The laboratory achieves this policy by implementing the quality management system defined in this manual, by following industry accepted protocol for analytical testing and quality assurance and quality control (QA/QC) activities, by conformance with published and industry accepted testing methodologies, and by compliance with international and national standards for the competency and/or accreditation of testing laboratories.

Intrinsic to this policy statement is each of the following principles:

- The laboratory will provide customers with reliable, consistent, and professional service. This is accomplished by making sure the laboratory has the resources necessary to maintain capability and capacity; that staff are trained and competent to perform the tasks they are assigned; that client-facing staff are trained and prepared to find solutions to problems and to assist customers with their needs for analytical services. Customer feedback, both positive and negative, is shared with personnel and used to identify opportunities for improvement.
- The laboratory maintains a quality program that complies with applicable, state, federal, industry standards for analytical testing and competency.

  ISO/IEC 17025 and the TNI (The NELAC Institute) Standard is used by PAS to establish the minimum requirements of the PAS quality program.

  ISO/IEC 17025 is a competency standard that outlines the general requirements for the management system for calibration and testing laboratories. It is the primary quality system standard from which other quality system standards, such as the TNI Standard, are based. The TNI Standard are consensus standards that provides management and technical requirements for laboratories performing environmental analysis.
- Laboratory management provides training to personnel so that all personnel are familiar with the quality management system outlined in this manual and that they understand that implementation of the quality management system is achieved by adherence to the organization's policies and procedures.
- Laboratory management continuously evaluates and improves the effectiveness of the quality management system by responding to customer feedback, and other measures of performance, such as but not limited to: the results of internal/external audits, proficiency testing, metrics, trend reports, and annual and periodic management reviews.

4.2.2.1 Ethics Policy / Data Integrity Program

PAS has established a comprehensive ethics and data integrity program that is communicated to all PAS employees in order that they understand what is expected of them. The program is designed to promote a mindset of ethical behavior and professional conduct that is applied to all work activities.

The key elements of the PAS Ethics / Data Integrity Program include:

- Ethics Policy (COR-POL-0004);
- Ethics Compliance Officer;
- Standardized data integrity training course taken by all new employees on hire and a yearly refresher data integrity training course for all existing employees;
- Policy Acknowledgement Statements that all PAS personnel, including contract and temporary, are required to sign at the time of employment and again during annual refresher training to document the employee's commitment and obligation to abide by the company's standards for ethics, data integrity and confidentiality;
- SOPs that provide instructions for how to carry out a test method or process to assure tasks are done correctly and consistently by each employee;
- On the Job Training;
- Data integrity monitoring activities which include, but are not limited to, secondary and tertiary data review, internal technical and system audits, raw data audits, data mining scans, and proficiency testing; and
- Confidential reporting process for alleged ethics and data integrity issues.

All laboratory managers are expected to provide a work environment where personnel feel safe and can report unethical or improper behavior in complete confidence without fear of retaliation. Retaliation against any employee that reports a concern is not tolerated.

PAS has engaged Lighthouse Services, Inc. to provide personnel with an anonymous reporting process available to them 24 hours a day/7 days per week. The alert line may be used by any employee to report possible violations of the company's ethics and data integrity program. When using the reporting process, the employee does need to specify the location of concern and when reporting by email, also include the company name. Messages are collected, documented, reviewed, and will be followed up on by the Ethics Compliance Officer to resolve the matter. Investigations concerning data integrity are kept confidential.

Lighthouse Compliance Alert Lines:

English Speaking US & Canada	+1 (844) 940-0003
Spanish Speaking North America	+1 (800) 216-1288
Internet	www.lighthouse-services.com/pacelabs
Email	reports@lighthouse-services.com

4.2.3 Management Commitment: Quality Management System

Evidence of management's commitment for the development, maintenance, and on-going improvement of the quality management system is provided by the application of their signature of approval to this manual. Their signature confirms they understand their responsibility to implement the quality management system outlined in this manual, to communicate the quality program to personnel, and to uphold requirements of the program during work activities.

4.2.4 Management Commitment: Customer Service

Management communicates the importance of meeting customer and regulatory requirements to personnel by training personnel on the quality management system outlined in this manual, implementing the quality management system outlined in this manual, and upholding these requirements for all work activities.

4.2.5 Supporting Procedures

Documents that support this manual and quality management system are referenced throughout this manual. The structure of the document management system is outlined in SOP ENV-SOP-CORQ-0015 *Document Management and Control* and summarized in the following subsections.

4.2.5.1 Quality Management System Document Structure

Documents associated with the quality management system are classified into document types that identify the purpose of the document and establish how the document is managed and controlled.

Document types are ranked to establish which documents takes precedence when there is an actual or perceived conflict between documents and to establish the hierarchal relationships between documents. The ranking system also provides information to document writers and reviewers to assure downline documents are in agreement with documents of higher rank. Project specific documents are not ranked because client specific requirements are not incorporated into general use documents in order to maintain client confidentiality.

PAS Quality Management System Documents: Internal

Document Type	Purpose
Quality Manual	Outlines the laboratory's quality management system and structure and how it works for a system including policy, goals, objectives and detailed explanation of the system and the requirements for implementation of system. Includes roles and responsibilities, relationships, procedures, systems and other information necessary to meet the objectives of the system described.
Policy	Provide requirements and rules for a PAS process and is used to set course of actions and to guide and influence decisions. Policy describes the "what", not the "how".
Standard Operating Procedure	Provide written and consistent set of instructions or steps for execution of a routine process, method, or set of tasks performed by PAS. Includes both fundamental and operational elements for implementation of the systems described in PAS manual(s). Assures that activities are performed properly in accordance with applicable requirements. Designed to ensure consistency, protect EHS of employees and environment, prevent failure in the process and ensure compliance with company and regulatory requirements. SOPs describes the "how" based on policy.
Standard Work Instruction	Provide step by step visual and/or written instruction to carry out a specific task to improve competency, minimize variability, reduce work injury and strain, or to boost efficiency and quality of work (performance). SWI are associated with an SOP unless the task described is unrelated to generation of or contribution to environmental data or analytical results.
Template	Pre-formatted document that serves as a starting point for a new document.
Guide	Provide assistance to carry out a task. Most often used for software applications.
Form	Used for a variety of purposes such as to provide a standardized format to record observations, to provide information to supplement an SOP.

PAS Quality Management System Documents: External

Certificate	Lists parameters, methods, and matrices for which the laboratory is certified/accredited to perform within the jurisdiction of the issuing regulatory agency or accreditation body.
Reference Document	Provide information, protocol, instructions, and/or requirements. Issued by the specifier. Examples include quality system standards such as ISO/IEC, TNI, DoD and published referenced methods such as Standard Methods, ASTM, SW846, EPA, and federal and state regulatory bodies.
Project Document	Provides requirements necessary to meet individual client expectations for intended use of data. Examples include: project quality assurance plans (QAPP), client-program technical specifications, contracts, and other agreements.

Document Hierarchy

Rank	Document
1	Reference Documents
2	Corporate Manual
3	Corporate Policy
4	Corporate SOP
5	Corporate SWI, Templates & Forms
6	Laboratory Manual
7	Laboratory SOP
8	Laboratory SWI, Templates, & Forms
NA	Project Documents[1]

4.2.6 Roles and Responsibilities

The roles and responsibilities of technical management and of the quality manager are provided in section 4.1.5.1.2.

4.2.7 Change Management

When significant changes to the quality management system are planned, these changes are managed by corporate quality personnel to assure that the integrity of the quality management system is maintained.

4.3 Document Control

4.3.1 General

The laboratory's procedures for document control are provided in SOP ENV-SOP-CORQ-0015 *Document Management and Control.*

The documents that support the quality management system include internally generated documents such as manuals, policies, standard operating procedures, standard work instructions, forms, guides, and templates and external source documents such as but not limited to, regulations, standards, reference methods, manuals, and project-specific documents.

The laboratory uses electronic document management software (eDMS) to carry out the procedures of the SOP. eDMS automates the process for unique document identification, version control, approval, access, and archival.

4.3.2 Document Approval and Issue

Documents that are part of the quality management system are reviewed by qualified personnel and approved by laboratory management prior to release for general use.

Local QA maintains a master list of controlled documents used at the laboratory. The master list includes the document control number, document title, and current revision status and is made available to personnel for their reference.

Only the approved versions of documents are available to personnel for use. The eDMS system does not allow user access to draft versions of documents except to personnel assigned to work on the draft. eDMS also restricts access to archived documents except to authorized users, such as local QA, in order to prevent the use of obsolete documents.

See SOP ENV-SOP-CORQ-0015 *Document Management and Control* for more information.

4.3.3 Document Review and Change

Unless a more frequent review is required by regulatory, certification or accreditation program, the laboratory formally reviews documents at least every two years to ensure the document remains current, appropriate, and relevant.

Documents are also informally reviewed every time the document is used. Personnel are expected to refer to and follow instructions in controlled documents when they carry out their work activities. Consequently, any concerns or problems with the document should be caught and brought to the attention of laboratory management on an on-going basis.

Documents are revised whenever necessary to ensure the document remains usable and correct. Older document versions and documents no longer needed are made obsolete and archived for historical purposes.

The laboratory does not allow hand-edits to documents. If an interim change is needed pending re-issue of the document, the interim change is communicated to those that use the document using a formal communication channel, such as SOP Change in Progress form, email, or memorandum.

The document review, revision, and archival process is managed by local QA at the location from which the document was released using the procedures established in SOP ENV-SOP-CORQ-0015 *Document Management and Control.*

4.4 Analytical Service Request, Tender, and Contract Review

The laboratory's management and/or client service personnel perform thorough reviews of requests and contracts for analytical services to verify the laboratory has the capability, capacity, and resources necessary to successfully meet the customer's needs. These review procedures are described in laboratory SOP Contract Review Procedure.

The procedures in this SOP(s) are established to ensure that:

- The laboratory understands the purpose of data collection in order to ensure the test methods requested are appropriate for the intended use of the data and capable of meeting the client's data quality objectives;
- The laboratory and any subcontractor has the capability, capacity, and resources to meet the project requirements and expectations within the requested time frame for delivery of work product;

- Any concerns that arise from review are discussed and resolved with the client; and
- The results of review and any correspondence with the client related to this process and/or any changes made to the contract are recorded and retained for historical purposes.

Capability review confirms that the in-network laboratories and any potential subcontractors hold required certification/accreditation for the test method, matrix, and analyte and verifies the laboratory can achieve the client's target compound list and data quality objectives (DQOs) for analytical sensitivity and reporting limits, QA/QC protocol, and hardcopy test report and electronic data deliverable (EDD) formats.

Capacity review verifies that the in-network laboratories and any potential subcontractors are able to handle the sample load and deliver work production within the delivery time-frame requested.

Resource review verifies that the laboratory and any potential subcontractors have adequate qualified personnel with the skills and competency to perform the test methods and services requested and sufficient and proper equipment and instrumentation needed to perform the services requested.

4.5 Subcontracting and In-Network Work Transfer

The terms 'subcontract' and "subcontracting" refers to work sent to a business external to Pace Analytical Services, LLC (PAS) and the term 'subcontractor' refers to these external businesses, which are also called vendors.

Work transferred within the PAS network is referred to as interregional work orders (IRWO) and network laboratories are referred to as IR or network laboratory.

The network of PAS laboratories offers comprehensive analytical capability and capacity to ensure PAS can meet a diverse range of client needs for any type of project. If the laboratory receives a request for analytical services and it cannot fulfill the project specifications, the laboratory's client services team will work with the client to place the work within the PAS network. When it is not possible to place the work within network, the laboratory will, with client approval, subcontract the work to a subcontractor that has the capabilities to meet the project specifications and can meet the same commitment agreed on between the laboratory and the client. Some client programs require client consent even for IRWO work transfer, and when this applies, the client services team obtains consent as required. The laboratory retains the record of client notification and their consent in the project record for historical purposes.

Whenever work is transferred to a subcontractor or an IRWO laboratory, the laboratory responsible for management of the project verifies each of these qualifications:

- The subcontractor or IRWO laboratory has the proper accreditation/certifications required for the project and these are current; and
- The use of the subcontractor or IRWO laboratory is approved by the client and/or regulatory agency, when approval is required. Record of approval is retained in the project record.

When possible, the laboratory selects subcontractors that maintain a quality management system similar to PAS and that comply with ISO/IEC 17025 and the TNI Standard(s).

PAS also evaluates and pre-qualifies subcontractors as part of the company's procurement program. The complete list of approved vendors is maintained by the corporate procurement department and is made available to all PAS locations. Pre-qualification of a subcontractor does not replace the

requirement for the placing laboratory to verify the capability, capacity, and resources of any selected subcontractor on a project-specific basis to confirm the subcontractor can meet the client's needs.

For both subcontracting and in-network work transfer, the project specifications are always communicated to the subcontractor or the IRWO laboratory by the project manager so that the laboratory performing the work is aware of and understands these requirements.

The procedures for subcontracting are outlined in laboratory SOP Purchasing/Subcontracting Procedure.

4.6 Purchasing Services and Supplies

Vendors that provide services and supplies to the laboratory are prequalified by corporate procurement personnel to verify the vendor's capability to meet the needs of PAS. These needs include but are not limited to: competitive pricing, capacity to fill purchase orders, quality of product, customer service, and business reputation and stability. The records of vendor evaluation and the list of approved vendors is maintained by the corporate procurement department.

The laboratory may purchase goods and services from any supplier on the approved vendor list.

The specifications (type, class, grade, tolerance, purity, etc.) of supplies, equipment, reagents, standard reference materials and other consumables used in the testing process are specified in SOPs. The SOP specifications are based on the governing requirements of the approved reference methods and any additional program driven regulatory specification, such as drinking water compliance. All requisitions for materials and consumables are approved by the department supervisor to confirm the purchase conforms with specified requirements. After approval the requisition is handled by the laboratory's designated purchasing agent. On receipt, the product is inspected and verified before use, when applicable.

The laboratory's procedure for the purchase of services and supplies is specified in laboratory SOP Purchasing/Subcontracting Procedure.

4.7 Customer Service

Project details and management are handled by the laboratory's customer service team. Each customer is assigned a Project Manager (PM) that is responsible for review of contract requirements and handling laboratory to customer communication about the project status.

4.7.1 Commitment to Meet Customer Expectations

The laboratory cooperates and works closely with our customers to ensure their needs are met and to establish their confidence in the laboratory's capability to meet their needs for analytical services and expectations for service.

Each customer's project is handled by a PM that is the customer's primary point of contact. The PM gathers information from the customer to ensure the details of their request are understood. After samples are received, the PM monitors the progress of the project and alerts the customer of any delays or excursions that may adversely impact data usability. Laboratory supervisors are expected to keep the PM informed of project status and any delays or major issues, so that the PM can keep the client informed.

PAS also has a team of subject matter experts (SME) available to provide customers with advice and guidance and any other assistance needed. SME are selected by top management based on their knowledge, experience, and qualifications.

The laboratory encourages customers to visit the laboratory to learn more about the laboratory's capabilities, observe performance and to meet laboratory personnel.

PAS customers expect confidentiality. Laboratory personnel will not divulge or release information to a third party without proper authorization unless the information is required for litigation purposes. See Section 4.1.5.3 of this manual and policy COR-POL-0004 *Ethics Policy* for more information on the laboratory's policy for client confidentiality.

4.7.2 Customer Feedback

The laboratory actively seeks positive and negative feedback from customers through surveys and direct communication. Information from the client about their experience working with the laboratory and their satisfaction with work product is used to enhance processes and practices and to improve decision making. Customer feedback is communicated to laboratory management and corporate personnel in monthly reports and analyzed yearly during management review (See 4.15) to identify risk and opportunity. Corrective, preventive, or continuous improvement actions are taken based on the nature of and/or feedback trends.

Also see sections 4.9, 4.10, 4.11, 4.12, 4.14, and 4.15 for more information about how customer feedback is managed by the laboratory and used to enhance the quality management system.

4.8 Complaints

Complaints provide opportunities to improve processes and build stronger working relationships with our clients.

The laboratory's complaint resolution process includes three steps. First, handle and resolve the complaint to mutual satisfaction. Second, perform corrective action to prevent recurrence (See 4.11). Third, record and track the complaint and use these records for risk and opportunity assessment and preventive action. (See 4.12)

4.9 Nonconforming Work

4.9.1 Definition of Nonconforming Work

Nonconforming work is work that does not conform to customer requirements, standard specifications, laboratory policies and procedures, or that does not meet acceptance criteria.

The discovery of non-conforming work comes from various sources which include, but are not limited to:

- results of quality control samples and instrument calibrations;
- quality checks on consumables and materials;
- general observations of laboratory personnel;
- data review;
- proficiency testing;

- internal and external audits;
- complaints and feedback;
- management review and reports; and
- regulatory and certification and accreditation actions.

The way in which the laboratory handles nonconforming work depends on the significance and impact (risk) of the issue. Some issues may simply require correction, others may require investigation, corrective action (See 4.11) and/or data recall (See 4.16). When the laboratory releases data and test results associated with nonconforming QC and acceptance criteria test results are qualified or non-conformances are noted in the final analytical report to apprise the data user of the situation. (See 5.10)

Nonconforming work also includes unauthorized departure from laboratory policies, procedures and test methods. Authorized departures are explained in the following subsections. Situations that do not conform to these conditions are considered unauthorized departure(s).

4.9.1.1 Authorized Departure from SOP

An authorized departure from a test method SOP is one that has been reviewed and approved by the Department Manager, Technical Manager, Acting Technical Manager for TNI, Quality Manager, or the General Manager. Review is conducted to confirm the departure does not conflict with regulatory compliance requirements for which the data will be used or does not adversely affect data integrity. The departure may originate from client request or may be necessary to overcome a problem.

An authorized departure from administrative or process-oriented SOP is typically necessary to correct an error in the SOP. These departure requests are reviewed and pre-approved by the local QA Manager. Documentation of SOP departures and approval decisions are retained by the laboratory as evidence that the departure was authorized. When necessary, approved departures from test method SOPs are noted in the final test report to advise the data user of any ramification to data quality.

4.9.1.2 Authorized Departure from Test Methods (Method Modifications)

When test results are associated to a published reference test method, the laboratory's test method SOP must be consistent with the test method. If the test method is mandated for use by a specific regulatory program such as drinking water or wastewater or a certification or accreditation program, such as TNI/NELAC, the SOP must also comply with or include these requirements. If the procedures in the SOP are modified from the test method, these modifications must be clearly identified in the SOP. The conditions under which the laboratory may establish an SOP that is modified from these reference documents, and what is considered a modification are specified in ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification*.

Modifications that do not meet the requirements of this SOP (ENV-SOP-CORQ-0011) are unauthorized. Client requests to deviate from the test method are handled as client requests to depart from the test method SOP since it is the SOP that the laboratory follows when performing work.

4.9.1.3 Stop Work Authority

Stop Work Authority provides laboratory personnel with the responsibility and obligation to stop work when there is a perceived unsafe condition or behavior that may result in an unwanted event.

All laboratory and corporate personnel have the authority to stop work when needed to preserve data integrity or safety of workers.

Once a stop work order has been initiated and the reason for doing so is confirmed valid; laboratory management is responsible for immediate correction and corrective action (see section 4.10) before resumption of work.

4.10 Continuous Improvement

The laboratory's quality management system is designed to achieve continuous improvement through the implementation of the quality policy and objectives outlined in this manual. Information about the laboratory's activities and performance is gained from many sources such as customer feedback, audits, QC, trend analysis, business analytics, management reports, proficiency testing, and management systems review. This information is subsequently used during the laboratory's corrective action (see section 4.11) and preventive action (see section 4.12) processes and to establish goals and objectives during annual review of the management system (see section 4.15).

PAS also promotes a continuous improvement culture based on the principles of lean manufacturing. These principles include 3P (Process, Productivity, Performance) and Kaizen. 3P is a platform used by Pace to share best practices and standardization across the network to achieve operational excellence. Kaizen is a team based process used to implement tools and philosophies of lean to reduce waste and achieve flow with the purpose of improving both external and internal customer satisfaction.

4.11 Corrective Action

Corrective action is a process used to eliminate the cause of a detected nonconformity. It is not the same as a correction. A correction is an action taken to fix an immediate problem. The goal of the corrective action process is to find the underlying cause(s) of the problem and to put in place fixes to prevent the problem from happening again. The corrective action process, referred to as CAPA by PAS, is one of the most effective tools used by the laboratory to prevent nonconforming work, identify risk and opportunity, and improve service to our customers.

The laboratory has two general processes for corrective action:

The process used for actions taken in response to day to day quality control (QC) and acceptance criteria exceptions (nonconformance) that occur during the day to day testing process are called corrections. These events do not usually include formal methods for cause analysis; instead the reason for the failure is investigated through troubleshooting or other measures. Required actions for correction of routine nonconformance is specified in laboratory SOPs. When corrective action is not taken, cannot be taken, or is not successful, test results associated with the nonconforming work are qualified in the final test report. Documentation of the nonconformance and corrective action taken is documented in the analytical record.

A formal 7 step corrective action process is used when there is a problem or departure from the quality management system, technical activities, or when the extent of a single problem has

significant impact on data, regulatory compliance or customer needs. These problems are identified through various activities such as but not limited to: quality control trends, internal and external audits, management review, customer feedback, and general observation.

The laboratory's 7 Step CAPA Process includes:

1) Define the Problem
2) Define the Scope of the Problem
3) Contain the Problem
4) Root Cause Analysis
5) Plan Corrective Action
6) Implement Corrective Action
7) Follow Up / Effectiveness Check

The formal CAPA process may be initiated by any employee. Once the process is initiated it is overseen and coordinated by laboratory management. The CAPA process is documented using an electronic or paper-based system. The CAPA record includes tracking information, dates, individuals involved, those responsible for action plan implementation and follow-up, and timelines and due dates.

For more information about the laboratory's procedure for corrective action, see laboratory SOP Corrective Action Procedure. Additional explanation about certain aspects of the laboratory's corrective action process are outlined in the next three subsections.

4.11.1 Root Cause Analysis

Root cause analysis (RCA) is the process of investigation used by the laboratory to identify the underlying cause(s) of the problem. Once causal factors are identified, ways to mitigate the causal factors are reviewed and corrective action(s) most likely to eliminate the problem are selected.

The laboratory uses different methods to conduct this analysis. The most common approach is 5-Why, but fishbone diagrams, or even brainstorming may be appropriate depending on the situation. The method used is documented in the CAPA record.

4.11.2 Effectiveness Review

Monitoring corrective actions for effectiveness is shared by laboratory supervisors and quality assurance personnel. Effectiveness means the actions taken were sustainable and appropriate. Sustainable means the change is still in place. Appropriate means the action(s) taken prevented recurrence of the problem since the time corrective action was taken.

The time-frame in which effectiveness review takes place depends on the event and is recorded in the CAPA record with any additional actions that need to be taken.

Corrective action trends are also monitored by laboratory management and used to identify opportunities for preventive action or to gain lessons learned when actions taken were not adequate to solve the problem. See Section 4.12 (Preventive Action) and 4.15 (Management Review) for more information.

4.11.3 Additional Audits

When non-conformances or other problems cast doubt on compliance with the laboratory's policies, procedures, or compliance to regulatory requirements; laboratory management schedules a special audit of the area of activity in accordance with Section 4.14.1 as soon as

possible. These special audits are used to determine the scope of the problem and to provide information for the CAPA process. Additional full-scale audits are done when a serious issue or risk to the laboratory's business is identified.

4.12 Preventive Action

Preventive action is an action taken to eliminate the cause of a potential nonconformity and to achieve improvement. Preventive action is a forward thinking process designed to prevent problems opposed to reacting to them (corrective action).

Some examples of preventive action include, but are not limited to:

- Scheduled instrument maintenance (Preventive maintenance)
- Addition of Staff and Equipment
- Professional Development Activities
- Implementation of New Technology

The laboratory looks for opportunities for preventive action from a variety of sources including but not limited to: employee idea's, customer feedback, business partners input, trend analysis, business analytics, management reviews, proficiency testing results, lean management events, and risk-benefit analysis.

The process for preventive actions follows the same 7 step process for corrective action except "problem" is replaced with "opportunity", "cause analysis" is replaced with "benefit analysis", and "corrective action" is replaced with "preventive action".

Laboratory management evaluates the success of preventive actions taken in any given year during annual management review. See Section 4.15 for more information.

4.12.1 Change Management

Preventive actions may sometimes result in significant changes to processes and procedures used by the laboratory. Laboratory management evaluates the risks and benefits of change and includes in its implementation of change process, actions to minimize or eliminate any risk. The types of changes for which risk are considered and managed include: infrastructure change, change in analytical service offerings, certification or accreditation status, instrumentation, LIMS changes, and changes in key personnel.

For more information about the laboratory's procedures for preventive action see laboratory SOP *Preventive Action Procedure*.

4.13 Control of Records

A record is a piece of evidence about the past, especially an account of an act or occurrence kept in writing or some other permanent form. Laboratory records document laboratory activities and provide evidence of conformity to the requirements established in the quality management system. These records may be hardcopy or electronic on any form of media.

4.13.1 General Requirements

4.13.1.1 Procedure

The laboratory's procedures for control of records is provided in laboratory SOP *Control of Records Procedure* as well as the corporate policy *ENV-POL-CORQ_0013 on Record Management* or equivalent replacements.

The procedures in the SOP are established to assure quality and technical records are identified, retained, indexed, and filed to allow for retrieval during the entire retention time frame. During storage, records are kept secure and protected from deterioration. At the end of the retention time, the records are disposed of properly in order to maintain client confidentiality and to protect the interests of the company.

In general, laboratory records fall into three categories: quality, technical, and administrative.

Examples of each are provided in the following table:

Record Type	Includes Records of:
Quality	Documents: Document Types listed in SOP ENV-SOP-CORQ-0016 Audits: Internal and External Certificates and Scopes of Accreditation Corrective & Preventive Action Management Review Data Investigations Method Validation Instrument Verification Training Records
Technical	Raw Data Logbooks Certificates of Traceability Analytical Record Test Reports & Project Information Technical Training Records & Demonstration of Capability
Administrative	Personnel Records Finance/Business

4.13.1.2 Record Legibility and Storage

Records are designed to be legible and to clearly identify the information recorded. Manual entries are made in indelible ink; automated entries are in a typeface and of sufficient resolution to be read. The records identify laboratory personnel that performed the activity or entered the information.

Records are archived and stored in a way that they are retrievable. Access to archived records is controlled and managed.

For records stored electronically, the capability to restore or retrieve the electronic record is maintained for the entire retention period. Hardcopy records are filed and stored in a suitable environment to protect from damage, deterioration, or loss. Hardcopy records may be scanned to PDF for retention. Scanned records must be checked against the hardcopy to verify the scan is complete and legible.

Records are kept for a minimum of 10 years unless otherwise specified by the client or regulatory program.

The date from which retention time is calculated depends on the record. In general, the retention time of technical records of original observation and measurement is calculated from the date the record is created. If the technical record is kept in a chronological logbook, the date of retention may be calculated from the date the logbook is archived. The retention time of test reports and project records, which are considered technical records, is calculated from the date the test report was issued. The retention time of quality records is usually calculated from the date the record is archived.

Refer to the laboratory's record management SOP for more information.

4.13.1.3 Security

The laboratory is a secure facility and access to records is restricted to laboratory personnel.

4.13.1.4 Electronic Records

The data systems used to store electronic records is backed up in accordance with laboratory SOP IT Procedures. Access to archived records stored electronically is maintained by personnel responsible for management of the electronic system.

4.13.2 Technical Records

In addition to the requirements identified in subsections 4.13.1.1 through 4.13.1.4, the requirements in the following subsections also apply to technical records.

4.13.2.1 Description

Technical records are the accumulation of data and information generated from the analytical process. These records may include forms, worksheets, workbooks, checklists, notes, raw data, calibration records, final test reports, and project records. The accumulated record essentially needs to provide sufficient detail to historically reconstruct the process and identify the personnel that performed the tasks associated with a test result.

4.13.2.2 Real Time Recordkeeping

Personnel are instructed and expected to always record observations, data, and calculations at the time they are made. Laboratory managers are responsible to assure that data entries, whether made electronically or on hardcopy, are identifiable to the task.

4.13.2.3 Error Correction

Errors in records must never be erased, deleted or made illegible. Use of correction fluid, such as white-out is prohibited. In hardcopy records, the error is corrected by a single-strike through the original entry and the new entry recorded alongside or footnoted to allow for readability. Corrections are initialed and dated by the person making the correction. If the correction is not self-explanatory, a reason for the correction is recorded.

For electronic records, equivalent measures of error correction or traceability of changes made is kept. For example, audit trails provide records of change.

Maintenance of proper practices for error correction is monitored through the tiered data review process described in Section 5.9.3. Laboratory records are reviewed throughout the data review process. Individuals performing these reviews flag errors that are not properly corrected and bring these to the attention of the department manager or supervisor of the work area in which the record was generated so that the problem may be addressed and corrected with the individual(s) that did not make the correction properly.

4.14 Audits

The laboratory performs internal systems and technical audits to assess compliance to this manual and to other laboratory procedures, such as policy, SOP and SWI. Since the processes in this manual are based on the relevant quality system standards and regulatory and accreditation/certification program requirements the laboratory provides services for, the internal audits also assess on-going compliance to these programs.

The laboratory is also audited by external parties such as regulatory agencies, customers, consultants and non-government assessment bodies (NGAB).

Information from internal and external audits is used by laboratory management to address compliance concerns and opportunities where improvement will increase the reliability of data.

Deficiencies, observations and recommendations from audits are managed by local QA using the laboratory's formal CAPA process. See Section 4.11 for more information.

4.14.1 Internal Audit

The laboratory's internal audit program is managed by local QA in accordance with a pre-determined audit schedule established at the beginning of each calendar year. The schedule is prepared to assure that all areas of the laboratory are reviewed over the course of the year. Conformance to the schedule is reported to both laboratory management and corporate quality personnel in a monthly QA report prepared by the quality manager.

Although the QA Manager creates the audit schedule, it is the shared responsibility of local QA and laboratory managers to assure the schedule is maintained. Laboratory supervisors cooperate with QA to provide the auditors with complete access to the work area, personnel, and records needed.

Internal audits are performed by personnel approved by the quality manager. In general, personnel may not audit their own activities unless it can be demonstrated that an effective and objective audit will be carried out. The auditor must be trained, qualified, and familiar enough with the objectives, principles, and procedures of laboratory operations to be able to perform a thorough and effective evaluation.

The laboratory's internal audit program includes:

- System Audits & Method Audits: The purpose of these audits is to determine if daily practice is consistent with laboratory's SOPs and if SOPs are compliant with adjunct policy and procedures. Auditing techniques include analyst interviews and observation and records review. These audits are performed per the pre-determined schedule.

- Raw Data / Final Test Report Audits: The purpose of these audits is to review raw data and/or final test reports to verify the final product is consistent with customer/project requirements and supported as compliant to SOPs, reference methods, with test results that are properly qualified when necessary, accurate, and of known and documented quality. The reviews should also identify opportunities for improvement and best practices.

- Special Audits: Special audits are those performed ad hoc to follow up on a specific issue such as a client complaint, negative feedback, concerns of data integrity or ethics, or a problem identified through other audits. Special audits may be scheduled or unscheduled. Unscheduled internal audits are conducted whenever doubts are cast on the laboratory's compliance with regulatory requirements or its own policies and procedures. These unscheduled internal audits may be conducted at any time and may be performed without an announcement to laboratory personnel.

When observations and findings from any audit (internal or external) cast doubt on the validity of the laboratory's testing results, the laboratory takes immediate action to investigate the problem and take corrective action. (Also see 4.11 and 4.16)

The laboratory's internal audit program and auditing procedures are further described in laboratory SOP Audit Procedure.

4.14.1.1 Corporate Compliance Audit

The laboratory may also be audited by corporate quality personnel to assess the laboratory's compliance to the company's quality management program and to evaluate the effectiveness of implementation of the policies and procedures that make up the quality management system. The purpose of the compliance audit is to identify risks and opportunities and to assist laboratory management achieve the goals and objectives of the company's quality program.

4.15 Management Review

The laboratory's management team formally reviews the management system on an annual basis to assess for on-going suitability and effectiveness and to establish goals, objectives, and action plans for the upcoming year.

At a minimum, the following topics are reviewed and discussed:

- The on-going suitability of policies and procedures including HSE (Health, Safety and Environment) and waste management;
- Reports from managerial and supervisory personnel including topics discussed at regular management meetings held throughout the year;
- The outcome of recent internal audits;
- Corrective and preventive actions;
- Assessments by external bodies;
- The results of interlaboratory comparisons or proficiency tests;
- Changes in the volume and type of the work;

- Customer and personnel feedback, including complaints;
- Effectiveness of improvements / preventive actions made since last review;
- Internal and external issues of relevance and risk identification;
- A review of the status of actions from prior management reviews; and
- Other relevant factors, such as quality control activities, resources, and staff training.

The discussion and results of this review are documented in a formal report prepared by laboratory management. This report includes a determination of the effectiveness of the management system and its processes, goals and objectives for improvements in the coming year with timelines and responsibilities, and any other need for change. See laboratory SOP Management Review Procedure for more information.

Goals and action items from annual management systems review are shared with employees to highlight focus areas for improvement in addition to areas in which the laboratory has excelled.

4.16 Data Integrity

The laboratory's procedures for data integrity reviews are described in SOP ENV-SOP-CORQ-0010 *Data Recall*.

Customers whose data are affected by these events are notified in a timely manner, usually within 30 days of discovery. Some accreditation programs also require notification to the accreditation body (AB) within a certain time-frame from date of discovery when the underlying cause of the issue impacts accreditation. The laboratory follows any program or project specific client notification requirements for notification, when applicable.

5.0 TECHNICAL REQUIREMENTS

5.1 General

Many factors contribute to the correctness and reliability of the technical work performed by the laboratory. These factors fall under these general categories:

- Human Performance
- Facility and Environmental Conditions
- Test Method Performance and Validation
- Measurement Traceability
- Handling of Samples

The impact of each of these factors varies based on the type of work performed. To minimize negative effects from each these factors, the laboratory takes into account the contribution from each of these categories when developing test method and process (administrative) SOPs, evaluating personnel qualifications and competence, and in the selection of equipment and supplies used.

5.2 Personnel

5.2.1 Personnel Qualifications

The laboratory's program for personnel management is structured to ensure personnel are selected, qualified, and competent to perform the roles and responsibilities of their position based on education, experience, and training.

Qualifications, duties, responsibilities, and authorities of each position are specified in job descriptions maintained by corporate HR (See Section 5.2.4). These job descriptions provide the general basis for the selection of personnel for hire and are used by the laboratory to communicate to personnel the duties, responsibilities, and authorities of their position.

The term "personnel" refers to individuals employed by the laboratory directly as full-time, part-time, or temporary, and individuals employed by the laboratory by contract, such as through an employment agency. The term "personnel" is used interchangeably with the term "employee" throughout this manual. For purposes of this manual, these terms are equivalent.

The personnel management program is structured to establish and maintain records for each of the following:

- Selection of personnel;
- Training of personnel;
- Supervision of personnel;
- Authorization of personnel; and
- Monitoring Competence of personnel.

5.2.1.1 Competence

Competence is the ability to apply a skill or series of skills to complete a task or series of tasks correctly within defined expectations.

Competence for technical personnel authorized by PAS to provide opinion and interpretation of data to customers also includes the demonstrated ability to:

- Apply knowledge, experience, and skills needed to safely and properly use equipment, instrumentation, and materials required to carry out testing and other work activities in accordance with manufacturer specifications and laboratory SOPs;
- Understand and apply knowledge of general regulatory requirements necessary to achieve regulatory compliance in work product; and
- Understand the significance of departures and deviations from procedure that may occur during the analytical testing process and the capability and initiative to troubleshoot and correct the problem, document the situation and decision making process, and to properly qualify the data and analytical results.

The laboratory's requirements for the competence of personnel (education, qualification, work experience, technical skills, and responsibilities) are specified in job descriptions created by management and kept by human resources (HR). The job description provides the basis for the selection of personnel for each position.

An employee is considered competent when he/she has completed required training.

The policies and standard operating procedures (SOPs) for the following topics are established by management as minimum required training for all personnel:

- Ethics and Data Integrity
- Quality Manual
- Safety Manual
- Quality Management System
- Technical Process and Procedure relevant to their job tasks
- Successful Demonstration of Capability (DOC) – Analytical Personnel Only

Personnel are initially authorized competent to independently carry out their assigned duties when required training is complete and documented.

Records of training and qualification provide the record of competence for the individual. Qualification records may include but are not limited to diploma, transcripts, and curriculum vitae (CV).

The on-going competence of each employee is monitored by laboratory management through on-the-job performance. Analytical employees are also required to successfully complete another demonstration of capability for each test method performed on an annual basis.

5.2.2 Training

Training requirements are outlined in policies COR-POL-0023 *Mandatory Training Policy*. COR-POL-0004 *Ethics Policy*, and laboratory SOP *Training Procedure*. Additional training requirements may also be specified in other documents, such as manuals.

5.2.2.1 Training Program and Goals

The laboratory's training program includes 4 elements:

- Identification of Training Needs
- Training Plan Development and Execution
- Documentation and Tracking
- Evaluation of Training Effectiveness

Laboratory management establishes goals and training needs for individual employees based on their role, education, experience, and on-the-job performance.

Training needs for all employees are based on business performance measures that include but are not limited to:

- Quality Control Trends
- Process Error / Rework Trends

- Proficiency Testing Results
- Internal & External Audit Performance
- Management Review Goals

Training is delivered using various methods that incorporate techniques that appeal to the main learning styles: visual, aural, linguistic, and kinesthetic. Techniques include, on-the-job, instructor-led, self-study, eLearning, and blended.

The employee's direct supervisor is responsible for oversight of the employee's training plan and for providing adequate time to the employee to complete training assignments. Both the supervisor and employee are responsible to make sure the employee's training status and training records are current and complete.

The laboratory's QA department monitors the training status of personnel and provides the status to the General Manager (GM or AGM) at least monthly or more frequently, if necessary. The status report is used by laboratory management to identify overdue training assignments, the reasons for the gaps, and to make arrangements for completion.

The following subsections highlight specific training requirements:

5.2.2.1.1 New Hire Training

New hire training requirements apply to new personnel and to existing employees starting in a new position or different work area.

Required new hire training includes each of the following:

- Ethics and Data Integrity (See 5.2.2.1.3)
- Quality Manual / Quality Management System (See 5.2.2.1.4)
- Safety Manual and any training requirements specified in the manual.
- Policies & SOPs relevant to their job tasks
- Technical personnel that test samples must also successfully complete an initial demonstration of capability (IDOC) for the test methods performed before independently testing customer samples. (See 5.2.2.1.5). Independent testing means handling of client samples without direct supervision of the work activity by the supervisor or a qualified trainer.

All required training must be current and complete before the employee is authorized to work independently. Until then, the employee's direct supervisor is responsible for review and acceptance of the employee's work product.

5.2.2.1.2 On-Going Training

Personnel receive on-going training in each of the following topics:

- Ethics and Data Integrity (See 5.2.2.1.3)
- Quality Manual / Quality Management System (See 5.2.2.1.4)
- Safety Training
- Changes to Policies & SOPs
- Specialized Training
- Technical employees that carry out testing must also successfully complete on-going demonstration of capability (ODOC) for all test methods performed on an annual basis. (See 5.2.2.1.5)

Personnel are expected to maintain their training status and records of training current and complete and to complete training assignments in a timely manner.

5.2.2.1.3 Ethics and Data Integrity Training

Data integrity training is provided to all new personnel and refresher data integrity training is provided to all employees on an annual basis. Personnel are required to acknowledge they understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, debarment, or civil/criminal prosecution.

The initial data integrity training and the annual refresher training is documented with a signature attendance sheet or other form of documentation to provide evidence that the employee has participated in training on this topic and understand their obligations related to data integrity.

The following topics and activities are covered:

- Policy for honesty and full disclosure in all analytical reporting;
- Prohibited Practices;
- How and when to report data integrity issues;
- Record keeping. The training emphasizes the importance of proper written documentation on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially nonconforming;
- Training Program, including discussion regarding all data integrity procedures;
- Data integrity training documentation;
- In-depth procedures for data monitoring; 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.

