Quality Assurance Project Plan (QAPP)

For

Ambient Air Quality Monitoring of Criteria Air Pollutants

KNOX COUNTY
TENNESSEE
HEALTH DEPARTMENT

Prepared For:
US EPA Region 4
Laboratory Services and Applied Science Division
980 College Station road
Athens, GA 30605

Submitted By
Amber Talgo, Knox County Department of Air Quality

1.0 Identification and Approval

Title: *Quality Assurance Project Plan for Ambient Air Quality Monitoring of Criteria Air Pollutants*, Revision 1, July 29, 2020.

The attached QAPP is hereby recommended for approval and commits the Knox County Department of Air Quality, hence forth referred to as Air Quality, to follow the elements described within.

1) Signature: _____ Date: 8/6/20
Brian Rivera, Director

2) Signature: _____ Date: 8/6/20
Amber Talgo, Air Monitoring Program Manager

3) Signature: _____ Date: 7/29/20
Rebecca Larocque, Environmental Specialist – Quality Assurance

US Environmental Protection Agency, Region 4

4) Signature: _____ Date: _____
Reviewing US Environmental Protection Agency Region 4 Staff

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2.2 Acronym Glossary

AAMP – Ambient Air Monitoring Program
APM – Air Program Manager
AMPM – Air Monitoring Program Manager
AQI – Air Quality Index
AQS – Air Quality System (EPA's air database)
ARD – Air and Radiation Division (EPA Region 4)
CAA – Clean Air Act
CFR – Code of Federal Regulations
DQA – Data Quality Assessment
DQI – Data Quality Indicator
DQO – Data Quality Objective
ERG – Eastern Research Group
EPA – Environmental Protection Agency
FEM – Federal Equivalent Method
FRM – Federal Reference Method
IML – Inter- Mountain Laboratories
LAN – Local Area Network
LSASD -Laboratory Services and Applied Sciences Division (EPA Region 4)
MQO – Measurement Quality Objective
NAAQS – National Ambient Air Quality Standards
NIST – National Institute of Science and Technology
NPAP – National Performance Audit Program
Pb - Lead
PEP – Performance Evaluation Program
PM – Particulate Matter
PQAO – Primary Quality Assurance Organization
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
QAO – Quality Assurance Officer
QC – Quality Control
QMP – Quality Management Plan
SD – Standard Deviation
SLAMS – State and Local Air Monitoring Station
SOP – Standard Operating Procedures
SPM – Special Purpose Monitor
TDEC – Tennessee Department of Environment and Conservation
TSA – Technical Systems Audit

2.3 Change Tracking

Change	Page #	Date of Change	Revision#
Style, department references, grammar	Throughout	3/19/20	1
Replaced all references to TEOM with T640XT	Throughout	3/19/20	1
2.2 Added Acronym glossary	7	3/19/20	1
4.0Updated org chart and responsibilities	10-12	3/19/20	1
6.1 Updated site charts, clarity remove redundancy	17-18	3/19/20	1
7.1.2.1-7.1.2.4 Added pollutant specific	21-23	3/19/20	1
Table 7.1 Shelter Temp range	24	3/19/20	1
Table 7.3 to PM continuous T640/X MQO	31-33	3/19/20	1
Table 11.1 updated references	48	3/19/20	1
11.3.3 Ozone probe design update to solenoid	49-50	3/19/20	1
Table 14.1 update for PM continuous	56	3/19/20	1
Table 14.5 Update for continuous PM	59	3/19/20	1
Table 14.7 update flow rates for PM continuous	60	3/19/20	1
Section 14.1.6 Rounding Convention inserted	61	3/19/20	1
15.2.2 Ozone qualification point updated 75 to 70	63	3/19/20	1
16.2 removed references to tetraCal	67	3/19/20	1
22.4 updated validation procedures	93, 96-103	3/19/20	1

3.0 Distribution List

Digital and/or paper copies of this QAPP have been distributed to the people listed in the following distribution list.

A PDF version of the document is maintained and available on Air Quality's shared drive with the location accessible and known to all air monitoring staff.

Name	Position	Department
Brian Rivera	Director	Air Quality 865-215-5913
Amber Talgo	Air Monitoring Program Manager	Air Quality 865-215-5942
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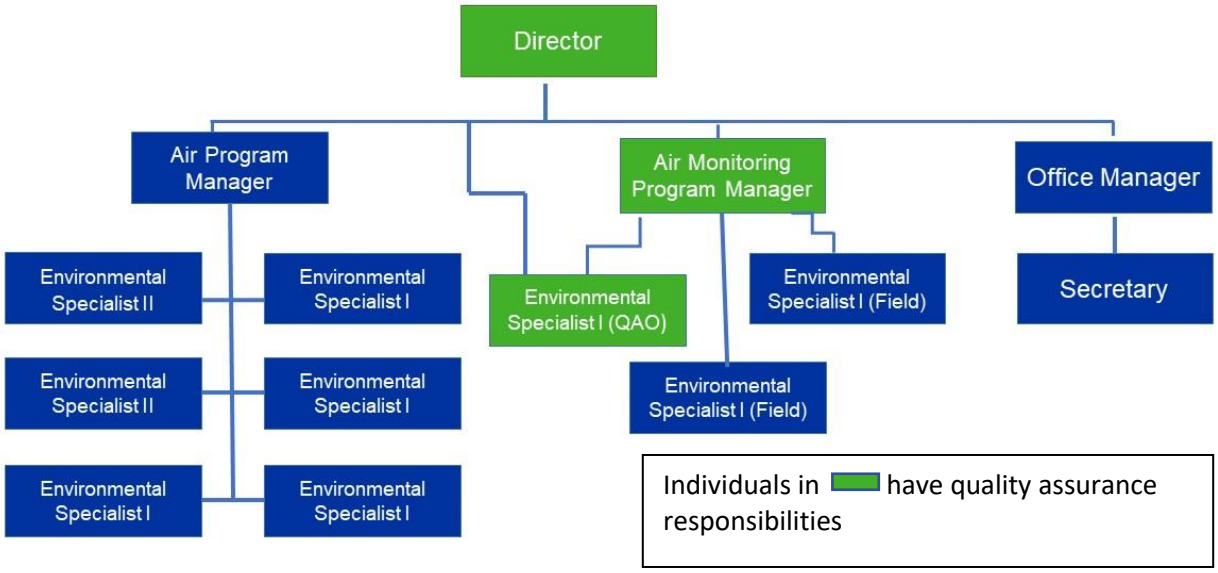
Laura Ackerman	Reviewing US Environmental Protection Agency Region 4	US EPA 706-355-8776

4.0 Project/Task Organization

The Knox County Department of Air Quality, hence forth referred to as Air Quality, is a small local organization which does not have an independent Quality Assurance Department. Many staff perform multiple duties or back up duties within the department. The Ambient Air Monitoring Program (AAMP) generates the monitoring data and provides the quality assurance for the data. The Air Program generates the emission inventory data, issues permits, and conducts compliance evaluations. Air Quality's quality management system for the Air Program and AAMP are described in the department's Quality Management Plan (QMP).

The Director has overall management responsibility, then delegates the responsibility and authority to develop, organize, and maintain quality programs to the program managers. The Ambient Air Monitoring staff are responsible for coordinating all aspects (quality assurance (QA), data collection, and data processing) of Air Quality's monitoring program. The organizational structure for Air Quality is shown in Figure 4.1.

Figure 4.1 Program Organizational Structure



The following are the specific responsibilities for the positions within Air Quality which operate under the guidelines of this document.

Director:

- Senior administrative official accountable for quality of all operations performed by Air Quality staff
- Maintains public relations
- Delegates QA management to Program Managers
- Manages and reviews grants, budgets, proposals, and allocation of resources
- Ensures the establishment of contracts for purchasing equipment and conducting sample analysis ($PM_{2.5}$ & lead analytical laboratories) necessary for the implementation of programs
- Ensures adequate staffing of Air Quality
- Working with the Air Monitoring Program Manager to resolve issues such as obtaining land use and building permits for siting monitoring stations, and corrective action response after significant data loss

- Certifies data on an annual basis in accordance with 40 CFR 58.15

Air Program Manager (APM):

- Ensures implementation of the permitting and compliance QA programs including developing/revising QMP, SOPs and QAPP
- Develops/ revises regulations for Air Board approval
- Prepares and reviews calculation and permitting templates for conformity
- Reviews all compliance evaluation reports
- Reviews developed emission inventory data to verify accuracy for modelling
- Conducts final quality review of permits and emissions inventory data for submittal to EPA
- Conducts annual review of stationary source and compliance data entered into EPA's ICIS-Air database
- Prepares reports for the department
- Liaison to the State of Tennessee Air Pollution Control & US EPA Region 4 permitting and enforcement staff

Air Monitoring Program Manager (AMPM):

- Ensures the air monitoring network is operated in accordance with the Code of Federal Regulations, 40 CFR part 58
- Ensures implementation of the Air Monitoring QA program including meeting data quality objectives (DQOs) and developing/ revising of the QMP, SOP and QAPP
- Conducts final review of environmental and QA data prior to submittal to AQS for precision and accuracy.
- Assesses software and hardware needs of monitoring program
- Ensures software and hardware comply with technical system requirements
- Facilitates the resolution to disputes arising from discrepancies in audit findings and other quality system issues
- Prepares reports for the department
- Supervises corrective actions
- Prepares the data certification for Director review
- Ensures completion of the 5-year Network Assessment and the Annual Network Plan (ANP)
- Liaison to the State of Tennessee Air Pollution Control & US EPA Region 4 staff for ambient air monitoring
- Assessing the effectiveness of the network system
- Supervisor of Environmental Specialist I positions: Quality Assurance Officer and Air Monitoring Field Staff

Environmental Specialist I – Air Monitoring Field Staff

- Operates the monitoring network in accordance with pollutant specific SOPs and QAPPs
- Maintains the equipment and inventories;
- Collecting, and calculating environmental data;
- Participates in training and certification activities;
- Performs all required QC activities to meet the measurement quality objectives prescribed in this QAPP;
- Performs site maintenance and repair

- Documents deviations from established procedures and methods;
- Assesses initial data quality and notifying Quality Assurance Officer of suspect data
- Tests new software and hardware

Environmental Specialist I – Quality Assurance Officer (QAO)

- Validates data by reviewing log books, monthly reports, and all QA and QC documentation.
- Reviews all contract lab reports ensuring QC/QA is within specifications
- Assesses QA/QC quality by tracking frequency of standard certifications
- Verifies all required QA/QC activities are performed in accordance with this QAPP and SOPs
- Reviews audits, both internal and external, for up to date standard certifications and data quality
- Discusses internal and external audit results with Air Monitoring Program Manager
- Notifies Air Monitoring Program Manager of QC problems and when large section of data will be invalidated
- Enters validated data and QA/ QC results into AQS
- Conducts Appendix E siting criteria evaluations
- Maintains QA records and documentation
- Organizing, collecting and processing the data produced by the monitoring network
- Writing and revising SOPs as needed or reporting the needed revision to the AMPM
- Assists with site maintenance and repair when requested
- Performs statistical analysis of quality control data
- Performs the internal performance audits of the Air Monitoring Program
- Reports results to the Director and Air Monitoring Program Manager
- Reports any issues with audits or air monitoring staff directly to Director

5.0 Problem Definition and Background

5.1 Statement of Problem & Purpose

In 1970 the Clean Air Act (CAA) was signed into law. Section 108 &109 of the CAA govern the establishment and revision of the National Ambient Air Quality Standard (NAAQS) for criteria pollutants. The CAA and its amendments provide the framework for protecting air quality. One major step in protecting air quality addressed in the CAA, is ambient air monitoring. 40 CFR 58 describes the requirements for a State or Local Air Monitoring Station (SLAMS). This Air Quality QAPP outlines the procedures used to ensure the quality and validity of the ambient air monitoring data collected by Air Quality. This QAPP prescribes requirements, procedures, and guidelines for Air Quality's Ambient Air Monitoring Program. It is intended to serve as a reference document for implementing and expanding the Quality Assurance (QA) program and provides detailed operational procedures for measurement processes used by Air Quality. It is designed to achieve a high percentage of valid data samples (75%) while maintaining integrity and accuracy. This QAPP clearly and thoroughly establishes QA protocols and Quality Control (QC) criteria required to successfully implement and maintain the state of Knox County's ambient air monitoring program. It is the responsibility of Air Quality to ensure that the quality assurance programs for field, laboratory and data processing phases of the monitoring program are implemented.

5.2 Background Information

The Environmental Protection Agency (EPA) has established criteria pollutants: particulate matter (PM), sulfur dioxide (SO_2), carbon monoxide (CO), nitrogen dioxide (NO_2), ozone (O_3) and lead (Pb). Criteria pollutants are air pollutants with National Ambient Air Quality Standards (NAAQS) which define allowable concentrations in ambient air. These standards are identified in Table 5.1 and can be found online at https://www.epa.gov/criteria-air-pollutants/naaqs-table.

In early 1965, the subject of air pollution in Knox County had garnered sufficient interest that the Technical Society of Knoxville and the Knox Area Health Services Study requested that Knoxville City Government and Knox County Government direct the Metropolitan Planning Commission to file an application with the Division of Air Pollution Control of the U.S. Department of Health, Education and Welfare for a grant to study air pollution in Metropolitan Knoxville. The grant was authorized in June of 1966 and funds were granted to the Metropolitan Planning Commission with the city and county governments matching the grant. A survey of emissions began in July of 1967. Once completed, the survey indicated the need for a local air quality management program.

A federal grant authorized under the Department of Health, Education and Welfare led to the establishment of the Department of Air Pollution Control on June 4, 1968. The State of Tennessee Chapter 37, Private Acts established the department on March 20, 1969 and with the assistance of the Knox Clean Air League, founded in September 1968, the Knox County Air Pollution Control Regulations were adopted on September 22, 1969 and became effective on October 1, 1969. The Air Pollution Control Board (The Board) was also created in the Chapter 37 Private Acts of 1969. The Board is separate from Air Quality and acts as the legislative arm with the power to adopt rules and regulations implemented by Air Quality.

In October 1971, the State of Tennessee Air Pollution Control Board granted Knox County the first certificate of exemption to operate an air pollution program. The Knox County program must be at least as stringent as the program operated by the State of Tennessee, which must in turn be as stringent as the federal program. The

State reviews the Knox County program every two years to determine if it meets all of the requirements to continue operating under the Certificate of Exemption.

In 2014, Air Quality began acting as its own Primary Quality Assurance Organization (PQAO). It samples ambient air at 7 sites across Knox County, TN. The network includes 1: PM_{10} continuous monitor, 5 $PM_{2.5}$ monitors, 2 O_3 monitors, 3 Pb monitors and 1 $PM_{2.5}$ continuous monitor.

Table 5.1 National Ambient Air Quality Standards

Pollutant		Primary/ Secondary	Averaging Time	Level	Form
Carbon Monoxide (CO)		primary	8 hours	9 ppm	Not to be exceeded more than once per year
			1 hour	35 ppm	
Lead (Pb)		primary and secondary	Rolling 3-month period	0.15 µg/m^3 (1)	Not to be exceeded
Nitrogen Dioxide (NO_2)		primary	1 hour	100 ppb	98th percentile of 1-hour daily max concentrations, averaged over 3 years
		primary and secondary	1 year	53 ppb (2)	Annual Mean
Ozone (O_3)		primary and secondary	8 hours	0.070 ppm (3)	Annual 4th highest daily maximum 8-hour concentration, averaged over 3 years
Particle Pollution (PM)	$PM_{2.5}$	primary	1 year	12.0 µg/m^3	Annual mean, averaged over 3 years
		secondary	1 year	15.0 µg/m^3	Annual mean, averaged over 3 years
		primary and secondary	24 hours	35 µg/m^3	98th percentile, averaged over 3 years
	PM_{10}	primary and secondary	24 hours	150 µg/m^3	Not to be exceeded more than once per year on average over 3 years
Sulfur Dioxide (SO_2)		primary	1 hour	75 ppb (4)	99th percentile of 1-hour daily max concentrations, averaged over 3 years
		secondary	3 hours	0.5 ppm	Not to be exceeded more than once per year

(1) In areas designated nonattainment for the Pb standards prior to the promulgation of the current (2008) standards, and for which implementation plans to attain or maintain the current (2008) standards have not been submitted and approved, the previous standards (1.5 µg/m^3 as a calendar quarter average) also remain in effect.

(2) The level of the annual NO_2 standard is 0.053 ppm. It is shown here in terms of ppb for the purposes of clearer comparison to the 1-hour standard level.

(3) Final rule signed October 1, 2015, and effective December 28, 2015. The previous (2008) O_3 standards additionally remain in effect in some areas. Revocation of the previous (2008) O_3 standards and transitioning to the current (2015) standards will be addressed in the implementation rule for the current standards.

(4) The previous SO_2 standards (0.14 ppm 24-hour and 0.03 ppm annual) will additionally remain in effect in certain areas: (1) any area for which it is not yet 1 year since the effective date of designation under the current (2010) standards, and (2)any area for which implementation plans providing for attainment of the current (2010) standard have not been submitted and approved and which is designated nonattainment under the previous SO_2 standards or is not meeting the requirements of a SIP call under the previous SO_2 standards (40 CFR 50.4(3)), A SIP call is an EPA action requiring a state to resubmit all or part of its State Implementation Plan to demonstrate attainment of the require NAAQS.

6.0 Project/ Task Description

6.1 Description of Work to Be Performed

Air Quality has 7 ambient air monitoring sites. Tables 6.1 through 6.7 describe the site location, pollutants monitored, method of monitoring, duration, frequency, objective and monitoring start date at each site for each pollutant.

This QAPP was developed to ensure that the air monitoring network collects ambient air monitoring data that meet or exceed EPA quality assurance requirements. Criteria pollutant data collected by Air Quality is used for regulatory decision-making purposes; Determination of compliance with the NAAQS, determining effects on air quality from adjustments to source emissions, determine trends over time and providing real-time monitoring data to the public.

In accordance with 40 CFR Part 58, Appendix D, Section 1.1, SLAMS monitoring networks must be designed to meet three basic monitoring objectives: provide air pollution data to the general public in a timely manner; support compliance with ambient air quality standards and emissions strategy development; and support for air pollution research studies. Air Quality's air monitoring program is designed to support these objectives. Additional goals of this program include:

- Determining the highest concentrations expected to occur in the area covered by the network
- Determining representative concentrations in areas with high population density or heavy congestion
- Determining the impact of significant sources or source categories on air quality
- Providing data to TDEC to assist the agency in forecasting air quality

In accordance with 40 CFR Part 58, Appendix D, Section 4.5 "State and, where appropriate, local agencies are required to conduct ambient air Pb monitoring near Pb sources which are expected to or have been shown to contribute to a maximum Pb concentration in ambient air. At a minimum, there must be one source-oriented SLAMS site located to measure the maximum Pb concentration in ambient air resulting from each non-airport Pb source which emits 0.50 or more tons per year. Knox County has one such source.

Data will be reported to AQS in accordance with the requirements stated in 40 CFR Part 58 Subpart B §58.16. Collocation of monitors will occur in accordance with 40 CFR Part 58, Appendix A requirements.

The types of data collected will include:

- Continuous pollutant concentration data collected by FEMs averaged by minute and hour
- 24-hour particulate matter samples for PM 2.5 and lead, collected by FRMs in the field and subsequently analyzed at independent laboratories' using the appropriate analytical method
- Continuous shelter temperature measurements for ensuring conformity to environmental requirements of the air monitoring equipment
- Quality control checks for statistical analysis

The work required to collect, document and report this data includes, but is not limited to:

- Establishing a monitoring network that has:
    - Appropriate density, location and sampling frequency
    - Accurate and reliable monitors, data recording equipment and software
- Developing encompassing documentation for

  - Data report format, content and schedule
  - Quality objectives and criteria
- Establishing standard operating procedures, with provide activities and schedules for
  - Equipment operation and preventative maintenance
  - Instrument calibrations, precision checks, and accuracy evaluations
- Establishing assessment criteria and schedules
- Verifying and validating the data product by network monitors in accordance with the criteria and schedules established
- Performing statistical analysis for quality control checks

Field Activities

Site operators will perform field activities that include, but are not limited to, conducting periodic preventative maintenance, calibrations, routing QC checks, and servicing equipment located at SLAMS and Special Purpose Monitoring (SPM) sites. Operational activities may include but are not limited to, collecting and shipping samples, recording field data, and restocking consumables, such as in-line filters at the monitoring sites. Additional field activities include locating, relocating and ground maintenance at monitoring sites.

Laboratory/ Office Activities

Air Quality's level II transfer standard for ozone is located in the laboratory (Lab) facility. Bench Level comparisons are completed at the Lab, as well as some maintenance to monitoring equipment. In the clean room area of the Lab, Air Quality prepares filters for use in the collection of lead and $PM_{2.5}$ as well as post sampling storage and shipment preparation to contract laboratories.