All PAS personnel, including contract and temporary, are required to sign an "Attestation of Ethics and Confidentiality" at the time of employment and during annual refresher training. This document clearly identifies inappropriate and questionable behavior. Violations of this document result in serious consequences, including prosecution and termination, if necessary.

Also see SOP-ENV-COR-POL-0004 *Ethics Policy* for more information.

5.2.2.1.4 Management System Documents Training

PAS Manuals, policies, and SOPs are the primary documents used by regulatory bodies and PAS customers to verify the laboratory's capability, competency. and compliance with their requirements and expectations.

In addition to on-the-job training, employees must have a signed Read and Acknowledgement Statement on record for the laboratory quality manual, and the policies and SOPs relating to his/her job responsibilities. This statement when signed by the employee electronically or by wet signature, confirms that the employee has received, read, and understands the content of the document, that the employee agrees to follow the document when carrying out their work tasks; and the employee understands that unauthorized change to procedures in an SOP is not allowed except in accordance with the SOP departure policy (See 4.9.9.1) and SOP ENV-CORQ-0016 *Standard Operating Procedures and Standard Work Instructions* for more information.

5.2.2.1.5 Demonstration of Capability (DOC)

Technical employees must also complete an initial demonstration of capability (IDOC) prior to independent work on client samples analyzed by the test methods they perform. After successful IDOC, the employee must demonstrate continued proficiency (CDOC) for the test method on an annual basis. If more than a year has passed since the employee last performed the method; then capability must be re-established with an IDOC.

Demonstration of capability (IDOC and DOC) is based on the employee's capability to achieve acceptable precision and accuracy for each analyte reported by the laboratory for the test method using the laboratory's test method SOP.

Records of IDOC and ODOC are kept in the employee's training file.

For more information, see laboratory SOP *Training Procedure*.

5.2.2.2 Effectiveness of Training

The results of the performance measures used to identify training needs are the same measures used by the laboratory to measure effectiveness of the training program. Improvement in key performance measures suggests the training program is successful. (See 5.2.2.1)

Effectiveness of individual employee training is measured by their demonstrated ability to comprehend the training material and apply knowledge and skills gained to their job task. Measurements include but are not limited to:

- Testing of the employee's knowledge of the quality management system, policies, and technical and administrative procedures through various mechanisms, such as quizzes, observation, and interviews.
- Demonstrated ability to convey information correctly and factually in written and verbal communication to internal and external parties.
- Demonstrated ability to carry out tasks in accordance with SOPs and other work instructions.
- Demonstrated ability to make sound decisions based on guidance and information available.
- Demonstrated initiative to seek help or guidance when the employee is unsure of how to proceed.

5.2.3 Personnel Supervision

Every employee is assigned a direct supervisor, however named, who is responsible for their supervision. Supervision is the set of activities carried out by the supervisor to oversee the progress and productivity of the employees that report to them.

General supervisory responsibilities may include but are not limited to:

- Hiring Employees
- Training Employees
- Performance Management
- Development, oversight, and execution of personnel training plans
- Monitoring personnel work product to assure the work is carried out in accordance with this quality manual, policies, SOPs, and other documents that support the quality management system.

5.2.4 Job Descriptions

Job Descriptions that define the required education, qualifications, experience, skills, roles and responsibilities, and reporting relationships for each PAS position are established by top management and kept by corporate HR. PAS laboratories use these job descriptions as the source of positions and job titles for the laboratory. The job descriptions apply to employees who are directly employed by PAS, part-time, temporary, technical and administrative and by those that are under contract with PAS through other means.

The job descriptions include the education, expertise, and experience required for the position and the responsibilities and duties, including any supervisory or managerial duties assigned to the position.

5.2.5 Authorization of Technical Personnel

Laboratory management authorizes technical personnel to perform the technical aspects of their position after it has been verified that the employee meets the qualifications for the position, has successfully completed required training, and has demonstrated capability. After initial authorization, technical personnel are expected to maintain a current and complete training record, demonstrate on-going capability at least annually for each test method performed, and produce reliable results through accurate analysis of certified reference materials, proficiency testing samples, and/or routine quality control samples in order to remain authorized to continue to perform their duties.

Records to support authorization including, education, experience, training, and other evaluations are kept by the laboratory.

5.3 Accommodations and Facilities

5.3.1 Facilities

The laboratory is designed to support the correct performance of procedures and to not adversely affect measurement integrity or safety. Access to the laboratory is controlled by various measures, such as card access, locked doors, main entry. Visitors to the laboratory are required to sign-in and to be escorted by laboratory personnel during their visit. A visitor is any person that is not an employee of the laboratory.

5.3.2 Environmental Conditions

The laboratory is equipped with energy sources, lighting, heating, and ventilation necessary to facilitate proper performance of calibrations and tests. The laboratory ensures that housekeeping, electromagnetic interference, humidity, line voltage, temperature, sound and vibration levels are appropriately controlled to ensure the integrity of specific measurement results and to prevent adverse effects on accuracy or increases in the uncertainty of each measurement.

Environmental conditions are monitored, controlled, and recorded as required by the relevant specifications, methods, and procedures. Laboratory operations are stopped if it is discovered that the laboratory's environmental conditions jeopardize the analytical results.

5.3.3 Separation of Incompatible Activities

The layout and infrastructure of each work area including air handling systems, power supplies, and gas supplies of each laboratory work area is specifically designed for the type of analytical activity performed. Effective separation between incompatible work activities is maintained. For example, sample storage, preparation, and chemical handling for volatile organic analysis (VOA) is kept separate from semi-volatile organic (SVOA).

The laboratory separates samples known or suspected to contain high concentration of analytes from other samples to avoid the possibility for cross-contamination. If contamination is found, the source of contamination is investigated and resolved in accordance with laboratory SOPs.

5.3.4 Laboratory Security

Security is maintained by controlled access to the building and by surveillance of work areas by authorized personnel. Access is controlled to each area depending on the required personnel, the sensitivity of the operations performed, and possible safety concerns. The main entrance is kept unlocked during normal business hours for visitors, and is continuously monitored by laboratory staff. All visitors must sign a visitor's log, and a staff member must accompany them during the duration of their stay.

5.3.5 Good Housekeeping

The laboratory ensures good housekeeping practices in work areas to maintain a standard of cleanliness necessary for analytical integrity and personnel health and safety. Minimally, these measures include regular cleaning of the work area. Where necessary, areas are periodically monitored to detect and resolve specific contamination and/or possible safety issues.

5.4 Test Methods

5.4.1 General Requirements

The laboratory uses test methods and procedures that are appropriate for the scope of analytical services the laboratory offers.

Instructions on the use and operation of equipment and sample handling, preparation, and analysis of samples are provided in SOPs. The instructions in SOPs may be supplemented with other documents including but not limited to, standard work instructions (SWI), manuals, guides, project documents and reference documents.

These documents are managed using the procedures described in SOP ENV-SOP-CORQ-0015 *Document Management and Control* and SOP ENV-SOP-CORQ-0016 *Standard Operating Procedures and Standard Work Instructions.*

Deviations to test methods and SOPs are allowed under certain circumstances. See sections 4.9.1.1 and 4.9.1.2 for more information.

5.4.2 Method Selection

The test methods and protocols used by the laboratory are selected to meet the needs of the customer, are appropriate for the item tested and intended use of the data, and to conform with regulatory requirements when regulatory requirements apply.

In general, the test methods offered are industry accepted methods published by international, regional, or national standards. The laboratory bases its procedure on the latest approved edition of a method unless it is not appropriate or possible to do so or unless regulatory requirements specify otherwise.

The laboratory confirms that it can perform the test method and achieve desired outcome before analyzing samples (see section 5.4.5). If there is a change in the published analytical method, then the confirmation is repeated.

When a customer does not specify the test method(s) to be used, the laboratory may suggest test methods that are appropriate for the intended use of the data and the type of samples to be tested. The laboratory will also inform customers when test methods requested are

considered inappropriate for their purpose and/or out of date. This discourse takes place during review of analytical service requests (See Section 4.4).

5.4.3 Laboratory Developed Methods

A laboratory developed method is a method developed from scratch (no published source method), a procedure that modifies the chemistry from the source method, or a procedure that exceeds the scope and application of the source method.

Laboratory developed methods must be validated prior to use (see section 5.4.5) and the procedure documented in a test method SOP.

The requirements for non-standard methods (Section 5.4.4) also apply to laboratory developed methods.

5.4.4 Non-Standard Methods

A non-standard method is a method that is not published or approved for use by conventional industry standards for the intended purpose of the data. Non-standard methods must be validated prior to use (see section 5.4.5) and the procedure developed and documented in a test method SOP.

At a minimum, the following information must be included in the procedure:

- Title / Identification of Method;
- Scope and Application;
- Description of the type of item to be analyzed;
- Parameters or quantities and ranges to be determined;
- Apparatus and equipment, including technical performance requirements;
- Reference standards and reference materials required;
- Environmental conditions required and any stabilization period needed; and
- Description of the procedure, including:
    - Affixing identification marks, handling, transporting, storing and preparing of items;
    - Checks to be made before the work is started;
    - Verifying equipment function and, where required, calibrating and/or adjusting the equipment before each use;
    - Method of recording the observations and results;
    - Any safety measures to be observed;
    - Criteria and/or requirements for approval/rejection;
    - Data to be recorded and method of analysis and presentation; and
    - Uncertainty or procedure for estimating uncertainty.

Use of a non-standard method for testing must be agreed upon with the customer. The agreement, which is retained by the laboratory in the project record, must include the

specifications of the client's requirements, the purpose of testing, and their authorization for use of the non-standard method.

5.4.5 Method Validation

5.4.5.1 Validation Description

Validation is the process of conformation and the provision of objective evidence that the stated requirements for a specific method/procedure are fulfilled.

The laboratory's requirements and procedures for method validation are outlined in SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.*

5.4.5.2 Validation Summary

All test methods offered by the laboratory are validated before use to confirm the procedure works and the data and results achieved meet the goals for the method. The extent of validation performed is based on technology and other factors as defined in the validation SOP (ENV-SOP-CORQ-0011).

Results of validation are retained in accordance with the laboratory's SOP *Control of Records Procedure* for retention of technical records.

The need to repeat validation is assessed by laboratory management when there are changes to the test method.

5.4.5.3 Validation of Customer Need

Laboratory management reviews the results of test method validation, which include accuracy, precision, sensitivity, selectivity, linearity, repeatability, reproducibility, robustness, and cross-sensitivity, against general customer needs to ensure the laboratory's procedure for the test method will meet those needs.

The review procedure is detailed in SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.*

The following subsections highlight some of these concepts:

5.4.5.3.1 Accuracy

Accuracy is the degree to which the result of a measurement, calculation, or specification conforms to the correct value or a standard. When the result recovers within a range from the known value (control limit); the result generated using the laboratory's test method SOP is considered accurate.

5.4.5.3.2 Precision

Precision refers to the closeness of two or more measurements to each other. It is generally measured by calculating the relative percent difference (RPD) or relative standard deviation (RSD) from results of separate analysis of the same sample. Precision provides information about repeatability, reproducibility, and robustness of the laboratory's procedure.

5.4.5.3.3 Limits of Detection (LOD) (Chemistry)

The LOD is the minimum result which can be reliably discriminated from a blank with a predetermined confidence level. The LOD establishes the limit of method sensitivity and is also known as the detection limit (DL) or the method detection limit (MDL).

Values below the LOD cannot be reliably measured and are not reported by the laboratory unless otherwise specified by regulatory program or test method.

The LOD is established during method validation and after major changes to the analytical system or procedure that affect sensitivity are made.

The laboratory's procedure for LOD determination is detailed in laboratory SOP *Method Detection Limit Procedure.* The SOP complies with 40 CFR 136 Appendix B or the current industry approved and accepted guidance for this process.

5.4.5.3.4 Limits of Quantitation (LOQ) and Reporting Limit (RL)

The LOQ is the minimum level, concentration, or quantity of a target analyte that can be reported with a specified degree of confidence. The LOQ is established at the same time as the LOD. The laboratory's procedure for determination and verification of the LOQ is detailed in laboratory SOP *Method Detection Limit Procedure.*

The Lower Limit of Quantitation (LLOQ) is the value of the lowest calibration standard. The LOQ establishes the lower limit of quantitation.

The LOQ and LLOQ represent quantitative sensitivity of the test method.

- The LOQ must always be equal to or greater than the LLOQ and the LLOQ must always be greater than the LOD.
- Any reported value (detect or non-detect) less than the LLOQ is a qualitative value.

The RL is the value to which the presence of a target analyte is reported as detected or not-detected. The RL is project-defined based on project data quality objectives (DQO). In the absence of project specific requirements, the RL is usually set to the LOQ or the LLOQ. Depending on the relationship of the RL to the LLOQ or LOQ, both the RL value may be or quantitative.

For more information, refer to laboratory SOP *Sample Receiving Procedure.*

5.4.5.3.5 Linearity

Linearity is a mathematical concept applied to calibration models that employ multiple points to establish a calibration range used for quantitative analysis. Linearity is measured differently based on the calibration model. In general, if linearity is demonstrated then the slope of the response of standards are sufficiently close to one another. The accuracy of the linear regression and non-linear curves is verified by checking percent error or relative standard error (RSE), which is the process of refitting calibration data back to the model to determine if the results are accurate. For linear curves that use average calibration or response factor, error is measured by relative standard difference (RSD).

Linearity also establishes the range of quantitation for the test method used which directly impacts the sensitivity of the test method and uncertainty in measurement results. As previously noted, the LLOQ establishes the lower limit of quantitation. Similarly, the upper range of linearity establishes the upper limit of quantitation. In general, results outside of this range are considered qualitative values. However, some inorganic methods allow for extension of the linear range above the upper limit of quantitation when accuracy at this value is verified.

Linearity can also be used to establish repeatability, reproducibility, and robustness of the laboratory's test method. When linearity is demonstrated using a specific calibration model during method validation, then use of this same calibration model to achieve linearity on a day to day basis confirms the laboratory's method is repeatable, reproducible, and robust.

5.4.5.3.6 Demonstration of Capability (DOC)

The DOC performed during method validation confirms that the test method is of acceptable precision and accuracy. The procedure used for DOC for method validation is the same as described in section 5.2.2.1.5 for demonstration of analyst capability.

5.4.6 Measurement Uncertainty

The laboratory provides an estimate of uncertainty in testing measurements when required or on client request. In general, the uncertainty of the test method is reflected in the control limits used to evaluate QC performance. (See 5.9.1.1.10) ISO/IEC supports this concept with language that reads when a well-recognized test method specifies limits to the values of the major source of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory has satisfied the requirements on analytical uncertainty by following the test method and reporting instructions.

When measurement uncertainty cannot be satisfied through control limits, the laboratory will provide a reasonable estimation of uncertainty. A reasonable estimation is based on knowledge of method performance and previous experience. When estimating the analytical

uncertainty, all uncertainty components which are of importance in the given situation are taken into account.

5.4.7 Control of Data

The laboratory has policies and processes in place to assure that reported data is free from calculation and transcription errors, that quality control is reviewed and evaluated before data is reported, and to address manual calculation and integration.

5.4.7.1 Calculations, Data Transfer, Reduction and Review

Whenever possible, calculations, transfer of data, and data reduction are performed using validated software programs. (See 5.4.7.2)

If manual calculations are necessary, the results of these calculations are verified during the data review process outlined in section 5.9.3.

5.4.7.1.1 Manual Integration

The laboratory's policy and procedures for manual integration are provided in SOP ENV-SOP-CORQ-0006 *Manual Integration*.

This SOP includes the conditions under which manual integration is allowed and the requirements for documentation.

Required documentation of manual integration includes:

- complete audit trail to permit reconstruction of before and after results;
- identification of the analyst that performed the integration and the reason the integration was performed; and
- the individual(s) that reviewed the integration and verified the integration was done and documented in compliance with the SOP.

5.4.7.2 Use of Computers and Automated Acquisition

Whenever possible the laboratory uses software and automation for the acquisition, processing, recording, reporting, storage, and/or retrieval of data.

Software applications developed by PAS are validated by corporate IT for adequacy before release for general use. Commercial off the shelf software is considered sufficiently validated when the laboratory follows the manufacturer or vendor's manual for set-up and use. Records of validation are kept by the corporate information technology (IT) group or by the local laboratory, whichever group performed the validation.

The laboratory's process for the protection of data stored in electronic systems includes:

- Individual user names and passwords for Laboratory Information Management Systems (LIMS) and auxiliary systems used to store or process data.
- Employee Training in Computer Security Awareness

- Validation of spreadsheets used for calculations to verify formulas and logic yield correct results and protection of these cells to prevent unauthorized change.
- Operating system and file access safeguards
- Protection from Computer Viruses
- Regular system backup; and testing of retrieved data

The laboratory's process for software development and testing process includes:

- Verification the software application works as expected and is adequate for use and fulfills compliance requirements, such as the need to record date/time of data generation.
- Change control to assure requests for changes are reviewed and approved by management before the change is made.
- Communication channels to assure all staff are aware of changes made.
- Version Control and maintenance of historical records.

These procedures are detailed in laboratory SOPs *Control of Records Procedure*, *Data Handling Procedure*, *Report Generation Procedure*, and *IT Procedures*.

5.5 Equipment

5.5.1 Availability of Equipment

The laboratory is furnished with all equipment and instrumentation necessary to correctly perform the tests offered in compliance with the specifications of the test method and to achieve the accuracy and sensitivity required.

5.5.2 Calibration

Equipment and instrumentation is checked prior to use to verify it performs within tolerance for its intended application.

Laboratory management is made aware of the status of equipment and instrumentation and any needs for either on a daily basis. This information is obtained during laboratory walkthroughs (LDM) that are conducted as part of the laboratory's lean program.

5.5.2.1 Support Equipment

The laboratory confirms support equipment is in proper working order and meets the specifications for general laboratory use prior to placement in service and with intermediate checks thereafter. Equipment that does not meet specifications is removed from service until repaired or replaced. Records of repair and maintenance activities are maintained.

Procedures used to carry out and record these checks are outlined in laboratory SOP *Equipment Procedure*.

5.5.2.2 Analytical Instruments

Analytical instruments are checked prior to placement in service in accordance with SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.* After the initial service date, the calibration of instruments and verification calibration is performed in accordance with local test method SOPs.

The calibration procedures in the test method SOPs comply with the requirements for acceptable calibration practices outlined in corporate document ENV-SOT-CORQ-0026 *Acceptable Calibration Practices*, the reference methods, and any applicable regulatory or program requirements.

5.5.3 Equipment Use and Operation

Equipment is operated and maintained by laboratory personnel that are trained on the test method SOP. Up-to-date instructions and procedures for the use and maintenance of analytical equipment are included in SOPs and/or supplemental documents such as standard work instructions (SWI) or instrument manuals which are made readily accessible in the work area to all laboratory personnel.

5.5.4 Equipment Identification

The laboratory uniquely identifies equipment by serial number or any other unique ID system, when practical. The identifier is included in the equipment list maintained by QA.

5.5.5 Equipment Lists and Records

5.5.5.1 Equipment List

The laboratory maintains a master list of equipment that includes information about the equipment including a description, manufacturer, serial number, date placed in service, condition when received, identity, and the current location in the laboratory. The date of purchase is tracked by the procurement record. The equipment list(s) for each location covered by this manual is provided in Appendix F.

5.5.5.2 Equipment Records

In addition to the equipment list, the laboratory maintains records of equipment that include:

- Verification that equipment conforms with specifications.
- Calibration records including dates, results, acceptance criteria, and next calibration dates.
- Maintenance plan and records.
- Records of damage, malfunction, or repair.

The laboratory follows an equipment maintenance program designed to optimize performance and to prevent instrument failure which is described in laboratory SOP *Equipment Procedure* or individual test method SOPs.

The maintenance program includes routine maintenance activities which are performed as recommended by the manufacturer at the frequency recommended and non-routine maintenance, which is performed to resolve a specific problem

such as degradation of peak resolution, shift in calibration relationship, loss of sensitivity, or repeat failure of instrument performance checks and quality control samples.

Maintenance is performed by laboratory personnel or by outside service providers.

All maintenance activities performed by laboratory personnel are recorded by the individual(s) that performed the activity at the time the maintenance was performed in an instrument maintenance log.

The maintenance record minimally includes the date of maintenance, the initials of the person(s) performing maintenance, a description of the activity performed, why (when the maintenance is non-routine), and the return to analytical control. When maintenance is performed by an external vendor, the laboratory staples the service record into hardcopy maintenance logs or scans the record easy retrieval. The laboratory provides unrestricted access to instrument maintenance logs in order to promotes good instrument maintenance and recordkeeping practices.

If an instrument must be moved, the laboratory will use safe practices for handling and transport to minimize damage and contamination.

5.5.6 Out of Service Protocol

Equipment that has been subjected to overloading, mishandling, gives suspect results, has been shown to be defective, or is performing outside of specified limits is taken out of service and either removed from the work area or labeled to prevent accidental use until it has been repaired and verified to perform correctly.

When analytical equipment is taken out of service, the laboratory examines the potential effect it may have had on previous analytical results to identify any non-conforming work. (See section 4.9).

5.5.7 Calibration Status

The laboratory labels support equipment to indicate calibration status, whenever practicable or otherwise maintains the calibration status in a visible location in the work area. These procedures are described in laboratory SOP *Balance Calibration Procedure* and *Thermometers and Monitoring Procedure.*

The calibration status of analytical instruments is documented in the analytical record. Analysts verify on-going acceptability of calibration status prior to use and with instrument performance check standards. These procedures are described in test method SOPs.

5.5.8 Returned Equipment Checks

When equipment or an instrument is sent out of the laboratory for service, the laboratory ensures that the function and calibration status of the equipment is checked and shown to be satisfactory before the equipment is returned to service. These procedures are outlined in SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.*

5.5.9 Intermediate Equipment Checks

The laboratory performs intermediate checks on equipment to verify the on-going calibration status. For example, most test methods require some form of continuing calibration verification check and these procedures are included in the test method SOP.

Periodic checks of support equipment are also performed; see appendix E for more information.

5.5.10 Safeguarding Equipment Integrity

The laboratory safeguards equipment integrity using a variety of mechanisms that include but are not limited to:

- Adherence to manufacturer's specification for instrument use so that settings do not exceed manufacturer's recommendation or stress the performance of the equipment.
- Established maintenance programs.
- Transparent maintenance records and unrestricted access to maintenance logs.
- Validation and approval of software before use.
- Audits to confirm instrument settings are consistent with SOPs.
- On-the-job training for safe and proper use of laboratory equipment.

5.6 Measurement Traceability

5.6.1 General

Measurement traceability refers to a property of a measurement result whereby the result can be related to a reference through an unbroken chain of calibration, each contributing to the measurement uncertainty. Traceability requires an established calibration hierarchy of equipment (instruments) used during testing including equipment used for subsidiary measurements. The laboratory assures this equipment is calibrated prior to being put into service and that the reference standard and materials used for calibration are traceable to the international standard of units (SI) or national measurement standard.

When strict traceability to SI units cannot be made, the laboratory establishes traceability with the use of reference standards and equipment obtained from competent suppliers that provide calibration certificates and/or certificates of analysis (COA).

5.6.2 Equipment Correction Factors

When correction factors are used to adjust results, the laboratory will assure that results in computer software are also updated. For example, if the direct instrument or reading output must be corrected based on preparation factor or concentration factors, laboratory management will assure the corrected result is also updated in the software, whenever possible.

5.6.3 Specific Requirements

5.6.3.1 Requirements for Calibration Laboratories

The laboratory does not offer calibration services to customers.

5.6.3.2 Requirements for Testing Laboratories

The laboratory has procedures in place to verify equipment is calibrated prior to being put into service (See 5.5.2) and ensures the reference standard and materials used for calibration are traceable to the international standard of units (SI) or national measurement standard. When strict traceability to SI units cannot be made,

the laboratory establishes traceability with the use of reference standards and equipment obtained from competent suppliers that provide calibration certificates and/or certificates of analysis (COA).

5.6.4 Reference Standards and Reference Materials

5.6.4.1 Reference Standards

The laboratory uses reference standards of measurement to verify adequacy of working weights and thermometers. The working weight is the weight(s) used for daily balance calibration checks and the working thermometers are used for temperature measurements on a daily basis.

Intermediate checks of the working reference measurement standards are performed to verify adequacy between calibrations from an external calibration laboratory. The measurements from working weights and thermometers are compared to measurements taken by the reference standard which is traceable to SI or a national standard. The reference weights and thermometers are used solely for verification purposes unless the laboratory can prove that daily use does not adversely affect performance of the reference standard.

The laboratory performs intermediate checks of the working weights at least annually.

Working thermometers (glass and digital) are checked against the reference thermometer prior to placement in service to establish a correction factor and then rechecked annually (glass) or quarterly (digital) thereafter.

The calibration of liquid in glass reference thermometers is verified every 5 years and the calibration of digital reference thermometers is verified annually by an ISO/IEC 17025 accredited calibration laboratory or service provider that provides traceability to a national standard.

The calibration of the reference weight(s) is verified every 5 years by an ISO/IEC 17025 accredited calibration laboratory.

If criteria for the intermediate checks or recertification is not acceptable, the impact on previously reported results is evaluated using the process for evaluation of nonconforming work. (See 4.9)

See laboratory *Balance Calibration Procedure* and *Thermometers and Monitoring Procedure* for more information about this process.

5.6.4.2 Reference Materials

The laboratory purchases chemical reference materials used (also known as stock standards) from vendors that are accredited to ISO 17034 or Guide 34. Purchased reference materials must be received with a Certificate of Analysis (COA) where available. If a reference material cannot be purchased with a COA, it must be verified by analysis and comparison to a certified reference material and/or there must be a demonstration of capability for characterization. COA are reviewed for adequacy and retained by the laboratory for future reference.

The laboratory procedure for traceability and use of these materials is provided in laboratory SOP *NIST Traceability Procedure*.

This SOP includes each of the following requirements:

- Procedures for documentation of receipt and tracking. The record of entry includes name of the material, the lot number, receipt date, and expiration date.
- Storage conditions and requirements. Reference materials must be stored separately from samples, extracts, and digestates.
- Requirements to assure that preparations of intermediate or working solutions are recorded and assigned a unique identification number for tracking. Records of preparation include the lot number of the stock standard(s) used, the type and lot number of the solvent, the formulation, date, expiration date, and the preparer's initials. The lot number of the working standards is recorded in the analytical record to provide traceability to the standard preparation record. The preparation record provides traceability to the COA, which is traceable to SI or the national measurement standard.
- A requirement that the expiration dates of prepared standards may not exceed the expiration date of the parent standard. Standards, reference materials, and reagents are not used after their expiration dates unless their reliability is thoroughly documented and verified by the laboratory. If a standard exceeds its expiration date and is not re-certified, the laboratory removes the standard and/or clearly designates it as acceptable for qualitative/troubleshooting purposes only. All prepared standards, reference materials, and reagents are verified to meet the requirements of the test method through routine analyses of quality control samples.
- The second source materials used for verification of instrument calibration are obtained from a different manufacturer or different lot from the same manufacturer.
- Procedures to check reference materials for degradation and replacement of material if degradation or evaporation is suspected.
- Procedures for labeling. At a minimum the container must identify the material, the ID of the material and the expiration date. Original containers should also be labeled with date opened.

5.6.4.3 Intermediate Checks

Checks to confirm the calibration status of standards and materials are described in laboratory SOPs. These checks include use of second source standards and reference materials reserved only for the purpose of calibration checks.

5.6.4.4 Transport and Storage

The laboratory handles and transports reference standards and materials in a manner that protects the integrity of the materials. Reference standard and material integrity is protected by separation from incompatible materials and/or minimizing exposure to degrading environments or materials. Standards and reference materials are

stored separately from samples, extracts, and digestates. All standards are stored according to the manufacturer's recommended conditions. Temperatures colder than the manufacturer's recommendation are acceptable if it does not compromise the integrity of the material (e.g. remains in liquid state and does not freeze solid). In the event a standard is made from more than a single source with different storage conditions, the standard will be stored according to the conditions specified in the analytical method.

See the applicable analytical SOPs for specific reference material storage and transport protocols.

5.7 Sampling

Sampling refers to the field collection of samples and to subsamples taken by the laboratory for analysis from the field collected sample.

Subsampling procedures are included in each test method SOP where applicable and generic guidance is included in SOP *Sample Receiving Procedure* to assure the aliquot used for testing is representative of the field collected sample.

The requirements in the following subsections apply when field sampling is performed by the laboratory.

5.7.1 Sampling Plans and SOPs

When the laboratory performs field collection of samples, sampling is carried out in accordance with a written sample plan prepared by the customer or by the laboratory and by relevant sampling SOPs. These documents are made readily accessible at the sampling location. Sampling plans and SOPs are, whenever reasonable, based on appropriate governing methods and address the factors to be controlled to ensure the validity of the analytical results.

5.7.2 Customer Requested Deviations

When the customer requires deviations, additions, or exclusions from the documented laboratory sampling plan and/or procedure, the laboratory records the client's change request in detail with the sampling record, communicates the change to sampling personnel, and includes this information in the final test report.

5.7.3 Recordkeeping

The laboratory assures the sampling record includes the sampling procedure used, any deviations from the procedure, the date and time of sampling, the identification of the sampler, environmental conditions (if relevant), and the sampling location.

5.8 Sample Management & Handling

5.8.1 Procedures

The laboratory's procedures for sample management and handling are outlined in laboratory SOPs *Sample Kit Preparation*, *Sample Receiving Procedure*, *Purchasing/Subcontracting Procedure*, and *Sample Disposal Procedure.*

The procedures in these SOPs are established to maintain the safe handling and integrity of samples from transport, storage, to disposal and during all processing steps in-between; to maintain client confidentiality, and to protect the interests of PAS and its customers.

5.8.1.1 Chain of Custody

All samples received by the laboratory must be accompanied with a Chain of Custody (COC) record. The COC provides information about the samples collected and submitted for testing and documents the possession of samples from time of collection to receipt by the laboratory.

The COC record must minimally include the following information:

- Client name, address, and phone number
- Project Reference
- Client Sample Identification (Client ID)
- Date, Time, and Location of Sampling
- Samplers Name or Initials
- Matrix
- Type of container, and total number collected in each sample
- Preservatives
- Analyses Requested
- Mode of collection
- Any special instructions
- The date and time and signature of each sample transfer from time of collection to receipt in the laboratory. When the COC is transported inside the cooler, independent couriers do not sign the COC. Shipping manifests and/or air bills are the records of possession during transport.

A complete and legible COC is required. If the laboratory observes that the COC is incomplete or illegible, the client is contacted for resolution. The COC must be filled out in indelible ink. Personnel correct errors by drawing a single line through the initial entry so the entry is not obscured, entering the correct information, and initialing, and dating the change.

5.8.1.2 Legal Chain of Custody

Legal chain of custody is a chain of custody protocol used for evidentiary or legal purposes. The protocol is followed by the laboratory when requested by customer or where mandated by a regulatory program.

Legal chain of custody (COC) protocol establishes an intact, continuous record of the physical possession*, storage, and disposal of "samples" which includes sample aliquots, and sample extracts/digestates/distillates.

Legal COC records account for all time periods associated with the samples, and identifies all individuals who physically handled individual samples. Legal COC

begins at the point established by legal authority, which is usually at the time the sample containers are provided by the laboratory for sample collect or when sample collection begins.

*A sample is in someone's custody if:

- It is in one's physical possession;
- It is in one's view after being in one's physical possession;
- It has been in one's physical possession and then locked or sealed so that no one can tamper with it; and/or
- It is kept in a secure area, restricted to authorized personnel only.

Refer to laboratory SOP *Sample Receiving Procedure Appendix A* for more information.

5.8.2 Unique Identification

Each sample is assigned a unique identification number by the laboratory (Lab ID) after the sample has been checked and accepted by the laboratory in accordance with the laboratory's sample acceptance policy (See 5.8.3). the Lab ID is affixed to the sample container using a durable label.

The unique identification of samples also applies to subsamples, and prepared samples, such as extracts, digestates, etc.

The lab ID is linked to the field ID (client ID) in the laboratory's record. Both IDs are linked to the testing activities performed on the sample and the documentation records of the test.

Also see 5.8.4.

5.8.3 Sample Receipt Checks and Sample Acceptance Policy

The laboratory checks the condition and integrity of samples on receipt and compares the labels on the sample containers to the COC record. Any problem or discrepancy is recorded. If the problem impacts the suitability of the sample for analysis or if the documentation is incomplete, the client is notified for resolution. Decisions and instructions from the client are maintained in the project record.

5.8.3.1 Sample Receipt Checks

The following checks are performed:

- Verification that the COC is complete and legible.
- Verification that each sample's container label includes the client sample ID, the date and time of collection and the preservative in indelible ink.
- The container type and preservative is appropriate for each test requested.
- Adequate volume is received for each test requested.
- Visual inspection for damage or evidence of tampering.
- Visual inspection for presence of headspace in VOA vials. (VOA = volatile organic analysis).

- Thermal Preservation: For chemical testing methods for which thermal preservation is required, temperature on receipt is acceptable if the measurement is above freezing but <6°C. For samples that are hand-delivered to the laboratory immediately after sample collection, there must be evidence that the chilling process has begun, such as arrival on ice. The requirements for thermal preservation vary based on the scope of testing performed. For example, for microbiology, temperature on receipt is acceptable if the measurement is <10°C. Refer to the laboratory's SOP for sample receipt for more information.
- Chemical Preservation.
- Holding Time: Sample receiving personnel are trained to recognize tests where the holding time is 48 hours or less and to expedite the log-in of these samples. Except for tests with immediate holding times (15 minutes from time of collection or less), when samples are received out of hold, the laboratory will notify the client and request instruction. If the decision is made to proceed with analysis, the final test report will include notation of this instruction.

5.8.3.2 Sample Acceptance Policy

The laboratory maintains a sample acceptance policy in accordance with regulatory guidelines to clearly establish the circumstances in which sample receipt is accepted or rejected. When receipt does not meet acceptance criteria for any one of these conditions, the laboratory must document the noncompliance, contact the customer, and either reject the samples or fully document any decisions to proceed with testing. In accordance with regulatory specifications, test results associated with receipt conditions that do not meet criteria are qualified in the final test report.

All samples received must meet each of the following:

- Be listed on a complete and legible COC.
- Be received in properly labeled sample containers.
- Be received in appropriate containers that identify preservative.
- The COC must include the date and time of collection for each sample.
- The COC must include the test requested for each sample.
- Be in appropriate sample containers with clear documentation of the preservatives used.
- Be received within holding time. Any samples received beyond the holding time will not be processed without prior customer approval.
- Have sufficient sample volume to proceed with the analytical testing. If insufficient sample volume is received, analysis will not proceed without customer approval.
- Be received within appropriate temperature ranges (not frozen but ≤6°C) unless program requirements or customer contractual obligations mandate otherwise. The cooler temperature is recorded directly on the COC. Samples that are

delivered to the laboratory immediately after collection are considered acceptable if there is evidence that the chilling process has been started. For example, by the arrival of the samples on ice. If samples arrive that are not compliant with these temperature requirements, the customer will be notified. The analysis will NOT proceed unless otherwise directed by the customer. If less than 72 hours remain in the hold time for the analysis, the analysis may be started while the customer is contacted to avoid missing the hold time. Data associated with any deviations from the above sample acceptance policy requirements will be appropriately qualified.

5.8.4 Sample Control and Tracking

The samples are controlled and tracked using the Laboratory Information Management System (LIMS). The LIMS stores information about the samples and project. The process of entering information into the LIMS is called login and these procedures are described in laboratory SOP *Sample Receiving Procedure*. After log-in, a label is generated and affixed to each sample container. Information on this label, such as the lab ID, links the sample container to the information in LIMS.

At a minimum, the following information is entered during log-in:

- Client Name and Contact Information;
- The laboratory ID linked to the client ID;
- Date and time of sample collection;
- Date and time of sample receipt;
- Matrix; and
- Tests Requested.

5.8.5 Sample Storage, Handling, and Disposal

The laboratory procedures for sample storage, handling and disposal are detailed in laboratory SOP *Sample Receiving Procedure*, *Purchasing/Subcontracting Procedure*, and *Sample Disposal Procedure*.

5.8.5.1 Sample Storage

The samples are stored according to method and regulatory requirements as per test method SOPs. Samples are stored away from all standards, reagents, or other potential sources of contamination and stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

Refrigerated storage areas are maintained at ≤6°C (but not frozen) and freezer storage areas are maintained at <-10°C (unless otherwise required per method or program). The temperature of each storage area is checked and documented at least once for each day of use. If the temperature falls outside the acceptable limits, then corrective actions are taken and appropriately documented.

The laboratory is operated under controlled access protocols to ensure sample and data integrity. Visitors must register at the front desk and be properly escorted at all times. Samples are taken to the appropriate storage location immediately after sample receipt and login procedures are completed. All sample storage areas have limited access. Samples are removed from storage areas by designated personnel and returned to the storage areas as soon as possible after the required sample quantity has been taken.

5.8.5.2 Sample Retention and Disposal

The procedures used by the laboratory for sample retention and disposal are detailed in laboratory SOP *Sample Receiving Procedure* and *Sample Disposal Procedure*.

In general, unused sample volume and prepared samples such as extracts, digestates, distillates and leachates (samples) are retained by the laboratory for the period of time necessary to protect the interests of the laboratory and the customer.

Samples may be stored at ambient temperature when all analyses are complete, the hold time is expired, the report has been delivered, and/or when allowed by the customer or program. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the laboratory has sufficient capacity and their presence does not compromise the integrity of other samples.

After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposition of hazardous samples is to return the excess sample to the customer.

5.9 Assuring the Quality of Test Results

5.9.1 Quality Control (QC) Procedures

The laboratory monitors the validity and reliability of test results using quality control (QC) samples that are prepared and analyzed concurrently with field samples in the same manner as field samples. QC results are always associated to and reported with the field samples they were prepared and analyzed with from the same preparation or analytical batch. See the glossary for definition of preparation and analytical batch.

The results of QC performed during the testing process are used by the laboratory to assure the results of analysis are consistent, comparable, accurate, and/or precise within a specified limit. When the results are not within acceptance criteria or expectations for method performance, correction and corrective action(s) are taken. These actions may include retesting or reporting of data with qualification to alert the end user of the situation.

Other QC measures performed include the use of certified reference materials (see 5.6.2), participation in interlaboratory proficiency testing (see 5.9.1.2), verification that formulae used for reduction of data and calculation of results is accurate (see 5.9.3), on-going monitoring of environmental conditions that could impact test results (see 5.3.2), and evaluation and verification of method selectivity and sensitivity (see 5.4.5).

QC results are also used by the laboratory to monitor performance statistical trends over time and to establish acceptance criteria when no method or regulatory criteria exist (see 5.9.1.4).

5.9.1.1 Essential QC

Although the general principles of QC for the testing process apply to all testing, the QC protocol used for each test depends on the type of test performed.

QC protocol used by the laboratory to monitor the validity of the test are specified in test method SOPs. The SOP includes QC type, frequency, acceptance criteria, corrective actions, and procedures for reporting of nonconforming work.

These requirements in the SOP conform to the reference method and any applicable regulations or certification and accreditation program requirement for which results of the test are used. When a project requires more stringent QC protocol than specified in the SOP, project specification is followed. When the project requires less stringent QC protocol, the project specification may be followed as an authorized departure from the SOP when the project specifications meet the requirements in the mandated method and any regulatory compliance requirements for which the data will be used.

The following are examples of essential QC for Chemistry:

5.9.1.1.1 Second Source Standard (ICV/QCS)

The second source standard is a standard obtained from a different vendor than the vendor of the standards used for calibration. It is a positive control used to verify the accuracy of a new calibration relative to the purity of the standards used for calibration. This check is referred to in test method and quality system standards as the initial calibration verification (ICV) or quality control sample (QCS). The second source standard is analyzed immediately after the calibration and before analysis of any samples. When the ICV is not within acceptance criteria, a problem with the purity or preparation of the standards may be indicated.

5.9.1.1.2 Continuing Calibration Verification (CCV)

CCV is to determine if the analytical response has significantly changed since initial calibration. If the response of the CCV is within criteria, the calibration is considered valid. If not, there is a problem that requires further investigation. Actions taken are technology and method specific.