Air Quality does not operate a gravimetric laboratory in support of the criteria pollutant program. These activities are completed by Inter-Mountain Laboratory (IML), in accordance with *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 Quality Assurance Project Plan Revision 14* which can be found on the LAN and Appendix G of this document. Activities performed by IML include PM2.5 filter conditioning, weighing, shipping, and archiving, among others. IML delivers an electronic data package to Air Quality on a monthly basis (approximate) that contains the results of the gravimetric analyses in delimited file format ready to be uploaded to AQS. Air Quality's Quality Assurance Officer (QAO) reviews and processes these data packages and completes the final validation of PM2.5 data in accordance with this QAPP. Any issues observed with the laboratory data packages received will be discussed with the Air Monitoring Program Manager, as well as communicated to IML.

Air Quality participates in the national contract for the analysis of lead. Exposed high volume TSP filters are shipped to Eastern Research Group (ERG) for analysis by Inductively Coupled Plasma/Mass Spectrometer (ICPMS) in accordance with *Support for the EPA National Contract for Lead Analysis, Contract No. EP-BPA-15-D-0004, 2015, Quality Assurance Project Plan Category 1*. This document can be found on the LAN as well as appendix H of this document. ERG delivers a monthly report to Air Quality containing an Excel file and a PDF file. Quarterly a PDF file of audit strip data is provided. The monthly PDF contains a certificate of analysis for each filter analyzed, certification of analysis for the quality control checks, a statement of compliance to the contract QAPP and the program manager's signature. The Excel file contains a list of filters analyzed, site number, sample date and results in micrograms of lead per filter and QA/QC checks performed. The monthly reports are reviewed by the operator.. The sample results are entered into the "Pb Concentration Calculations" Excel spreadsheet by the operator and results are computed. The QAO review the final data package provided

by the operator and ERD then enters data into AQS. This process is covered in the *Volumetric-Flow-Control (VFC) High Volume TSP/Pb Monitors Standard Operating Procedures* in Appendix B of this document. The Air Monitoring Program Manager reviews the quarterly audit strip report and puts the results into AQS.

Data handling software, AirVision and MTL are located in the Lab office space. Data validation activities, AQS entry and data input in Air Quality's QA Access database occur in the Lab office space.

Sites

Figure 6.1 illustrates the distribution of sites within the County and Table 6.2 below provides detailed descriptions of the sites operated by Air Quality and the parameters collected at each.

Figure 6.1 Site Locations Knox County

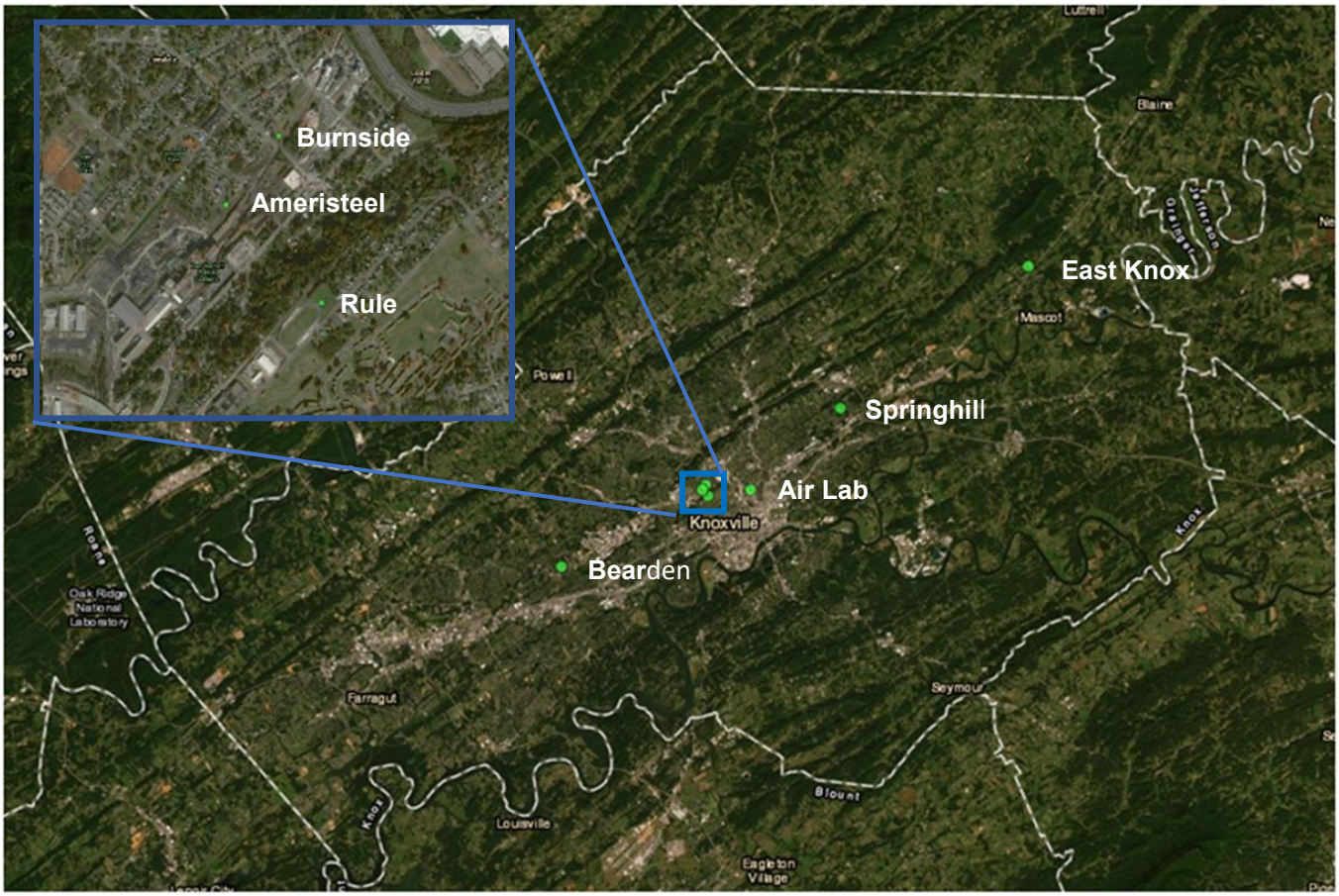


Table 6.2 Site Details

Site Name	AQS ID	Address	Pollutant	Monitor Type	Collection Frequency	Method	Instrument	Analysis	Reference Method	Monitor Objective	Dominant Source	Measurement Scale	Location Setting	Date Established
Ameristeel	470930023	1526 New York Ave, 37921	Lead	SLAMS	1:6	193	Hi Vol Pb-TSP	ICP - Mass Spectroscopy	RFLA-0813-813	Population Exposure	Point	Micro Scale	Urban Center	2011 0101
Air Lab	470931013	939 Stewart St, 37917	PM2.5	SLAMS	1:6	145	Thermo Partisol Plus 2025	Gravimetric	RFPS-0498-145	Population Exposure	Mobile	Middle Scale	Urban Center	2011 0101
			PM2.5 Continuous	SPM	hourly	238	Teledyne T640x	Light Scattering	EQPM-0516-238	AQI	Mobile	Middle Scale	Urban Center	2017 1001
			PM10 Continuous	SLAMS	hourly	239	Teledyne T640x	Light Scattering	EQPM-0516-239	Population Exposure	Mobile	Middle Scale	Urban Center	2020 0101
Bearden	470930028	1000 Francis St, 37909	PM 2.5	SLAMS	1:3	145	Thermo Partisol Plus 2025	Gravimetric	RFPS-0498-145	Population Exposure	Mobile	Neighborhood	Suburban	1999 0101
			PM2.5	SLAMS	1:6	145	Thermo Partisol Plus 2025	Gravimetric	RFPS-0498-145	Collocated	Mobile	Neighborhood	Suburban	2011 0401
Burnside	470930027	2522 Burnside St, 37921	Lead	SLAMS	1:6	193	Hi Vol Pb-TSP	ICP -Mass Spectroscopy	RFLA-0813-813	Population Exposure	Point	Neighborhood	Urban Center	1994 1204
			Lead	SLAMS	1:6	193	Hi Vol Pb-TSP	ICP - Mass Spectroscopy	RFLA-0813-813	Collocated	Point	Neighborhood	Urban Center	1994 1204
East Knox	470930021	9315 Rutledge Pike, 37806	Ozone	SLAMS	hourly	87	Teledyne 400 series	Ultra-violet	EQOA-0992-087	Highest Concentration	Area	Urban	Rural	1981 0601
Rule	470931017	1613 Vermont Ave, 37917	PM2.5	SLAMS	1:3	145	Thermo Partisol Plus 2025	Gravimetric	RFPS-0498-145	Population Exposure	Mobile	Neighborhood	Urban Center	1999 0101
Springhill	470931020	4711 Mildred Dr, 37914	Ozone	SLAMS	hourly	87	Teledyne 400 series	Ultra-violet	EQOA-0992-087	Population Exposure	Mobile	Neighborhood	Suburban	1981 0101
			PM2.5	SLAMS	1:3	145	Thermo Partisol Plus 2025	Gravimetric	RFPS-0498-145	Population Exposure	Mobile	Neighborhood	Suburban	1999 0101

6.2 Project Assessment Techniques

An assessment is an evaluation process used to measure the performance or effectiveness of a system and its elements. As detailed in this QAPP, an assessment is an all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance. Section 20.0 will discuss the details of assessments. Information on the parties implementing assessments and their frequency is provided in table 6.8 below.

Table 6.3 Assessment Schedule

Assessment Type	Assessment Agency	Frequency
Network Review		
5-Year Network Assessment	Air Quality	5 years
Annual Network Plan	EPA Region 4, State	Annually
Annual Site Evaluations	Air Quality	Annually
Quarterly data validation checks	Air Quality	Quarterly
Technical System Audit	EPA Region 4	Every 3 Years
Performance Evaluation		
Internal Performance Audit	Air Quality	Quarterly
EPA Performance Evaluation Program	EPA Designated Contractor	5 per year
National Performance Audit Program	EPA Designated Contractor	20% of monitoring sites per year
State Performance Audit	State	Semi-Annually
Data Quality Assessment		
Annual Data Certification	Air Quality	Annually
Air Quality Quarterly Data Quality Report	Air Quality	Quarterly
Annual Performance Report	Air Quality	Annually
Employee Competency Assessment	Air Quality	Annually

6.3 Project Records

Air Quality will establish and maintain procedures for the timely preparation, review, approval, issuance, use control, revision, and maintenance of documents and records. The categories and types of records and documents which are applicable to document control for ambient air quality information are presented in Table 6.9 below. Information on key documents in each category are explained in more detail in Section 9 of this document.