5.9.1.1.3 Method Blank (MB) / Other Blanks

A method blank is a negative control used to assess for contamination during the prep/analysis process. The MB consists of a clean matrix, similar to the associated samples that is known to be free of analytes of interest. The MB is processed with and carried through all preparation and analytical steps as the associated samples.

In general, contamination is suspected when the target analyte is detected in the MB above the reporting limit. Some programs may require evaluation of the MB to ½ the reporting limit or the

detection limit. When contamination is evident, the source is investigated and corrections are taken to reduce or eliminate it. Analytical results associated with MB that does not meet criteria are qualified in the final test report.

Other types of blanks that serve as negative controls in the process may include:

- Trip Blanks (VOA)
- Storage Blanks
- Equipment Blanks
- Field Blanks
- Calibration Blanks
- Cleanup Blanks
- Instrument Blanks

5.9.1.1.4 Laboratory Control Sample (LCS)

The LCS is positive control used to measure the accuracy of process in a blank matrix. The LCS is spiked by the laboratory with a known amount of analyte. The spike is a standard solution that is pre-made or prepared from a certified reference standard. The LCS is processed with and carried through all preparation and analytical steps as the associated samples.

When the percent recovery (%R) of the LCS is within the established control limit, sufficient accuracy has been achieved. If not, the source of the problem is investigated and corrected and the procedure may be repeated. Analytical results associated with LCS that does not meet criteria are qualified in the final test report.

5.9.1.1.5 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Matrix spikes measure the effect the sample matrix has on precision and accuracy of the determinative test method. The MS and MSD are replicates of a client sample that is spiked with known amount of target analyte.

Due to the heterogeneity of matrices even of the same general matrix type, matrix spike results mostly provide information on the effect of the matrix to the client whose sample was used and on samples of the same matrix from the same sampling site. Therefore, MS should be client-specific when the impact of matrix on accuracy and precision is a project data quality objective. When there is not a client-specified MS for any sample in the batch, the laboratory randomly selects a sample from the batch; the sample selected at random is called a "batch" matrix spike.

The MS/MSD results for percent recovery and relative percent difference are checked against control limits. Because the performance of matrix spikes is matrix-dependent, the result of the matrix spike is not used to determine the acceptability of the test.

5.9.1.1.6 Sample Duplicate (SD)

A sample duplicate is a second replicate of sample that is prepared and analyzed in the laboratory along another replicate. The SD is used to measure precision.

The relative percent difference between replicates are evaluated against the method or laboratory derived criteria for relative percent difference (RPD), when this criterion is applicable. If RPD is not met, associated test results are reported with qualification.

5.9.1.1.7 Surrogates

Surrogates are compounds that mimic the chemistry of target analytes but are not expected to occur naturally in real world samples. Surrogates are added to each sample and matrix QC samples (MS, MSD, SD) at known concentration to measure the impact of the matrix on the accuracy of method performance. Surrogates are also added to the positive and negative control samples (MB, LCS) to evaluate performance in a clean matrix, and included in the calibration standards and calibration check standards.

The percent recovery of surrogates is evaluated against method-specified limits or statistically derived in-house limits. Project-specific limits and/or program-specific limits are used when required. Results with surrogate recovery out of limits in samples are reported with qualification. Samples with surrogate failures can also be re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error.

5.9.1.1.8 Internal Standards

Internal Standards are compounds not expected to occur naturally in field samples. They are added to every standard and sample at a known concentration prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. The laboratory follows specific guidelines for the treatment of internal standard recoveries and further information can be found in the applicable laboratory SOP.

5.9.1.1.9 QC Acceptance Criteria and Control Limits

The QC acceptance criteria are specified in test method SOPs. The criteria in the SOP are based on the requirements in the published test method or regulatory program. When there are no established acceptance criteria, the laboratory develops acceptance criteria in accordance with recognized industry standards.

Some methods and programs require the laboratory to develop and use control limits for LCS, MS/MD and surrogate evaluation. In-laboratory developed limits are referred to as "in-house" control

limits. In-house control limits represent ± 3 Standard Deviations (99% confidence level) from the average recovery of at least 20 data points generated using the same preparation and analytical procedure in a similar matrix.

See laboratory SOP *Measurement Uncertainty Procedure* for more information.

5.9.1.2 Proficiency Testing (PT)

The laboratory participates in interlaboratory proficiency testing (PT) studies to measure performance of the test method and to identify or solve analytical problems. PT samples measure laboratory performance through the analysis of unknown samples provided by an external source.

The PT samples are obtained from accredited proficiency testing providers (PTP) and handled as field samples which means they are included in the laboratory's normal analytical processes and do not receive extraordinary attention due to their nature.

The laboratory does not share PT samples with other laboratories, does not communicate with other laboratories regarding current PT sample results during the duration of the study, and does not attempt to obtain the assigned value of any PT sample from the PT provider.

The laboratory initiates an investigation and corrective action plan whenever PT results are deemed unacceptable by the PT provider.

The frequency of PT participation is based on the certification and accreditation requirements held by the laboratory.

5.9.2 QC Corrective Action

When the results of QC are not within acceptance criteria or expectations for method performance, correction and corrective action(s) are taken per the specifications in the test method SOP. These actions may include retesting or reporting of data with qualification to alert the end user of the situation.

5.9.3 Data Review

The laboratory uses a tiered system for data review. The tiered process provides sequential checks to verify data transfer is complete; manual calculations, if performed, are correct, manual integrations are appropriate and documented, calibration and QC requirements are met, appropriate corrective action was taken when required, test results are properly qualified, process and test method SOPs were followed, project specific requirements were met, when applicable, and the test report is complete.

The sequential process includes three tiers referred to as primary review, secondary review, and administrative/completeness review.

Detailed procedures for the data review process are described in laboratory SOP *Report Generation Procedure*. The general expectations for the tiered review process are described in the following sections:

5.9.3.1 Primary Review

Primary review is performed by the individual that performed the task. All laboratory personnel are responsible for review of their work product to assure it is complete, accurate, documented, and consistent with policy and SOPs.

Checks performed during primary review include but are not limited to:

- Verification that data transfer and acquisition is complete
- Manual calculations, if performed, are documented and accurate
- Manual integrations, if performed, are documented and comply with SOP ENV-SOP-CORQ-006 *Manual Integration*
- Calibration and QC criteria were met, and/or proper correction and corrective actions were taken, and data and test results associated with QC and criteria exceptions are properly qualified
- Work is consistent with SOPs and any other relevant instructional document such as SWI, program requirements, or project QAPP

5.9.3.2 Secondary Review

Secondary review is performed by qualified peer or supervisor. Secondary review is essentially a repeat of the checks performed during primary review by another person. In addition to the checks of primary review, secondary review includes chromatography review to check the accuracy of quantitative analyte identification.

5.9.3.3 Completeness Review

Completeness review is an administrative review performed prior to release of the test report to the customer. Completeness review verifies that the final test report is complete and meets project specification. This review also assures that information necessary for the client's interpretation of results are explained in the case narrative or footnoted in the test report.

5.9.3.4 Data Audits

In addition to the 3 tier data review process, test reports may be audited by local QA to verify compliance with SOPs and to check for data integrity, technical accuracy, and regulatory compliance. These audits are not usually done prior to issuance of the test report to the customer. The reports chosen for the data audits are selected at random.

If any problems with the data or test results are found during the data audit, the impact of the nonconforming work is evaluated using the process described in Section 4.9.

Also see Section 4.14 for internal audits.

5.10 Reporting

5.10.1 General Requirements

The laboratory reports results of testing in a way that assures the results are clear, and unambiguous. All data and results are reviewed prior to reporting to assure the results reported are accurate and complete.

Test results are summarized in test reports that include all information necessary for the customer's interpretation of the test results. Additional information necessary to clarify the data or disclose nonconformance, exceptions, or deviations that occurred during the analytical process are also reported to the customer in the test report.

The specifications for test reports and electronic data deliverables (EDD) are established between the laboratory and the customer at the time the request for analytical services is initiated. The report specifications include the test report format, protocol for the reporting limit (RL), conventions for the reporting of results less than the limit of quantitation (LOQ), and specification for the use of project or program specific data qualifiers. Information about review of analytical service requests is provided in Section 4.4.

5.10.2 Test Reports: Required Items

Test Reports are prepared by the laboratory at the end of the testing process. The format of the report depends on the level of reporting requested by the customer. The laboratory offers a variety of standardized test report formats and also can provide custom test report formats, when necessary.

The level of detail required in the test report depends on the customer's needs for data verification, validation, and usability assessments that occur after the laboratory releases the test report to the customer. The test report formats offered by the laboratory provide gradient levels of detail to meet the unique needs of each customer. The laboratory project manager helps the customer select the test report format that best meets their needs. When a specific report format or protocol is required for a regulatory or program compliance, the laboratory project manager must ensure the test report selected meets those requirements.

Every test report issued by the laboratory includes each of the following items:

a) Title

b) Name and phone number of a point of contact from the laboratory issuing the report.

c) Name and address of the laboratory where testing was performed. When testing is done at multiple locations within network (IRWO), the report must clearly identify which network laboratory performed each test and must include the physical address of each laboratory.

d) Unique identification of the test report and an identifier on each page of the report to link each page to the test report and clear identification of the end of the report.

e) The name and address of the customer

f) Identification of test methods used

g) Cross reference between client sample identification number (Sample ID) and the laboratory's identification number for the sample (Lab ID) to provide unambiguous identification of samples.

h) The date of receipt of samples, condition of samples on receipt, and identification of any instance where receipt of the samples did not meet sample acceptance criteria.

i) Date and times of sample collection, receipt, preparation, and analysis.

j) Test results and units of measurement, and qualification of results associated with QC criteria exceptions, and identification of reported results outside of the calibration range.

k) Name, title, signature of the person(s) authorizing release of the test report and date of release.

l) A statement that the results in the test report relate only to the items tested.

m) Statement that the test report may not be reproduced except in full without written approval from the laboratory.

5.10.3 Test Reports: Supplemental Items

5.10.3.1 Supplemental Requirements

The following items are included in the test report when required or relevant:

a) Explanation of departure from test method SOPs including, what the departure was and why it was necessary.

b) Statistical methods used. (Required for Whole Effluent Toxicity)

c) For solid samples, specification that results are reported on a dry weight or wet weight basis.

d) Signed Affidavit, when required by client or regulatory agency.

e) A statement of compliance / non-compliance with requirements or specifications (client, program, or standard) that includes identification of test results that did not meet acceptance criteria.

f) When requested by the client, statement of estimated measurement uncertainty. In general, for environmental testing, estimated uncertainty of measurement is extrapolated from LCS control limits. Control limits incorporate the expected variation of the data derived from the laboratory's procedure. When the control limits are specified by the test method or regulatory program, the control limits represent the expected variation of the test method and/or matrices for which the test method was designed.

g) Opinions and Interpretations.

h) If a claim of accreditation/certification is included in the test report, identification of any test methods or analytes for which accreditation/certification is not held by the laboratory if the accrediting body offers accreditation/certification for the test method/analyte. The fields of accreditation/certification vary between agencies and it cannot be presumed that because accreditation/certification is not held that it is offered or required.

i) Certification Information, including certificate number and issuing body.

5.10.3.2 Test Reports: Sampling Information

The following items are included in the test report when samples are collected by the laboratory or when this information is necessary for the interpretation of test results:

a) Date of Sampling.

b) Unambiguous identification of material samples.

c) Location of sampling including diagrams, sketches, or photographs.

d) Reference to the sampling plan and procedures used.

e) Details of environmental conditions at time of sample that may impact test results.

f) Any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

5.10.4 Calibration Certificates

The laboratory does not perform calibration activities for its customers and calibration certificates are not offered or issued.

5.10.5 Opinions and Interpretations

The laboratory provides objective data and information to its customers of sufficient detail for their interpretation and decision making. Objective data and information is based solely on fact and does not attempt to explain the meaning (interpret) or offer a view or judgement (opinion). Sometimes the customer may request the laboratory provide opinion or interpretation to assist them with their decisions about the data.

When opinions and interpretations are included in the test report, the laboratory will document the basis upon which the opinions and interpretations have been made and clearly identify this content as opinion or interpretation in the test report.

Examples of opinion and interpretation include but are not limited to:

- The laboratory's viewpoint on how a nonconformance impacts the quality of the data or usability of results.
- The laboratory's judgment of fulfillment of contractual requirements.
- Recommendations for how the customer should use the test results and information.
- Suggestions or guidance to the customer for improvement.

When opinions or interpretations are verbally discussed with the customer, the content of these conversations is summarized by the laboratory and kept in the project record.

5.10.6 Subcontractor Reports

When analytical work has been subcontracted to an organization external to PAS, the test report from the subcontractor is included in its entirety as an amendment to the final test report.

Note: Test results for analytical work performed within the PAS network may be merged into a single test report. The test report issued clearly identifies the location and address of each network location that performed testing and which tests they performed. (See 5.10.2)

5.10.7 Electronic Transmission of Results

When test results and/or reports are submitted to the customer through electronic transmission, follow the procedures established in this manual for confidentiality and protection of data.

5.10.8 Format of Test Reports

The test formats offered by the laboratory are designed to accommodate each type of analytical test method carried out by the laboratory and to minimize the possibility of misunderstanding or misuse of analytical results. The format of electronic data deliverables (EDD) follow the specifications for the EDD.

5.10.9 Amendments to Test Reports

Test reports that are revised or amended by the laboratory after date of release of the final test report to the customer are issued as a new test report that is clearly identified as an amendment or revision and that includes a reference to the originally issued final test report.

The customer is the organization doing business with PAS external to PAS.

Changes made to test results and data before the final test report is issued to the customer are not amendments or revisions, these are corrections to errors found during the laboratory's data verification and review process.

The laboratory's procedure for report amendments and revision are outlined in laboratory SOP *Report Generation Procedure*.

6.0 REVISION HISTORY

This Version:

Section	Description of Change
All	This version is a complete rewrite of the document this version supersedes.

This document supersedes the following documents:

Document Number	Title	Version
	Quality Assurance Manual for Inter-Mountain Laboratories, Inc.	8.21

7.0 APPENDICES

7.1 Appendix A: Certification / Accreditation Listing

The certifications / accreditation lists provided in this manual represent those that were held by the named location on the effective date of this manual. This information is subject to change without notice and must not be considered valid proof of certification or accreditation status. Current certificates are maintained by Local QA and a copy of the certificate is posted to PAS's eDMS Portal for access by all PAS employees. External parties should contact the laboratory for the most current information.

7.1.1 PAS-Sheridan

Authority	Certificate Number	Authority	Certificate Number
Arizona Department of Health Services	AZ0774	Oklahoma Dept. of Environmental Quality	2019-089
USEPA Region 8	WY00005	South Dakota Dept. of Env. And Natural Resources	WY00005
Idaho Department of Health and Welfare	EPA ID: WY00005	Texas Commission on Env. Quality	T104704507-20-14
Montana Dept. of Public Health	CERT00001	Railroad Commission of Texas	File Ref No: 1423801
Nevada Dept. of Conservation and Natural Resources	WY000052020-1	USDA Animal and Plant Health Insp Serv	P330-19-00351
North Dakota State Dept. of Health	R-199	USNRC	49-29405-01 Amendment No. 3
North Dakota State Dept. of Health	R-199A	Utah Dept of Health	WY000052020-9

7.2 Appendix B: Capability Listing

The capabilities listed in this Appendix were held by the location referenced on the effective date of this manual. This information is subject to change without notice. External parties should contact the laboratory for the most current information.

Table Legend:

- Air = Whole gas or vapor
- DW = Drinking Water
- NPW = Non-Potable Water
- SCM = Solid and Chemical Materials
- Waste = Non-Aqueous Phase Liquid (NAPL), Oil
- Tissue = Biota and Tissue

7.2.1 PAS-Sheridan

Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Isotopic Uranium	ACW01			X	X		
Th 230 and Isotopic Th	ACW10	X		X	X		X
Hot Water Boron	ASA 9 25 9.1				X		
Ammonium	ASA 9 33-3.2				X		
$NaHCO_3$ Extraction for P	ASA 9 73-4.4				X		
Diffusive Releases	ASTM C1308-08				X		
Particle Size	ASTM D 422				X		
Specific Gravity	ASTM D1429-08			X			
Oxidation Reduction Potential	ASTM D 1498-08			X			
Sulfur Forms	ASTM D 2492-84				X		
As Hydride Generation	ASTM D 2972-88				X		
Se Hydride Generation	ASTM D 3859-93				X		
Suspended Sediment Concentration	ASTM D 3977-97 (2013)ε1			X			
Radon 222	ASTM D5072-09		X	X			
Humidity Cell Test	ASTM D 5744-13				X		
Aquatic Free CN	ASTM D 7237-06			X			
Total UV CN	ASTM D7511-09			X			
CN Mine Waste	ASTM D 7572-09				X		
Analysis of Metal Bearing Ores	ASTM E 1915-11				X		
Meteoric Water	ASTM E 2242-13				X		
TCLP	EPA 1311			X	X		

Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
SPLP	EPA 1312				X		
Metals Digest	EPA 200.2		X	X			
ICP Metals	EPA 200.7		X	X			
ICP-MS Metals	EPA 200.8		X	X			
Mercury Water	EPA 245.1		X	X			
Mercury Tissue	EPA 245.6						X
Bromide	EPA 300.0		X	X	X		
Chloride	EPA 300.0		X	X	X		
Sulfate	EPA 300.0	X	X	X	X		
Ortho-Phosphate	EPA 300.0		X	X	X		
Total Cyanide	EPA 335.4		X	X			
Ammonia as N	EPA 350.1 (discrete Analyzer)			X	X		
Total Kjeldahl Nitrogen	EPA 351.2			X			
Nitrate as N	EPA 300.0		X	X	X		
Nitrate as N	EPA 353.2		X	X			
Nitrite as N	EPA 300.0	X	X	X	X		
Nitrite as N	EPA 353.2		X	X	X		
Total Phenolics	EPA 420.4			X			
Radon Flux	EPA-520/5-85-029	X					
Paste pH	EPA 600/2-78-054 section 3.2.2				X		
Neutralization Potential	EPA 600/2-78-054 section 3.2.3				X		
Total Sulfur	EPA 600/2-78-054 section 3.2.4				X		
Extractable Sulfur	EPA 600/2-78-054 section 3.2.6				X		
Gamma Emitting Radionuclides	EPA 901.1 Modified				X		
Pb210	EPA 909.0 Modified				X		
Pb210	OTW01	X		X	X		
HEM/SGT-HEM	EPA 1664A			X			
Pb in Air Filters	EQL-0310-189 (IML 2009)	X					
Analysis of Ra 226 and Ra 228 by Gamma Spec	Georgia Tech Method		X	X			
Sulfide	HACH 8131			X			
Cr VI	HACH 10218			X			
DO/BOD	HACH 10360			X			

Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Meteoric Water	MWMP BMRR, NDEP				X		
Lead in Ambient Air	IO 3.1	X					
Lead in Ambient Air	IO 3.4	X					
Lead in Ambient Air	IO-3.5	X					
Prep of pH Specific Leachates	ISO/TS 21268-4				X		
Cyanide	OIA-1677 09		X	X			
Pb210	OTW01	X		X	X		X
Color	SM2120B		X	X			
Turbidity	SM2130B		X	X			
Odor	SM2150B		X				
Acidity	SM2310B			X			
Alkalinity as $CaCO_3$	SM2320B		X	X			
Hardness	SM2340B		X	X			
Electrical Conductivity	SM2510B		X	X			
Total Dissolved Solids	SM2540C		X	X			
Total Suspended Solids	SM2540D		X	X			
Volatile Solids	SM2540E			X			
Settleable Matter	SM2540F			X			
Temperatue	SM2550B			X			
Cr VI	SM3500-Cr B			X			
Chloride	SM4500-Cl^- B			X			
Free Chlorine	SM4500-Cl G (Hach 8167)		X	X			
Total Residual Chlorine	SM4500-Cl G (Hach 8021)		X	X			
WAD Cyanide	SM4500-CN I, E			X			
Fluoride	SM4500-F- C		X	X			
Fluoride	EPA 300.0		X	X	X		
pH	SM4500-H+ B		X	X			
Total Nitrogen	SM4500-N C			X			
Dissolved Oxygen	SM4500-O G			X			
BOD/CBOD	SM5210B Modified			X			
COD	SM5220D			X			
Total Organic Carbon	SM5310B		X	X			

Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Gross Alpha/Beta	SM7110B	X	X	X	X		
Radium 226	SM7500-Ra B	X	X	X	X		X
Heterotrophic Plate Count Pour Plate	SM9215 B		X	X			
Heterotrophic Palate Count SimPlate	SM9215 E		X	X			
Fecal Coliform Multiple Tube Fermentation	SM9221E			X	X		
Fecal Coliform Membrane Filtration	SM9222D			X			
E. coli	SM9222G			X			
Total coliform/E.coli	SM9223B Colilert P/A		X	X			
Total coliform/E.coli	SM9223B Colisure P/A		X	X			
Total coliform/E.coli	SM9223B Colilert MPN		X	X			
Metals Extraction	SW-846 3010A			X			
Metals Extraction	SW-846 3020A			X			
Metals Extraction (Solid)	SW-846 3050B				X	X	
ICP Metals	SW-846 6010C			X			
ICPMS Metals	SW-846 6020A			X			
Mercury	SW-846 7470A			X			
Mercury	SW-846 7471A/B				X		
Paint Filter	SW-846 9095B			X	X	X	
Cation Exchange Capacity	USDA 60 19				X		
Paste pH	USDA 60 21a				X		
Gypsum	USDA 60 22a				X		
Gypsum	USDA 60 22b				X		
Organic Matter	USDA 60 24				X		
Saturation Percentage	USDA 60 6(27a)				X		

7.3 Appendix C: Glossary

This glossary provides common terms and definitions used in the laboratory. **It is not intended to be a complete list of all terms and definitions used.** The definitions have been compiled mostly from the TNI Standard and DoD QSM. Although this information has been reproduced with care, errors cannot be entirely excluded. Definitions for the same term also vary between sources. When the meaning of a term used in a laboratory document is different from this glossary or when the glossary does not include the term, the term and definition is included or defined in context in the laboratory document.

Term	Definition
3P Program	PAS-The continuous improvement program used by PAS that focuses on Process, Productivity, and Performance.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. DoD- Refers to accreditation in accordance with the DoD ELAP.
Accreditation Body (AB)	TNI- The organization having responsibility and accountability for environmental laboratory accreditation and which grants accreditation under this program. DoD- Entities recognized in accordance with the DoD-ELAP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies.* The AB must be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.
Accuracy	TNI- 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.
Activity, Absolute	TNI- Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time. NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.
Activity, Areic	TNI- Quotient of the activity of a body of material and its associated area.
Activity, Massic	TNI- Quotient of the activity of a body of material and its mass; also called specific activity.
Activity, Volumic	TNI- Quotient of the activity of a body of material and its volume; also called activity concentration. NOTE: In this module [TNI Volume 1, Module 6], unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.
Activity Reference Date	TNI- The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated. NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
American Society for Testing and Materials (ASTM)	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems and services.
Analysis	DoD- A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI- 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	TNI- A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. DoD- 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 are analyzed together.

Analytical Method	DoD- A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Annual (or Annually)	Defined by PAS as every 12 months ± 30 days.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- An all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- 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	TNI- 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 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 or the time-frame specified by the regulatory program. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.
Batch, Radiation Measurements (RMB)	TNI- An RMB is composed of 1 to 20 environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameter, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last in an RMB is 14 calendar days.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- 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 and is sometimes used to adjust or correct routine analytical results (See Method Blank). DoD- Blank samples are negative control samples, which typically include field blank samples (e.g., trip blank, equipment (rinsate) blank, and temperature blank) and laboratory blank samples (e.g., method blank, reagent blank, instrument blank, calibration blank, and storage blank).
Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.
Calibration	TNI- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.

Calibration Range	DoD- 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.
Calibration Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of Custody Form (COC)	TNI- 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 type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100
Confirmation	TNI- 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. DoD- Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.
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.
Congener	A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a Calibration Verification Standard (CVS) in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.
Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Continuous Improvement Plan (CIP)	The delineation of tasks for a given laboratory department or committee to achieve the goals of that department.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)

Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Correction	DoD- Action taken to eliminate a detected non-conformity.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
Corrective and Preventive Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Critical Value	TNI- Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level). NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence (1 – α) that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.
Customer	DoD- Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Integrity	TNI- The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- 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 usable form.
Definitive Data	DoD- Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.
Demonstration of Capability (DOC)	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).
Department of Defense (DoD)	An executive branch department of the federal government of the United States charged with coordinating and supervising all agencies and functions of the government concerned directly with national security.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.
Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance	TNI- Laboratories that analyze drinking-water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25.c as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).
Deuterated Monitoring Compounds (DMCs)	DoD- SIM specific surrogates as specified for GC/MS SIM analysis.
Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in 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.
Documents	DoD- Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).

Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.
Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
Facility	A distinct location within the company that has unique certifications, personnel and waste disposal identifications.
False Negative	DoD- A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	DoD- A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and sPAS to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT)	TNI- Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the PTPEC.
Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard, ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.

Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
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, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Improper Actions	DoD- Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (e.g., DoD or DOE).
Incremental Sampling Method (ISM)	Soil preparation for large volume (1 kg or greater) samples.
In-Depth Data Monitoring	TNI- 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.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.
Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Injection Internal Standard Analyte	Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples and batch QC after extraction and prior to analysis.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.

Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.
Internal Standard	TNI and DoD- 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.
International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element 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.
Laboratory	A body that calibrates and/or tests.
Laboratory Control Sample (LCS)	TNI- (also known as laboratory fortified blank (LFB), 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 and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	DoD- The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.
Limit(s) of Detection (LOD)	TNI- The minimum result, which can be reliably discriminated from a blank with predetermined confidence level. DoD- The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
Linear Dynamic Range	DoD- Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	TNI- A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.
Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.

Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurement Performance Criteria (MPC)	DoD- Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.
Measurement Quality Objective (MQO)	TNI- The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. MQOs are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.
Measurement System	TNI- A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the sample preparation and test and the operator(s).
Measurement Uncertainty	DoD- An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.
Method Blank	TNI- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	TNI- One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
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.
Minimum Detectable Activity (MDA)	TNI- Estimate of the smallest true activity that ensures a specified high confidence, $1 - \beta$, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05. NOTE 1: The MDS is a measure of the detection capability of a measurement process and as such, it is an a priori concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold. NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.
MintMiner	Program used by PAS to review large amounts of chromatographic data to monitor for errors or data integrity issues.

Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
National Environmental Laboratory Accreditation Conference (NELAC)	See definition of The NELAC Institute (TNI).
National Institute of Occupational Safety and Health (NIOSH)	National institute charged with the provision of training, consultation and information in the area of occupational safety and health.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
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.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Operator Aid	DoD- A technical posting (such as poster, operating manual, or notepad) that assists workers in performing routine tasks. All operator aids must be controlled documents (i.e., a part of the laboratory management system).
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Physical Parameter	TNI- A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical and biological components.
Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
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.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI- 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	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	TNI- The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing (PT)	TNI- A means to evaluate 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 (PT Program)	TNI- The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Testing Provider (PT Provider)	TNI- A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT Program.

Proficiency Testing Provider Accreditor (PTPA)	TNI- An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.
Proficiency Testing Reporting Limit (PTRL)	TNI- A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.
Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Proficiency Testing (PT) Study	TNI- a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants; b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard [TNI] but that does not have a pre-determined opening date and closing date.
Proficiency Testing Study Closing Date	TNI- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider; b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.
Proficiency Testing Study Opening Date	TNI- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT Provider; b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	DoD- Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	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 Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.
Quality Control (QC)	TNI- 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 (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality Manual	TNI- 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	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.

Quality System Matrix	TNI and DoD- These matrix definitions shall be used for purposes of batch and quality control requirements and may be different from a field of accreditation matrix: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution and final volume) lies within the calibration range.
Quantitative Analysis	DoD- Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.
Reagent Blank (method reagent blank)	A sample consisting of reagent(s), without the target analyte 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.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Records	DoD- The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).
Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized 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	TNI- A published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a "standard method", that term is equivalent to "reference method"). When a laboratory is required to analyze by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is no regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.

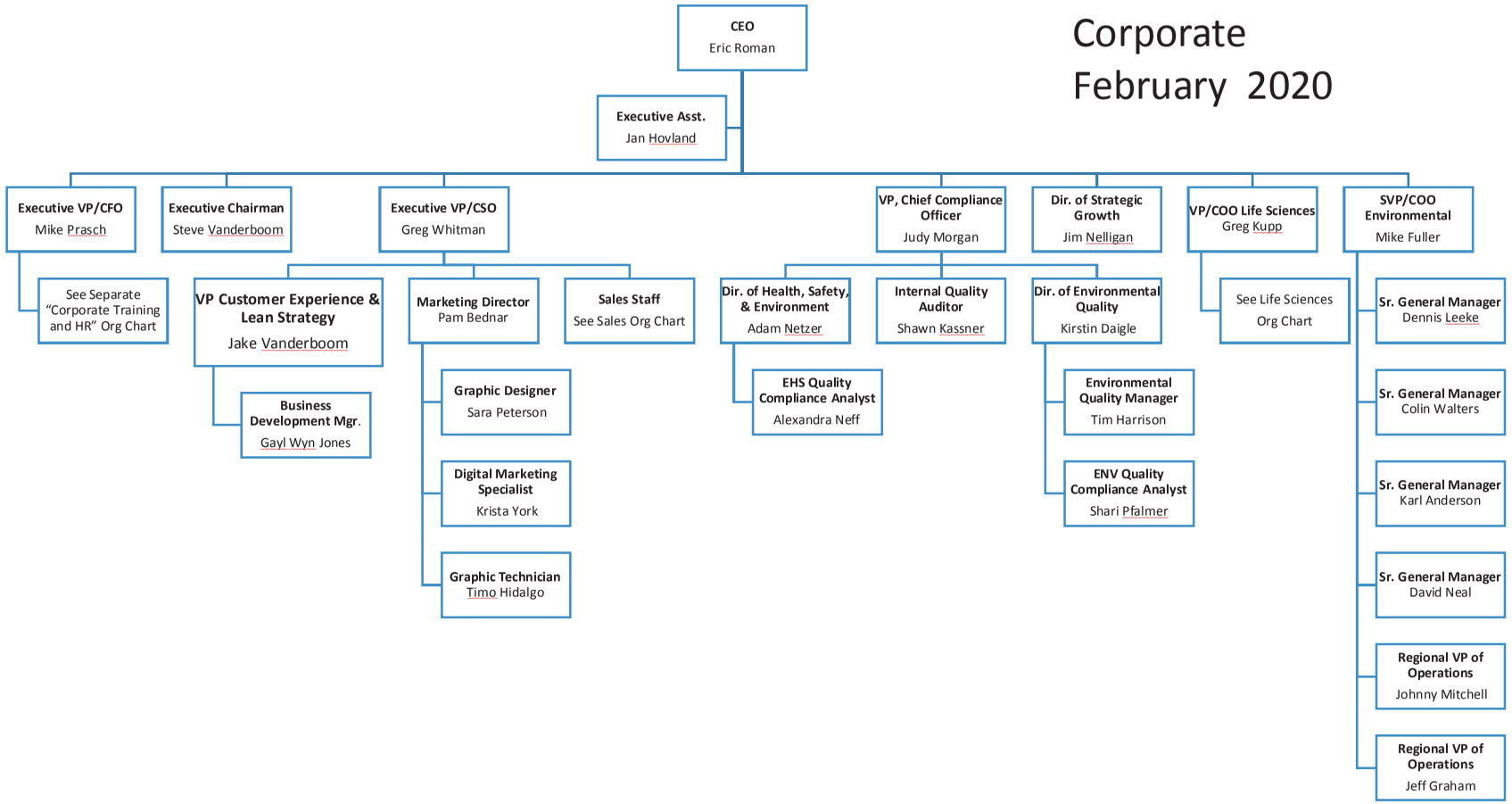
Reporting Limit Verification Standard (RLVS)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	Denotes a mandatory specification; often designated by the term "shall".
Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Revocation	TNI- The total or partial withdrawal of a laboratory's accreditation by an accreditation body.
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 Condition Upon Receipt Form (SCURF)	Form used by sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
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.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selected Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector. DoD- Using GC/MS, characteristic ions specific to target compounds are detected and used to quantify in applications where the normal full scan mass spectrometry results in excessive noise.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled.
Should	Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
Signal-to-Noise Ratio (S/N)	DoD- A measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.
Source Water	TNI- When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.
Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	A test method issued by an organization generally recognized as competent to do so.

Standard Operating Procedure (SOP)	TNI- 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.
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	DoD- A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
Supervisor	The individual(s) 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	DoD- 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.
Suspension	TNI- The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed 6 months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes or chemicals of primary concern identified by the customer on a project-specific basis.
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	DoD- A definitive procedure that determines one or more characteristics of a given substance or product.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.
Test Source	TNI- A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.
The NELAC Institute (TNI)	A non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. Previously known as NELAC (National Environmental Laboratory Accreditation Conference).
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- 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 conditions 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.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.

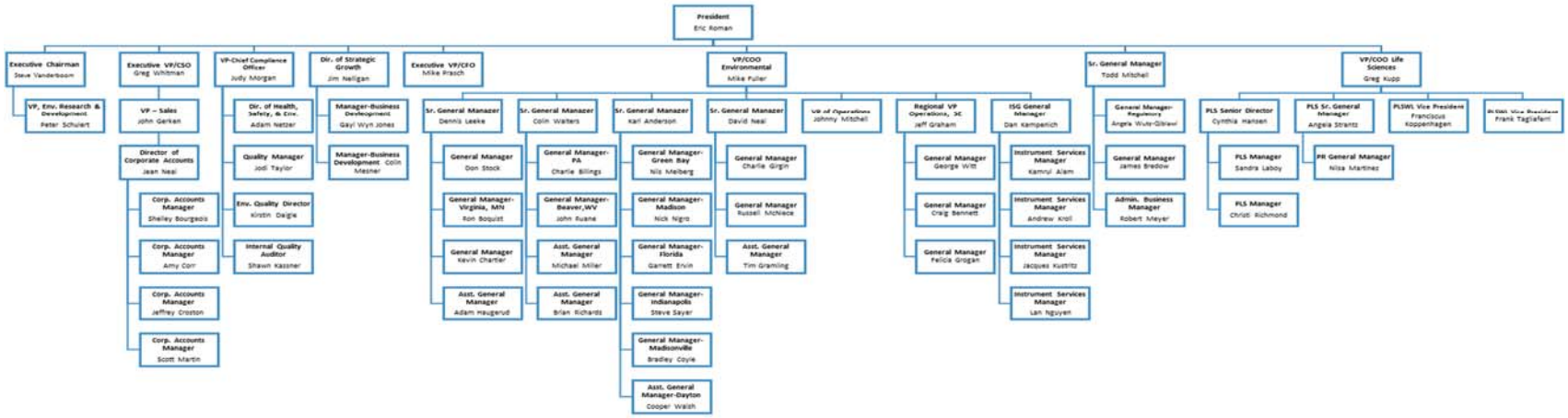
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty, Counting	TNI- The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts (MARLAP). Older references sometimes refer to this parameter as Error, Counting Error or Count Error (c.f., Total Uncertainty).
Uncertainty, Expanded	TNI- The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty). NOTE: Radiochemical results are generally reported in association with the Total Uncertainty. Either if these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or as an Expanded Uncertainty (k-sigma, where k > 1).
Uncertainty, Measurement	TNI- Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
Uncertainty, Standard	TNI- An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).
Uncertainty, Total	TNI- An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar items (c.f., Counting Uncertainty).
Unethical actions	DoD- Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.
United States Department of Agriculture (USDA)	A department of the federal government that provides leadership on food, agriculture, natural resources, rural development, nutrition and related issues based on public policy, the best available science, and effective management.
United States Geological Survey (USGS)	Program of the federal government that develops new methods and tools to supply timely, relevant, and useful information about the Earth and its processes.
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	TNI- 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.
Voluntary Action Program (VAP)	A program of the Ohio EPA that gives individuals a way to investigate possible environmental contamination, clean it up if necessary and receive a promise from the State of Ohio that no more cleanup is needed.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

7.4 Appendix D: Organization Chart(s)

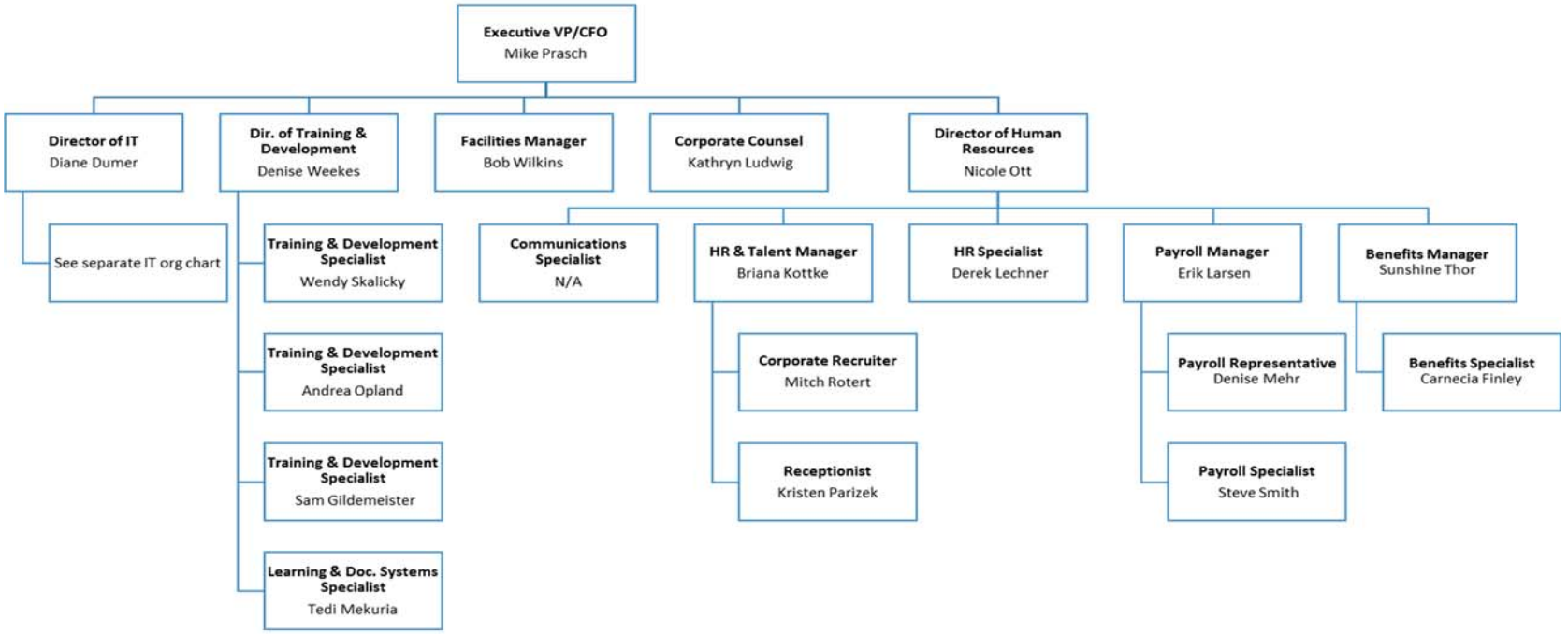
7.4.1 PAS - Corporate



Management Staff
December 2019



Corporate Staff
Training and HR
December 2019



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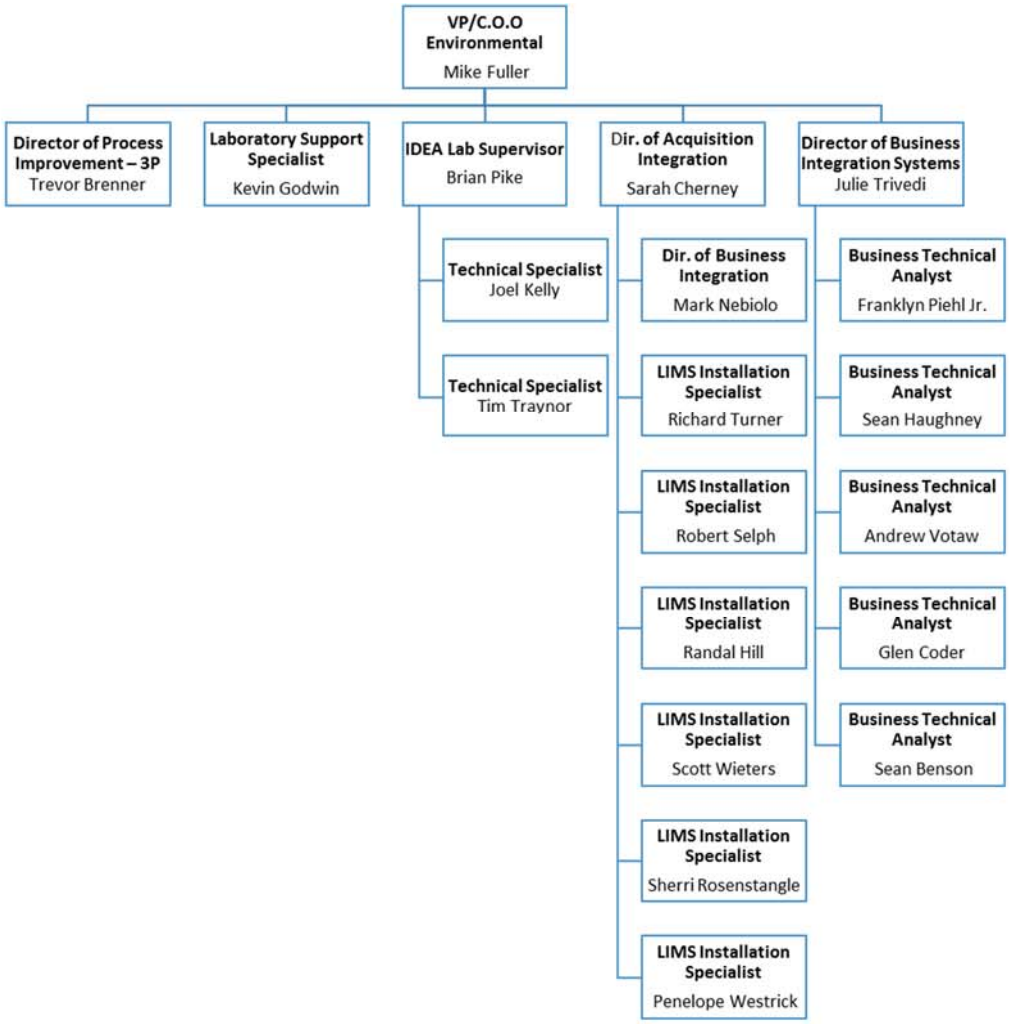
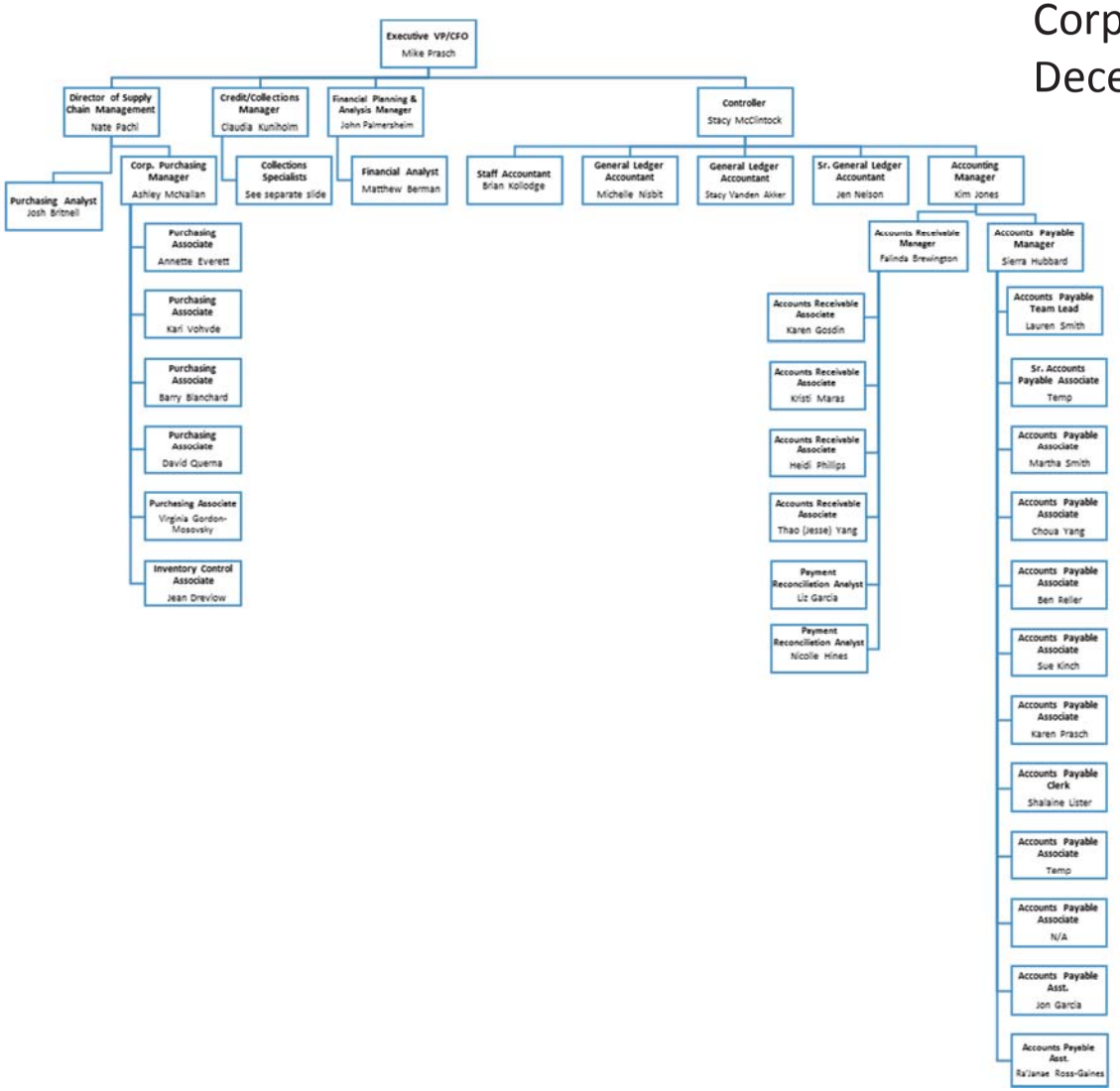
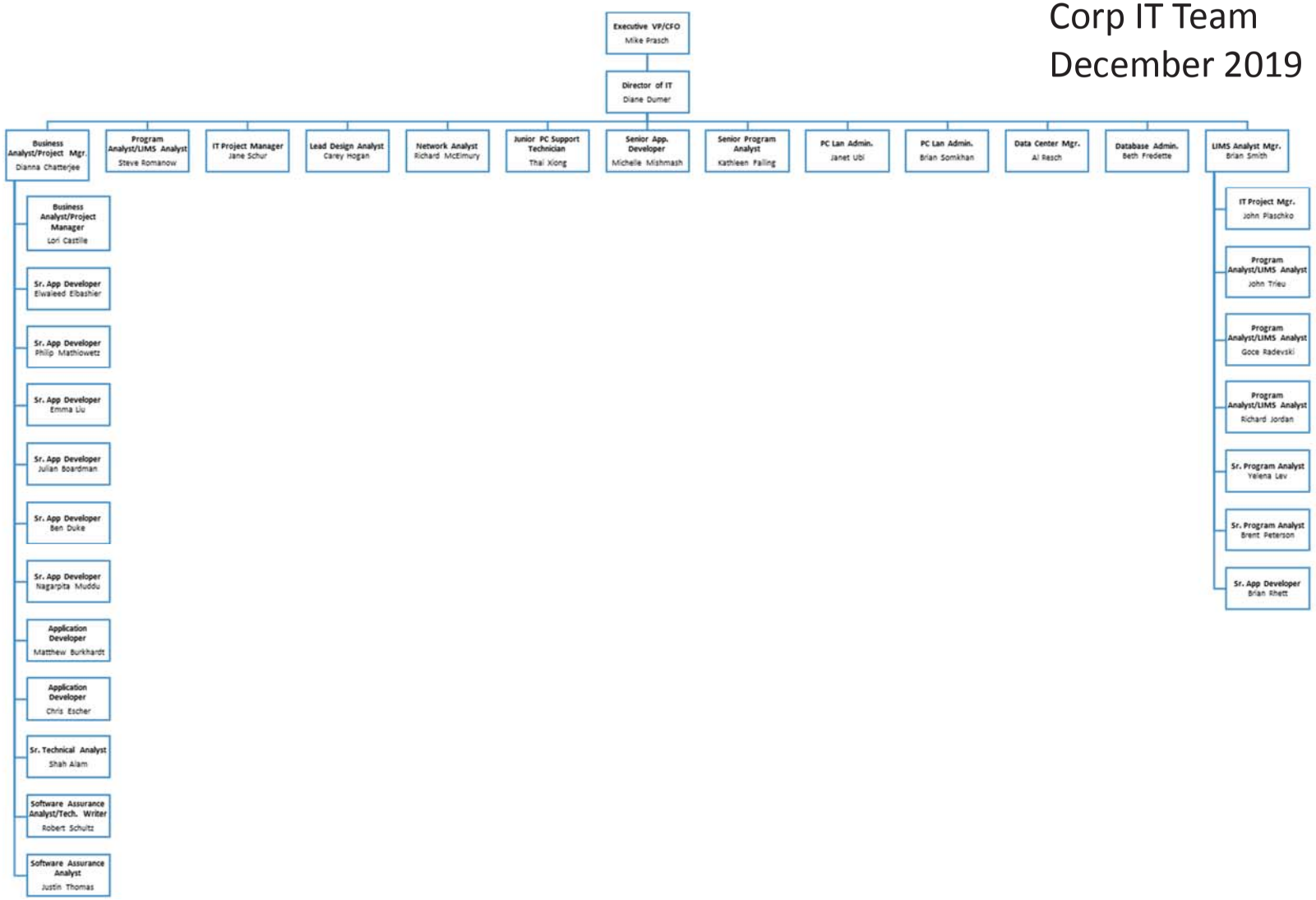


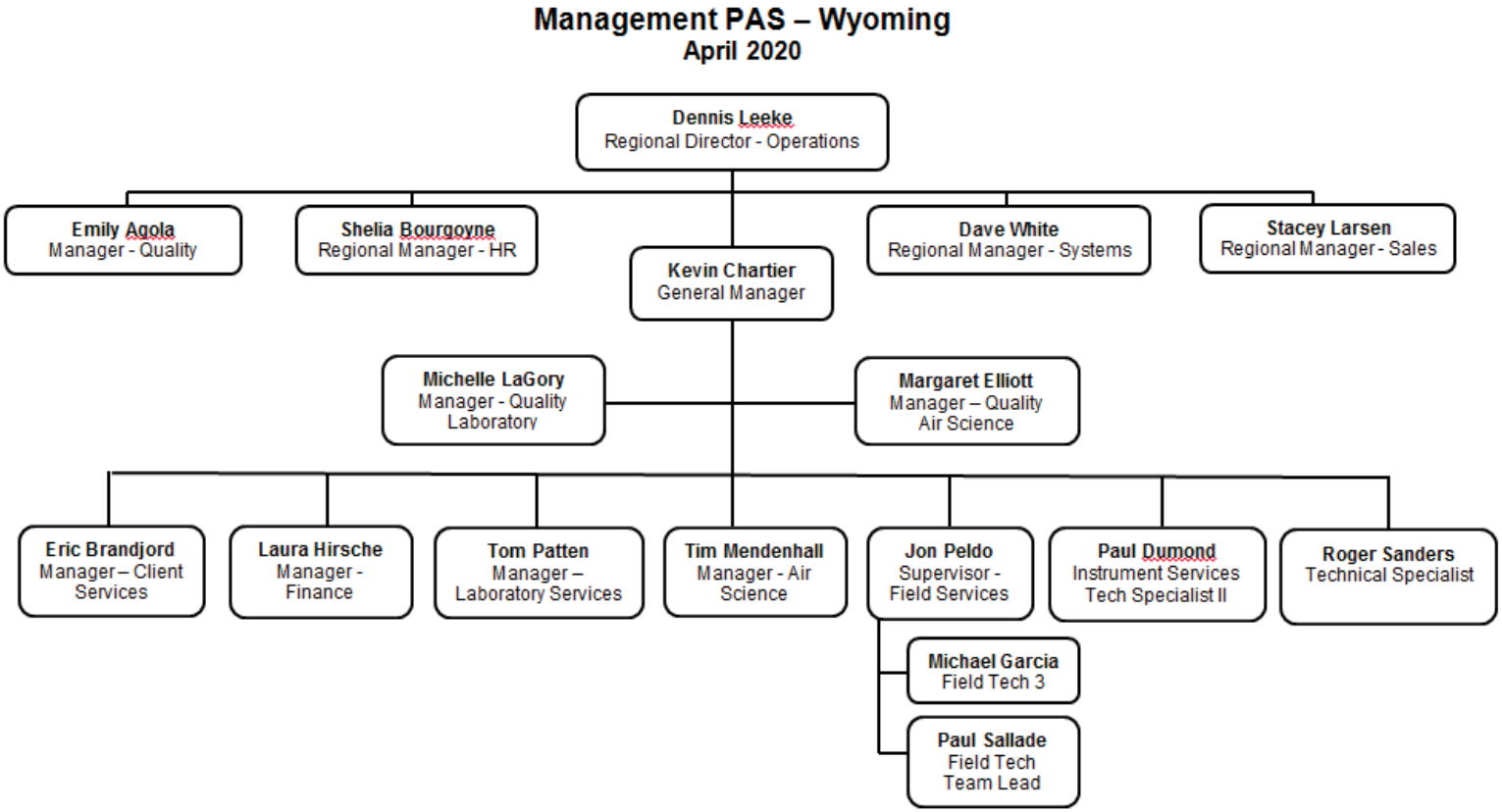
Corp. Finance Team
December 2019



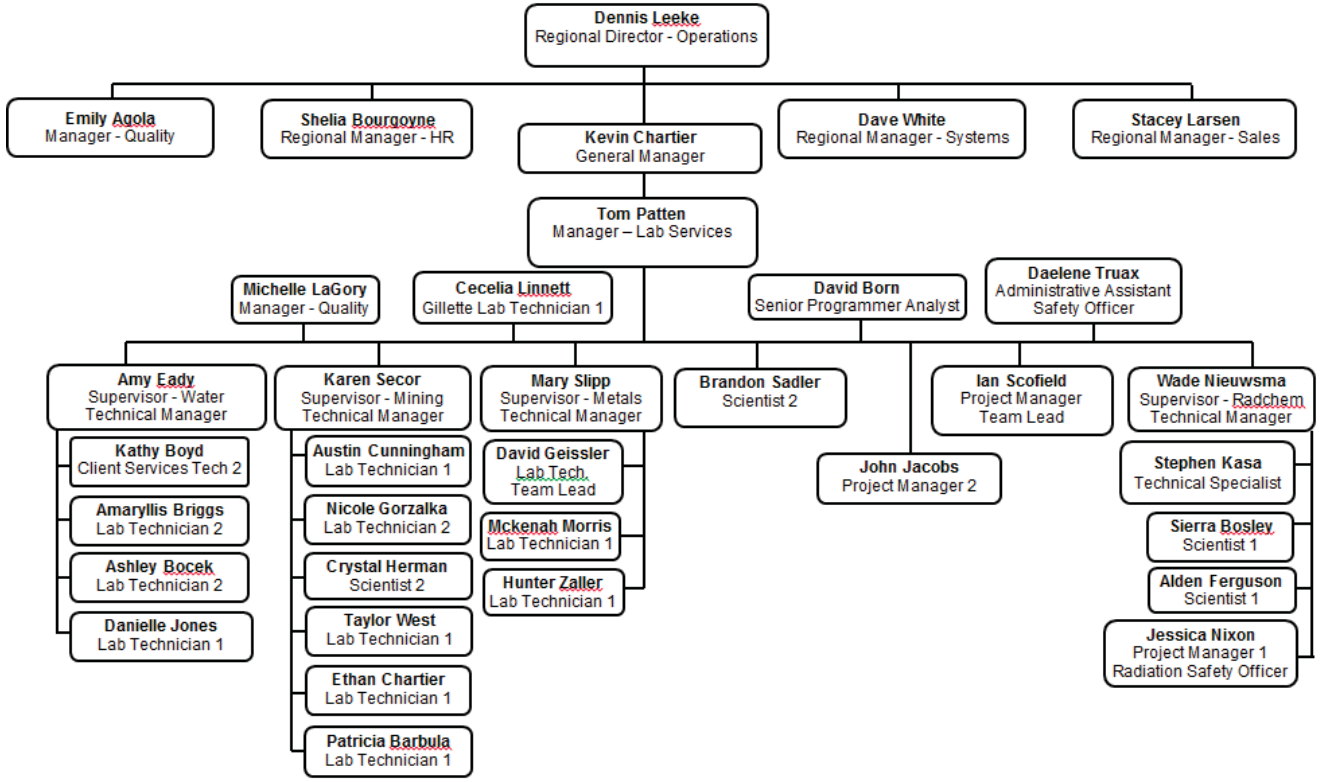
Corporate Credit & Collections Team
December 2019



7.4.2 PAS-Wyoming



Laboratory Services PAS - Wyoming
March 2020



7.5 Appendix E: Equipment Listing

The equipment listed represents equipment were held by each location on the effective date of this manual. This information is subject to change without notice. External parties should contact the location for the most current information.

7.5.1 PAS-Sheridan

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Manual Location
Balance	Mettler Toledo	AB 104-S	1120022615	10/4/19	Unknown	Water	737	TBD
Balance	AND	FR-200MKII	8300463	10/4/19	Unknown	Water	439	TBD
Balance	Denver Inst.	TR-2102	T-0117266	10/4/19	Unknown	Water	643	TBD
Balance	OHaus	AV4101	8029011171	10/4/19	Unknown	Water	NA	Water Lab
Reference weights	Troemner	Class I	63919	5/31/2018	Unknown	Water	NA	NA
Oven	Binder	115FED	14-10794	Unknown	Unknown	Water	1202	Bench
Oven	Binder	720	11-10331	Unknown	Unknown	Water	1142	Bench
Dessicator	Fisher Sci.	NA	NA	Unknown	Unknown	Water	NA	TBD
Dessicator	Fisher Sci.	NA	NA	Unknown	Unknown	Water	NA	TBD
Dessicator	NA	NA	NA	Unknown	Unknown	Water	NA	TBD
Dessicator	NA	NA	NA	Unknown	Unknown	Water	NA	TBD
Dessicator	Fisher Sci.	NA	NA	Unknown	Unknown	Water	NA	TBD
Dessicator	Nalgene	5317-0180	NA	Unknown	Unknown	Water	NA	TBD
Digestion Block	AndrewGlass	110-19-R	A4N0209	Unknown	Unknown	Water	122	TBD
Digestion Block	Foss Tecator	2000	2850	Unknown	Unknown	Water	NA	TBD
COD Reactor	Hach	45600-00	000600020662	Unknown	Unknown	Water	NA	TBD
Hot Block TKN System	Environmental Express	TKN 100 (controller)	2015TKNBC108	Unknown	New	Water	NA	Water Lab
Stir Plate	Corning	PC-410	350401284084	Unknown	Unknown	Water	NA	TBD
Stir Plate	Corning	PC-410	350401284141	Unknown	Unknown	Water	NA	TBD
Stir Plate	Cimarec I	54645	621910366626	Unknown	Unknown	Water	NA	TBD
Stir Plate	IKA			Unknown	Unknown	Water	NA	TBD
Stir/Hot Plate	Curtin Matheson	267-914	43900528	Unknown	Unknown	Water	NA	TBD
Hot Plate	Corning	PC-300	NA	Unknown	Unknown	Water	NA	TBD
Hot Plate	Cimarec I	HP46515	46500153	Unknown	Unknown	Water	NA	TBD
SPE-Extractor	Environmental Express	StepSaver G1103	NA	Unknown	New	Water	1158	Water Lab
mV Meter	Allied	805 MP	3164	Unknown	Unknown	Water	NA	Instrument Room
Conductivity Meter	Control Co.	4163	192308491	10/4/2019	New	Water	NA	Bench
pH/Cond Meter	Oakton	510	1424881	Unknown	Unknown	Water	NA	Bench
Turbidimeter	HACH	2100N	06100C021726	Unknown	New	Water	NA	Bench
Spectrophotometer	HACH	DR3900	1437665	Unknown	New	Water	1155	Bench
Incubator	Blue M	100A	6104	Unknown	Unknown	Water Micro	395	Bench
Incubator	Forma Sci.	3740	32210-490	Unknown	Unknown	Water Micro	NA	Bench
Incubator	Fisher	625F	105N0006	Unknown	Unknown	Water Micro	NA	Bench
Water Bath	Precision	253	9603-006	Unknown	Unknown	Water Micro	396	Bench
Water Bath	Neslab	GP-300	198205045	Unknown	Unknown	Water Micro	NA	
Autoclave	Tuttnauer	2540E B/L	1210225	Unknown	Unknown	Water Micro	745	Bench
Ultraviolet Lamp	Spectroline	EA-160	933105	Unknown	Unknown	Water Micro	NA	TBD
Quanti-Tray Sealer PLUS	IDEXX	89-0003936-00	QTP13191205087	Unknown	Unknown	Water Micro	NA	Bench
DO Meter	Hach	HQ440dMulti	110200051502	Unknown	New	Water	NA	Bench

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Manual Location
						Micro		
DO Probe	Hach	LBOD10101	110423032024	Unknown	New	Water Micro	NA	Bench
Autotitrator	ManTech	PC-1000-102	MSOHO-256	Unknown	New	Water	774	At Instrument
Autotitrator	ManTech	PC-1000-102/4	MS-OF6-427	Unknown	New	Water	NA	At Instrument
IC	Thermo/Dionex	ICS-1100	12030735	Unknown	New	Water	1143	Bookshelf Water Lab
IC	Thermo/Dionex	ICS-1000	7080942	Unknown	New	Water	1007	Bookshelf Water Lab
TOC Analyzer	Shimadzu	TOC -V CPH	638-91062-32	Unknown	Unknown	Water	1133	At Instrument
Distillation Unit	Lachat	MicroDist	A2000-829	Unknown	Unknown	Water	872	Instrument Room
Flow Analyzer	Alpkem	RFA-300		Unknown	Unknown	Water	527	Instrument Room
Flow Analyzer	OI Analytical	FS3000	15804554	Unknown	Unknown	Water	1025	Bookshelf Water Lab
UV Digester	OI Analytical	321201	403813593	Unknown	Unknown	Water	NA	Bookshelf Water Lab
Flow Analyzer/Detector	OI Analytical	(FS3100) 322689	212831028	Unknown	New	Water	1145	Bookshelf Water Lab
Discrete Analyzer	Unity Scientific	399-W170_02 SmartChem 170	W1602055	Unknown	New	Water	NA	At Bench
Lab pure water System	Deionizer	NA	NA	4/10/20	New	Mechanical Room	NA	TBD
Dishwasher	LG	MEZ64589004	3091879	Unknown	Unknown	Water	NA	TBD
ICP-OES	Varian	Vista Pro	3098093	Unknown	New	Metals	0833	Software
ICP-OES	Varian	720-ES	IP0909M050	Unknown	New	Metals	1138	Software
ICP-MS	Agilent	7800	SG18193130	Unknown	New	Metals	NA	Software and Instrument Room
ICP-MS	Agilent	7700x(G3281A)	JP11231114	Unknown	New	Metals	1132	Software and Instrument Room
Hg Analyzer	Teledyne/Leeman	Hydra II	1029	Unknown	New	Metals	826	Instrument Room
AA	Varian	SpectraAA 220FS	EL02103375	Unknown	Unknown	Metals	NA	Unknown
Vapor Generator	Varian	VGA-76	8061581	Unknown	Unknown	Metals	NA	Instrument Room
Balance	OHaus	GT-2100	2518	10/4/19	Unknown	Mining	449	TBD
Balance	OHaus	TP4KD	1239	10/4/19	Unknown	Mining	317	TBD
Balance	OHaus	AX8201/E	B512728832	10/4/19	Unknown	Mining	NA	TBD
Balance	Mettler Toledo	AE100	22715	10/4/19	Unknown	Mining	9719	TBD
Balance	Mettler Toledo	PB3002-SDR	1121330461	10/4/19	Unknown	Mining	NA	TBD
Balance	Denver I C	TL-4101	T-0111653	10/4/19	Unknown	Mining	NA	TBD
Balance	Denver Inst.	TL-104	T-0111761	10/4/19	Unknown	Mining	NA	TBD
Balance	Sartorius	A200S D20	10502468	10/4/19	Unknown	Mining	96	TBD
Weights	Troemner	Class I	63920	10/17/18	Unknown	Mining	NA	TBD
Stir Plate	Thermolyne Cimarec I	S4 6415	621940351162	Unknown	Unknown	Mining	441	TBD
Stir Plate	Fisher	120MR	NA	Unknown	Unknown	Mining	587	TBD
Stir Plate	Corning	PC-610	310701066411	Unknown	Unknown	Mining	NA	TBD
Stir Plate	Corning	PC-103	NA	Unknown	Unknown	Mining	NA	TBD
Stir Plate	IKA	LabChief	NA	Unknown	Unknown	Mining	NA	TBD
Stir Plate	Fisher Scientific	120S	905N1138	Unknown	Unknown	Mining	NA	TBD

Pace Analytical®

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Manual Location
Stir Plate	Thermolyne Barnstead	HP46825	639950140968	Unknown	Unknown	Mining	NA	TBD
Vortex	Fisher	Vortex-Genie	26772	Unknown	Unknown	Mining	NA	TBD
Vortex	Barnstead	M16715	1329040799154	Unknown	Unknown	Mining	NA	TBD
pH Meter	ThermoOrion	3 Star	15179	Unknown	Unknown	Mining	NA	At Bench
pH Meter	ThermoOrion	520 A+	1623	Unknown	Unknown	Mining	318	At Bench
pH Meter	ThermoOrion	520 A+	70566	Unknown	Unknown	Mining	NA	At Bench
Cond Meter	Accumet	AB30	AB92312695	10/4/2019	Unknown	Mining	NA	At Bench
Hydrometer	Ertco	N16B	5169100	10/4/2019	Unknown	Mining	NA	TBD
Hydrometer	Fisherbrand	NA	3979	10/4/2019	Unknown	Mining	NA	TBD
Hydrometer	Fisherbrand	NA	15-105-6	10/4/2019	Unknown	Mining	NA	TBD
Hydrometer	Fisherbrand	NA	32982	10/4/2019	Unknown	Mining	NA	TBD
Hydrometer	NA	NA	124686	10/4/2019	Unknown	Mining	NA	TBD
Testing Screen	Gilson	TS-1	8423	Unknown	Unknown	Mining	NA	TBD
Open Air Shaker	Amerex Instruments	Gyromax SK818	AI14106501-40	Unknown	Unknown	Mining	NA	TBD
Centrifuge	IEC	K	AA0602	Unknown	Unknown	Mining	NA	TBD
Incubator	Pegasus Scientific	Orbit Enviro-Shaker	NA	Unknown	Unknown	Mining	NA	TBD
Humidifier	LG	LD450EAL	NA	Unknown	Unknown	Mining	NA	TBD
Humidifier	LG	NA	NA	Unknown	Unknown	Mining	NA	TBD
Hood	BICO	NA	70694	Unknown	Unknown	Mining	808	TBD
Hood	BICO	NA	70695	Unknown	Unknown	Mining	809	TBD
Splitter	Gilson	SP-1	NA	Unknown	Unknown	Mining	334	TBD
Jaw Crusher	Allis	Morse Jaw	2000-003	Unknown	Unknown	Mining	333	TBD
Soil Grinder	Nasco Asplin	SG	7792	Unknown	Unknown	Mining	326	TBD
Wyly Mill	Thomas Sci.	3383-L10	090107	Unknown	Unknown	Mining	766	TBD
Pulverizer	BICO	976-XX1410	781030A-28	Unknown	Unknown	Mining	328	TBD
Pulverizer	Holmes	350A1BS	516	Unknown	Unknown	Mining	NA	TBD
15 Bar Plate Extractor	Soil Moisture Equip	Unknown	Unknown	Unknown	Unknown	Mining	536	TBD
Bench Top Ring and Puck Mill	Rock Labs	1A	460	Unknown	Unknown	Mining	1130	TBD
Oven	VWR	NA	Unknown	Unknown	Unknown	Mining	NA	TBD
Oven	Fisher	338-F	Unknown	Unknown	Unknown	Mining	NA	TBD
Furnace	Thermolyne/ Sybron	30420	304-00294	Unknown	Unknown	Mining	305	TBD
Microwave	GE	JES1136WL01	SL 934271 B	Unknown	Unknown	Mining	NA	TBD
Microwave	GE	JES738WJ02	ML 913611 U	Unknown	Unknown	Mining	NA	TBD
C/S Analyzer	Leco	606-000-RFB	4522	Unknown	Unknown	Mining	1131	Drawer near instrument
C/S Analyzer	Leco	SC-144DR	3296	Unknown	Unknown	Mining	735	Drawer near instrument
Spectrophotometer	HACH	DR2010	970100001571	Unknown	Unknown	Mining	NA	TBD
Pancake Griddles (5)	Varied	Varied	Varied	Unknown	Unknown	Mining	NA	TBD
Hotplate	Cimarec 2	Varied	Varied	Unknown	Unknown	Mining	NA	TBD
Hotplate	VWR	130	Varied	Unknown	Unknown	Mining	NA	TBD
Autotitrator	Man-Tech	PC-1000-400	MS-OH-685	Unknown	New	Mining	NA	At Instrument
Balance	Ohaus	AV4101	8033221228		New	Kinetics	NA	TBD
Reference Weights	Not Available	Class 1	55365	Unknown	Unknown	Kinetics	NA	TBD
Hygrometer/Thermometer	Dwyer	485B-1	MOZAA	Unknown	Unknown	Kinetics	NA	TBD
Hygrometer/Thermometer Probe	Dwyer	RPI	0067DV	Unknown	Unknown	Kinetics	NA	TBD
Bench Top Bottle Roller	Legend Inc.	98989	NA	Unknown	Unknown	Kinetics	1160	TBD
pH/ORP/EC Meter	Accumet	XL20	XL94005458	Unknown	Unknown	Kinetics	NA	TBD
pH/ORP Meter	Hanna	HI98190	G0053707	Unknown	Unknown	Kinetics	NA	TBD
Conductivity Meter	Oakton	Con 510 Series	582392	Unknown	Unknown	Kinetics	NA	TBD
DO Meter	YSI	YSI 5000	96K0501AF	Unknown	Unknown	Kinetics	NA	TBD

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Manual Location
BOD Probe	YSI	YSI 5010	06M1674	Unknown	Unknown	Kinetics	NA	TBD
Oven	Fisher IsoTemp	300	NA	Unknown	Unknown	Kinetics	388	TBD
Digital Fractional Caliper	General Tools	147	NA	Unknown	Unknown	Kinetics	NA	TBD
Liquid Scintillation Counter	Beckman Coulter	LS 6500	7070183	Unknown	Used	Radchem	NA	At Instrument
Gas Proportional Counter	Tennelec	LB4110	185	Unknown	New	Radchem	781	At Instrument
Gas Proportional Counter	Gamma Products	G542 MQuad	121001	Unknown	New	Radchem	NA	At Instrument
Alpha Spec	Canberra	7200-08	13000018	Unknown	New	Radchem	1115	At Instrument
Vacuum Pump	Welch	8905B-24	13000023	Unknown	New	Radchem	NA	Radchem
Vacuum Pump	JB	C55JXKPK-5060	1836033100973	Unknown	New	Radchem	NA	Radchem
Gamma Spec (HPGe)	Ortec	GEM40P4-83-SMP	50-TP-50712C	Unknown	New	Radchem	1112	At Instrument
Controller	Ortec	DSPEC jr 2.0	10131748	Unknown	New	Radchem	NA	Radchem
X-Cooler	MMR	CC2402	100414CT	Unknown	New	Radchem	1111	Radchem
Gamma Spec (HPGe)	Ortec	GEM40P4-83-SMP	P13717A	Unknown	New	Radchem	NA	At Instrument
Controller	Ortec	DSPEC-JR-2.0	16096493	Unknown	New	Radchem	1113	TBD
Lead Shield	Ortec	NA	NA	NA	New	Radchem	NA	TBD
X-Cooler	MMR	Smart-1-POS	16057787	Unknown	New	Radchem	NA	TBD
Automatic Sample Changer	Gamma Products	G3200W	071202	Unknown	New	Radchem	1159	Radchem
Gamma Spec (NaI)	Canberra	707	10974890	Unknown	Used	Radchem	NA	TBD
Balance	Mettler	AJ100	I-23325	10/4/19	Unknown	Radchem	NA	TBD
Balance	Mettler Toledo	PL-602-S	1125492939	10/4/19	Unknown	Radchem	NA	Radchem
Balance	Sartorius	1602	3303147	10/4/19	Unknown	Radchem	NA	TBD
Reference Weights	Christian Becker	Class 1	54106		Unknown	Radchem	NA	NA
Oven	Fisher	116G	309	Unknown	Unknown	Radchem	NA	NA
Incubator/oven	Yamato	IC600	B1300035	Unknown	Unknown	Radchem	810	NA
pH Meter	CMS LabCraft	pH102	1134	Unknown	Unknown	Radchem	134	At Instrument
Conductivity Meter	Accumet	AB 30	AB92312240	10/4/19	Unknown	Radchem	NA	At Instrument
Hotplate	Thermolyne/Cimarec 3	HP 47135	611960704345	Unknown	Unknown	Radchem	123	TBD
Hotplate	Thermolyne/Cimarec 3	HP 47135	611950467482	Unknown	Unknown	Radchem	NA	TBD
Hotplate	Thermolyne/Cimarec 3	HP 47135	1073981151142	Unknown	Unknown	Radchem	NA	TBD
Hotplate	Thermolyne/Cimarec 3	HP 47135	1073990756777	Unknown	Unknown	Radchem	NA	TBD
Hotplate	Thermolyne/Cimarec 3	HP 47135	NA	Unknown	Unknown	Radchem	NA	TBD
Hotplate	Thermolyne/Cimarec 3	HP 47135	NA	Unknown	Unknown	Radchem	NA	TBD
Hotplate	Thermolyne/Cimarec 3	HP 47135	NA	Unknown	Unknown	Radchem	NA	TBD
Hotplate	Corning	PC-310	NA	Unknown	Unknown	Radchem	NA	TBD
Hotplate/Stirrer	Corning	PC-320	03027082	Unknown	Unknown	Radchem	NA	TBD
Hotplate/Stirrer	Torrey Pines Scientific	HS19	7131018	Unknown	Unknown	Radchem	NA	TBD
Hotplate/Stirrer	Torrey Pines Scientific	HS19	02161105	Unknown	Unknown	Radchem	NA	TBD
Hotplate/Stirrer	Torrey Pines Scientific	HS19	09210904	Unknown	Unknown	Radchem	NA	TBD
Hotplate/Stirrer	Torrey Pines Scientific	HS19	09210905	Unknown	Unknown	Radchem	NA	TBD
Digestion Block	CPI	MOD Block	NA	Unknown	Unknown	Radchem	NA	Radchem
Vortex Genie	Scientific	G560	2-419789	Unknown	Unknown	Radchem	NA	TBD

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Manual Location
	Industries							
Vortex Genie	Scientific Industries	G560	2-415262	Unknown	Unknown	Radchem	NA	TBD
Vortex Genie	Scientific Industries	G560	2-415322	Unknown	Unknown	Radchem	NA	TBD
Vacuum Pump	Emerson	SA55NXGTE	l017	Unknown	Unknown	Radchem	NA	TBD
Vacuum Pump	Fisher Scientific	SA55JXGTD-4144	602145093	Unknown	Unknown	Radchem	NA	TBD
Vacuum Pump	Gast	0523-V4F-G588DX	K08J180087	Unknown	Unknown	Radchem	NA	TBD
24-place Vacuum Box	Eichrom	NA	NA	Unknown	Unknown	Radchem	NA	TBD
24-place Vacuum Box	Eichrom	NA	NA	Unknown	Unknown	Radchem	NA	TBD
Centrifuge	Garver Mfg	Babcock	6924	Unknown	Unknown	Radchem	NA	TBD
Centrifuge	International Centrifuge	EXD	50037P	Unknown	Unknown	Radchem	NA	TBD
Centrifuge	International Centrifuge	K	71652704	Unknown	Unknown	Radchem	NA	TBD
Dessicator	Boekel	1344	NA	Unknown	Unknown	Radchem	33	TBD

7.5.2 PAS-Gillette

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Manual Location
Dry Air Incubator	Napco	330	7-76-0103	Unknown	Unknown	Micro	NA	TBD
Quantitray Sealer	Idexx	2X	3968	Unknown	Unknown	Micro	NA	TBD
Autoclave	Tuttnauer	EZ-9	2908785	Unknown	Unknown	Micro	1120	Lab
UV Lamp	Spectrolite	EA-160	728992	Unknown	Unknown	Micro	NA	TBD

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the analysis of elements in an aqueous solution using inductively coupled plasma mass spectrometry (ICPMS).

This method is appropriate for drinking water, surface water, ground water, wastewater, aqueous extractions of solids, biosolids, filter extractions, and select biological fluids.

Performance of this procedure requires a working knowledge of the Agilent ICPMS.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The typical reporting limits for aqueous samples are listed below for the following elements:

Element	Reporting Limit (mg/L)
Aluminum	0.050
Antimony	0.005
Arsenic	0.005
Barium	0.100
Beryllium	0.002
Bismuth	0.010
Cadmium	0.002
Chromium	0.010
Cobalt	0.002
Copper	0.010
Gallium	0.010
Lanthanum	0.002
Lead	0.002
Manganese	0.010
Molybdenum	0.020
Nickel	0.010
Selenium	0.005
Silver	0.003
Strontium	0.010
Thallium	0.001
Tin	0.010
Titanium	0.010
Uranium	0.001
Vanadium	0.020
Zinc	0.010

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and

non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

2.0 SUMMARY OF METHOD

ICP-MS technology makes possible the simultaneous multi-element determination of trace elements. Sample material in solution is introduced by pneumatic nebulization into a radio-frequency plasma, where energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer having a minimum resolution capability of 1 amu peak width at 5% peak height. The ions transmitted through the quadrupole are detected by an electron multiplier, or Faraday detector, and the ion information processed by a data handling system. Interferences relating to the technique must be recognized and corrected for. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents, or sample matrix. Instrumental drift as well as suppressions or enhancements of instrument response caused by the sample matrix must be corrected for by the use of internal standards.

3.0 INTERFERENCES

Isobaric elemental interferences: These are caused by isotopes of different elements which form singly or doubly-charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrophotometer. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method, only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative analytical isotopes having higher natural abundance are selected in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest.

Abundance sensitivity: This is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operation pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

Isobaric polyatomic ion interferences: These are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer. These ions are commonly formed in the plasma or interface system from support gasses or sample components. Most of the common interferences have been identified, and these are listed in Table 1. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Equations for the correction of data should be established at the time of the analytical run sequence, because the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions. In particular, the common 82Kr interference that affects the determination of both arsenic and selenium can be greatly reduced with the use of high purity krypton-free argon.

Physical interferences: These are associated with the physical processes that govern the transportation of sample into the plasma, sample conversion process in the plasma, and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the extraction and /or skimmer cones reducing the effective diameter of the orifices and therefore ion transmission. Dissolved solids levels not exceeding 0.2% (w/v) are recommended. Internal standards may be used effectively to compensate for many physical interference effects.

Memory interferences: These interferences result when isotopes of elements in the previous sample contribute to the signals measured in a new sample. Memory effects can be a result of sample deposits on sampler and skimmer cones, plasma torch, and spray chamber. These interferences can be minimized by flushing with a rinse blank between samples

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

- Linear Dynamic Range: The linear dynamic range is the concentration range over which the instrument to an analyte is linear. The linear dynamic range is performed every six (6) months. The successful linear dynamic range result is a recovery of ± 10% of the expected value. The final linear dynamic range value is 10% less than the solution concentration used

5.0 HEALTH AND SAFETY

ICP-MS instruments operate at high currents of electricity and care should be taken to avoid placement of liquids upon the instrument.

Contact your supervisor or local safety coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure

The following sections provide general health and safety information about chemicals and materials that may be present in the laboratory.