Table 6.4 Critical Documents and Records

Categories	Record/Document Type
Site Information	Network Descriptions Site Files (including maps and pictures)
Environmental Data Operations	Quality Assurance Project Plans, Standard Operating Procedures Field and Temperature tracking Logbooks Sample Handling/Custody Records Inspection/Maintenance Records
Raw Data	Any Original Data (routine and quality control) Data Entry Forms
Data Reporting	Air Quality Index Reports

Categories	Record/Document Type
	Data/Summary Reports (AQS)
Data Management	Data Algorithms Data Management Plans/Flowcharts Data Management Systems
Quality Assurance	Control Charts Quality Assurance Reports Technical System Audits, Site Audits Response/Corrective Action Reports

7.0 Data and Measurement Quality Objectives and Criteria

Air Quality operates under an EPA-approved Quality Management Plan (QMP) that describes the department's system for implementing and communicating quality within Air Quality. Air Quality will meet the QA/QC requirements outlined in 40 CFR 50 and 58. Additional quality assurance beyond CFR are outlined in this document. Quality Assurance (QA) is a system of management activities designed to ensure that the data produced by the operation will be of the type and quality needed and expected by the data user. Quality Control (QC) defines the procedures implemented to assure that acceptable precision, bias, completeness, representativeness, comparability and sensitivity are obtained and maintained in the generated data set. Quality control procedures when properly executed, provide data that meet or exceed the minimally acceptable criteria established. It is the policy of Air Quality to implement a QA program and QC procedures to assure that data of known and acceptable precision, bias, completeness, comparability, sensitivity and representativeness are collected in all monitoring projects.

Precision: "Precision is a measure of agreement between two replicate measurements of the same property, under prescribed similar conditions. This agreement is calculated as either the range or as the standard deviation" (US EPA QA/G-5, Appendix D). This is the random component of error.

Bias: "Bias is the systematic or persistent distortion of a measurement process that causes errors in one direction." (US EPA QA/G-5 Appendix D) Bias is determined by estimating the positive or negative deviation from the true value as a percentage of the true value.

Completeness: Completeness is a metric quantifying the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions. Completeness can be expressed as a ratio or a percentage. Data completeness requirements are included in the reference methods found in 40 CFR Part 50.

Comparability: "Comparability is the qualitative term that expresses the confidence that two data sets can contribute to a common analysis and interpolation. Comparability must be carefully evaluated to establish whether two data sets can be considered equivalent in regard to the measurement of a specific variable or groups of variables" (US EPA QA/G-5 Appendix D)

Sensitivity or Detectability: is defined as the lowest value that a method procedure can reliably discern a measured response above background noise. There is always variation in any measurement process, therefore, the level of detectability depends on how much precision error is in the process. Detection limits for Air Quality's instruments are consistent with the requirements listed in 40 CFR 53. For Federal Reference Method (FRM) and Federal Equivalent Method (FEM), the detection limits are specified with the EPA FRM/FEM designation.

7.1 Data Quality Objectives (DQOs)

DQOs are qualitative and quantitative statements derived from the DQO process that:

- Clarify the monitoring objective
- Define the type of data needed
- Specify the tolerable limits on the probability of decision error due to uncertainty in the data

7.1.1 Intended Use of Data

The intended use of data for the Air Quality Program are to:

- Determine compliance with the NAAQS for the criteria pollutant
- Determine ambient concentrations of criteria pollutants
- Establish historical baseline concentrations of air pollutants
- Monitor progress toward meeting ambient air quality standards
- Provide data to be used toward developing long-term control strategies

- Observe pollution trends in the monitoring network
- Provide data for forecasting the air quality index (AQI) values
- Determine the highest concentrations expected to occur in the area covered by the network
- Provide credible evidence for source-oriented enforcement
- Determine the impact of significant sources or source categories on ambient pollution levels,
- Determine the extent of regional pollutant transport among populated areas and in support of secondary standards, and
- Determine the welfare-related impacts in rural and remote areas (such as visibility impairment and effects on vegetation).

7.1.2 Data Types

The data type needed is determined by the intended use of the data. The primary use of the air monitoring data is comparison to the NAAQS. For this reason, data must be collected in accordance with 40 CFR Part 50, 53 and 58. The monitoring data compiled by Air Quality consists of 4 criteria pollutants: particulate matter (PM2.5 & PM10), lead and ozone. 40 CFR 58.16 specifies the data reporting requirements that must be followed, and appendices to 40 CFR Part 50 explain the data handling computations for determining whether the NAAQS are met for each pollutant.

Criteria pollutant data will be collected for comparison to the NAAQS using hourly concentration data for continuous monitors (valid if 45, 1-minute readings are valid) and 24-hour non-continuous particulate samples. Quarterly data capture will need to be ≥ 75% complete. The collection of precision and bias data is also required. In addition to these requirements, the data needed for this monitoring project will meet the following principle quality objectives:

- All transfer standards and therefore data, should be traceable to a National Institute of Science and Technology (NIST) primary standard.

- All data shall be of known and documented quality. The two major quantitative measurements used to define quality are precision and bias.

- All data shall be comparable. This means all data shall be produced in a similar and scientific manner. The use of the standard methodologies for sampling, calibration and auditing found in the QAPP should achieve this goal.

- All data shall be representative of the parameters being measured with respect to time, location and the conditions from which the data are obtained. The use of the standard methodologies contained in the QAPP should ensure that the data generated are representative.

- The QAPP and associated SOPs, must be dynamic to continue to achieve its stated goals as techniques, systems, concepts and project goals change.

7.1.2.1 $PM_{2.5}$

The following types of data are needed for NAAQS comparison and is integral to meeting the goals of this project. $PM_{2.5}$ is collected using both continuous and intermittent filter based sampling.

Continuous:

- Hourly data with at least one decimal place in units of $\mu g/m^3$ (valid if 45, 1-minute readings are valid).
- Calculate a 24-hr period as midnight to midnight for the daily average.
- 24-hr average considered valid if at least 75 % of the hourly averages for the 24-hr period are available.

- Incomplete 24-hr periods shall be considered valid if, after substituting zero for all missing/ invalid concentrations, the 24-hr average daily value result is greater than the NAAQS.
- Instrument specific data such as temperature, pressure, and flow used to calculate concentration data.
- Twenty four hour average PM2.5 mass concentrations that are averaged in AQS from hourly values will be truncated to one decimal place.

Intermittent Filter Based:
- Sampler must have operated for 23-25 hours with scheduled start and stop times of midnight to midnight. Samples run less than 23 hours shall be valid if the concentration exceeds the NAAQS.
- Daily instrument specific data such as run time, temperature, pressure, and flow used to calculate concentration data.

Both Methods:
- The 3- year average of $PM_{2.5}$ annual mean mass concentrations for each eligible monitoring site is referred to as the "Annual $PM_{2.5}$ NAAQS DV" and compared to the annual standard.
- The 3- year average of the annual 98th percentile 24-hour average $PM_{2.5}$ mass concentration values recorded at each eligible monitoring site is referred to as the "Daily $PM_{2.5}$ NAAQS DV" and compared to the daily standard.

7.1.2.2 PM_{10}

The project utilizes FEM low-volume, continuous PM10 data. 40 CFR 50 Appendix K references the high volume sampling described in 40 CFR 50 Appendix J. Air Quality will use the following for determining valid hours and days.
- Hourly data to at least whole number in units of µg/m^3 (valid if 45, 1-minute readings are valid). Adjusted to standard temperature and pressure (25C, 101.3kPa)
- Calculate a 24-hr period as midnight to midnight for the daily average.
- 24-hr average considered valid if at least 75 % of the hourly averages for the 24-hr period are available.
- Incomplete 24-hr periods shall be considered valid if, after substituting zero for all missing/ invalid concentrations, the 24-hr average daily value result is greater than the NAAQS.
- Hourly Instrument specific data such as temperature, pressure, and flow used to calculate concentration data.
- Twenty four hour average mass concentrations that are averaged in AQS from hourly values will be rounded to nearest 10 µg/m^3.
- An exceedance is a 24-hr average that is greater than the standard after rounding to the nearest 10 µg/m^3.

7.1.2.3 Lead

- Sampler must have operated for 23-25 hours with scheduled start and stop times of midnight to midnight. Samples run less than 23 hours or more than 25 are not sent to analyzing laboratory.
- Credible daily values include valid samples on scheduled sampling days and valid make-up samples.
- Daily average in µg/m^3 at local conditions of temperature and pressure, to three decimal places, further decimal places are truncated.
- Daily average temperature and pressure from a PM continuous monitor for calculating concentration.
- Stagnation pressure before and after run, for calculating flow rate and concentration.

- The design value is gathered from the 3 month arithmetic mean concentration for the most recent 3- year calendar period plus two previous months. Three month means are computed on a rolling, overlapping basis.

7.1.2.4 Ozone

- Keep each hourly data point (≥ 45 minutes of the hour to be used) to at least 3 decimal places in the units of ppm, with additional digits to the right being truncated.
- Calculate average values for every rolling 8 hour period in the day, averages are calculated rolling forward. An 8 hour average is valid when at least 6 hourly concentrations are valid. Or If less than 6 valid hours: Sum of 5 or less hours >.0567ppm
- Daily maximum 8 hour average ozone concentrations are determined for each day as the highest of the 17 consecutive 8 hour averages beginning with the 8 hour period from 7:00 a.m. to 3:00 p.m. and ending with the 8 hour period from 11:00 p.m. to 7:00 a.m. the following day.
- The daily maximum 8 hour average is valid if 13 of 17 consecutive 8 hour periods starting at 7:00 a.m. are valid.
- The highest 8 hour averages in each year are rank, and the fourth highest value is used,
- 3 consecutive years of 4th highest values are averaged and compared for a design value and to the standard.

7.1.3 Specify Tolerable Data Limits

Data quality objective (DQO) error limits are promulgated in 40 CFR 58 App A sec 2. Regarding the quality of the measurement system, the objective is to control precision and bias in order to reduce the probability of decision errors. Air Quality refers to the validation templates in the “Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II” as a guide to establish criteria for precision and bias limits that are used to determine the validity of data. Air Quality has also set corrective action limits that in some cases are more strenuous than the validation criteria. These limits are outlined in tables 14.1- to 14.4 and outline at what point corrective action should be taken to reduce the loss of data. Data quality objectives are also outlined in pollutant specific Standard Operating Procedure (SOP) which are included as Appendix C-F of this document. SOPs are reviewed annually and updated every 5 years or when significant changes occur.