- The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (EHS) policies and procedures specified in this SOP and in the Pace® Chemical Hygiene / Safety Manual (COR-MAN-0001)
- Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

- Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. For procedures that require use of acids, use acids in a fume hood whenever possible with PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. For procedures that that emit large volumes of solvents (evaporation/concentration processes), these activities must be performed in a fume hood or apparatus that reduces exposure.

6.0 .SAMPLE COLLECTION, PRESERVATION, HOLDING TIME & STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory will provide containers for the collection of samples upon client request for analytical services. The bottle kits provided by the laboratory should include field test kits and treatment reagent.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding time
Aqueous	Glass, fluoropolymer, or polyethylene containers	250 mL (lab supplies these along with a small vial containing 2mL of 1:1 nitric acid for preservation)	Preserved to pH < 2 with nitric acid	Preserved aqueous samples: 6 months from collection
Solid	Glass, plastic bag	10 g	Unprocessed samples are refrigerated	6 months from collection
Air Filter	Glassine envelope, manilla envelope for TSP and glass filters and plastic container for PM Teflon filters	1 filter	NA	180 days from collection

[1]*Minimum amount needed for each discrete analysis.*

Field / Matrix QC

Trip Blank	Field Blank	MS/MSD	Field Duplicate
Not applicable	Not applicable	Not applicable	Not applicable

Samples for analysis as dissolved metals are filtered in the laboratory if they have not been filtered prior to receipt. After filtering with a 0.45µm filter, the samples are acidified with 1+1 nitric acid to a pH < 2. When samples are filtered in the lab, the technician records the date and time of filtering, as well as their initials on the bottle label.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory procedures. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at room temperature until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at room temperature until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, aqueous samples are retained for 5 months from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT & SUPPLIES

7.1 Equipment

Item	Vendor	Model ID	Catalog ID	Description
Inductively Coupled Plasma Mass Spectrometer (ICPMS)	Agilent	7700 ICPMS and 7800 ICPMS	NA	ICPMS
Data handling system	Agilent	7700: MassHunter Workstation Software Version A.01.02 Patch 5 7800: MassHunter Workstation Software Version C.01.04 Patch 3	NA	Proprietary software for instrument operation and analyte detection
Instrument compatible chiller	Agilent	G3292A	NA	chiller
Autosampler	Cetac Agilent	7700: ASX-500 7800: SPS4	NA	autosampler

7.2 Supplies

Item	Vendor	Model ID*	Catalog ID*	Description*
Analytical Balance	Mettler	AE100	NA	Capable of weighing to 0.0001g
Centrifuge with guard bowl	Clay Adams	Dynac	NA	
Air displacement pipettes	Variety of Vendors	Variety of Models	Variety of catalog numbers	Variety of volumes
Volumetric flasks	Variety of sources and sizes	NA	NA	Variety of volumes Class A
Graduated cylinders	Variety of sources and sizes	NA	NA	Variety of sizes Class A
Pipette Tips	Variety of Vendors	Variety of Models	Variety of catalog numbers	Variety of volumes
Argon gas supply	American Welding	Dewar	AR-180LIQ-5.0	99.999% Ultra High Purity Argon dewar
Hydrogen gas supply	American Welding	T/K tank	HYK-5.0	High Purity Hydrogen

Item	Vendor	Model ID*	Catalog ID*	Description*
Helium gas supply	American Welding	T/K tank	HE HPK	High Purity Helium
Plastic test tubes (single-use)	Mold Pro	MP-100	17mm x 100mm test tubes	1200 per case
Disposable pipette tips	Eppendorf or equivalent	Various sizes	Various catalog IDs	Variety of sizes
Plastic containers	QEC	8oz Oblong plastic	6212-0008	Various sizes as needed

*Alternate consumable supplies may be used providing the quality is equivalent to that listed.

8.0 REAGENTS & STANDARDS

8.1 Reagents

Refer to SOP for Standard and Reagent Management and Traceability for storage requirements and expiration information. Unless otherwise noted, stock reagents and standard must be stored per manufacturer's recommendations.

Reagent	Description/Concentration	Vendor*	Catalog #*
Reagent water	Deionized (DI) water	N/A	N/A
Nitric acid	Concentrated; metals grade	Fisher	A509P212
Hydrochloric acid	Concentrated; metals grade	Fisher	A508-P212
Rinse blank	2% nitric acid	Prepped on site	Prepared from Nitric Acid, concentrated, trace metals grade

*Alternate reagent supplies may be used providing the quality is equivalent to that listed.

8.2 Standards

Refer to SOP for Standard and Reagent Management and Traceability for storage requirements and expiration information. Unless otherwise noted, stock reagents and standard must be stored per manufacturer's recommendations.

Standard	Description/Concentration	Vendor*	Catalog #*
Tuning solution	10ppm Varian Tuning Solution MS plus individual analyte additions of 1000ppm Li and 1000ppm Y	Inorganic Ventures and Absolute Standards for individual Standards	VAR-TS-MS Li: 58003 Y: 58039
Internal standard solution	20ppm Internal Standard Solution 1000ppm Sc 1000ppm In 1000ppm Th	Inorganic Ventures and Absolute Standards for individual Standards	IML-MSISS-1A Sc: 58021 In: 58049 Th: 58090
Interference check standard	Interference Check Standard A for 6020 MS	Inorganic Ventures	6020ICS-0A
Calibration standards	20ppm ICPMS Standard-2B 20ppm ICPMS Standard-1C	Inorganic Ventures	IML-MSCAL-2B IML-MSCAL-1C
Second Source Standard	100ppm Spex Second Source	Spex	CL-CAL-2

*Alternate standard sources may be used providing the quality is equivalent to that listed.

9.0 PROCEDURE

9.1 **Calibration and Standardization**:

9.1.1 Calibration standards are handled in accordance with the appropriate method being run. The standards are diluted to working solutions from stock supply. Preservation is to match normal sample acid concentrations for analysis. Multi-element solutions are used and obtained from reputable commercial sources. Details about calibration concentrations and requirements for each method are listed in Appendices A-D.

9.1.2 The instrument must be calibrated at the beginning of each run.

9.1.3 Calibration data are accepted based upon acceptance criteria outlined in the appropriate method being used as detailed in Appendices A-D.

9.1.4 The lab routinely uses an external calibration (as opposed to a standard additions calibration). This can be checked by viewing the settings in the Online ICPMS Mass Hunter software and choosing the Batch section → Data Analysis Method tab → Full Quant tab → Basic Calibration Parameters → Calibration Method → External Calibration.

9.2 **Procedure**:

9.2.1 Sample preparation and digestion (if applicable) are detailed in lab SOPs for digestion by EPA methods 200.2, 3020A, 3050, IO-3.1, and FEM 2009 (Appendix A).

9.2.2 See Appendices E-F for detailed instrument operation instructions and screen shots of software operations.

9.2.3 Maintenance procedures are followed as recommended by the instrument's manufacturer and outlined in the appropriate method. Peristaltic pump tubing and 2% HNO_3 rinse water are changed daily, or as needed. Pump oil is changed according to instrument manufacturer's recommendation. Chiller fluid level must be monitored periodically and changed as needed. The ICPMS sampler and skimmer cones, and Extraction Lens-Omega Lens assembly are cleaned or replaced as needed. Periodic cleaning or changing of the nebulizer, spray chamber, auto-sampler tubing, torch, and instrument tubing will be necessary. Any time the operator feels that the instrument is not operating properly; the problem will be found and rectified before processing samples.

9.2.4 Initiation of the ICPMS begins with checking for adequate argon, helium and hydrogen gas, changing and engaging peristaltic pump tubing, emptying waste, and filling 2% HNO_3 water and 2% HNO_3 + 2% HCl rinse reservoirs. Uncap all tuning solutions and the P/A solution. Place the internal standard line in a solution of 2% HNO_3 DI. Turn on the computer monitor and computer if not already on. Open the Agilent operating software labeled ICPMS Instrument Control. Create the day's working batch file by copying the previous day's batch using "Save Batch As…" function under "File" and give it the current date as a file name saving it to the D drive. Delete previous data from the queue and any samples in the Sample List tab in the Unknown Samples table. Check to make sure Start Up has tests checked that you want to have run at the end of the warm-up period. Put the auto-sampler probe in the 1ppb Tuning Solution by going to the auto-sampler section of the task bar and telling the auto-sampler the location of the 1ppb Tuning Solution. Press the "Plasma On" icon. At this point you can choose to let the software start the preset Performance Test after a 30-minute instrument warm-up, or you can manually start the Performance Test if needed. Allow the instrument to warm-up for at least 30 minutes.

9.2.5 After warm-up, the Start-Up program will run automatically, or must be started manually depending on the operator's choice to optimize, align and check instrument performance. The normal start-up sequence includes: Torch Axis, EM, Plasma Correction, Standard Lens Tune, Resolution/Axis, Performance Report, and P/A factor. When this is finished,

check to assure all operations are successful and check the Performance Report to check sensitivity and cone condition. Put the auto-sampler probe in the 10ppb Tune Solution. Ensure that the instrument is in the "no gas" mode and perform a "Signal Monitor" to detect when the instrument is in steady state and is ready for the instrument tune. Click the "Stop" icon. To start the instrument tune, click the "Start Auto Tune" green arrow icon. When this is complete click the "Save" icon. Then perform the USEPA tune check by clicking the "Generate Tune Report" icon in the Report section of the operations bar. The report will print automatically and save a pdf in the day's batch as well. Next tune the gas modes by clicking on the appropriate tab (no gas, He, or H2) and "Send to ICPMS" in the tool bar. Then time scan to equilibrate and when in a steady state click the "Start Auto Tune" green arrow icon. When complete, save settings. Continue to the next mode until all modes have been tuned. When finished, put the auto-sampler in the 2% HNO_3 rinse solution and the internal standard line in the internal standard. Start the "Signal Monitor" program in the Tune tab and monitor the reading until a steady state is achieved.

9.2.6 Enter a sample run table by going to the Batch section, then the Sample list tab, and highlight in the Sequence Flow table the Unknown Samples option and fill in the Unknown Samples table appropriately. Place prepared samples in the appropriate racks and then onto the auto-sampler. To start the run, click "Add to Queue" in the Batch (Queued) bar.

9.2.7 The auto-sampler table should be established following procedures according to EPA 200.8 (or appropriate method) and lab standard analysis procedures.

9.2.8 In the Online/Offline Data Analysis Mode, which auto initiates, run progress may be monitored in this mode.

9.2.9 Evaluate all data according to standards presented in all methods being run in that day's batch. Table 2 contains valuable guidance for isotope selection of select sample types and methods being utilized.

9.2.10 After the run is completed, export the data file by going to "File", "Export Table". Name the file and change the XLSX type to XLS to be compatible with Excel 97-2003 Workbook. Save the file in the day's batch and also export onto a flash drive to transfer the data to a LIMS enabled system.

9.2.11 The Agilent ICPMS can be set to auto-shut off by clicking the "Plasma Off at End" icon in the Queue. It can be manually shut down by clicking the "Plasma Off" icon in the task bar.

9.2.12 After the instrument has shut off, release the peristaltic pump tubing, send the auto-sampler home, and cap all solutions and rinses.

10.0 DATA ANALYSIS & CALCULATIONS

10.1 Calculations

See the Laboratory Quality Assurance Manual for equations for common calculations.

10.1.1 ICPMS data is generated in µg/L.

10.1.2 Dilution factors, which are multiplied by the instrument software, must be reviewed to take instrument detection limits into account.

10.1.3 Air Filter Calculation:

Air filter conversion from µg/L to ng/filter for PM Teflon style filter using IO-3.1 digestion:

ng/filter = µg/L * 0.02 L/filter * 1000 ng/µg

Air filter conversion from µg/L to ng/filter for TSP/PM10 style filter using IO-3.1 digestion

ng/filter = µg/L * 0.02 L/strip * 12 strips/filter * 1000 ng/µg

Air filter conversion from µg/L to µg/filter for TSP/PM 10 style filter using 40 CFR Appendix G Section 50 digestion:

µg/filter = µg/L * 0.1 L/strip * 12 strips/filter

10.1.4 Conversion of µg/L to mg/kg using a 3050 digestion with 1 g of sample and 100mL final volume:

mg/kg = µg/g = µg/L * 0.1 L/1 g sample

10.2 Manual Integration

Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument maintenance and/or method set-up. To assure that all manual integrations are justified and proper all manual integrations must be performed, documented, reviewed, and approved in accordance with corporate SOP ENV-SOP-CORQ-0006, *Manual Integration.* Refer to this SOP for guidance on manual integration techniques and required procedures.

11.0 QUALITY CONTROL & METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendices A-D for acceptance criteria and required corrective action.

QC Check	Acronym	Frequency
Method Blank	MB	1 per batch of 20 or fewer samples-All methods
Laboratory Control Sample	LCS	1 per batch of 20 or fewer samples-All methods
Sample Duplicate	DUP	1 DUP every 10 samples 200.8; other methods 1/20
Matrix Spike and Matrix Spike Duplicate	MS/MSD	1 set every 10 samples 200.8; other methods 1/20 (see Appendices A-D)

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendices A-D for acceptance criteria and required corrective action:

Instrument Check	Acronym	Frequency
Initial Calibration	ICAL	Daily
Initial Calibration Verification	ICV	Mid-range standard (second source) analyzed immediately after ICAL
Initial Calibration Blank	ICB	After ICV
Continuing Calibration Verification	CCV	CCV after every 10 samples
Continuing Calibration Blank	CCB	After every CCV

Data quality objectives for the analysis of QA/QC samples for each ICPMS method are summarized in Appendices A-D.

Internal standards may be changed from initial instrument settings due to presence in the sample or internal standard drift.

Bioassay samples that are greater than the reporting limit will be verified by repeat analysis.

If the method allows the use of an LDR, use the most recent acceptable LDR level. An acceptable LDR is one that, when run, read at ± 10% of the solution concentration used. The acceptable LDR level is then 10% less than the solution concentration used.

11.3 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file

11.4 Method Performance

11.4.1 Method Validation

Refer to corporate SOP ENV-SOP-CORQ-0011 for general requirements and procedures for method validation.

Establish detection limits (DL) and limits of quantitation (LOQ) at initial method set up and verify the DL and LOQ on an on-going basis thereafter. Refer to corporate policy and/or SOP for DL and LOQ requirements and procedures.

12.0 Data Review & Corrective Action

12.1 Data Review

The data review process of Pace® Analytical Services includes a series of checks performed at different stages of the process by different people to ensure that SOPs were followed, the analytical record is complete, and properly documented, QC criteria were met, proper corrective actions were taken for QC failure and other nonconformance(s), and test results are reported with proper qualification, when necessary.

The review and checks that are performed by the employee performing the task is called primary review.

All data and test results are also peer reviewed. This process, known as secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented, and approved in accordance with the Pace® Analytical Services SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

Lastly, a third-level review, called a completeness check, is performed by reporting or project management staff to verify the test report is complete.

Refer to laboratory Data Review SOP for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is required when QC or sample results are not within acceptance criteria.

Refer to Appendices A-D for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention & Waste Management

Pace® proactively seeks ways to minimize waste generated during work processes. Some examples of pollution prevention include but are not limited to reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practices comply with all applicable federal and state laws and regulations. Excess reagents, samples, and method process wastes are characterized and disposed of in an acceptable manner in accordance with the Pace® Chemical Hygiene Plan / Safety Manual. Refer to this manual for these procedures.

14.0 Modifications

The procedures in this SOP have not been modified from the reference test method(s) cited.

When applicable, comparability and/or equivalency studies necessary to validate the modification as required per corporate SOP ENV-SOP-CORQ-0011 are retained by local quality personnel for historical reference.

15.0 RESPONSIBILITIES

- All employees of Pace® Analytical Services that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file for the version(s) of the SOP that were in effect during the time the employee performed the activity.
- Local quality personnel are responsible for tracking the currency of the R&A on this SOP for employees at the locations they are assigned to and for notifying the General Manager (GM), however named, when R&A are overdue or outstanding. The GM and the employee's direct supervisor are responsible for ensuring the employee completes the R&A assignments as required.
- The supervisors and managers of Pace® Analytical Services, however named, are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP the work area(s) they oversee.
- All employees of Pace® Analytical Services are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local Quality Manager and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual change to the SOP are not permitted. If a change is desired or necessary, Pace® employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015 *Document Managemen*t and ENV-SOP-CORQ-0016 *SOP for Creation of SOP and SWI*.
- Local quality personnel are responsible for monitoring conformity to this SOP during routine internal audits of work areas that utilize this SOP and for communicating gaps and deviations found during monitoring to the work area supervisor, who is responsible for correction of the situation.

16.0 ATTACHMENTS

- Appendix A: QC Summary- Method 200.8
- Appendix B: QC Summary- Method 6020A
- Appendix C: QC Summary- IO-3.5
- Appendix D: QC Summary- FEM 2009 (EQL-0310-189)
- Table 1: Common Polyatomic Ion Interferences in ICPMS
- Table 2: Recommended Isotopes
- Appendix E: Step by Step Operation Instructions for Agilent 7700 ICPMS
- Appendix F: Step by Step Operation Instructions for Agilent 7800 ICPMS

17.0 REFERENCES

- ENV-SOP-CORQ-0006, *Manual Integration*, current version.
- ENV-SOP-CORQ-0011, *Method Validation*, current version.
- ENV-SOP-CORQ-0015, *Document Management*, current version.
- ENV-SOP-CORQ-0016, *SOP for SOP and SWI,* current version.
- ENV-TMP-CORQ-0007, *Quality Manual Template*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- COR-MAN-001, *Pace® Safety Manua*l, current version.
- USEPA. May 1994. Determination of Trace Elements in Waters and Waste by Inductively Coupled Plasma Mass Spectrometry, Method 200.8, Revision 5.4
- Agilent 7700 Series ICPMS Mass Hunter Workstation User Guide, Manual Part Number G7201-90200, Rev. A, September 2010
- Agilent 7800 Series ICPMS Mass Hunter Workstation User Guide, Manual Part Number G7201-90403, Rev. A, June 2017
- Code of Federal Regulations Title 40, Part 136.3, Table II
- USEPA. February 2007. SW-846. Chapter Three, Inorganic Analytes, Revision 4
- USEPA. February 2007. Inductively Coupled Plasma-Mass Spectrometry, Method 6020A, Revision 1
- USEPA. June 1999. Compendium Method IO-3.5 Determination of Metals in Ambient Particulate Matter Using Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)
- Inter-Mountain Labs. 2009. IML 2009 Procedure for the Determination of Lead in Ambient Air TSP by Hot Plate Acid Extraction and ICP-MS Analysis, EQL-0310-189
- 40 CFR Part 136 Appendix B. Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11

18.0 REVISION HISTORY

Authorship

Primary Author[1]	Job Title	Date Complete
Mary Slipp	Supervisor	04/07/2022

[1]The primary author is the individual / role responsible for the content of this SOP. Send questions or suggestions for content to the primary author. See the Quality Manager for questions or concerns related to implementation of this SOP.

Revisions Made from Prior Version

Section	Description of Change
All	Conversion to new SOP format

Document Succession: This version replaces the following documents:

Document Number & Version	Document Title	Effective Date:
ENV-SOP-SHRT-0015 v01	Metals by ICPMS	04/30/2022

Appendix A: QC Summary- Method 200.8

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Performance Test	Daily	RSD < 5%	Make necessary changes and rerun performance test.	None
ICAL; five standards minimum	Daily, prior to sample analysis	Correlation coefficient ≥ 0.995; %RE ≤50% for low std; ≤30% for mid-std	Problem must be corrected; no samples may be run until ICAL passes	None
ICV; second source	Immediately after ICAL	90-110%	If ICV is out of acceptance, take corrective action and repeat analysis	None
CCV	After every 10 samples and at end of analytical run	90-110%	If CCV is out of acceptance, take corrective action and repeat analysis	Qualify as needed
Internal Standards	Added online to each analysis	60-125%	Increased dilution may be needed	Qualify as needed
ICB	After ICV	< project detection limits	Correct problem. Reanalyze all samples < 10x the RL	Qualify as needed
CCB	After every CCV	< project detection limits	Correct problem. Reanalyze all samples < 10x the RL	Qualify as needed
Method Blank	1 per batch of 20 or fewer samples	< project detection limits	If MB is above the RL, samples are either reanalyzed or reported with qualifier	Qualify if blank fails
LCS	1 per batch of 20 or fewer samples	85-115%	Correct problem and rerun LCS. If that fails, correct problem and rerun ICAL.	Qualify as needed
Duplicate	1 DUP every 10 samples	RPD ≤ 20%	None	Qualify if DUP fails
MS/MSD	1 MS/MSD set every 10 samples	70-130%: RPD for MSD ≤ 20%	None	Qualify
Linear Dynamic Range Study	Annually	Determinations must be within ± 10% of the stated value	None	None

Appendix B: QC Summary- Method 6020A

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Performance Test	Daily	RSD < 5%		
ICAL five standards minimum	Daily, prior to sample analysis	Correlation coefficient ≥ 0.998; %RE ≤50% for low std; ≤30% for mid-std	Problem must be corrected; no samples may be run until ICAL passes	None
ICV (same as QCS, second source)	Immediately after ICAL	90-110%	Correct problem and rerun ICV. If that fails, correct problem and rerun ICAL.	None
LL ICV (low level ICV)	Beginning of each run	± 30% at lowest calibration level		
CCV	After every 10 samples and at end of analytical run	90-110%	Correct problem and rerun CCV. If that fails, correct problem and rerun ICAL. Reanalyze all samples since last acceptable CCV	Qualify as needed
LL CCV (low level CCV)	End of each run	± 30% of known value		
Internal Standards		≥ 70%		
ICB	After ICV	< project detection limits	Correct problem. Reanalyze all samples < 10x the RL	Qualify as needed
CCB	After every CCV	< project detection limits	Correct problem. Reanalyze all samples < 10x the RL	Qualify as needed
ICSA/ICSAB-Interference Check Solutions	Beginning of each run			
Method Blank	1 per batch of 20 or fewer samples	< project detection limits	If MB is above the RL, samples are either reanalyzed or reported with qualifier	Qualify if blank fails
LLQC	Following establishment of lower RLs, and as needed	± 30% of true value		
LCS	1 per batch of 20 or fewer samples	80-120% For solids, use manufacturer's established limits.	Correct problem and rerun LCS. If that fails, correct problem and rerun ICAL.	Qualify as needed
Duplicate	1 DUP every 20 samples	RPD ≤ 20%	None	Qualify if DUP fails
MS/MSD	1 MS/MSD set every 20 samples	75-125%: RPD for MSD ≤ 20%	If the digested spike fails, a post digestion spike should have recoveries of ±20%. If this spike fails, a dilution test should be run on this sample. If both digested and post digested spikes fail, matrix effects are confirmed	Qualify
Linear Dynamic Range Study	Annually	Determinations must be within ± 10% of the stated value	None	None
IDL- Instrument Detection Limit	(Blanks x7) x3 Quarterly, non-consecutive days	None	None	None

Appendix C: QC Summary- IO-3.5

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Performance Test	Daily	RSD < 5%		
ICAL; five standards minimum	Daily, prior to sample analysis	Correlation coefficient ≥ 0.995; %RE ≤50% for low std; ≤30% for mid-std	Problem must be corrected; no samples may be run until ICAL passes	None
ICV (same as QCS, second source)	Immediately after ICAL	90-110%	Correct problem and rerun ICV. If that fails, correct problem and rerun ICAL.	None
CCV	After every 10 samples and at end of analytical run	90-110%	Correct problem and rerun CCV. If that fails, correct problem and rerun ICAL. Reanalyze all samples since last acceptable CCV	Qualify as needed
Internal Standards		60-125%		
ICB	After ICV	< project detection limits	Correct problem. Reanalyze all samples < 10x the RL	Qualify as needed
CCB	After every CCV	< project detection limits	Correct problem. Reanalyze all samples < 10x the RL	Qualify as needed
HSV- High Standard Verification	Beginning of each run	95-105%		
ICSAB- Interference Check Standard	Beginning and end of each run	80-120%		Qualify as needed
Reagent Blank				
Method Blank (equal to Filter Blank)	1 per batch of 20 or fewer samples	< RL	If MB is above the RL, samples are either reanalyzed or reported with qualifier	Qualify is blank fails
LCS	1 per batch of 20 or fewer samples	80-120%	Correct problem and rerun LCS. If that fails, correct problem and rerun ICAL.	Qualify as needed
Duplicate	1 DUP every 20 samples	RPD ≤ 20%	None	Qualify if DUP fails
MS	1 MS every 20 samples	75-125%	None	Qualify
Linear Dynamic Range Study	Annually	Determinations must be within ± 10% of the stated value	None	None
Serial Dilution	1 per 20 or fewer samples	90-110%		Qualify as needed

Appendix D: QC Summary- FEM 2009 (EQL-0310-189)

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Performance Test	Daily	RSD < 5%		
ICAL; five standards minimum	Daily, prior to sample analysis	Correlation coefficient ≥ 0.995; %RE ≤50% for low std; ≤30% for mid-std	Problem must be corrected; no samples may be run until ICAL passes	None
Calibration readbacks	Beginning of each run	± 30% for lowest standard; ± 10% for all other standards		
ICV (same as QCS, second source)	Immediately after ICAL	90-110%	Correct problem and rerun ICV. If that fails, correct problem and rerun ICAL.	None
CCV	After every 10 samples and at end of analytical run	90-110%	Correct problem and rerun CCV. If that fails, correct problem and rerun ICAL. Reanalyze all samples since last acceptable CCV	Qualify as needed
LLOQ	Beginning of each run	± 50% at lowest RL concentration		
Internal Standards		60-125%		
ICB	After ICV	< project detection limits	Correct problem. Reanalyze all samples < 10x the RL	Qualify as needed
CCB	After every CCV	< project detection limits	Correct problem. Reanalyze all samples < 10x the RL	Qualify as needed
HSV- High Standard Verification	Beginning of each run	95-105%		
Interference Check Standard	Beginning and end of each run	80-120%		
Method Blank (equal to Filter Blank)	1 per batch of 20 or fewer samples	< RL	If MB is above the RL, samples are either reanalyzed or reported with qualifier	Qualify if blank fails
LCS	1 per batch of 20 or fewer samples	80-120%	Correct problem and rerun LCS. If that fails, correct problem and rerun ICAL.	Qualify as needed
Duplicate	1 DUP every 20 samples	RPD ≤ 20%	None	Qualify if DUP fails
MS	1 MS every 20 samples	75-125%	None	Qualify
Linear Dynamic Range Study	Annually	Determinations must be within ± 10% of the stated value	None	None
Serial Dilution	1 per 20 or fewer samples	90-110%		

Table 1: Common Polyatomic Ion Interferences in ICPMS

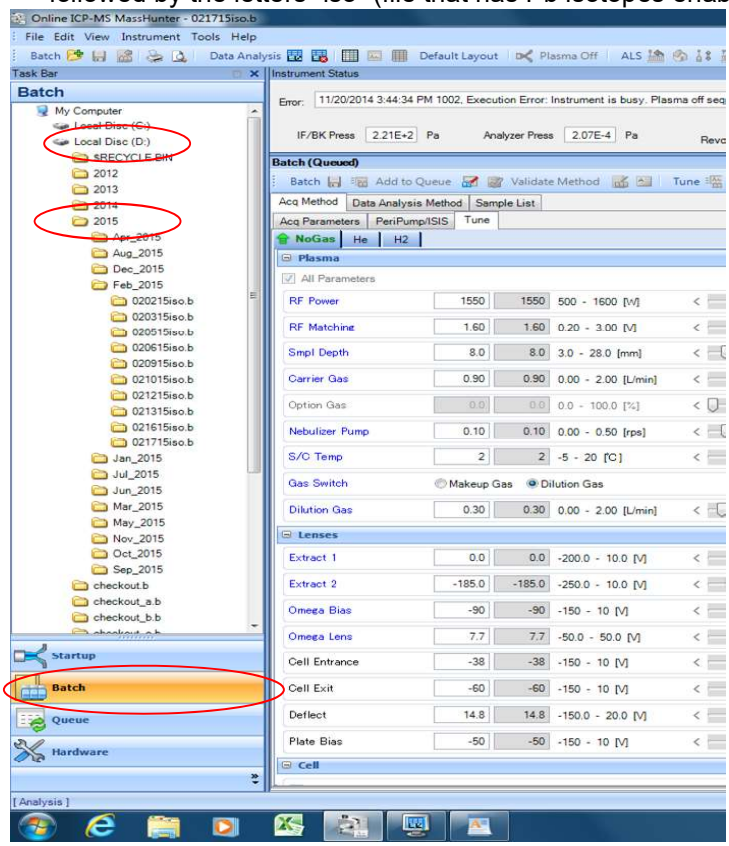
Background Molecular Ions		
Molecular Ion	**Mass**	**Element Interference**
NH^+	15	
OH^+	17	
OH_2^+	18	
C_2^+	24	
CN^+	26	
CO^+	28	
N_2^+	28	
N_2H^+	29	
NO^+	30	
NOH^+	31	
O_2^+	32	
O_2H^+	33	
$^{36}ArH^+$	37	
$^{38}ArH^+$	39	
$^{40}ArH^+$	41	
CO_2^+	44	
CO_2H^+	45	Sc
ArC^+, ArO^+	52	Cr
ArN^+	54	Cr
$ArNH^+$	55	Mn
ArO^+	56	
$ArOH^+$	57	
$^{40}Ar^{36}Ar^+$	76	Se
$^{40}Ar^{38}Ar^+$	78	Se
$^{40}Ar_2^+$	80	Se

Table 2: Recommended Isotopes

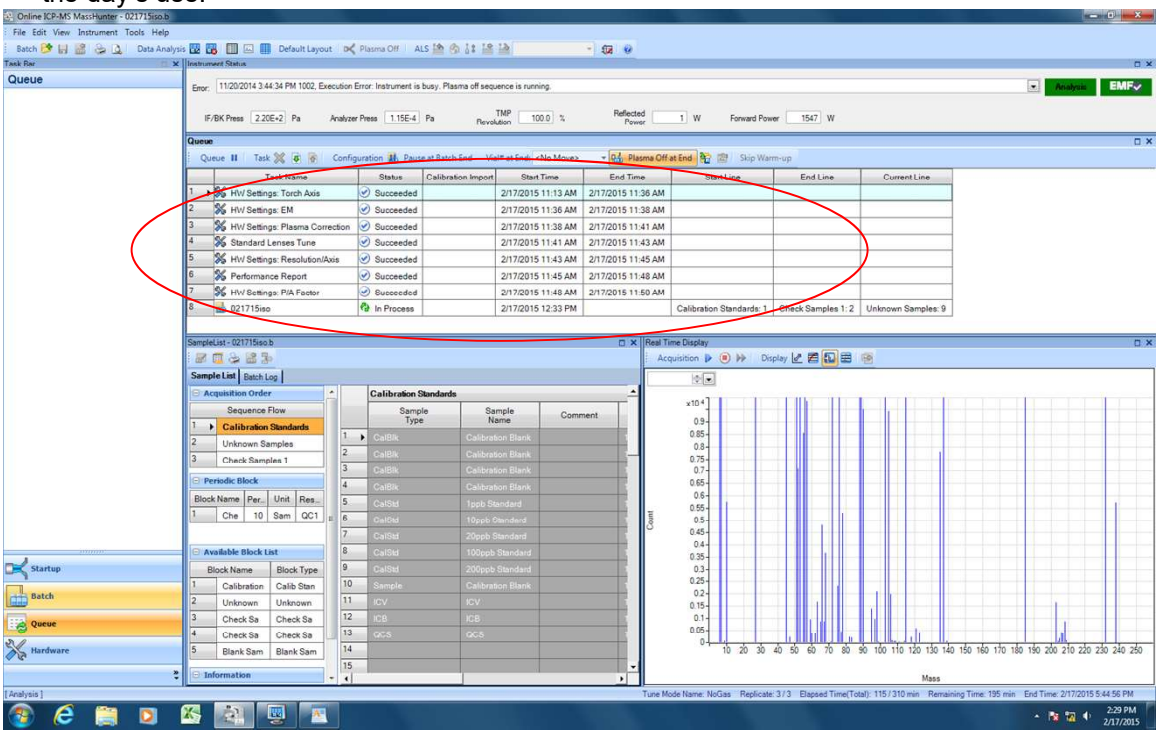
Recommended Analytical Isotopes *				
Element	**EPA 200.8**	**6020A**	**IO-3.5**	**FEM 2009**
Aluminum	**27**	**27**	**27**	
Antimony	121, **123**	121, **123**	**121**, 123	
Arsenic	**75**	**75**	**75**	
Barium	135, **137**	138, 137, 136, **135**, 134	135, **137**	
Beryllium	**9**	**9**	**9**	
Bismuth		**209**		
Cadmium	106, 108, **111**, 114	**114**, 112, **111**, 110, 113, 116, 106	106, 108, **111**, 114	
Chromium	**52**, 53	**52, 53, 50**, 54	**52**, 53	
Cobalt	**59**	**59**	**59**	
Copper	**63**, 65	**63**, 65	**63**, 65	
Lead	**206**, **207**, **208**	**206**, **207**, **208**, 204	**206**, **207**, **208**	**206**, **207**, **208**
Manganese	**55**	**55**	**55**	
Molybdenum	95, 97, **98**	98, 96, 92, **97**, 94	95, 97, **98**	
Nickel	**60**, 62	58, **60**, 62, **61**, 64	**60**, 62	
Selenium	77, **82**	80, **78**, **82**, **76**, **77**, 74	**77**, **82**	
Silver	**107**, 109	**107**, **109**	**107**, 109	
Thallium	203, **205**	203, **205**	203, **205**	
Thorium	**232**		**232**	
Tin	118	120, **118**	118	
Uranium	**238**		**238**	
Vanadium	**51**	**51**, **50**	**51**	
Zinc	**66**, 67, 68	64, **66**, **68**, **67**, 70	**66**, 67, 68	
Krypton	**83**			
*Isotopes recommended for analytical determination are bolded				
*Drinking Water analysis does not allow the use of gas mode determinations				
*Certain matrices may require the use of alternative isotopes				
*A recommendation is not a limitation				

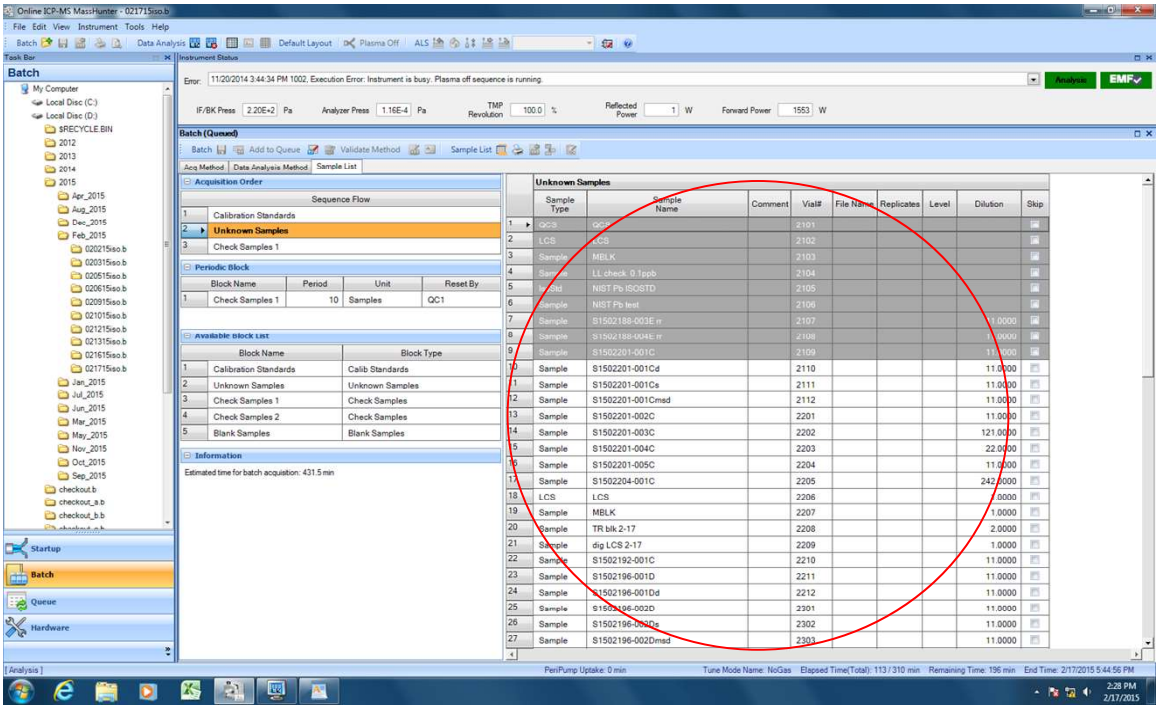
Appendix E: Step by Step Operation Instructions for Agilent 7700 ICPMS

1. Open “ICP-MS Instrument” icon.
2. Create an operating file for the day. Open the last existing batch file. Go to “File”, “Save Batch As”, save new batch in the “D:” drive in the appropriate year and month giving it the current date followed by the letters “iso” (file that has Pb isotopes enabled).