7.2 Measurement Quality Objectives

Tables 7.1-7.5 contain the Data Validations Tables modified by Air Quality to be representative of the program’s practices. For each criteria pollutant listed in the tables that follow, three validation criteria are listed: critical, operational, and systematic. The tables discriminate between criteria that must be met to ensure the quality of data (critical criteria in pink), criteria that indicates there may be issues and further investigation is warranted before making a determination about the validity of the data (operational criteria in yellow), and criteria that indicates a systematic problem with the data collection that may impact the decision process with a large group of data points (systematic criteria in blue). For each criterion, the tables include the requirement (1), the frequency of evaluation (2), the acceptance criteria (3), and reference and guidance information (4).

Table 7.1 Ozone Measurement Quality Objectives

1)Requirement (O3)	2) Frequency	3) Acceptance Criteria	4) Information/ Action
CRITICAL CRITERIA-OZONE			
Monitor	NA	Meets requirements listed in FRM/FEM designation	1)40 CFR par 58 App C Sec 2.1 3)40 CFR Part 53 & FRM/FEM method list
One-point QC Check	Every 14 days	<±7.1% percent difference or<1.5ppb difference whichever is greater	1) and 2) 40 CFR Part 58 App A sec 3.1 3) Recommendation based on DQO in 40 CFR Part 58 App A Sec 2.3.1.2 QC check Conc range 0.005-0.08ppm
Zero/ Span check	Every 14 days	Zero Drift <±3ppb (24 hr) <±5.1ppb (>24hr-14 day) Span drift <±7.1%	1) and 2) QA Handbook Volume 2 Sec 12.3 3) recommendation and related to DQO
OPERATIONAL CRITERIA - OZONE			
Shelter Temp Range	Daily (hourly values)	15-35 °C (hourly avg)	1,2 and 3) QA handbook volume 2 sec 7.2.2, Air Quality's Ozone SOP and manufacturer's recommendations
Shelter Temp control	Daily (hourly values)	<2.1°C SD over 24 hours	1,2 and 3) QA handbook volume 2 sec 7.2.2
Shelter Temperature Device Check	Every 182 days and 2/ calendar year	≤2.1 ° of Standard	1,2 and 3) QA handbook volume 2 sec 7.2.3
Annual Performance Evaluation	Every Site every quarter within period of monitor operation	Percent difference of audit levels 3-10 < ±15.1% Audit levels 1 &2 <±1.5ppb difference or < ±15.1%	1 and 2) 40 CFR Part 58 App A Sec 3.1.2 3) Recommendation 3- audit concentrations not including zero AMTIC guidance 2/17/2011
Federal Audits (NPAP)	20% of sites audited in Calendar year	Audit levels 1&2 <±1.5 pp difference all other levels <±10.1%	1 and 2) 40 CFR Part 58 App A Sec 3.1.3 3) NPAP QAPP/SOP
Verification/ Calibration	Upon receipt/ adjustment/ repair installation/moving and recalibration of standard of higher level. Every 182 day and 2/ calendar year.	All points <± 2.1% or ≤±1.5 pp difference of best fit straight line whichever is greater and Slope 1 ±.05	1)40 CFR Part 50 App D 2)Recommendation 3)40 CFR part 50 app d Sec 4.5.5.6 Multi-point calibration (0 and 4 upscale points) Slope criteria is a recommendation
Ozone level 2			
Certification/ recertification to Level 1 SRP	Every 365 days and 1/ calendar year	single point difference <± 3.1%	1)40 CFR Part 50 App D Sec 5.4 2)and 3) Transfer Standard guidance EPA-454/B-10-001 Level 2 Standard usually transported to EPA Regions SRP for comparison
Level 2 and > Transfer Standard Precision	Every 365 days and 1/ calendar year	Standard Deviation less than 0.005ppm or 3.0% whichever is greater	1 and 3) 40 CFR part 50 App D Sec 4.3.1 2) Recommendation, part of reverification
(if recertified via a transfer standard)	Every 365 days and 1 calendar year	regression slopes = 1.00 ± 0.003 and two intercepts are 0 ± 3 ppb	1,2 and 3) Transfer Standard Guidance EPA - 545/B- 10-001
Ozone Level 3 and greater			
Qualifications	Upon receipt of transfer standard	<± 4.1% or < ± 4 ppb (whichever is greater)	1,2 and 3) Transfer Standard Guidance EPA - 545/B- 10-001

1)Requirement (O3)	2) Frequency	3) Acceptance Criteria	4) Information/ Action
Certification	after qualification and upon receipt / adjustment/repair	RSD of six slopes ≤ 3.7% Std. Dev of 6 intercepts ≤ 1.5	1,2 and 3) Transfer Standard Guidance EPA - 545/B- 10-001
recertification to higher level standard	Beginning, middle and end of O3 season	New slope - ±0.05 of previous and RSD of six slopes ≤ 3.7% Std. Dev of 6 intercepts ≤ 1.5	1,2 and 3) Transfer Standard Guidance EPA - 545/B- 10-001 and Ozone SOP
SYSTEMATIC CRITERIA -OZONE			
Standard Reporting units	all data	ppm (final units in AQS)	1,2 and 3) 40 CFR part 50 app U section 3(a)
Rounding convention for design value calculation	all routine concentration data	3 places after decimal with digits to right truncated	1,2 and 3) 40 CFR part 50 app U section 3(a) the rounding convention is for averaging values for comparison to NAAQS not for reporting individual hourly values
Completeness Seasonal	3 - year comparison	≥90% (avg) daily max available in ozone season with min of 75% in any one year	1,2 and 3) 40 CFR part 50 app U section 4(b)
	8-hour average	>if at least 6 of the hourly concentrations for the 8 – hour period are available	1) 40 CFR part 50 app U 2 and 3) 40 CFR part 50 app U section 3(b)
	Valid Daily Max	≥ if valid 8-hour averages are available for at least 13 of the 17 consecutive 8-hour period starting from 7 am to 11pm	1) 40 CFR part 50 app U 2 and 3) 40 CFR part 50 app U section 3(d)
Sample Residence Time Verification	every 365 days and 1/ calendar year	≤ 20 seconds	1) 40 CFR Part 58 app E Sec 9 (c) 2) Recommendation 3) 40 CFR Part 58 app E Sec 9 (c)
Sample Probe, inlet, sampling train	all sites	Borosilicate glass or Teflon	1) and 3) 40 CFR Part 58 App E Sec 9(a) 2) Recommendation FEP and PFA have been accepted as equivalent to Teflon. Replacement or cleaning is suggested as 1/ year and more frequent if pollutant load or contamination dictate
Siting	Every 365 days and 1/ calendar year	meets siting criteria or waiver documented	1 and 3) 40 CFR part 58 App E Sec 2-6 2) Recommendation
Precision (using 1 pt QC Checks)	Calculated annually and as appropriate for design value estimates	90% CL CV <7.1%	1) 40 CFR Part 58 App A 2.3.1.2 & 3.1.1 2) 40 CFR Part 58 App A Sec 4(b) 3) 40 CFR Part 58 App A Sec 4.1.2
Bias (using 1-point QC checks)	Calculated annually and as appropriate for design value estimates	95% CL < ± 7.1%	1) 40 CFR Part 58 App A 2.3.1.2 & 3.1.1 2) 40 CFR Part 58 App A Sec 4(b) 3) 40 CFR Part 58 App A Sec 4.1.3

Table 7.2 $PM_{2.5}$ Filter Based Measurement Quality Objectives

1)Requirement (PM2.5 LC)	2) Frequency	3) Acceptance Criteria	4) Information/ Action
CRITICAL CRITERIA- PM2.5 Filter Based Local Conditions			
Field Activities			
Sampler/Monitor	NA	meets requirements listed in FRM/FEM designation	1) 40 CFR Part 58 App C Sec 2.1 2) NA 3) 40 CFR Part 53 & FRM/FEM method list
Filter holding Times			
Pre-sampling	all filters	≤ 30 days before sampling	1,2 and 3) 40 CFR Part 50 App L Sec 8.3.5
Sample recovery	all filters	≤ 7 days 9 hours from sample end date	1,2 and 3) 40 CFR Part 50 App L Sec 10.10
Sampling Period	all filters	1380-1500 minutes or if value <1380 and exceedance of NAAQS midnight to midnight local standard time	1,2 and 3) 40 CFR Part 50 App L Sec 3.3 and 40 CFR Part 50 App N sec 1 for the midnight to midnight local standard time requirement see details if less than 1380 min sampled
Sampling Instrument			
Average Flow Rate	every 24 hours of op	average within 5% of 16.67 LPM	1,2 and 3) 40 CFR Part 50 App L Sec 7.4.3.1
Variability in Flow Rate	Every 24 hours of op	CV ≤ 2%	1,2 and 3) 40 CFR Part 50 App L Sec 7.4.3.2
One-Point Flow Rate Verification	Monthly each separated by 14 days	< ± 4.1% of transfer standard < ± 5.1% of design flow rate	1,2 and 3) 40 CFR Part 50 App L Sec 9.2.5 and 7.4.3.1 and 40 CFR part 58 App 58 App A Sec 3.2.1
Design Flow Rate Adjustment	after multi-point calibration or failed verification	< ± 2.1% of design flow rate	1,2 and 3) 40 CFR Part 50 App L Sec 8.3.5
Individual Flow Rates	every 24 hours of op	no flow rate excursions > ± 5% for > 5 min	1,2 and 3) 40 CFR Part 50 App L Sec 9.2.6
Filter Temp Sensor	every 24 hours of op	no excursions of > 5° C lasting longer than 30 min	1,2 and 3) 40 CFR Part 50 App L Sec 7.4.11.4
External Leak Check	Before each flow rate verification/calibration and before and after PM2.5 separator maintenance	≤ 25mmHg	1)40 CFR Part 50 App L Sec 7.4.6.1 2)40 CFR Part 50 App L Sec 9.2.3 and method 2-12 3)Sec 7.4.3 40 CFR Part 50 App L 7.4.6.2, Air Quality PM2.5 SOP
Internal Leak Check	If failure of external leak check	140mmHg	1)40 CFR Part 50 App L Sec 7.4.6.2 2) Method 2-12 Sec 7.4.4 3)40 CFR Part 50 App L 7.4.6.2, Air Quality PM2.5 SOP
Laboratory Activities			