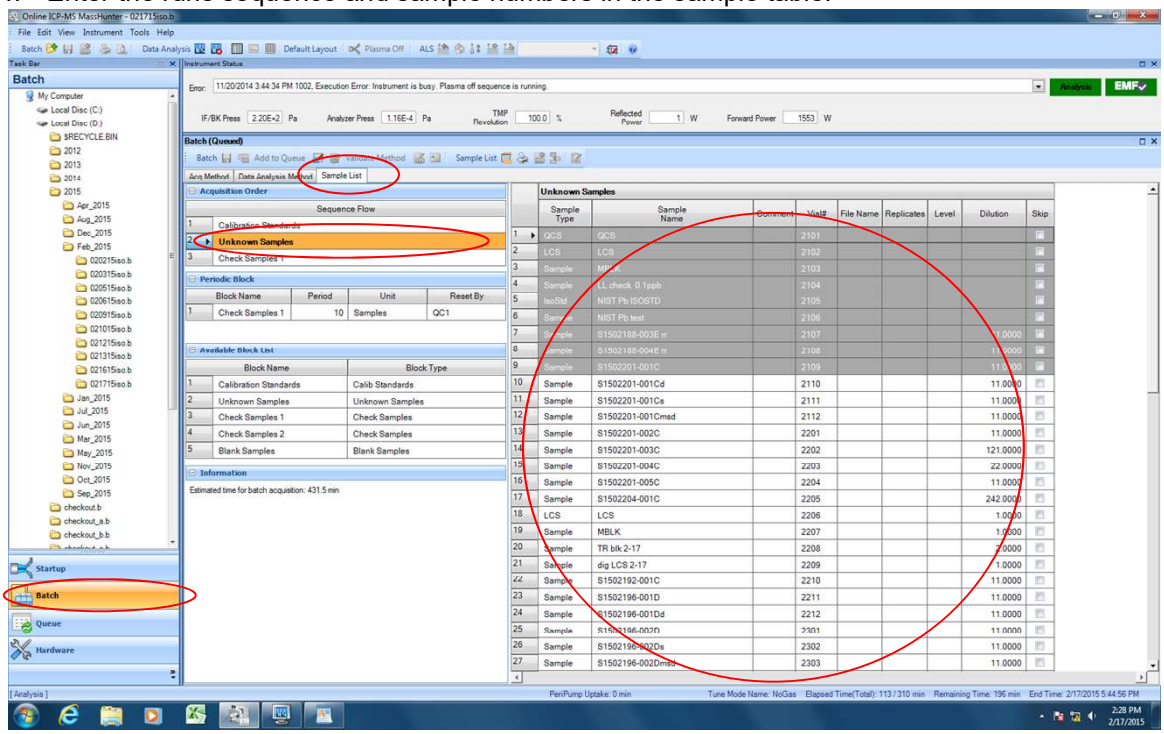


3. Highlight and delete the data from the queue and the sample list to create an empty template for the day's use.

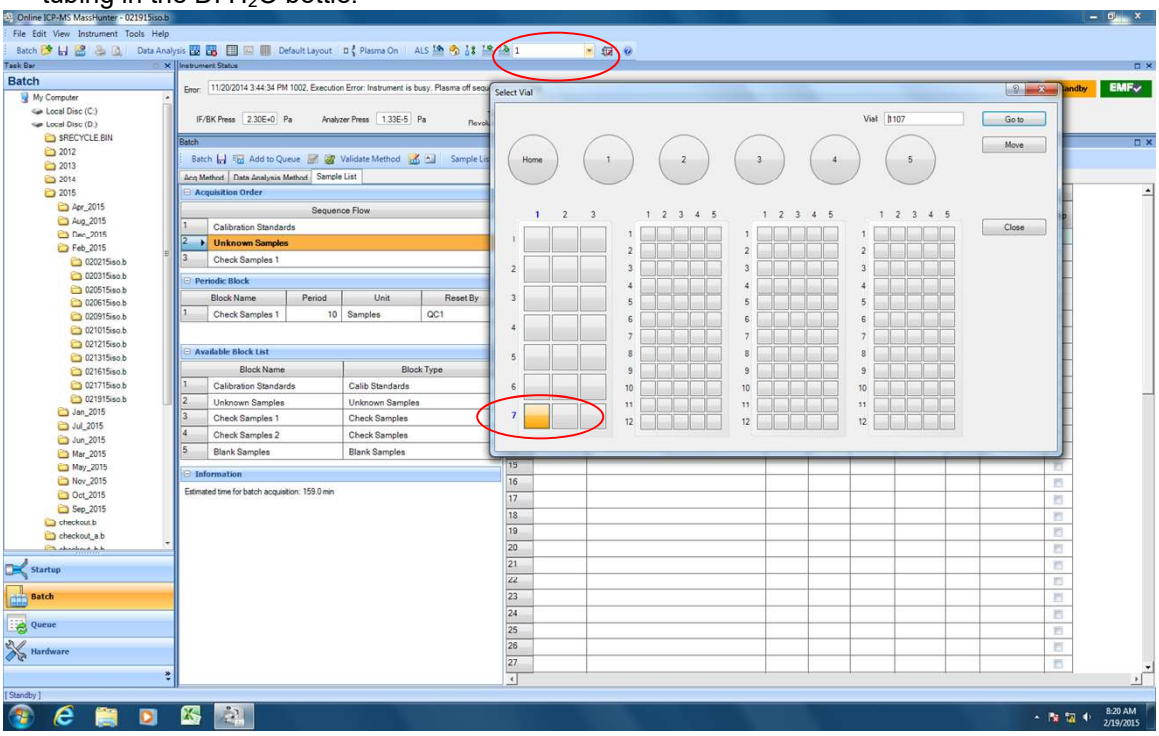




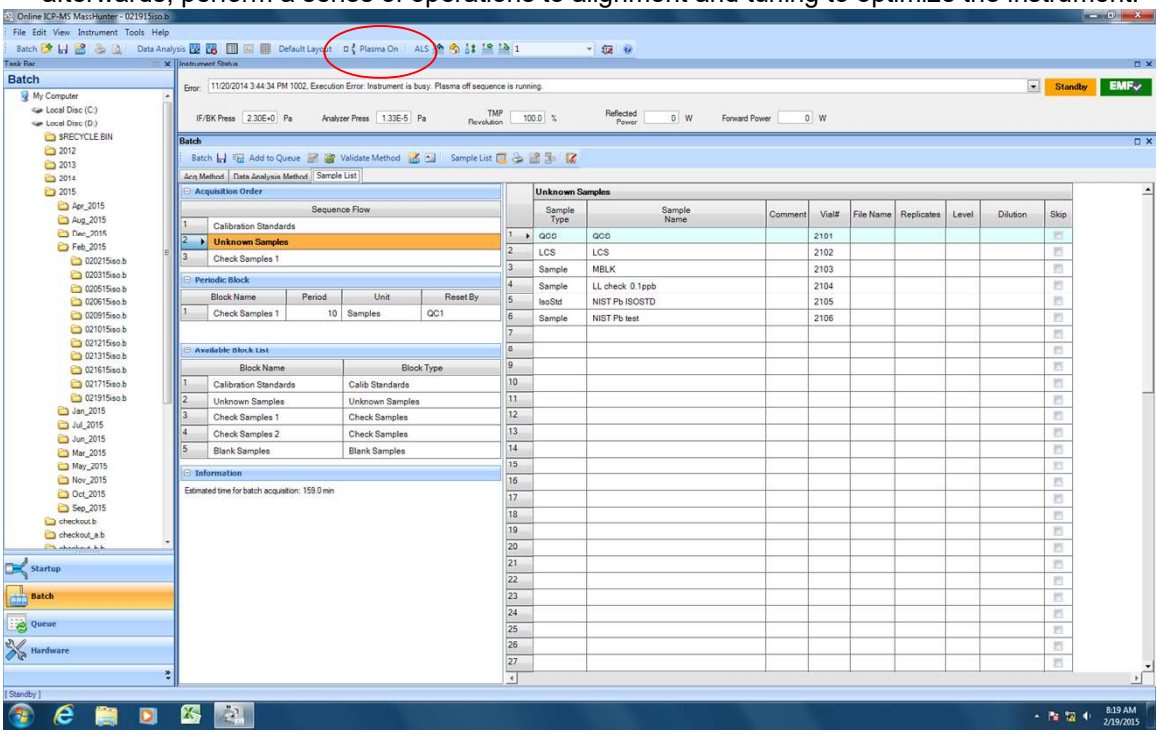
4. Enter the runs sequence and sample numbers in the sample table.



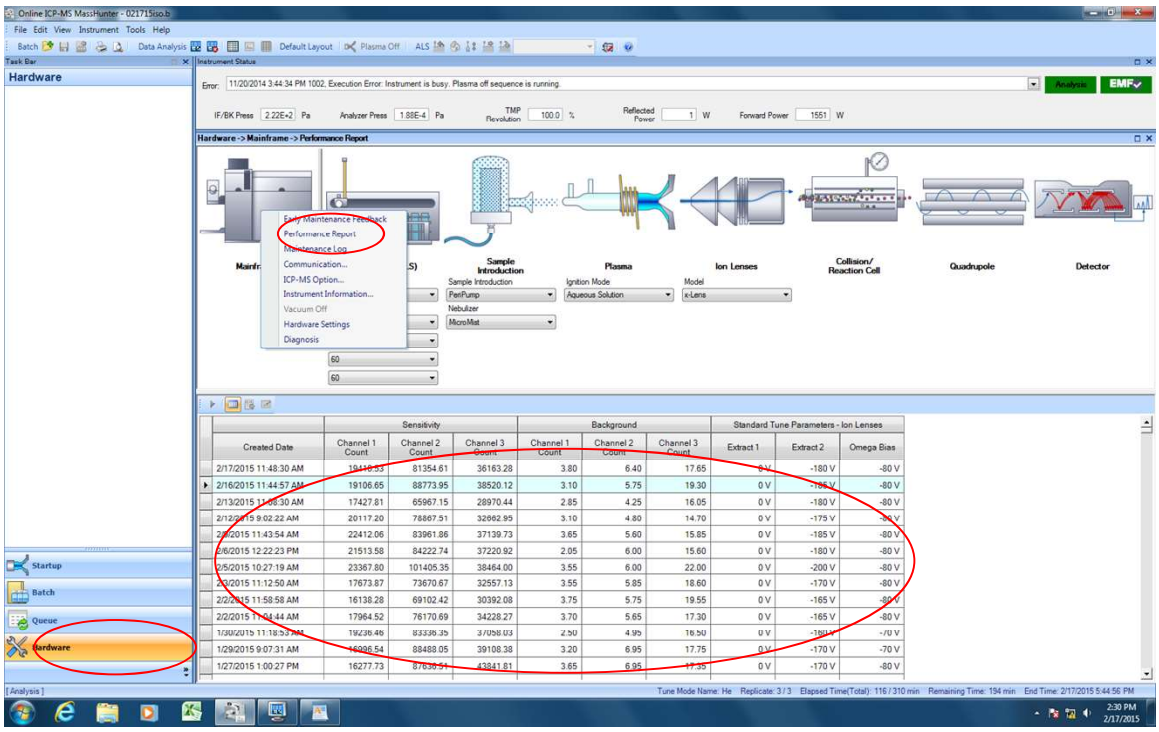
5. Prepare the instrument for startup and warm-up. Put new pump tubing on daily. Fill all rinse solutions, internal standard solution container, and empty waste. Pour fresh blank and 20ppb standards daily and all standards are poured fresh weekly. Open the 1ppb tuning solution, 10ppb tuning solution, and the PA solution vials. Direct the auto-sampler probe to go to the 1ppb tuning solution by using the dropdown box in the “Select ALS Vial #” box. Put the internal standard tubing in the DI H_2O bottle.

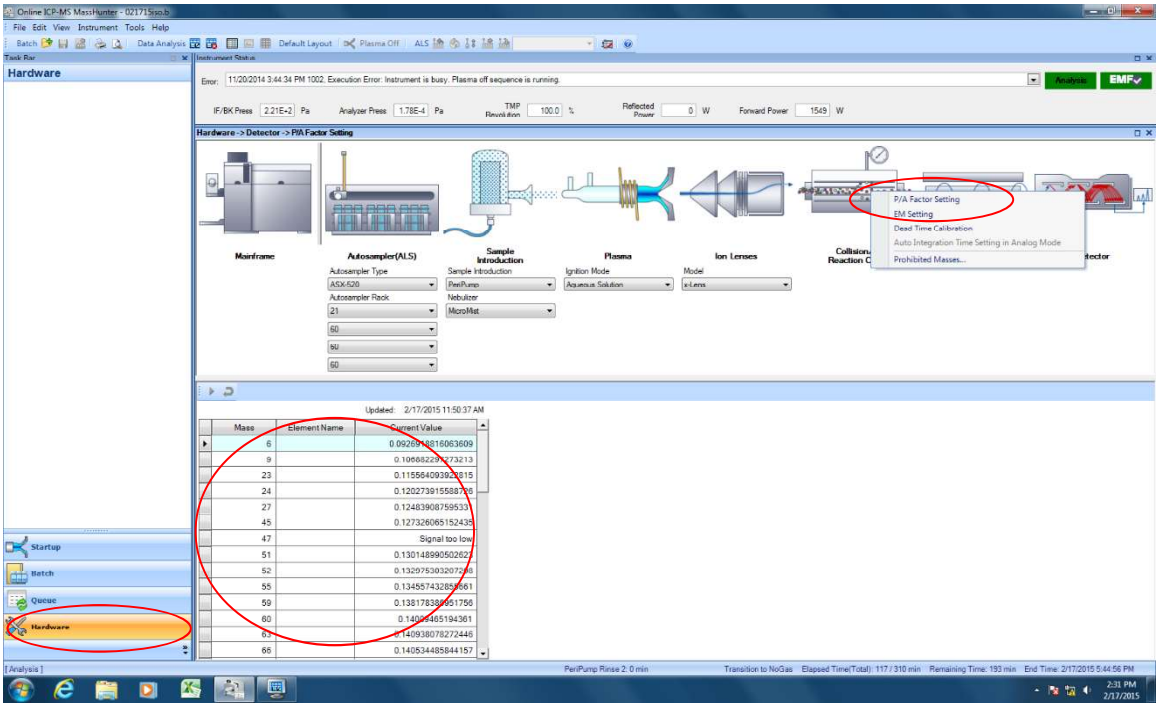


6. Click the “Plasma On” icon. The instrument software will warm up the instrument, and afterwards, perform a series of operations to alignment and tuning to optimize the instrument.

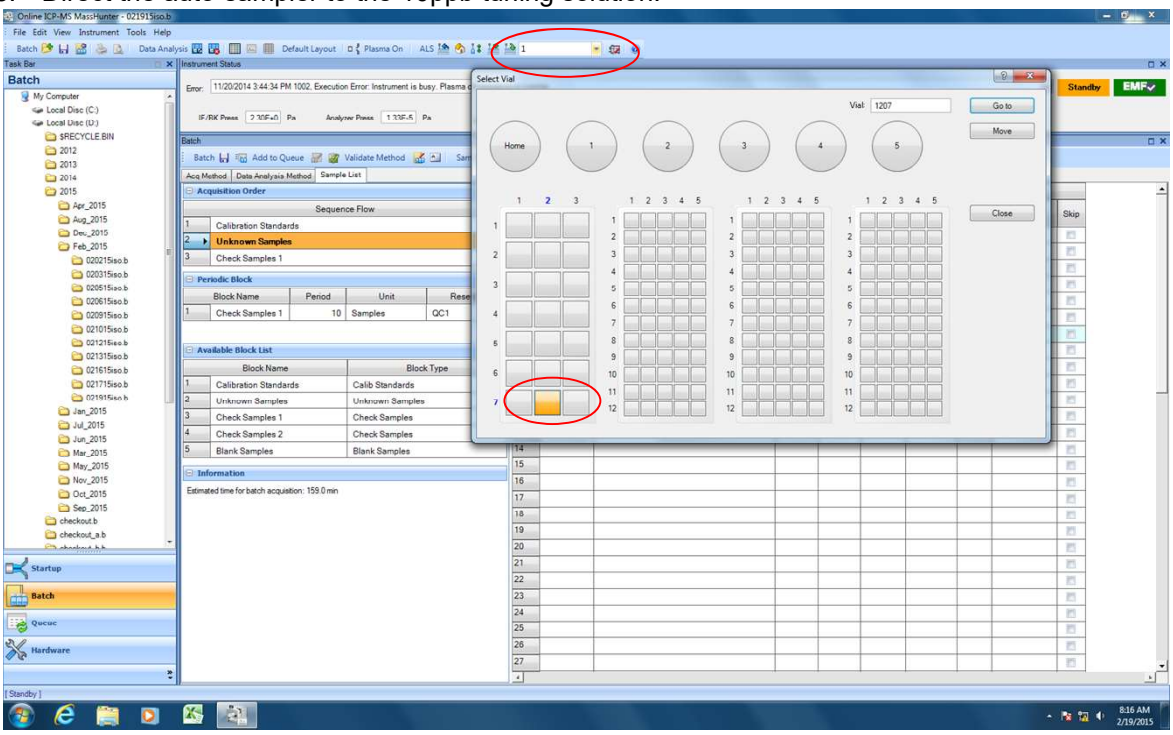


7. Check the “Performance Report” to make sure the sensitivity looks appropriate and similar to previous determinations. To access this report, go to the “Hardware” section and right click on the “Mainframe”. A list will appear so that you may select “Performance Report”. Check the P/A Report. To access this report, go to the “Hardware” section and right click on the “Detector”. A list will appear so that you may select “P/A Factor Setting”. Ensure that most masses have a P/A factor.

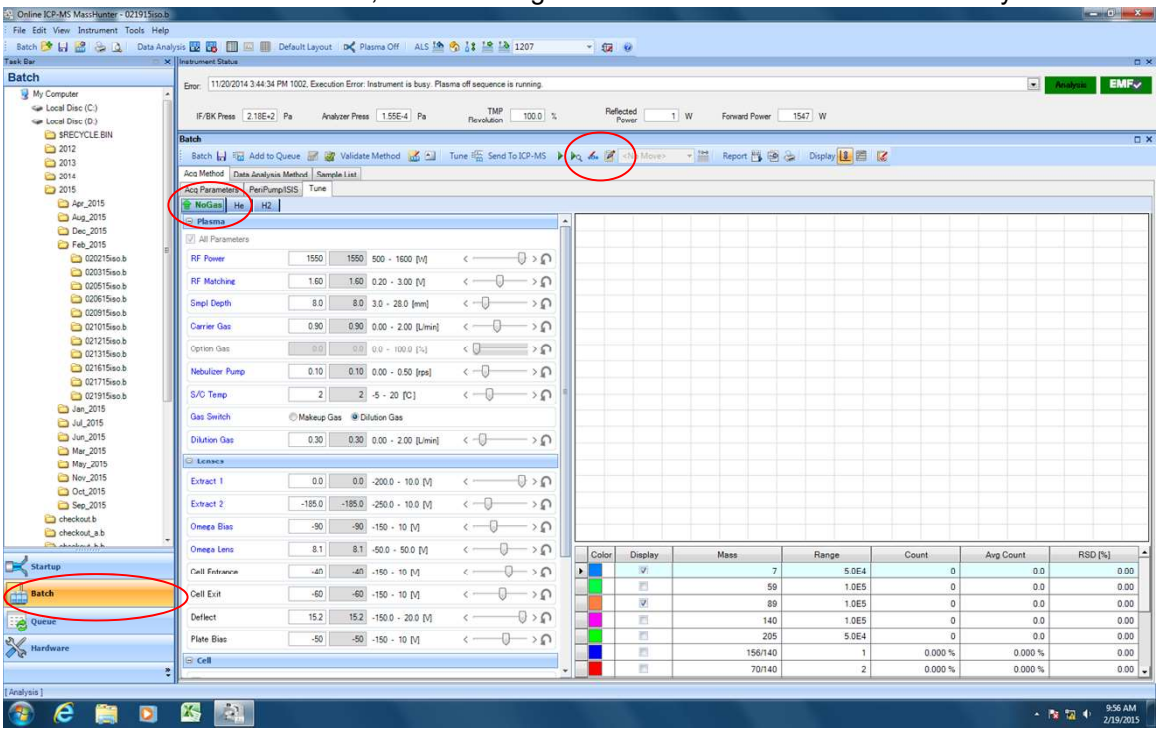


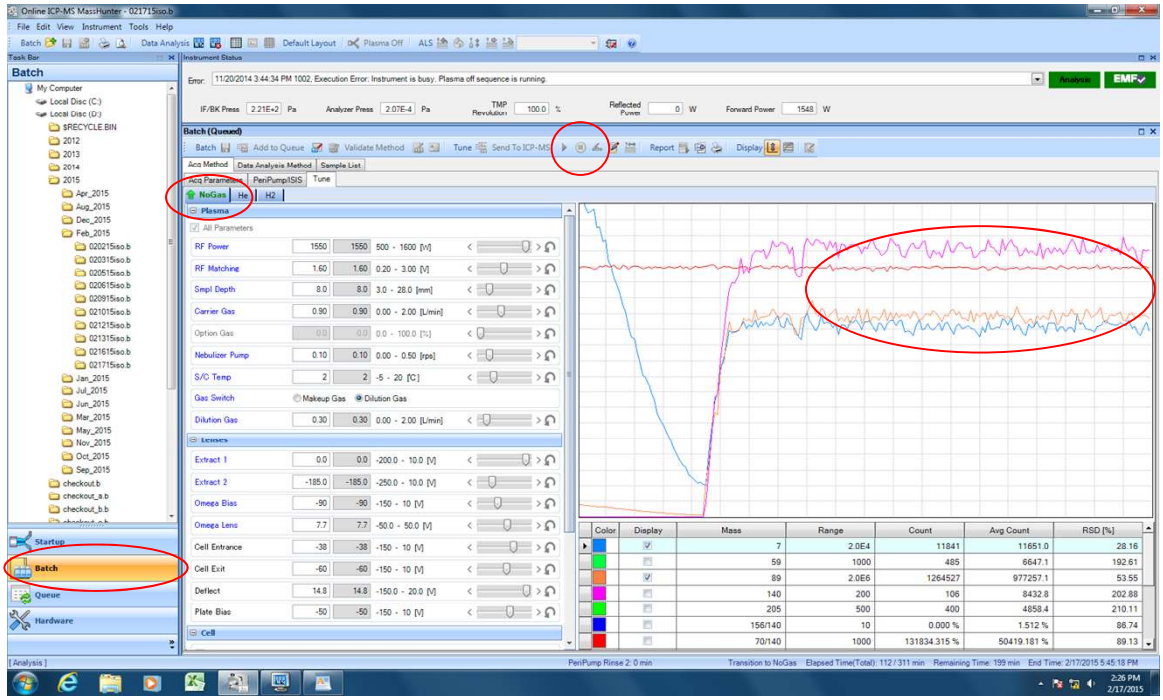

Hardware
Hardware -> Detector -> P/A Factor Setting
Mainframe
Autosampler(ALS)
Sample Introduction
Plasma
Ion Lenses
P/A Factor Setting
EM Setting
Dead Time Calibration
Auto Integration Time Setting in Analog Mode
Prohibited Masses...
Mass
Element Name
Current Value
Signal too low
Startup
Batch
Queue
Hardware

8. Direct the auto-sampler to the 10ppb tuning solution.

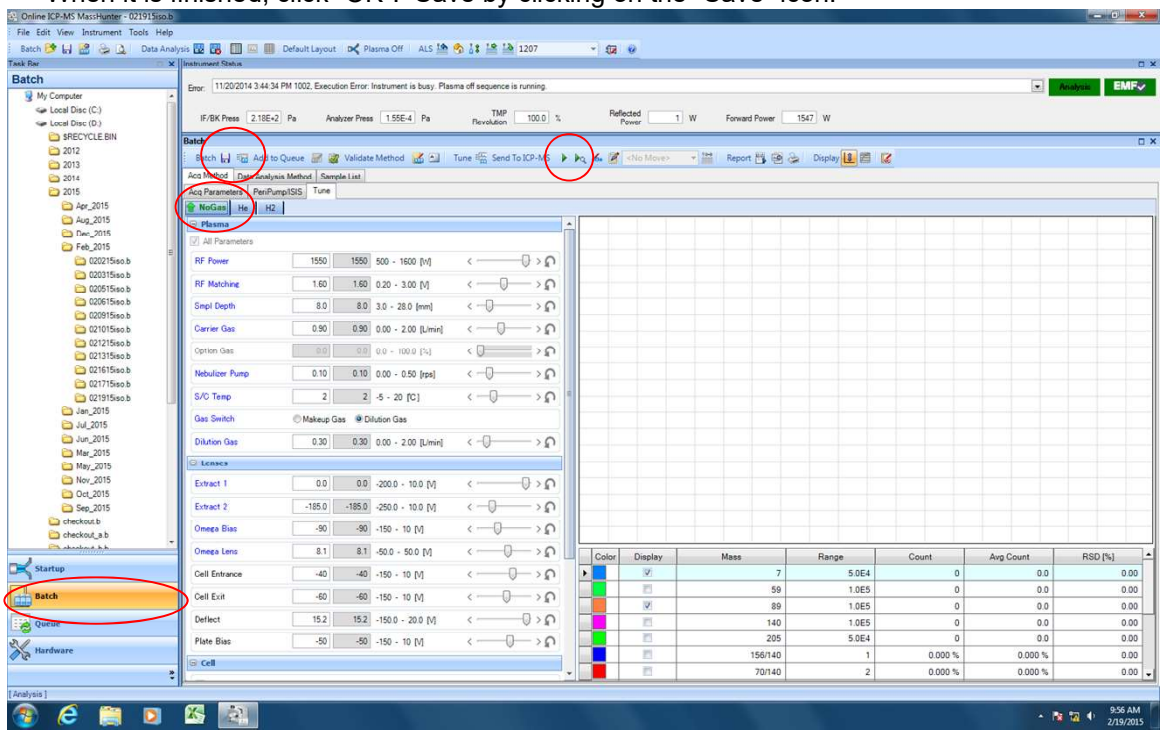


9. While in the “No Gas Mode”, enter the “Signal Monitor” mode to check for a steady state.

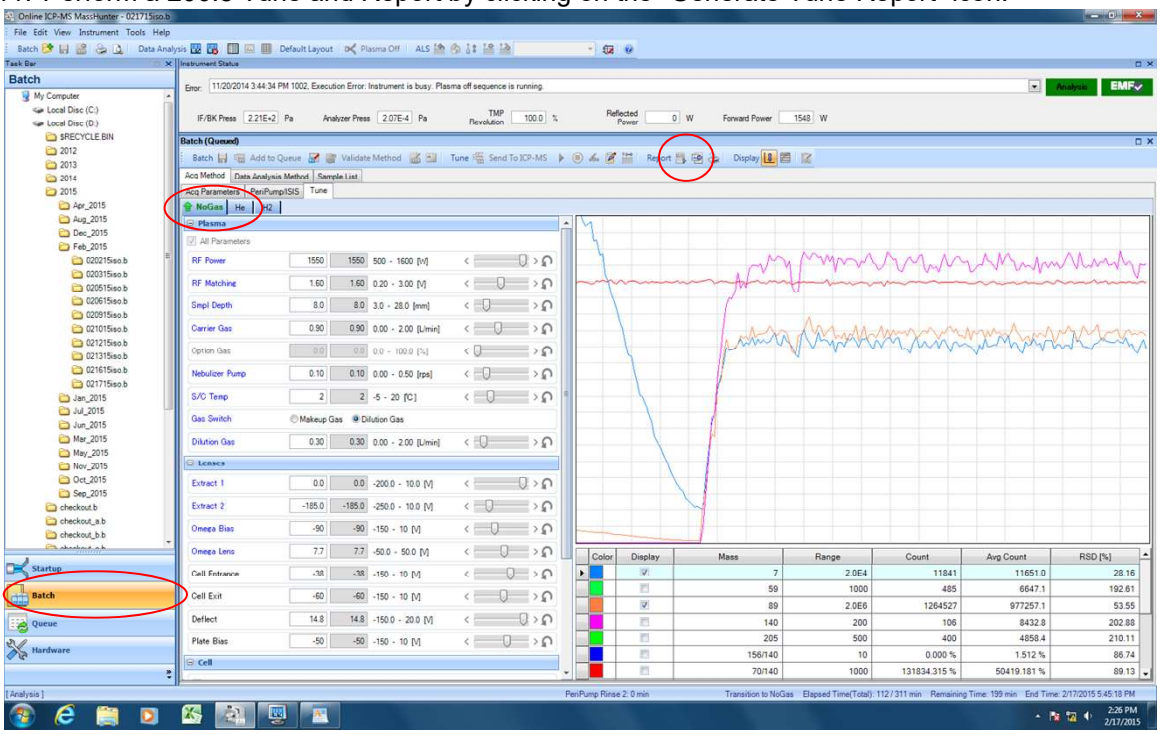




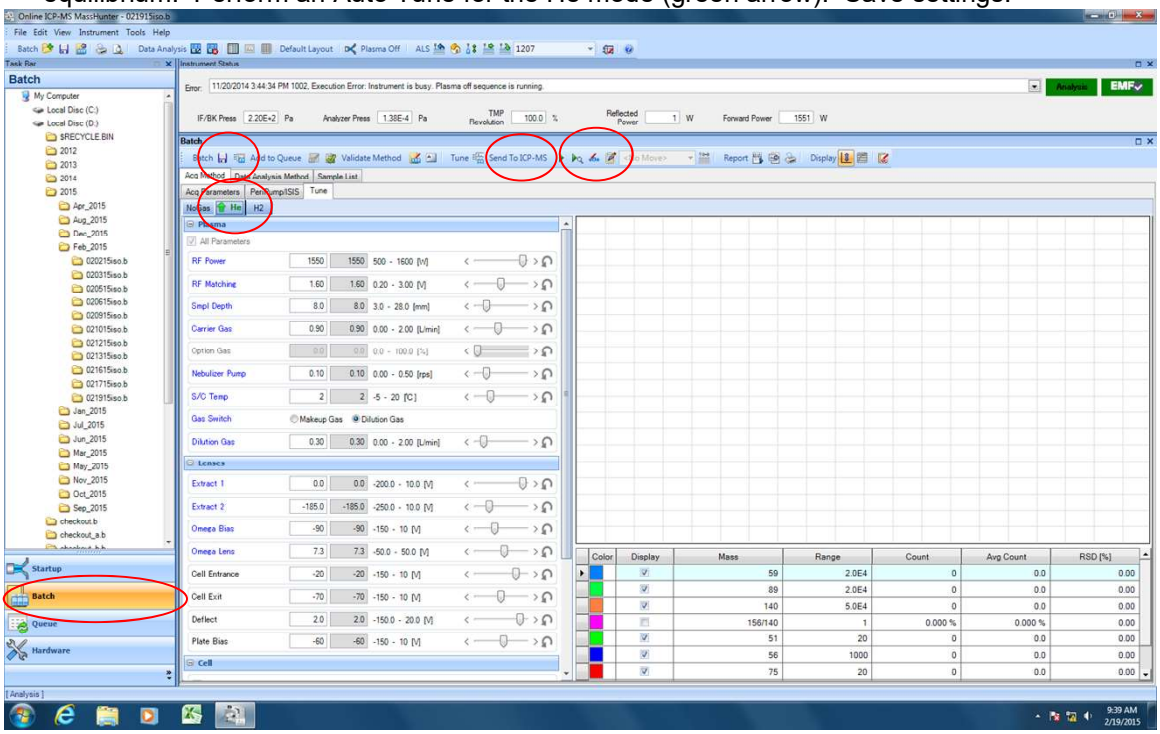
10. Perform an Auto Tune for the “No Gas Mode”. Click the green arrow icon to “Start Auto Tune”. When it is finished, click “OK”. Save by clicking on the “Save” icon.



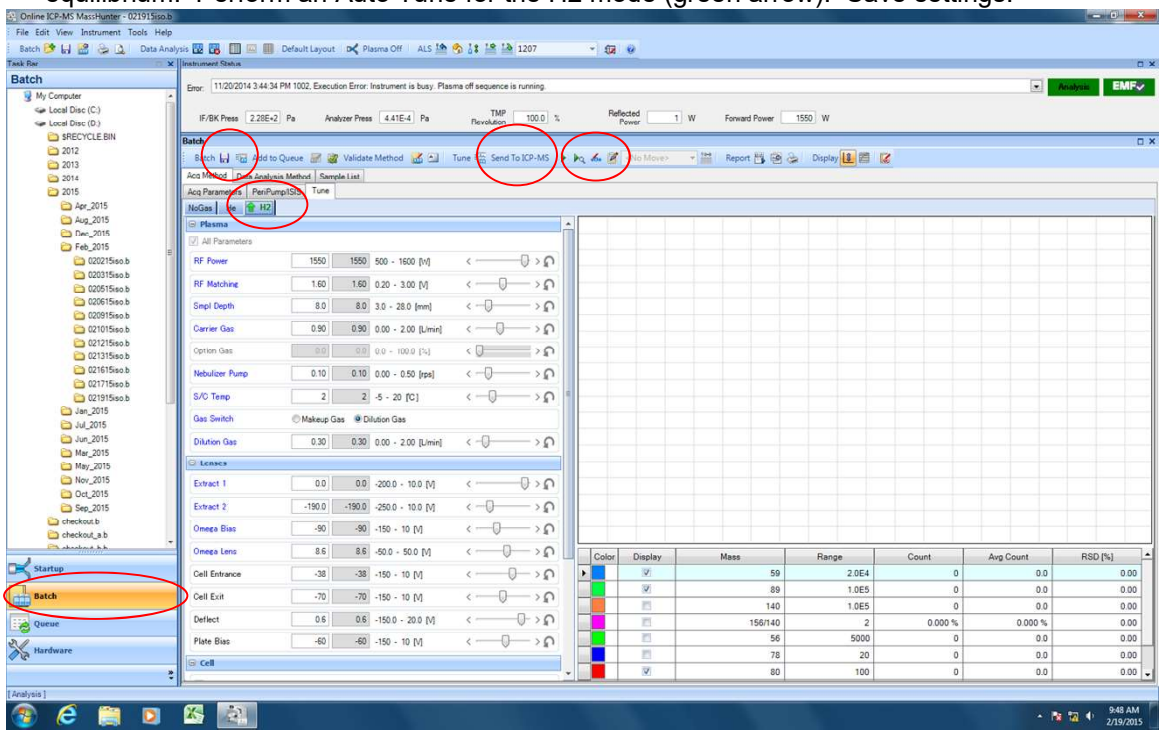
11. Perform a 200.8 Tune and Report by clicking on the “Generate Tune Report” icon.



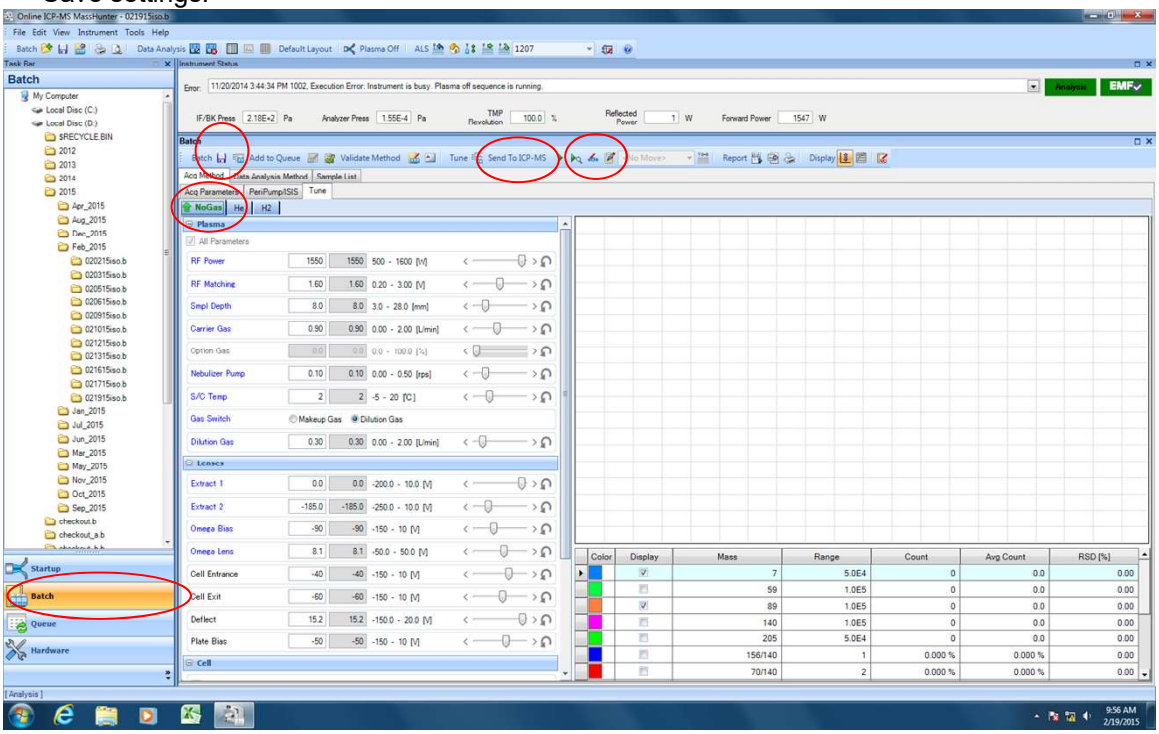
12. Click on the He tab. Click “Send to ICPMS”. Click “Signal Monitor” to let the He mode come to equilibrium. Perform an Auto Tune for the He mode (green arrow). Save settings.



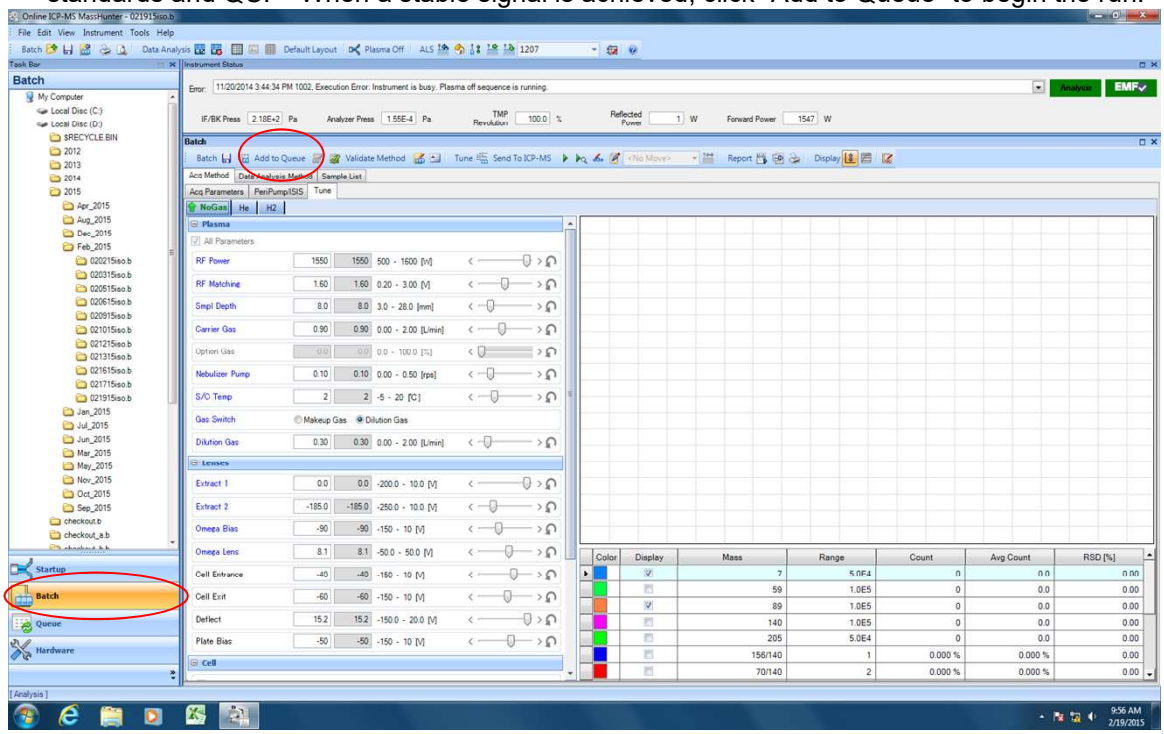
13. Click the H2 tab. Click “Send to ICPMS.” Click “Signal Monitor” to let the H2 mode come to equilibrium. Perform an Auto Tune for the H2 mode (green arrow). Save settings.



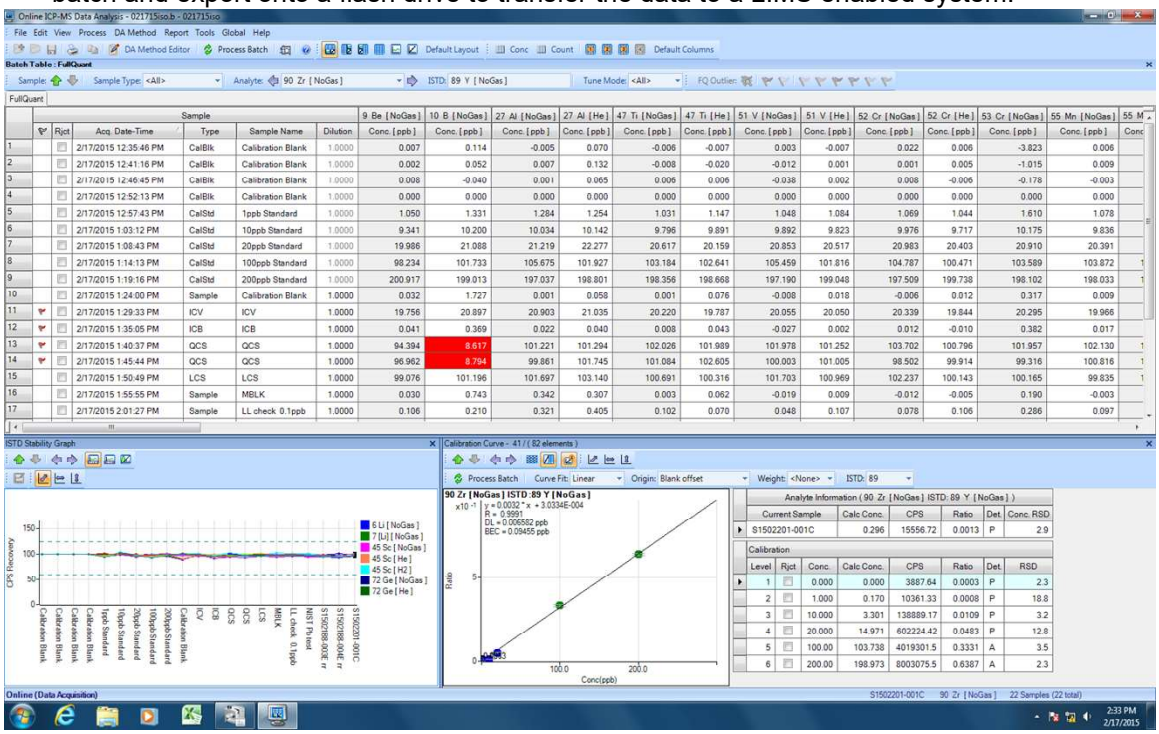
14. Click the No Gas mode. Click “Send to ICPMS”. Redo the Auto Tune for the No Gas mode. Save settings.