1)Requirement (PM2.5 LC)	2) Frequency	3) Acceptance Criteria	4) Information/ 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)	1)40 CFR Part 50 App L Sec. 8.3.6 and L Sec.10.13. 2)Method 2-12 Sec 7.4.5 and table 6- 3)Recommendation
Filter Visual Defect Check	all filters	Correct type & size and for pinholes, particles or imperfections	1, 2 and 3) 40 CFR Part 50 App L Sec 10.2
Filter Conditioning Environment			
Equilibration	all filters	24 hour minimum	1, 2 and 3) 40 CFR Part 50 App L Sec 8.2.5
Temp Range	all filters	24-hr mean 20.0-23.0°C	1, 2 and 3) 40 CFR Part 50 App L Sec 8.2.1
Temp Control	all filters	<2.1 ° C Std dev over 24 hr.	1, 2 and 3) 40 CFR Part 50 App L Sec 8.2.2 Std dev use is recommendation
Humidity Range	all filters	24-hr mean 30.0%-40.0% RH or within ± 5.0% sampling RH but ≥ 20.0 % RH	1, 2 and 3) 40 CFR Part 50 App L Sec 8.2.3
Humidity Control	all filters	< 5.1% std dev over 24 hr	1, 2 and 3) 40 CFR Part 50 App L Sec 8.2.4 Std dev use is recommendation
Pre/post Sampling RH	all filters	difference in 24-hr mean < ± 5.1% RH	1, 2 and 3) 40 CFR Part 50 App L Sec 8.3.3
Balance	all filters	located in filter conditioning environment	1, 2 and 3) 40 CFR Part 50 App L Sec 8.3.2
Microbalance Auto-Calibration	prior to each weighing session	manufacturer's specification	1)40 CFR Part 50 App L Sec 8.1 2)40 CFR Part 50 App L Sec 8.1 and Method 2.12 Sec 10.6 3)NA
OPERATIONAL CRITERIA – PM2.5 Filter Based			
Field Activities			
One- point Temp Verification	Monthly	< ± 2.1 ° C	1)40 CFR Part 50 App L Sec 9.3 2)Method 2.12 Sec 7.4.5 and Table 6-1 3) Recommendation
Pressure Verification	Monthly	< ± 10.1 mmHg	1)40 CFR Part 50 App L Sec 9.3 2)Method 2.12 Sec 7.4.6 and Table 6-1 3)Recommendation
Annual Multi-point verifications/Calibrations			
Temperature Calibration	on installation, then every 365 days / once a calendar year	< ± 2.1 ° C	1)40 CFR Part 50 App L Sec 9.3 2)Method 2.12 Sec 7.4.6 and Table 6-1 3)Recommendation
Pressure Calibration	on installation and on one-point verification failure	< ± 10.1 mmHg	1) 40 CFR Part 50 App L Sec 9.3 2 and 3) Method 2.12 Sec 6.5

1)Requirement (PM2.5 LC)	2) Frequency	3) Acceptance Criteria	4) Information/ Action
Flow rate multi-point calibration	electromechanical maintenance or transport or every 365 days / once a calendar year	< ± 2.1 % of transfer standard	1)40 CFR Part 50 App L Sec 9.2 2)40 CFR Part 50 App L Sec 9.1.3 Method 2.12 sec 6.3and Table 6-1 3) Recommendation
Other monitor Calibrations	per manufacture's op manual	Per-manufacturer's operating manual	1, 2, 3) Recommendation
Precision & Accuracy			
Collocated Samples	every 12 days for 15% of sites by method designation	CV <10.1% of samples ≥ 3.0µg/m3	1 and 2) Part 58 App A Sec 3.2.3 3) Recommendation based on DQO in 40 CFR Part 58 App A Sec 2.3.1.1
Temperature Audit	every 180 days and at the time of flow rate audit	< ± 2.1 ° C	1,2 and 3) Method 2.12 Sec 11.2.2
Pressure Audit	every 180 days and at time of flow rate audit	< ± 10.1 mmHg	1,2 and 3) Method 2.12 Sec 11.2.3
Semi Annual Flow Rate Audit	Quarterly	< ± 4.1% of transfer standard and <± 5.1% of design flow rate	1 and2) 40 CFR Part 58 App A Sec3.2.2 3) Method 2.12 Sec 11.2.1
Monitor Maintenance			
Pm $_{2.5}$ separator (VSCC)	Monthly	cleaned/changed	1,2 and3) Method 2.12 Sec 8.3.3
Inlet Cleaning	Monthly	cleaned	1,2 and3) Method 2.12 Sec 8.3
Downtube Cleaning	Monthly	cleaned	1,2 and3) Method 2.12 Sec 8.4
Filter housing Assembly Cleaning	Monthly	cleaned	1,2 and3) Method 2.12 Sec 8.3
Fan Filter Cleaning	Monthly	cleaned/changed	1,2 and3) Method 2.12 Sec 8.3
Recommended Maintenance	per manufacturer SOP	Per manufacturer's SOP	
Laboratory Activities			
Filter Checks			
Lot Blanks	9 filters per lot	< ± 15.1 µg change between weighings	1,2 and 3) recommendation and used to determine filter stability of the lot of filters received from EPA or vendor. Method 2.12 Sec 10.5
Exposure Lot Blanks	3 filters per lot	< ± 15.1 µg change between weighings	1,2 and 3) Method 2.12 Sec 10.5 Used for preparing a subset of filters for equilibration
Filter integrity (exposed)	each filter	no visual defects	1,2 and 3) Method 2.12 Sec 10.7 and 10.3
Lab QC Checks			
Field Filter Blank	10 % or 1 per weighing session	< ± 30.1 µg change between weighings	1) 40 CFR Part 50 App L Sec 8.3.7.1 2 and 3) Method 2.12 Table 7-1 Sec 10.5

1)Requirement (PM2.5 LC)	2) Frequency	3) Acceptance Criteria	4) Information/ Action
Lab filter Blank	10% or 1 per weighing session	< ± 15.1 µg change between weighings	1) 40 CFR Part 50 App L Sec 8.3.7.2 2 and 3) Method 2.12 Sec 10.5
Balance Check (working standards)	beginning, 10th sample, end	< ± 3.1 µg from certified value	1,2 and 3) Method 2.12 Sec 10.6 Standards used should meet specifications in Method 2.12 Sec 4.3.7
Routine Filter re-weighing	1 per weighing session	< ± 15.1 µg change between weighings	1, 2 and 3) Method 2.12 Sec 10.8
microbalance Audit	every 365 days / once a calendar year	< ±0.003mg or manufacture's specs, whichever is tighter	1,2 and 3) Method 2.12 11.2.7
Lap Temp Check	Every 90 days	< ± 2.1 ° C	1,2 and 3) Method 2.12 Sec 10.10
Lab humidity check	Every 90 days	< ± 2.1 %	1,2 and 3) Method 2.12 Sec 10.10
Verification/ Calibration			
Microbalance Calibration	At installation every 365 days / once a calendar year	Manufacturer's specification	1) 40 CFR Part 50 App L Sec 8.1 2) 40 CFR Part 50 App L Sec 8.1 and Method 2.12 Sec 10.11 3) NA
Lab Temperature Certification	every 365 days / once a year	< ± 2.1 ° C	1,2 and 3) Method 2.12 Sec 4.3.8 and 9.4
Lab Humidity Certification	every 365 days / once a year	< ± 2.1 %	1,2 and 3) Method 2.12 Sec 4.3.8 and 9.4
Calibration & Check Standards			
Working Mass Stds. Verification compared to primary standards	Every 90 days	< ± 2.1 µg	1,2 and 3) Method 2.12 Sec 9.7
Primary Standards Certification	every 365 days / once a calendar year	0.025 mg tolerance (Class 2)	1,2 and 3) Method 2.12 Sec 4.3.7
Systematic Criteria - PM 2.5 Filter Based			
Siting	every 365 days and / a calendar year	meets siting criteria or waiver documented	1)40 CFR Part 58 App E Sec 2-5 2)Recommendation 3)40 CFR Part 58 App E Sec 2-5
Data Completeness	Annual Standard	≥75% scheduled sampling days in each quarter	1,2 and 3) 40 CFR Part 50 app N Sec 4.1(b) 4.2(a)
	24- Hour Standard	≥ 75% scheduled sampling days in each quarter	1,2 and 3) 40 CFR Part 50 app N Sec 4.1(b) 4.2(a)
Reporting Units	all filters	µg/m3 at ambient temp/pressure	1, 2 and 3) 40 CFR Part 50 App N Sec 3.0 (b)
Rounding Convention for design value calculation	all filters	to one decimal place, with additional digits to the right being truncated	1, 2 and 3) 40 CFR Part 50 App N Sec 3.0 (b) The rounding convention is for averaging values for comparison o NAAQS not for reporting individual values.

1)Requirement (PM2.5 LC)	2) Frequency	3) Acceptance Criteria	4) Information/ Action
Annual 3- yr. average	all concentrations	nearest 0.1 µg/m3 (≥0.05 round up)	1,2 and 3) 40 CFR Part 50 App N Sec 3 and 4 Rounding convention for data reported to AQS is a recommendation
24-hr, 3-year average	all concentrations	nearest 0.1 µg/m3 (≥0.05 round up)	1,2 and 3) 40 CFR Part 50 App N Sec 3 and 4 Rounding convention for data reported to AQS is a recommendation
Detection Limit			
Lower DL	all filters	≤ 2 µg/m3	1,2 and 3) 40 CFR Part 50 App L Sec 3.1
Upper Conc. Limit	all filters	≥ 200 µg/m3	1,2 and 3) 40 CFR Part 50 App L Sec 3.2
Precision			
Single Analyzer (collocated monitors)	every 90 days	Coefficient of variation (CV) < 10.1% for values ≥ 3.0 µg/m3	1,2 and 3) Recommendation in order to provide early evaluation of achievement of DQOs
Primary Quality Assurance Org	Annual and 3-year estimates	90 % CL of CV < 10.1% for values ≥ 3.0 µg/m3	1,2 and 3) 40 CFR Part 58 App A 4.2.1 and 2.3.1.1
Bias			
Performance Evaluation Program	5 audits for PQAOs with ≤ 5 sites 8 audits for PQAOs with > 5 sites	< 10.1% for values ≥ 3.0 µg/m3	1,2 and 3) 40 CFR Part 58 App A Sec 3.2.4, 4.2.5 and 2.3.1.1
Field Activities			
Verification/Calibration Standards Recertification - All standards should have multi-point certifications against NIST Traceable Standard			
Flow rate Transfer Std.	every 365 days and once a calendar year	< ± 2.1% of NIST Traceable Std	1)40 CFR Part 50 App L Sec 9.1 & 9.2 2) Method 2.12 Sec 4.2.2 & 6.4.3 3) 40 CFR Part 50 App L Sec 9.1 & 9.2
Field Thermometer	every 365 days / once a calendar year	±0.1° C resolution ,± 0.5° C accuracy	1,2 and3) Method 2.12 Sec 4.2.2
Field Barometer	every 365 days / once a calendar year	±1 mmHg resolution, ±5mmHg accuracy	1,2 and3) Method 2.12 Sec 4.2.2
Clock/Timer Verification	every 30 days	±1min	1 and 2) Method 2.12 Sec 4.2.1 3)40 CFR Part 50 App L Sec 7.4.12
Laboratory Activities			
Microbalance Readability	at purchase	1µg	1,2 and 3) 40 CFR Part 50 App L sec 8.1
Microbalance Repeatability	at purchase	1µg	1) Method 2.12 Sec 4.3.6 2) Recommendation 3) Method 2.12 Sec 4.3.6
Primary / working mass Verification/Calibration Standards	at purchase	0.025mg tolerance (class 2)	1,2 and3) Method 2.12 Sec 4.3.7