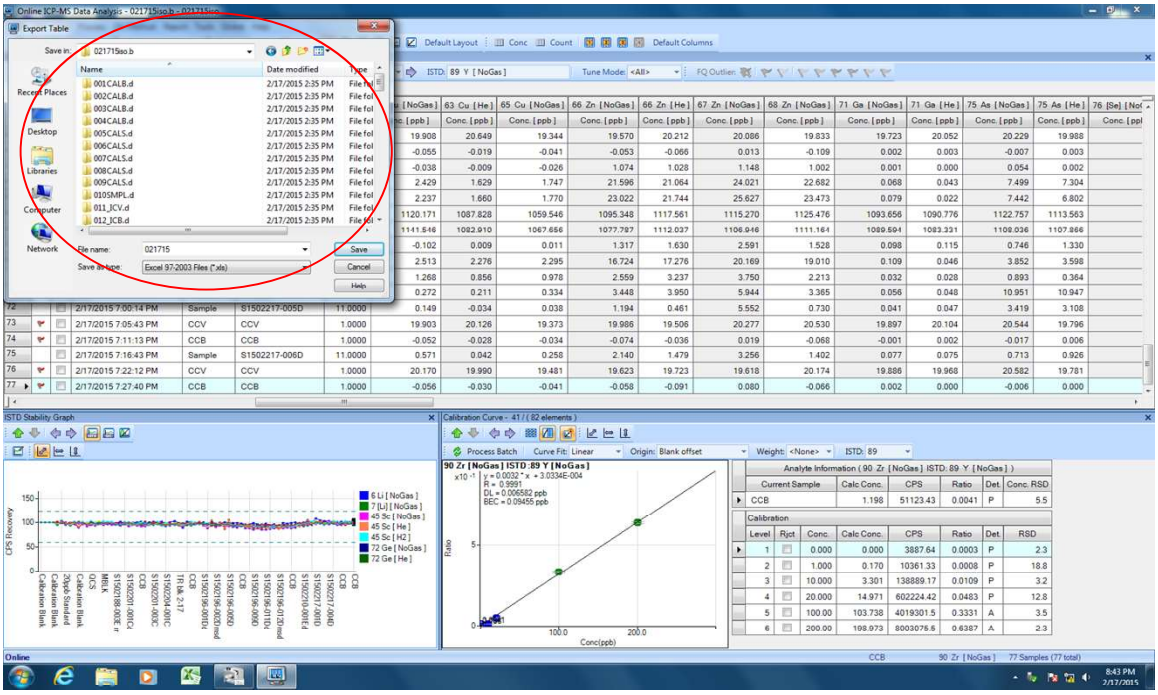


15. Direct the auto-sampler probe to the rinse position 1. Put the internal standard tubing into the internal standard. Click “Signal Monitor” and watch graph for a stable signal. Uncap all standards and QC. When a stable signal is achieved, click “Add to Queue” to begin the run.



16. After the run is complete, check all QC for acceptability. Export the data by going to "File" then "Export Table". Name the file and change the XLSX file default to XLS. Save the file in the day's batch and export onto a flash drive to transfer the data to a LIMS enabled system.

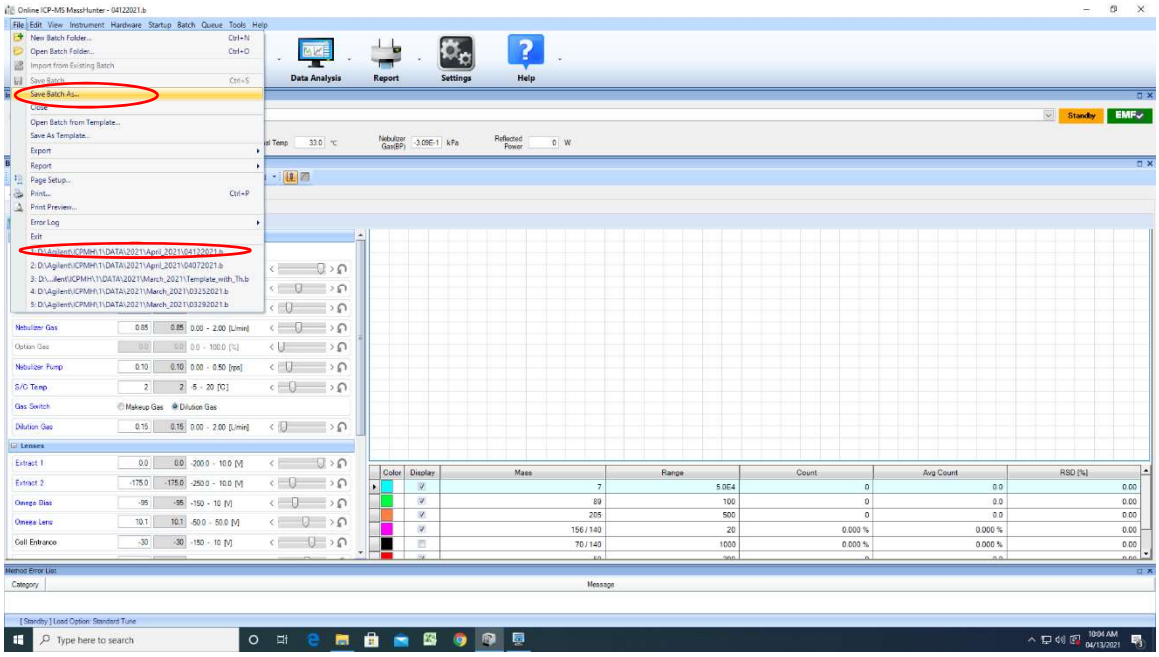


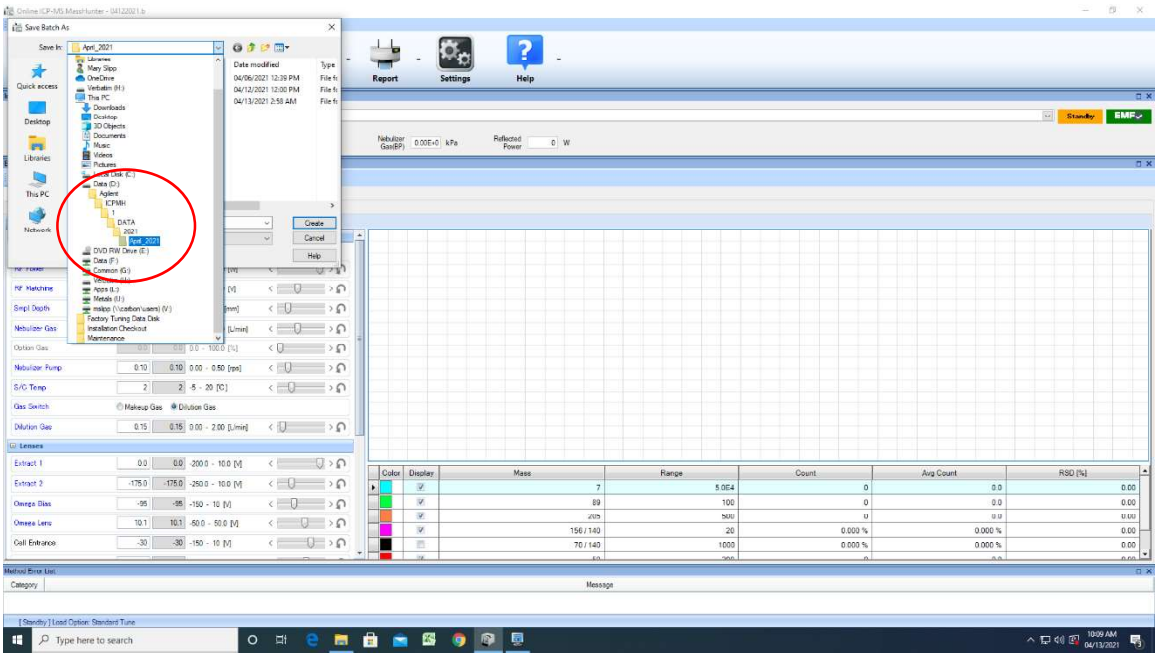


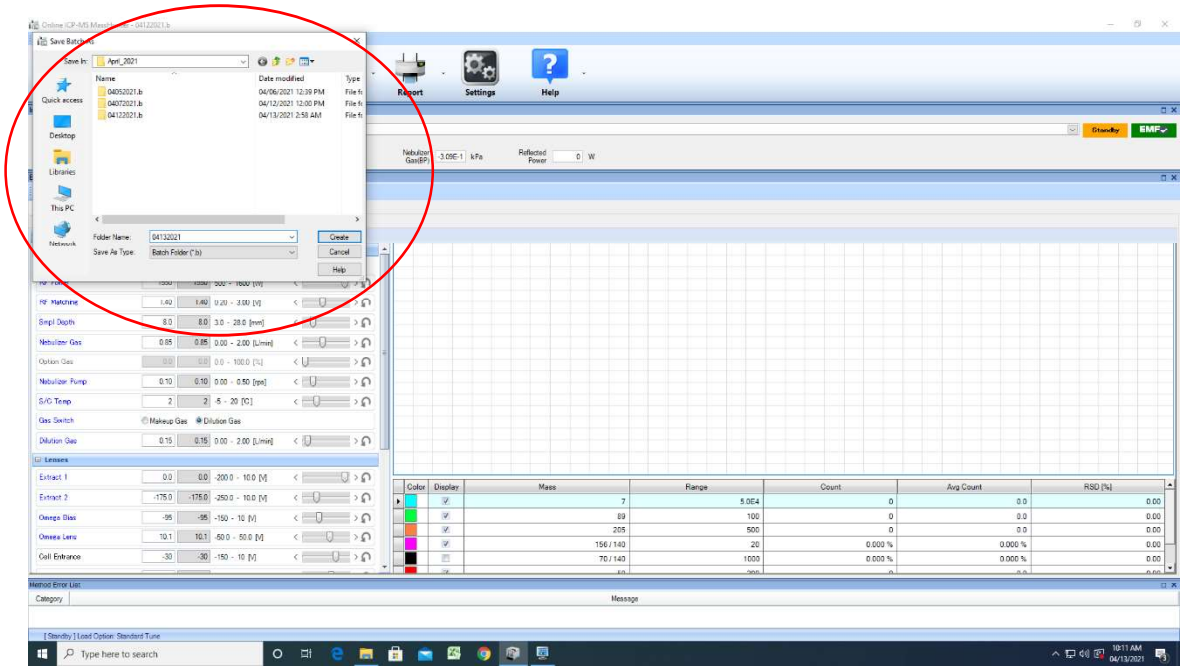
Appendix F: Step by Step Operation Instructions for Agilent 7800 ICPMS

1. Open “ICP-MS Instrument” icon.

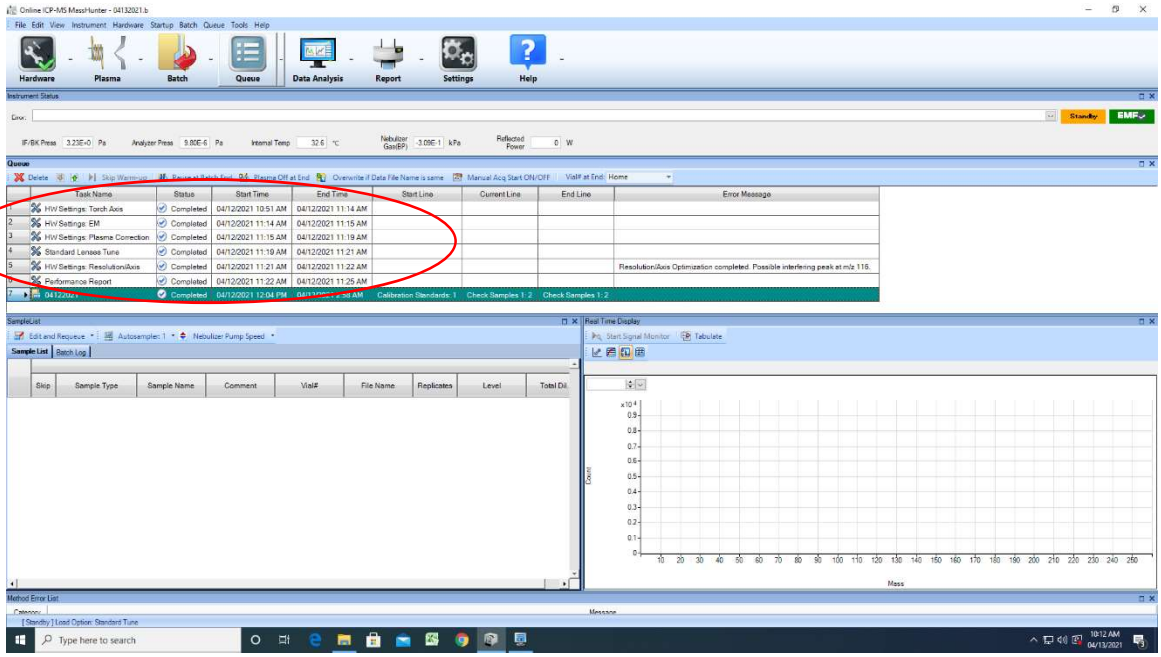
2. Create an operating file for the day. Open the last existing batch file. Go to “File”, “Save Batch As”, save new batch in the “D:” drive in the appropriate month, day, and year giving it the current date.

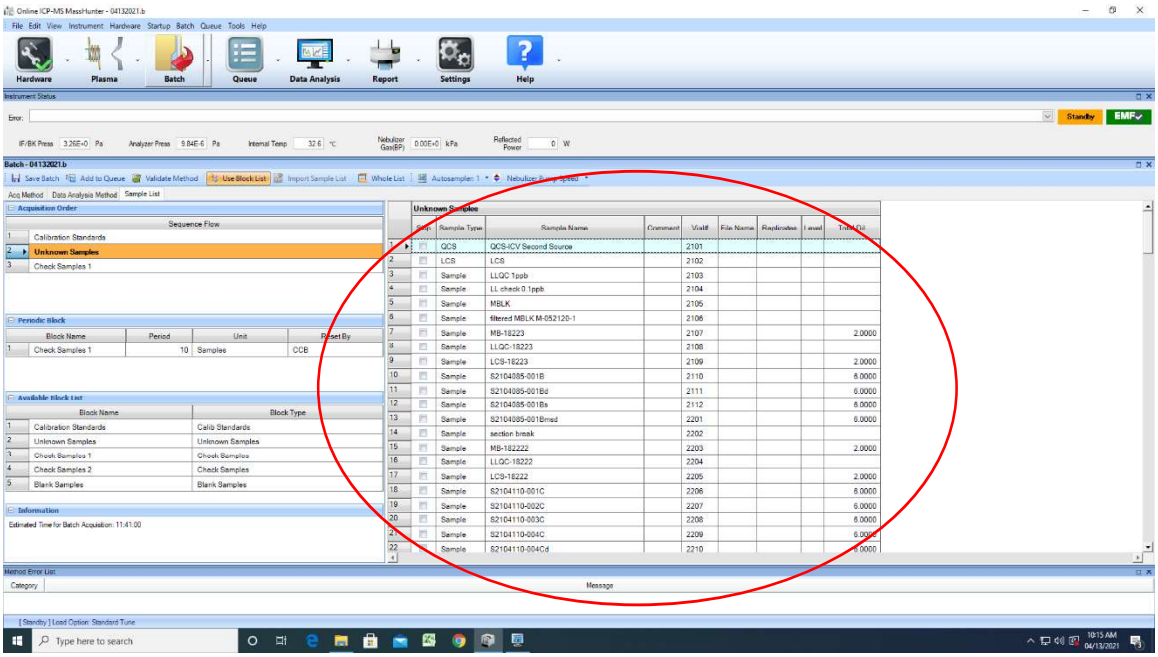




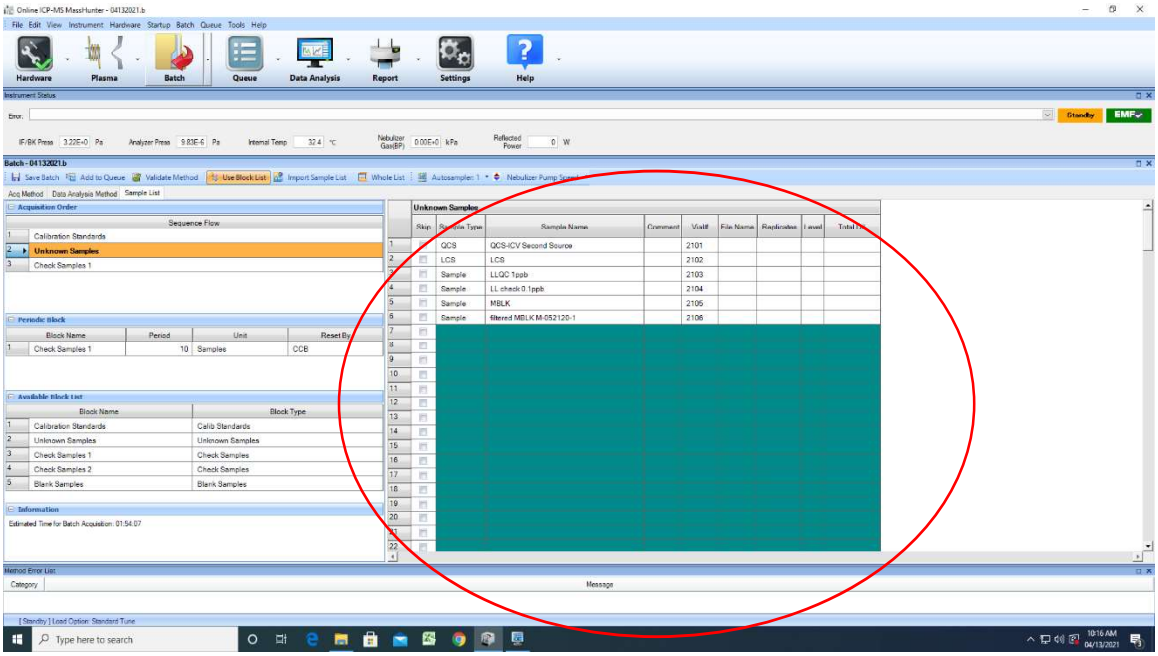


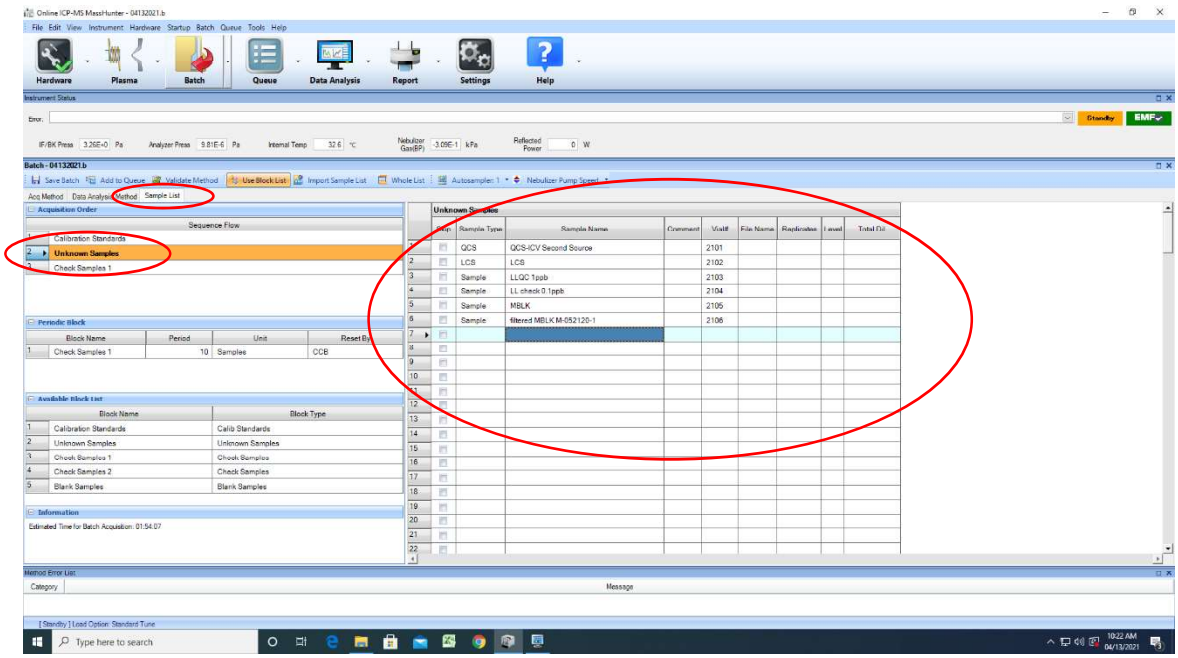
3. Highlight and delete the data from the queue and the sample list to create a clean template for the day's use.



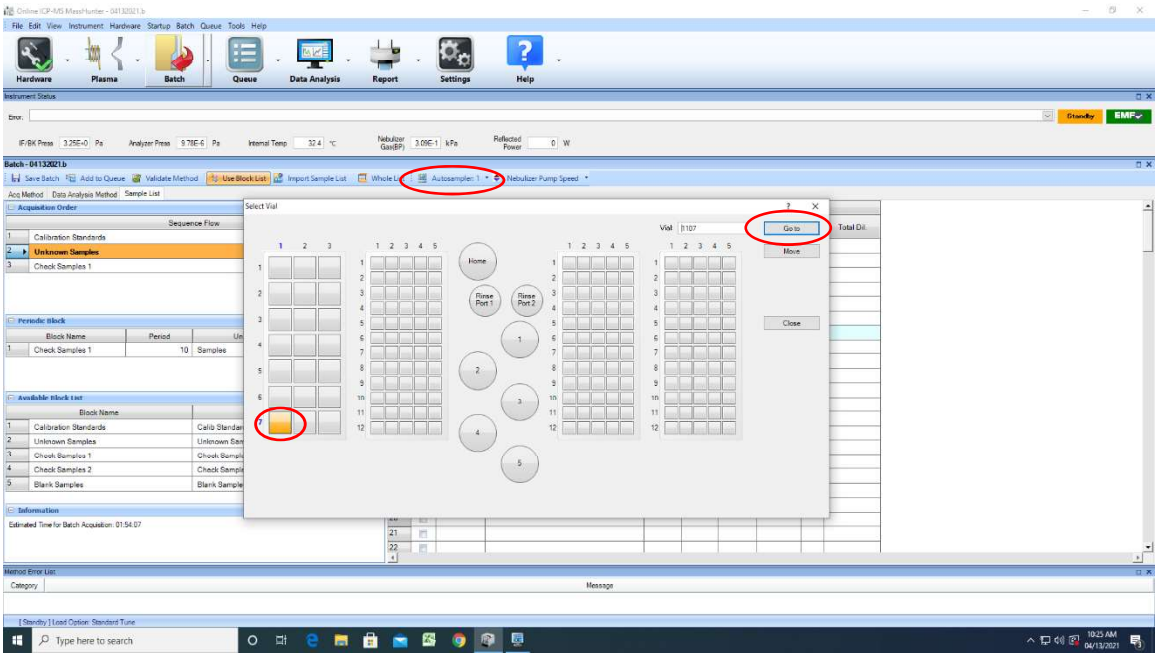


4. Enter the run's sequence and sample numbers in the sample table.

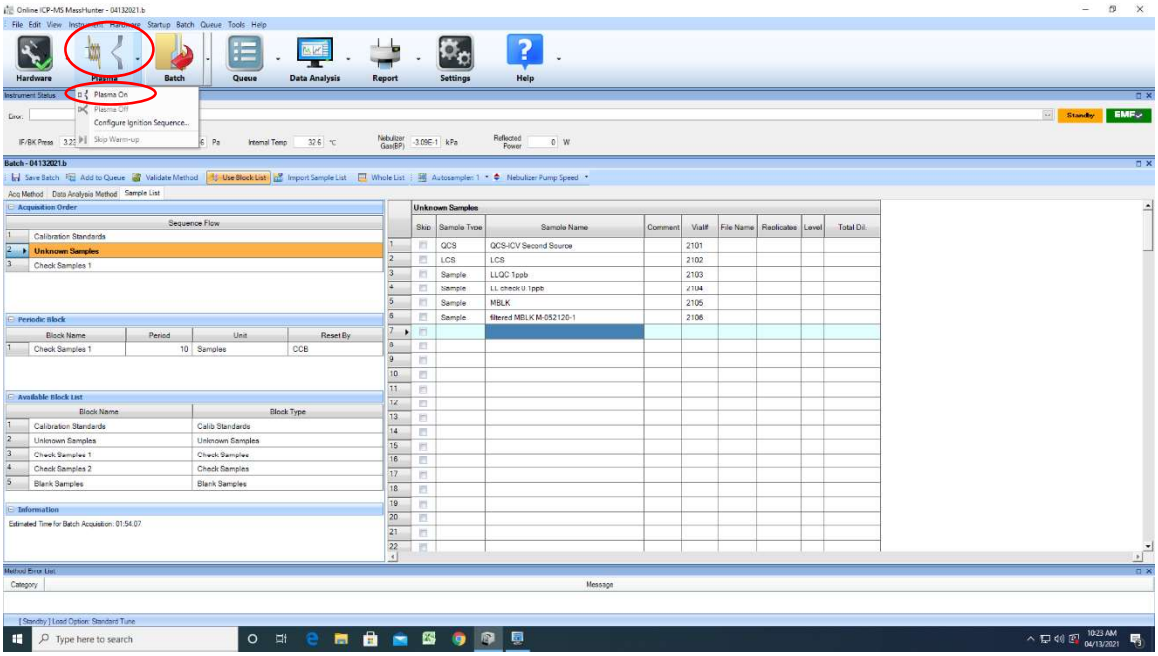




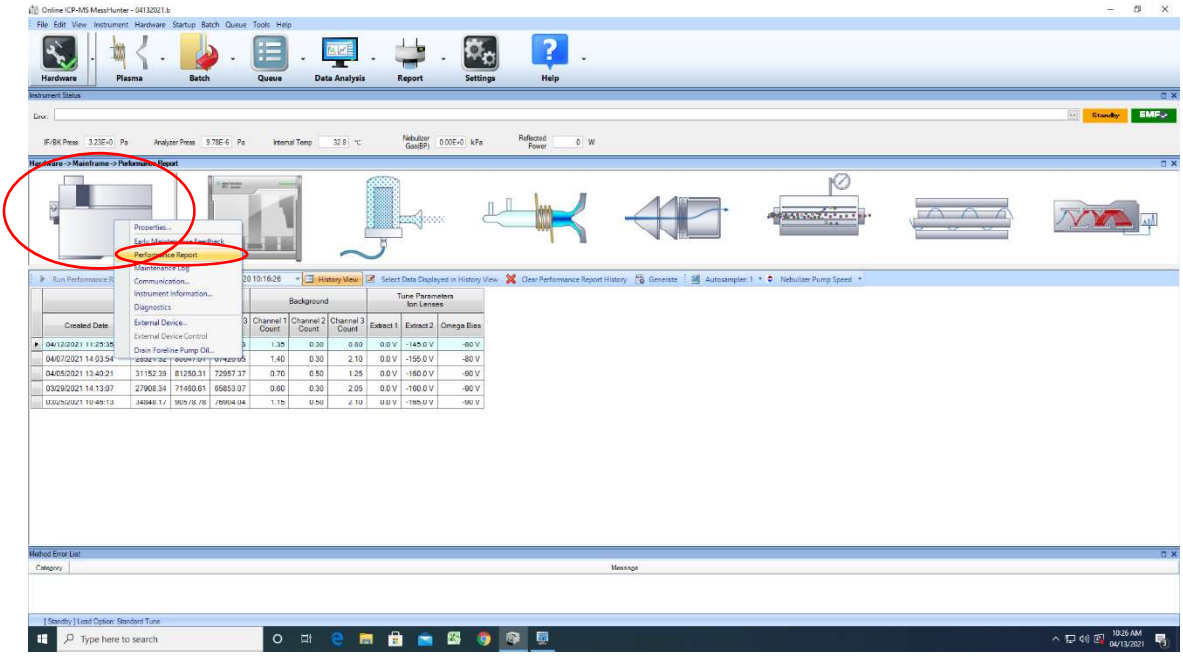
5. Prepare the instrument for startup and warm-up. Put new pump tubing on daily. Fill all rinse solutions, internal standard solution container, and empty waste. Pour fresh 2% HNO_3-DI blank and 20ppb standards daily and all standards are poured fresh weekly. Open the 1ppb tuning solution and the 10ppb tuning solution. Direct the autosampler probe to go to the 1ppb tuning solution using the autosampler button. Put the internal standard tubing into the DI H_2O bottle.



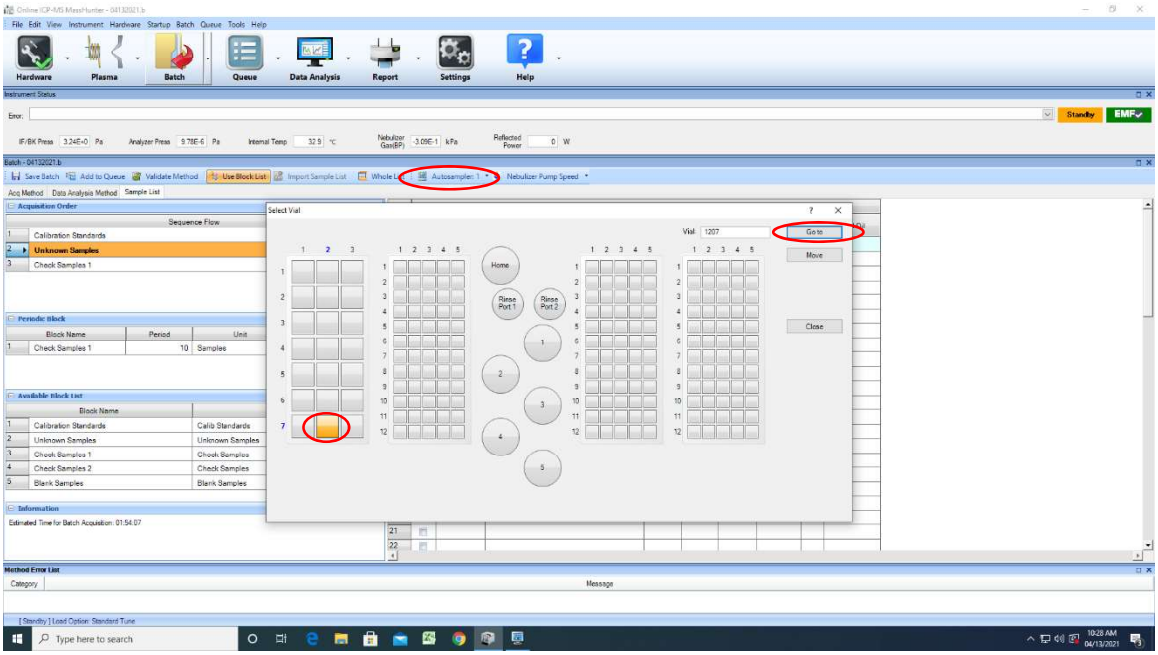
6. Click the Plasma icon. Respond “ok” to the prompt about performing a warmup routine. The instrument software will warm up the instrument for 30 minutes, then perform a series of operations to align and tune and optimize the ICPMS.



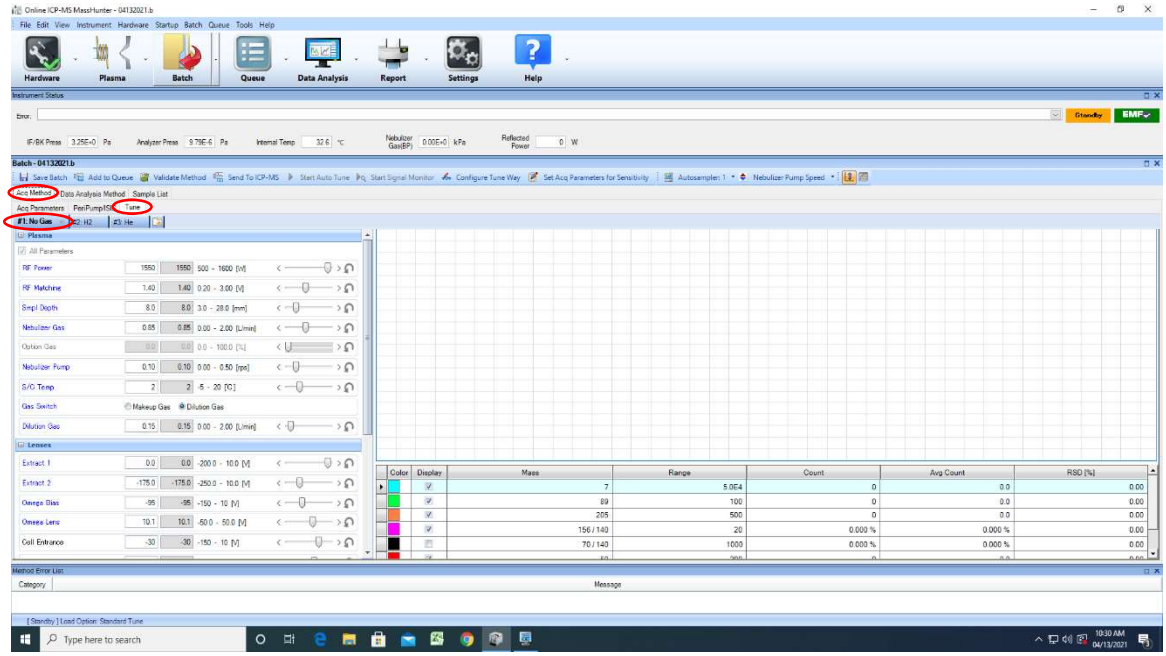
7. Check the “Performance Report” to ensure the sensitivity appears appropriate and similar to previous determinations. To access this report, go to “Hardware”, right click on the drop down box and click “Performance Report”.

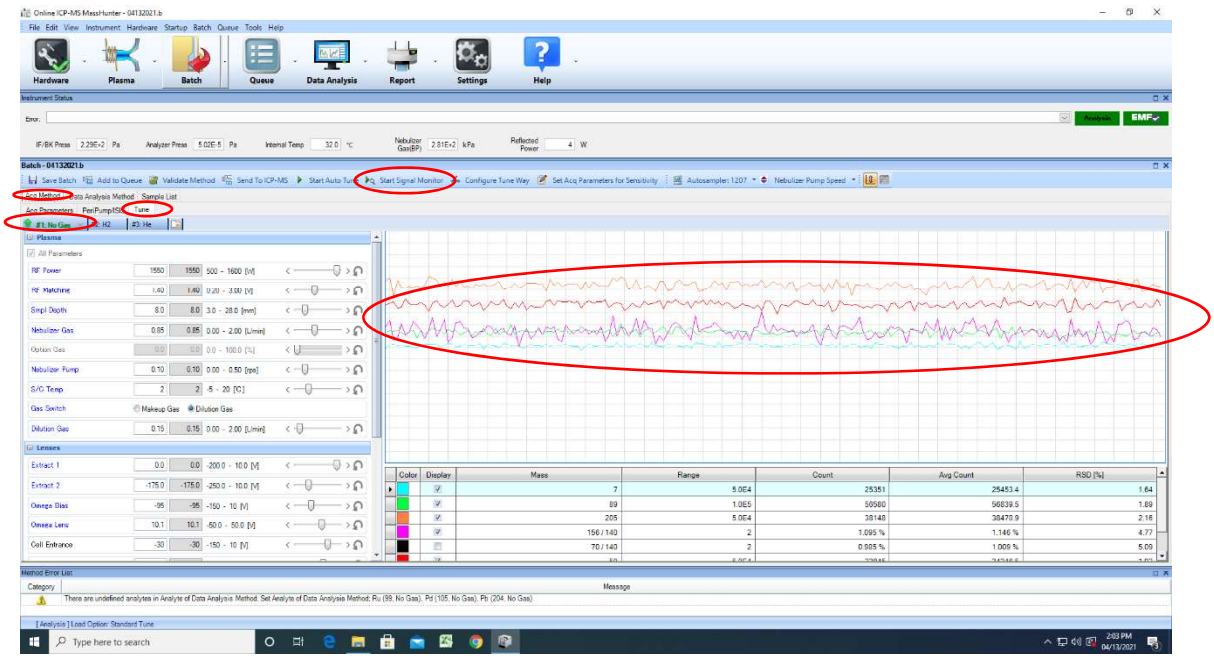


8. Direct the autosampler to the 10ppb tuning solution.

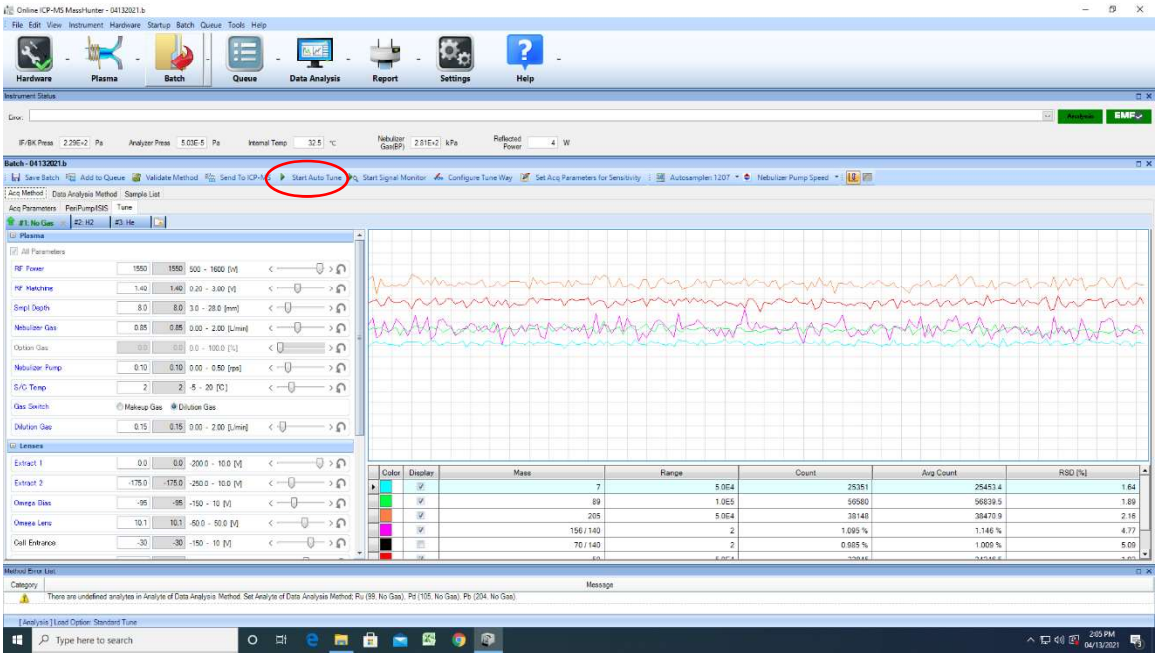


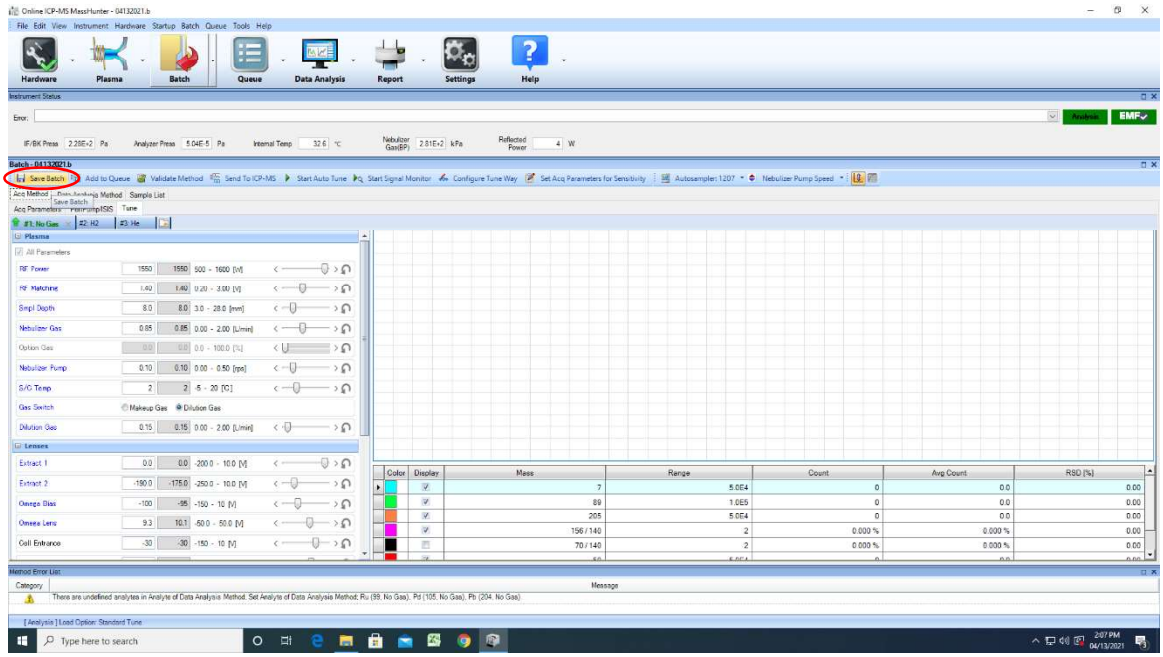
9. While in the “No Gas Mode”, enter the “Signal Monitor” mode. Monitor the instrument to reach stability.



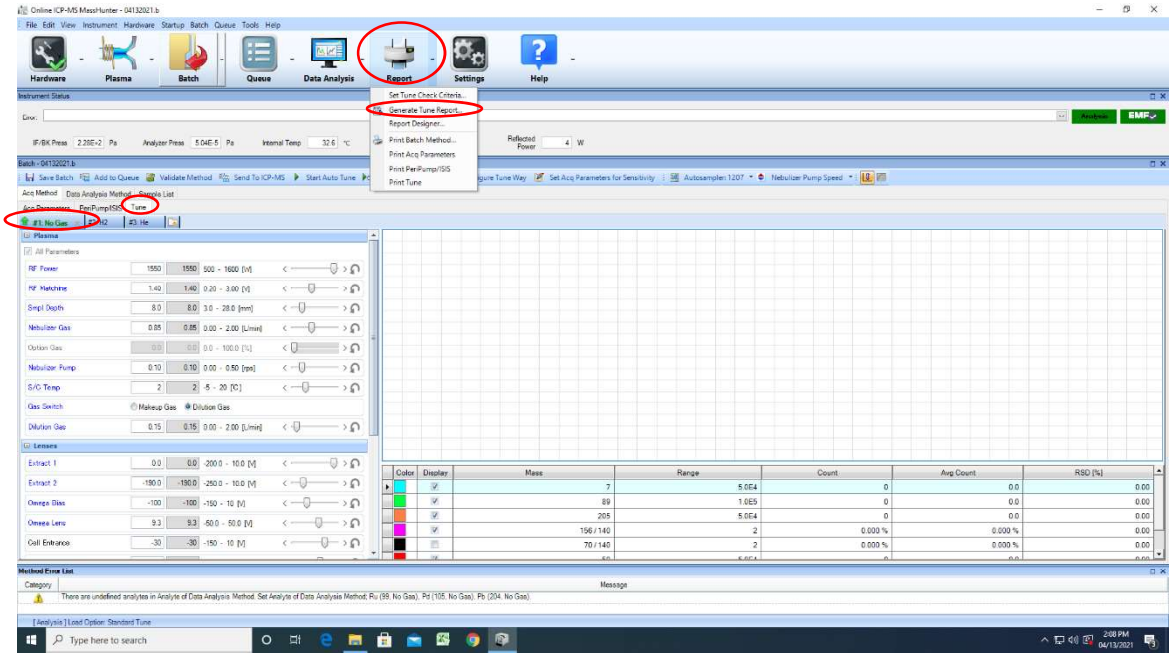


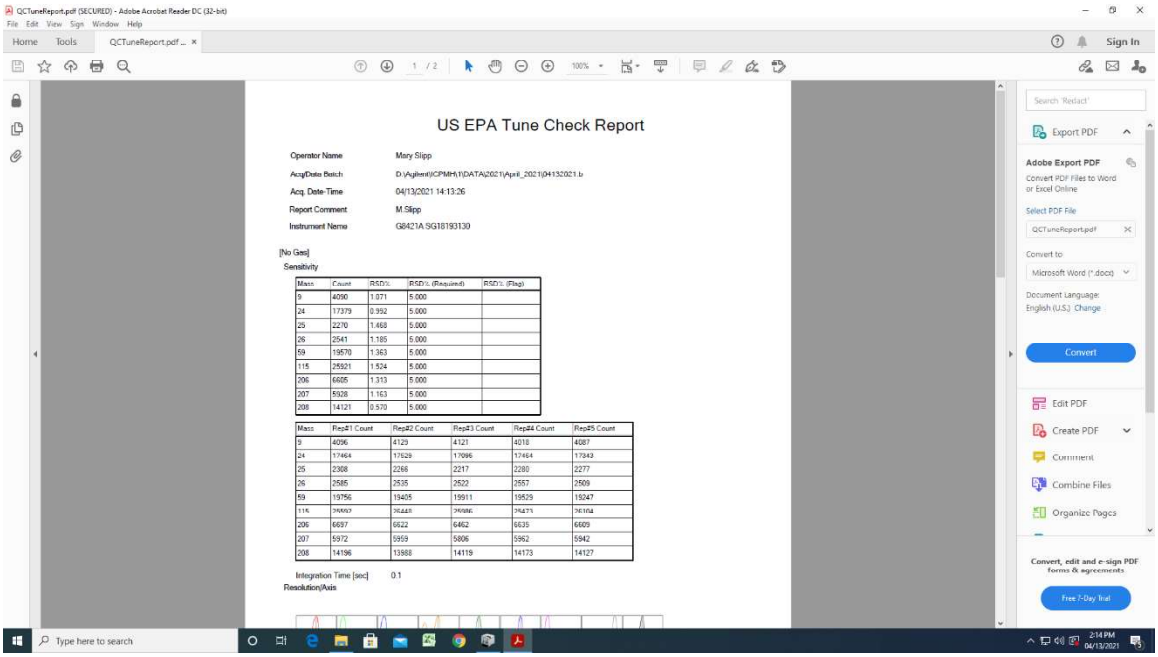
10. Perform an “Auto Tune” for the No Gas mode. Click the green arrow, start auto tune. When it is finished, respond “OK”, then save the settings by clicking the save icon.



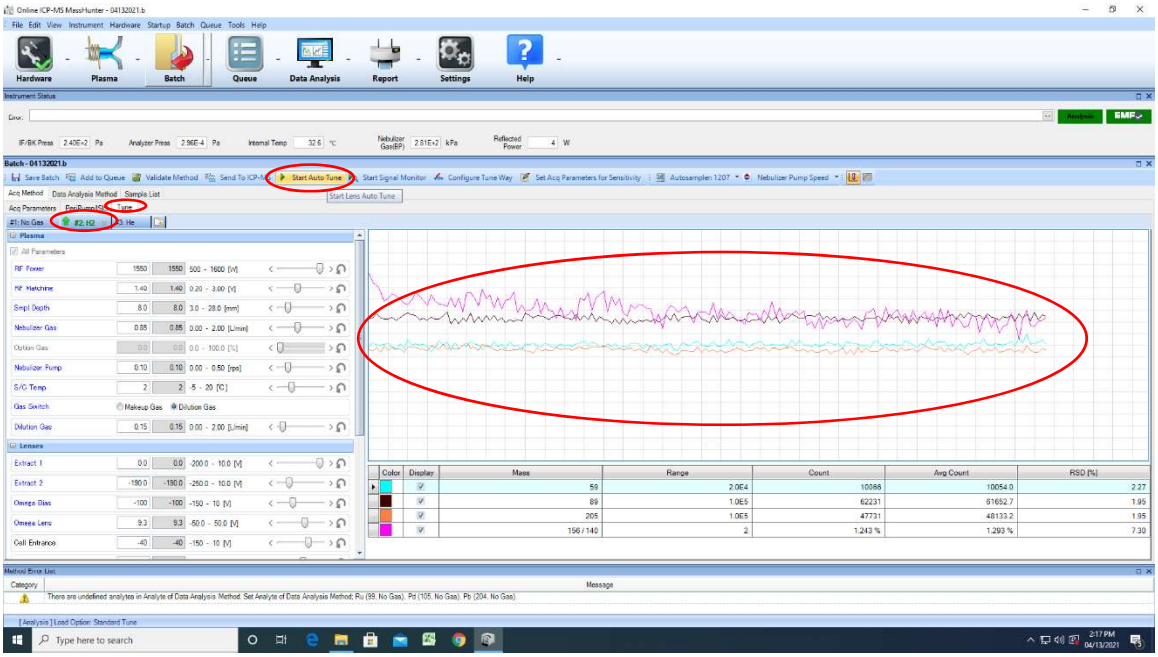


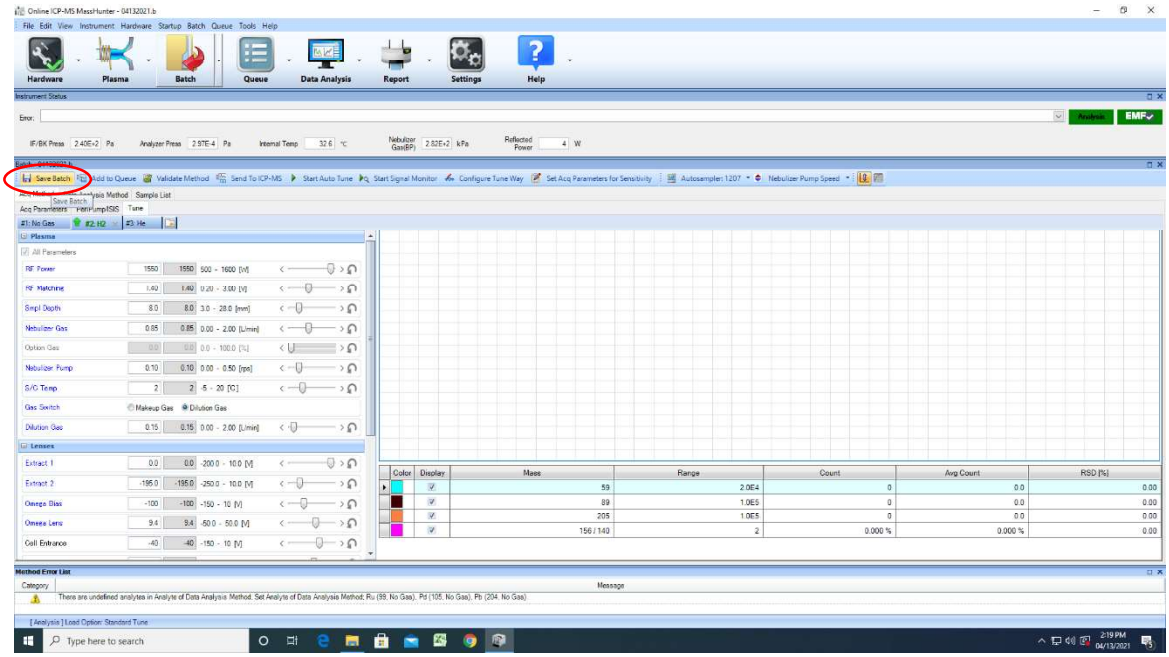
11. Perform a 200.8 tune report by clicking on the Report Icon and selecting the Generate Tune Report option.

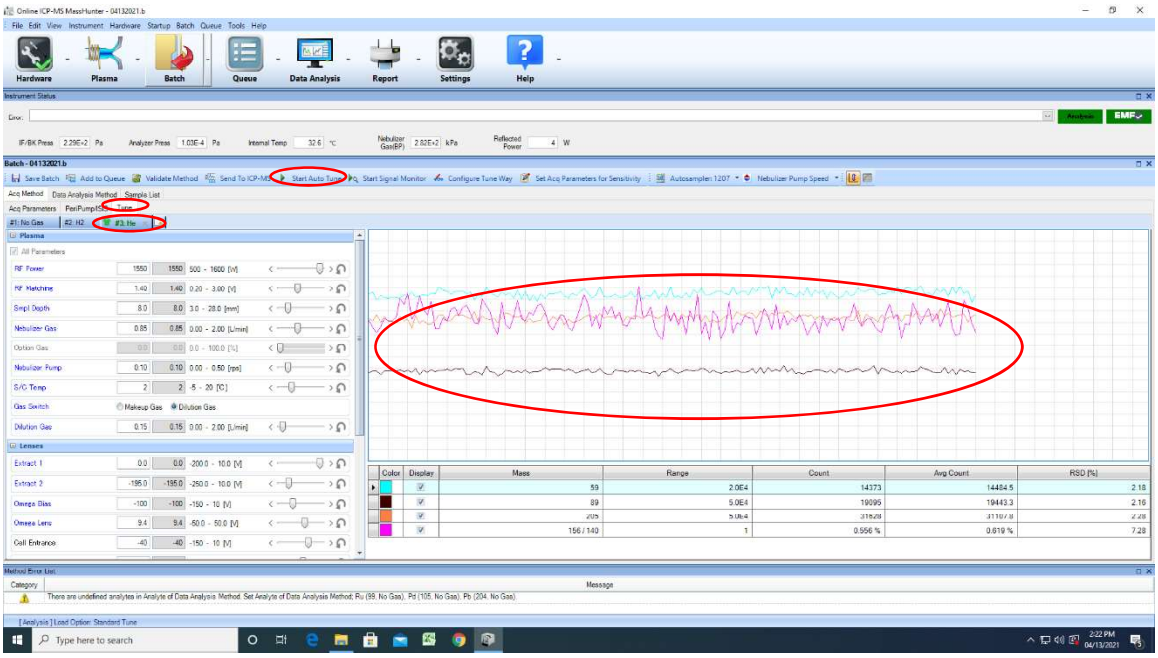




12. Click the H2 tab. Click the “Signal Monitor” function. Let the H2 mode come to equilibrium. Perform the auto tune for the H2 mode. Save the settings. Repeat the process for He mode.






Hardware
Plasma
Batch
Queue
Data Analysis
Report
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Help
Start Auto Tune
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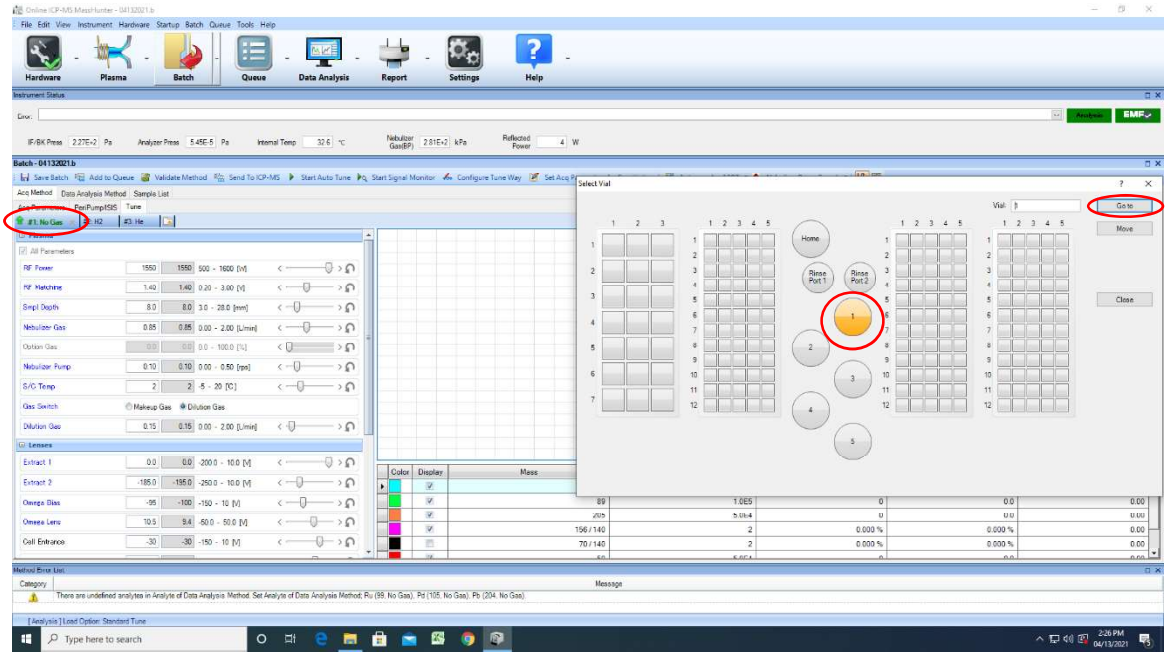
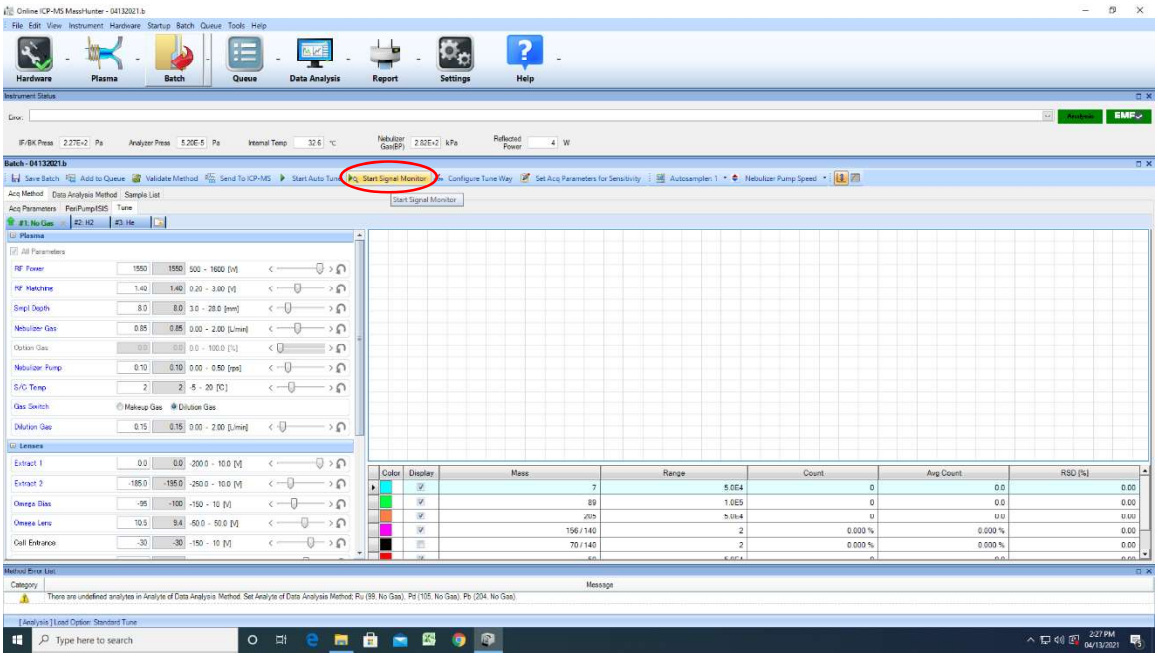
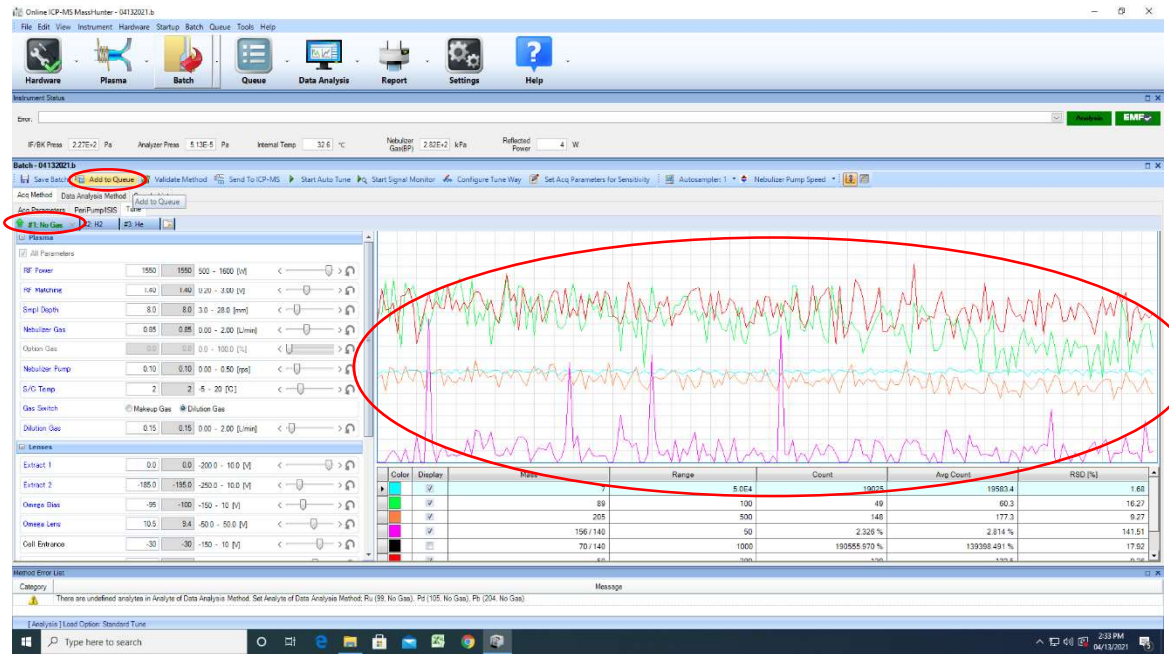
RF Power
RF Matching
Smpl Depth
Nebulizer Gas
Nebulizer Pump
S/C Temp
Gas Switch
Dilution Gas
Lenses
Extract 1
Extract 2
Omega Bias
Omega Lens
Cell Entrance
Color
Display
Mass
Range
Count
Avg Count
RSD (%)
Category
Message
Type here to search

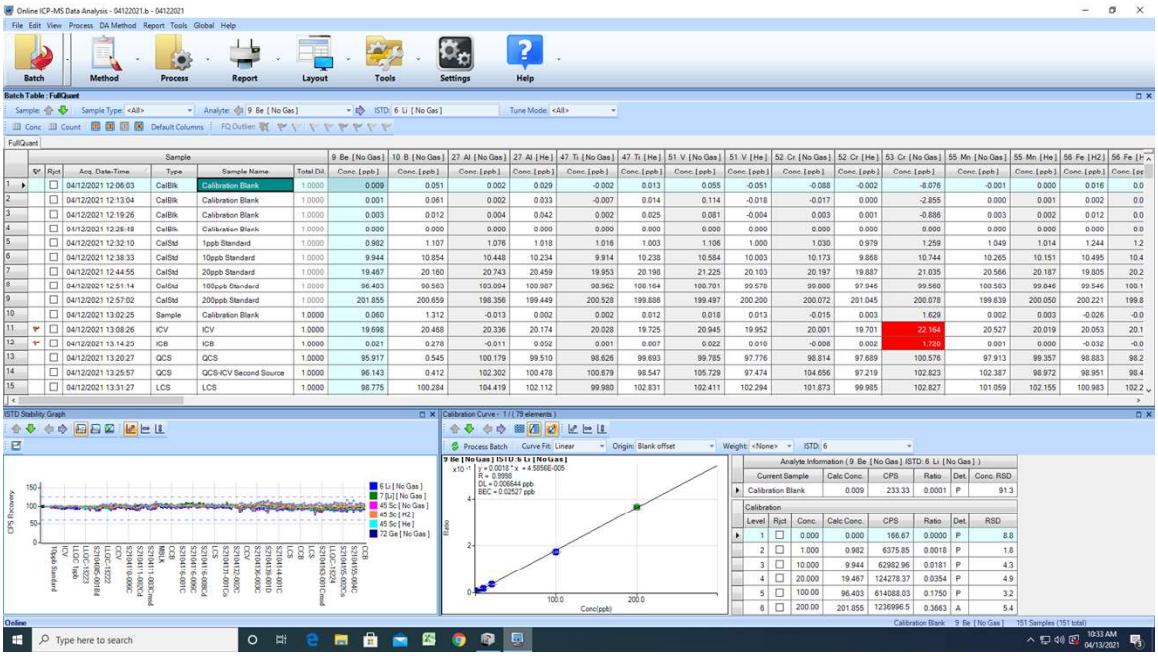
13. Return to the No Gas mode tab. Repeat the No Gas mode tune. Direct the auto sampler probe to rinse position 1. Put the internal standard tubing into the internal standard. Click “Signal Monitor” and monitor the graph for stability. Uncap all standards and QC. When a stable signal is achieved, click “Add to Queue” to begin the run.

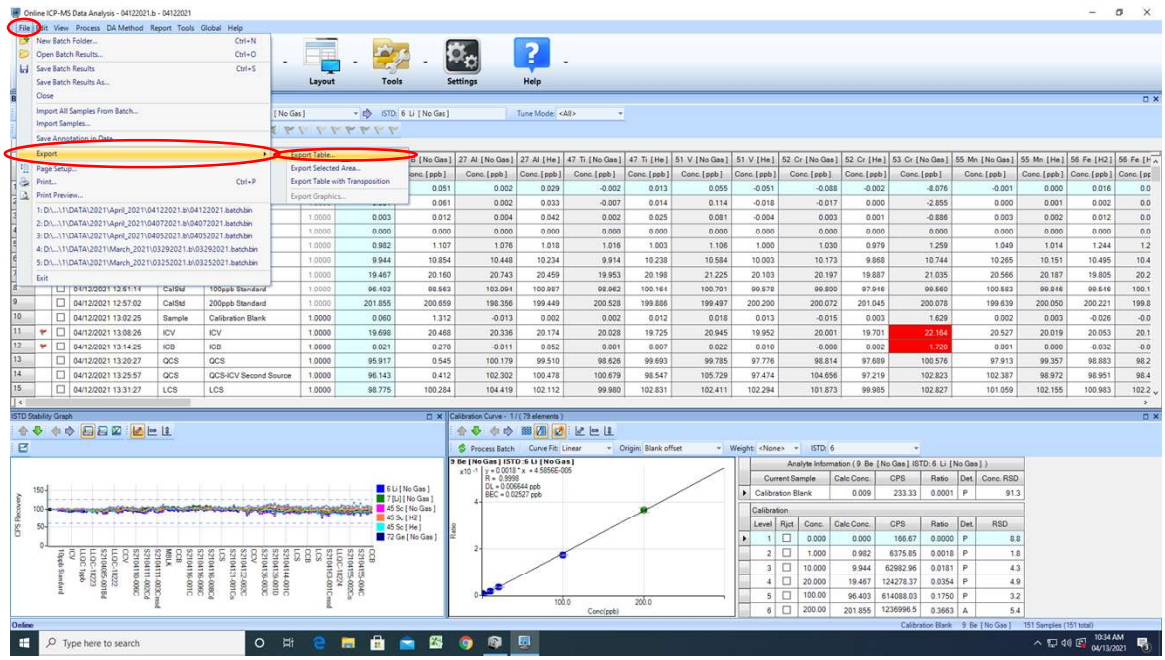


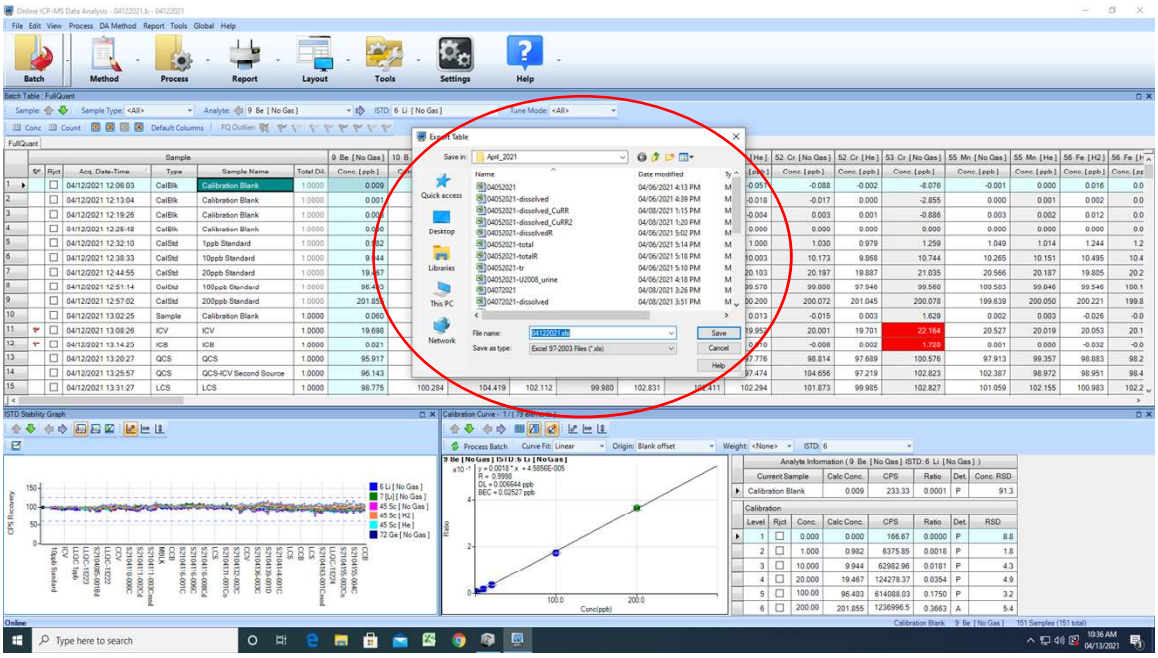




14. After the run is complete, check all QC and sample data for acceptability. Export data by going to File, then to Export Table. Name the file and change the format to XLS. Save the file in the day's batch and export to the U drive and the appropriate Agilent ICPMS 7800 directory.







Document Information

Document Number: ENV-SOP-SHRT-0020 **Revision:** 01

Document Title: Acid Digestion Filters IO 3.1

Department(s): Metals

Date Information

Effective Date: 18 Oct 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-SHRT-0020 **Revision:** 01

Title: Acid Digestion Filters IO 3.1

All dates and times are in Central Time Zone.

ENV-SOP-SHRT-0020

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Michelle LaGory (990324)	Manager - Quality	15 Oct 2021, 10:21:59 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Michelle LaGory (990324)	Manager - Quality	15 Oct 2021, 10:21:38 AM	Approved
Thomas Patten (990330)	Manager	18 Oct 2021, 10:43:34 AM	Approved

Pace Analytical™

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Acid Digestion of Filters
TEST METHOD IO-3.1
ISSUER: Pace ENV – Quality office – SHRT

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the procedure for preparation of air filter samples for analysis by spectrochemical determination of metals.

This digestion procedure is used for the preparation of air filter samples for analysis by Inductively Coupled Plasma (ICP) spectroscopy and ICPMS for metals. This procedure is used to determine the total amount of metals in the sample.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

Not applicable.

2.0 SUMMARY OF METHOD

A mixture of trace metals grade nitric acid, trace metals grade hydrochloric acid, deionized (DI) water, and the filter to be digested and analyzed are refluxed in a covered digestion vial in a block digester. If the sample contains suspended solids, it must be centrifuged, filtered or allowed to settle prior to analysis.

3.0 INTERFERENCES

Interferences are discussed in the referring analytical method.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

Concentrated acids should be handled inside an approved fume hood. Protective clothing, gloves, and safety glasses should be worn.

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory will provide containers for the collection of samples upon client request for analytical services. The bottle kits provided by the laboratory should include field test kits and treatment reagent.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding time
Air filters	Ambient large fiber filters should be received folded in half lengthwise with particulate material inward and enclosed in protective envelopes	N/A	Store at 15-30°C	180 days from collection

[1]*Minimum amount needed for each discrete analysis.*

Field / Matrix QC

Trip Blank	Field Blank	MS/MSD	Field Duplicate
Not applicable	Not applicable	Not applicable	Not applicable

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory procedures. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at approximately 15-30°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at approximately 15-30°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, sample digestates are retained for 30 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations. The remaining filter, if any, is returned to the client or returned to Air Quality Section.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Item	Vendor*	Model ID*	Catalog ID*	Description*
Digestion Block	CPI	ModBlock	4370-011023	48 position
Centrifuge	IEC International	Model K	Size 2	Variable capacity

*or equivalent

7.2 Supplies

Item	Vendor*	Model ID*	Catalog ID*	Description
Digestion vials	Environmental Express	70 mL Digestion Vials-Poly Snap Cap	SC601	Hinged caps, polypropylene, 70mL
Watch glasses	Environmental Express	Ribbed Watch Glass 77 mm	SC505	Polypropylene, ribbed
Thermometer	Midland Control Company	Traceable Waterproof Thermometer	CONTROL 4039	ISO 17025 traceable, waterproof
Die (Cutter)	NA	NA	NA	Custom Made Die
Template (board) and hammer	Various	Various	Various	Any lead-free, non-metallic material
Air displacement pipettor with tips	Eppendorf	Various	Various	Dispensing device,10 mL capacity
Graduated Cylinder	Various	Various	Various	2 L; Class A or volume verified quarterly
PP plastic container with lid	Various	Various	Various	Final container for digested filter solution

*or equivalent

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Refer to SOP for Standard and Reagent Management and Traceability for storage requirements and expiration information. Unless otherwise noted, stock reagents and standard must be stored per manufacturer's recommendations.

Pace Analytical™

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Acid Digestion of Filters
TEST METHOD IO-3.1
ISSUER: Pace ENV – Quality office – SHRT

Reagent	Description/Concentration	Vendor*	Catalog #*
Reagent water	DI water	N/A	N/A
Nitric acid	Concentrated; trace metals grade	Fisher	A509P212
Hydrochloric acid	Concentrated; trace metals grade	Fisher	A508-P212
IO3.1 Extraction solution	Into a 1L volumetric flask, add 500mL of reagent water, 55.5mL concentrated nitric acid and 167.5mL concentrated hydrochloric acid. Dilute to volume with reagent water. Final conc = 5.55% nitric acid and 16.75% hydrochloric acid.	N/A	N/A

*or equivalent

8.2 Standards

Refer to ENV-SOP-SHRT-0026 Standards Preparation Procedure for preparation of IO-3.1 20 ppm Spiker Solution.

Refer to SOP for Standard and Reagent Management and Traceability for storage requirements and expiration information. Unless otherwise noted, stock reagents and standard must be stored per manufacturer's recommendations.

Standard	Description/Concentration	Vendor	Catalog #
Not Applicable	Not Applicable	Not Applicable	Not Applicable

9.0 PROCEDURE

9.1 **Calibration and Standardization**:

9.1.1 Not applicable.

9.2 **Procedure**:

9.2.1 Prior to use, wash with DI water all non-consumable laboratory equipment that will come into contact with the filter samples to prevent contamination.

9.2.2 Using gloves, wipe the template base and cutting blade with a clean, dry Kimwipe to prevent sample cross-contamination.

9.2.3 Unfold the 8" x 10" filter to be sectioned and carefully place exposed side up on the cutting surface. Use a die cutting blade and hammer to cut ¾" X 8" strip.

9.2.4 Label the digestion vial with the lab ID. Carefully cut with ceramic scissors or otherwise non-contaminating implements and place (without disturbing sampled area of filter) the filter pieces down into the lower portion of the digestion vial to ensure the IO-3.1 Extraction Solution will cover the entire filter.

9.2.5 In the event that a smaller filter is used, use the entire filter in the digestion process. If needed, cut the smaller filter so that the pieces placed in the digestion vial will be submerged in the IO-3.1 Extraction Solution.

9.2.6 If the filter is a Teflon filter, snip the outer supporting plastic ring with ceramic scissors in a few places so that the filter will fit into a digestion vial and be submerged in IO-3.1 Extraction Solution. Add spiking solution to appropriate vessels. Using a repeating Eppendorf pipettor or similar device, add 10mL of IO-3.1 Extraction Solution. The IO-3.1 Extraction Solution should cover the filter material completely.

9.2.7 Place the digestion vial in the ModBlock, contained in a fume hood, and reflux gently while covered with a ribbed watch glass for 30 minutes. At least twice during the digestion process check to ensure that the filter is submerged. Do not allow samples to go dry. Remove vials from the digester and allow the sample to cool.

9.2.8 Rinse the vial walls and watch glass with DI water. Decant the solution from the filter into a clean screw top vial that has been weighed and labeled with the filter sample identification.

9.2.9 Add approximately 10mL DI water to the remaining filter material in the digestion vial and allow it to stand for at least 30 minutes. This critical step must not be deleted. It allows the acid to diffuse from the filter into the rinse.

9.2.10 Transfer the extraction fluid from the digestion vial to the second screw top vial. Rinse the digestion vial and any remaining solid material with DI water and add the rinses to the second screw top vial. Dilute to 20mL and mix well.

9.2.11 The final extraction volume is 20mL based on the above procedure. The final extraction solution concentration is 3% nitric acid/8% hydrochloric acid. The sample must be allowed to settle. Centrifuge the sample prior to analysis if needed.

9.2.12 Detailed digestion data is logged in LIMS including sample IDs; identification of person performing procedure; date and time at start and end of digestion; sample volumes; beginning and ending temperatures; solutions used; and IDs of pipettes, and thermometer used.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 Calculations

See the Laboratory Quality Assurance Manual for equations for common calculations.

10.1.1 Samples dilutions must be taken into account when reporting data.

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix A for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 MB per prep batch of 20 or fewer samples
Laboratory Control Sample (LCS)	1 LCS per prep batch of 20 or fewer samples

Sample Duplicate	1 DUP every 20 samples
Matrix Spike	1 MS every 20 samples

11.2 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory Data Review SOP for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range. Samples analyzed after a high concentration sample must be checked for carryover and

reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix A for a complete summary of QC associated with this test method.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A: QC Summary

17.0 REFERENCES

Methods for the Analysis of Water and Waste Water, Method 200.7 USEPA 600

Compendium Method IO-3.1 Selection, Preparation, and Extraction of Filter Material. Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. EPA/625/R-96/010a. June 1999.

40 CFR 50.1. Environmental Protection Agency, National Primary and Secondary Ambient Air Quality Standards, Appendix G to Part 50, Reference Method for the Determination of Lead in Total Suspended Particulate Matter.

Pace Quality Manual, most current version.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
All	Conversion to new SOP format
All	Removed use of balance to determine sample volume used in procedure. Removed option to use filter if solids present post-digestion, prior to analysis.
Attachments	Removed SOP method training record attachment

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-SHRT-0020 00	Acid Digestion of Filters	00

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Acid Digestion of Filters
TEST METHOD IO-3.1
ISSUER: Pace ENV – Quality office – SHRT

Appendix A: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Method Blank	1 per prep batch of 20 or fewer samples	See analytical SOPs	See analytical SOPs	See analytical SOPs
LCS	1 per prep batch of 20 or fewer samples	See analytical SOPs	See analytical SOPs	See analytical SOPs
Duplicate	1 DUP every 20 samples	See analytical SOPs	See analytical SOPs	See analytical SOPs
MS	1 MS every 20 samples	See analytical SOPs	See analytical SOPs	See analytical SOPs

APPENDIX E. SITE CHECK FORM

SITE VISIT CHECKLIST

Trinity Consultants

Operator _____ Date _____

Site Name _____ Stn ID _____

Project _____ Time _____

MET TOWER AND SENSORS:

	YES	NO	N/A	Comments
Solar Panel Clean				
Tower straight, guy wires taut, secured				
Wind Sensors Intact (Propeller, Cups, Vane)				
Temperature Aspirators Operational				
Pyranometer Domes Clean				
Rain Gauge Funnel Clear of Debris/Snow/Ice				
Signs of Sensor Damage				
Sensors Level and Oriented Correctly				
Boom Orientation OK				
Sensor Outputs Check and Functioning Correctly				
Grounding System Intact				
Cellular Antenna Correctly Oriented				
Area Free of Vandalism				
Shelter Integrity				
Fence Integrity				

AQ SHELTER, ANALYZERS, PM:	YES	NO	N/A	
Analyzer intake tubing unobstructed (ice, water, bugs)				
Zero Air System at 30 PSI				
Calibration Gas Cylinder at 30 PSI				**CC:** **Exp:** **Conc:**
Calibration Gas Tank Pressure Above 500 PSI				**Tank Pressure:**
Check for Analyzer(s) Warnings				
Check for Calibrator Warnings				
Check for PM Warnings				
Analyzer Filters Exchanged				
MFC checks Performed				
Inlet Teflon Exchanged				
Zero Air Media Replaced				
RCEL Checked				**Value:**
Slopes Checked (between 0.7 and 1.3)				
Inlet Manifold Fittings Checked and Tightened				
Inlet Cleaning				
Downtube Cleaning				
Filter Houting Assembly Cleaning				
Circulating Fan Filter Cleaning				
Shelter Temp Checked with Reference Standard	Shelter T	Ref T		(+/- 2 degrees C)
				Reference s/n:

Site Visit By: _____