Table 7.3 $PM_{2.5}$ and PM_{10} Continuous Measurement Quality Objectives

1)Requirement	2) Frequency	3) Acceptance Criteria	4) Information/ Action
CRITICAL CRITERIA- PM Continuous			
Sampler/Monitor	NA	Meets requirements listed in FRM/FEM designation	1)40 CFR par 58 App C Sec 2.1 3)40 CFR Part 53 & FRM/FEM method list
Firmware of monitor	At Setup	1. Must be the firmware (or Later version) as identified in the published method designation summary 2. Firmware settings must be set for flowrate to operate and report at "local" conditions (i.e. not STP)	40 CFR Part 50 App N sec 1(c)
Data Reporting Period	Report every hour	1. Hour valid if at least 45 minutes of hour valid.. 2. A 24- hour period is calculated in AQS if 18 or more valid hours are reported for a day	See operator's manual, Air Vision manual. Hourly data are always reported as the start of the hour on local standard time 40 CFR part 50 App N Sec 3.c
Sampling Instrument			
Pm10 Inlet (if applicable to method designated)	At Setup	Must be Louvered PM10 size selective inlet as specified in 40 CFR 50 Appendix L, Figures L2 through L-19	
Average Flow Rate	every 24 hours of operation; alternatively, each hour can be checked	average within 5% of design flow rate at local conditions	1,2 and 3) Part 50 App L Sec 7.4.3.1
One-point Flow Rate Verification (Total and Main Flow Rate)	Monthly each separated by 14 days	<± 4.1% of transfer standard <± 5.1% of flow rate design value	1,2, and 3) 40 CFR Part 50 App L Sec 8.2.5, 40 CFR Part 58 App A Sec 3.2.3 & 3.3.2
Flow Rate Adjustment Verification	After calibration	<± 2.1% of design and transfer standard flow rate	1,2 and 3) 40 CFR Part 50 App L Sec 9.2.6 and Air Quality Teledyne T640 SOP
Leak Check	Before each flow rate verification/calibration and before and after maintenance	0.0-0.2 $\mu g.m^3$	1)40 CFR Part 50 App L Sec 7.4.6.1 2)40 CFR Part 50 app L Sec 9.2.3 and Method 2-12 Sec 7.4.3 3) Teledyne APT T640 Manual section5.2.8
Operational Criteria - PM Continuous (Teledyne T640 / T640X)			
Annual Multi-Point Verifications/ Calibrations			
Temperature multi-point Verification/Calibration	on installation, then Every 365 days and 1/ calendar year	< ± 2.1 ° C	1) 40 CFR Part 50 App L Sec 9.3 2 and 3) Method 2.12 Sec 6.4.4
One-point Temp Verification	Monthly each separated by 14 days	< ± 2.1 ° C	1) 40 CFR Part 50 App L Sec 9.3 2) Method 2.12 Sec 7.4.5 and Table 6-1 3) Recommendation
Pressure Verification/Calibration	on installation, then Every 365 days and 1/ calendar year	< ± 10.1 mmHg	1) 40 CFR Part 50 App L Sec 9.3 2 and 3) Method 2.12 Sec 6.5

1)Requirement	2) Frequency	3) Acceptance Criteria	4) Information/ Action
Flow rate Multi-point verification/calibration	After maintenance or transport or Every 365 days and 1/ calendar year	<± 2.1% of transfer standard	1) 40 CFR Part 50 App L Sec 9.2 2) 40 CFR Part 50 App L sec 9.1.3. Method 2.12 Sec 6.3 & Table 6-1 3) Recommendation
Check/Adjust PMT with SpanDust™	Quarterly or as needed e.g., high dust load	Measured peak, limit: 0.0 ± 0.5)	1)per manufactures' operating manual 2) Air Quality Teledyne T640 SOP 3) Operating manual and T640 SOP
Precision and Accuracy			
Collocated Samples	every 12 days for 15% of sites by method	CV < 10.1 % of samples ≥ 3µg/m3	1 and 2) Part 58 App A Sec 3.2.3 3) Recommendation based on DQO in 40 CFR Part 58 app A Sec 2.3.1.1
Temperature Audit	Quarterly	<± 2.1 ° C	1, 2 and 3) Method 2.12 Sec 11.2.2
Pressure Audit	Quarterly	<± 10mmHg	1, 2 and 3) Method 2.12 Sec 11.2.3
Semi Annual Flow Rate Audit	Quarterly	<± 4.1% of audit standard < ± 5.1% of design flow rate	1 and 2) P4- CFR Part 58 App A Sec 3.3.3 3) Method 2.12 Sec 11.2.1
Shelter Temperature			
Temperature Range	At Setup	0-50°C	per manufactures' operating manual.
Temp Device Check	every 180 days and twice a calendar year	<±2.1°C	1,2 and 3) QA Handbook Volume 2 Sec 7.2.2
Monitor Maintenance			
PM 10 Well/ Inlet Cleaning	Monthly	cleaned	1, 2 and 3) Method 2.12 Sec 8.3 and Air Quality Teledyne T640 SOP
Downtube Cleaning	Semi–annually	cleaned	1, 2 and 3) Air Quality Teledyne T640 SOP
Optics Chamber Cleaning	Semi-annually	cleaned	1, 2 and 3) Air Quality Teledyne T640 SOP
Manufacturer- Recommended Maintenance	per manufacturer's SOP	per manufacturers' SOP	na
Systematic Criteria			
Siting	every 365 days and once a calendar year	meets siting criteria or waived documented	1) 40 CFR Part 58 App E Sections 2-5 2) Recommendation 3) 40 CFR Part 58 App E Sections 2-5
Data Completeness	Annual Standard	≥ 75% scheduled sampling days in each quarter	1,2 and 3) 40 CFR Part 50 app N Sec 4.1(b) 4.2(a)
	24-Hour Standard	≥ 75% scheduled sampling days in each quarter	1,2 and 3) 40 CFR Part 50 app N Sec 4.1(b) 4.2(a)
Reporting Units	all filters	µg/m3 at ambient temp/pressure for Pm2.5 and STP for PM10	1, 2 and 3) 40 CFR Part 50 App N Sec 3.0 (b)

1)Requirement	2) Frequency	3) Acceptance Criteria	4) Information/ Action
Rounding convention for data reported in AQS	all filters	to one decimal place or as reported by instrument	1, 2 and 3) 40 CFR Part 50 App N Sec 3.0 (b) The rounding convention is for averaging values for comparison o NAAQS not for reporting individual values.
Annual 3- yr average	all concentrations	nearest 0.1 µg/m3 (≥0.05 round up)	1,2 and 3) 40 CFR Part 50 App N Sec 3 and 4 Rounding convention for data reported to AQS is a recommendation
24-hour, 3 year average PM2.5	all concentrations	nearest 0.1 µg/m3 (≥0.05 round up)	1,2 and 3) 40 CFR Part 50 App N Sec 3 and 4 Rounding convention for data reported to AQS is a recommendation
24-hour, 3Year Average PM10	quarterly	nearest 10 µg/m3 (≥5 round up)	1,2, and 3) 40 CFR Part 50 App K Sec 1. Rounding convention is for averaging values for comparison to NAAQS not for reporting individual values
Verification/Calibration Standards Recertification- All standards should have multi-point certifications against NIST Traceable standards			
Flow rate transfer std	every 365 days and once a calendar year	<±2.1% of NIST traceable std	1)40 CFR Part 50 App L Sec 9.1 & 9.2 2) Method 2.12 Sec 4.2.2 & 6.4.3 3) 40 CFR Part 50 App L Sec 9.1 & 9.2
Field Thermometer	every 365 days and once a calendar year	±0.1° C resolution, ± 0.5° C accuracy	1,2 and3) Method 2.12 Sec 4.2.2
Field Barometer	every 365 days and once a calendar year	±1 mmHg resolution, ±5mmHg accuracy	1,2 and3) Method 2.12 Sec 4.2.2
Clock/Timer Verification	Monthly	1min	1 and 2) Method 2.12 Sec 4.2.1 3) 40 CFR Part 50 App L Sec 7.4.12
Precision/Bias			
Single Analyzer	every 90 days and 4 times a calendar year	CV < 10.1% of samples ≥ 3 µg/m3 (cutoff value)	1, 2 and 3) Recommendation in order to provide early evaluation of achievement of DQO
Primary Quality Assurance Org	annual and 3-year estimates	90% CL of CV < 10.1% of samples ≥ 3 µg/m3	1,2 and 3) 40 CFR Part 58 App A Sec 4.2.1 and Sec 2.3.1.1
Performance Evaluation Program (PEP)	5 audits for PQAOs with ≤ sites 8 audits for PQAOs with > 5 sites	< 10.1% for values ≥ 3.0 µg/m3	1,2 and 3) 40 CFR Part 58 App A Sec 3.2.4, 4.2.5 and2.3.1.1

Table 7.4 Lead in Hi Volume TSP Measurement Quality Objectives

1)Requirement	2) Frequency	3) Acceptance Criteria	4) Information/ Action
CRITICAL CRITERIA-Pb			
Field Activities			
Sampler/Monitor	NA	meets requirements listed in FRM/FEM designation	1)40 CFR par 58 App C Sec 2.1 2) NA 3)40 CFR Part 53 & FRM/FEM method list
Sample Recovery	all filters	ASAP	1,2, and 3) 40 CFR Part 50 App B Sec 6.3
Sample Period	all filters	1440 minutes ± 60 min midnight to midnight local standard time	1,2, and 3) 40 CFR Part 50 App B Sec 8.15
Average Flow Rate	every 24 hours of op	1.1-1.70 m3/min (varies with instrument) in actual condition	1,2, and 3) 40 CFR Part 50 App B Sec 8.8
One-Point Flow Rate Verification	once per month > 14 days apart, after motor change	<± 7.1% from transfer standard	1 and 2) 40 CFR Part 58 App A Sec 3.4.2 3) Method 2.2 sec 2.6
Laboratory Activities			
Visual Defect Check	all filters	Initial backlight inspection - no pinholes or imperfections. Visual inspection prior to shipping to lab	1,2, and 3) 40 CFR Part 50 App B Sec 8.2
Collection Efficiency	all filters	99%	1,2, and 3) 40 CFR Part 50 App B Sec 7.1.4
Pressure Drop Range	all filters	42-54 mmHg	1,2, and 3) 40 CFR Part 50 App B Sec 7.1.5
pH	all filters	6-10	1,2, and 3) 40 CFR Part 50 App B Sec 7.1.6
Pb Content	all filters pre-sampling batch check	< 75 µg/ filter	1,2, and 3) 40 CFR Part 50 App G Sec 6.1.1.1, Method 2.8 Sec 6.2.1. More information relative to whether filters should be corrected for blanks
Calibration Reproducibility Checks	Beginning, every 10 samples and end	± 5% of value predicted by calibration curve	1,2 and 3) 40 CFR Part 50 App G Sec 9.3 May be FEM dependent
Initial Calibration Blank	before first sample	<0.001 µg/mL	1,2, and 3) 40 CFR part 50 G Sec 8.8
Reagent Blank	Every analytical batch	< LDL	1,2, and 3) Recommendation
Operational Criteria (Pb)			
Field Activities			
Verification/Calibration			
System Leak Check	during pre-calibration check	Visual and Auditory inspection with faceplate blocked	1,2 and 3) recommendation

1)Requirement	2) Frequency	3) Acceptance Criteria	4) Information/ Action
Multi-point Verification/Calibration	After receipt, or failure of 1 - point check and every 365 days and once a calendar year	5 points over range of 1.1 to 1.7 m^3/min <± 5.1% limits of linearity	1,2 and 3) Method 2.2 Sec 2.6
Precision			
Collocated Samples	15% of each method code in PQAO Frequency every 12 days	CV < 20.1% of samples ≥ 0.02µg/m^3 (cutoff value)	1 and 2) 40 CFR Part 58 App A Sec 3.3.4.3 3) Recommendation for early evaluation of DQOs
Semiannual flow Rate Audit	every 180 days and twice a calendar year	≤± 7.1% of audit standard	1 and 2) 40 CFR Part 58 App A Sec 3.4.3 3) Method 2.2 Table 8.2
Monitor Maintenance			
Inlet Cleaning	every 90 days and 4 times a calendar year	cleaned	1,2 and 3) recommendation
Motor/housing gaskets	600 hours	inspected replaced	1,2 and 3) Air Quality Lead SOP &*IO Compendium Method IO-2.1
Blower motor brushes	600 hours	replace	1,2 and 3) Air Quality Lead SOP& *IO Compendium Method IO-2.1
Manufacturer- Recommended Maintenance	per manufacturers' SOP	per manufacturers' SOP	NA
Lab Activities			
Analysis Audits	6 strips/quarter 3 at each concentration range	< 10.1% (percent difference)	1 and 2) 40 CFR Part 58 App a Sec 3.4.6 3) Recommendation
Field Filter Blank	1/ quarter	<LDL	1,2 and 3) recommendation
Lab blanks	1/ sample run	< LDL	1,2 and 3) recommendation
Control Standards	1st, every 10 samples and last sample	Deviation of < 5.1% from value predicted by calibration curve	1,2 and 3) Method 2.8 Sec 5.7.3
Systematic Criteria - Pb (HiVol)			
Siting	Every 365 days and 1/ calendar year	Meets siting criteria or waiver documented	1)40 CFR Part 58 App E Sec 2-5 2)Recommendation 3)40 CFR Part 58 App E Sections 2-5
Data Completeness	3- year standard	average of the 3 consecutive monthly means ≥ 75%	1,2 and 3) 40 CFR Part 50 App R Sec 4 In addition there are substitution test that can be used for data not meeting completeness criteria
Reporting Units	all filters	µg/m3 at local temperature and pressure	1,2 and 3) 40 CFR Part 50 App R Sec 3(b)

1)Requirement	2) Frequency	3) Acceptance Criteria	4) Information/ Action
Rounding convention for design value calculation (3-month arithmetic mean)	quarterly	Report data to 3 decimal places (data after 3 are truncated)	1,2 and 3) 40 CFR Part 50 App R Sec 3(b) The rounding convention is for averaging values for comparison to NAAQS not for reporting individual values
Lower Detectable Limit (AA)	all samples	0.07 µg/m3	1,2 and 3) 40 CFR Part 50 App G Sec 2.3
Precision/Bias			
Single Analyzer	every 90 days and 4 times a calendar year	CV < 20.1% of samples ≥ 0.02µg/m3 (cutoff value)	1 and 2) 40 CFR Part 58 App A Sec 3.4.4 3) Recommendation related to DQO
Primary Quality Assurance Org	annual and 3-year estimates	90% CL of CV < 20.1% of samples ≥ 0.02 µg/m3	1,2 and 3) 40 CFR Part 58 App A Sec 3.4.4 and Sec 2.3.1.3
Performance Evaluation Program (PEP)	5 audits for PQAOs with ≤ sites 8 audits for PQAOs with > 5 sites	95% CL Absolute bias <± 15.1% of samples ≥ 0.02 µg/m3	1,2 and 3) 40 CFR Part 58 App A Sec 3.4.7 and Sec 2.3.1.3 The Pepe include 1 or independent collocated audits and 4 or 6 samples from the monitoring organizations collocated monitor sent to the independent national PEP lab
Field Activities			
Verification/Calibration Standards and Recertification - All Standards should have multi-point certifications against NIST Traceable Standards			
Flow Rate Transfer Std	Every 365 days and 1/ calendar year	Resolution 0.02m3/min ±2% reproducibility	1 40 CFR Part 50 App B Sec 7.8 2)Method 2.2 Sec 2.540 3) CFR Part 50 App B Sec 7.8
Field Thermometer	Every 365 days and 1/ calendar year	2°C resolution	1)40 CFR Part 50 App B Sec 7.5 2)Recommendation 3)40 CFR Part 50 App B Sec 7.5
Field Barometer	Every 365 days and 1/ calendar year	± 5 mmHg resolution	1 40 CFR Part 50 App B Sec 7.6 2)Recommendation 3)40 CFR Part 50 App B Sec 7.6
Clock/Timer Verification	every 90 days and 4 times a calendar year	± 2 min/24-hour	1,2 and 3) Method 2.2 Sec 2.3
Lab Activities			
Analytical Standards			
Reagents (HNO3 and HCL)	all	ACS reagent grade	1,2 and 3) 40 CFR Part 50 App G Sec 6.2.1
Pb nitrate Pb (NO3)2	all	ACS reagent grade (99.0% purity)	1,2 and 3) 40 CFR Part 50 App G Sec 6.2.8

*** Air Quality has historically ran the motors for 600 hours with no additional problem.**

7.3 Network Scale

Representativeness is defined as a measure of the degree to which data accurately and precisely represents a selected characteristic of a monitored system. Support in achieving representativeness is provided through adhering to the requirements provided in:

- 40 CFR Part 58, Appendix D (Network Design Criteria for Ambient Air Quality Monitoring)
- 40 CFR Part 58, Appendix E (Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring)

Each monitor operated is assigned a scale of representativeness based upon the definitions of 40 CFR Part 58, Appendix D.

- **Micro Scale** – describes air volumes associated with area dimensions ranging from several meters up to about 100 meters (m).
- **Middle Scale** – describes air volumes associated with area dimensions up to several city blocks in size with dimensions ranging from about 100 meters to 500 meters.
- **Neighborhood Scale** – describes air volumes associated with an area of a city that has relatively uniform land use with dimensions in the 500 meters to 4,000-meter range.
- **Urban Scale** – describes air volumes within cities with dimensions on the order of 4,000-meters to 50,000-meters. This scale would usually require more than one site for definitions.
- **Regional Scale** – describes air volumes associated with rural areas of reasonably homogeneous geography, without large sources, that extends for tens to hundreds of kilometers.

8.0 Training Requirements

All Air Quality employees involved in the collection, handling and analysis of environmental data must possess the minimum level of education, training and experience necessary to meet the demands of their position. These are reflected in the class specifications for the job position or the employee position description.

8.1 Personnel Qualifications

Qualifications of Air Quality personnel are assured through the requirements and operation of the Knox County Government hiring process. If the applicant meets the minimum criteria for the position, their information (copy of application, resume) is forwarded to Air Quality for review. Interviews will be set up with qualified candidates. During the interview process, the applicants are questioned not only about their technical expertise but also about their organizational, interpersonal & communication skills as well as work habits. References may be contacted for confirmation of work experience.

8.2 Training

New employee training includes at minimum: reviewing of quality documents (SOP's, QAPP, Quality Bulletins, etc), reviewing of applicable local and federal regulations, and on the job mentoring with experienced staff. Depending on the tasks they perform, they may be required to participate in a 40-hour HAZWOPER training and Visible Emissions Evaluation training. New employees are partnered with senior employees so that new employees understand their job responsibilities; both the quantity and quality of work to be completed. Air Quality relies heavily on on-the-job training (OJT). Employees must familiarize themselves with the CFR requirements, QA procedures, SOPs and QAPP. Additionally, how-to videos for technical and QC/QC functions are available on the shared drive for the air monitoring staff. Program Managers are responsible for notifying new employees of all required training and document competition of training. New staff are evaluated at six and twelve months after initial hire by the appropriate program manager. A plan for achieving performance objectives are included in the employee development portion of the evaluation.

Methods employed in the collection and analysis of environmental samples and environmental data are subject to continual review and improvement. Continuing educational courses offered by vendors, EPA, APTI and Metro 4 are available as funding allows. Additionally, all employees are actively encouraged to pursue online training opportunities whenever possible. These courses and seminars may be provided as videotapes, closed circuit transmission, and/ or web based real-time interactive formats. Any certifications issued for completed trainings will be placed in the employee's training folder.

The monitoring field staff will follow the specific directions within the Operations Manual supplied by the monitor vendor when servicing or performing QC and/or remedial checks on the monitors. All staff has accessibility to SOPs, operation manuals, and technical service contacts and will follow directions as specified in the approved Standard Operations Procedures. Technical and QA/QC how to videos are available on the shared drive for monitoring staff. These training videos are assigned by the AMPM for staff to view according to their job responsibilities. Employees are aware of the EPA website http://www.epa.gov/ttn/amtic/ where QA/QC and technical guidance documents can be obtained. All air monitoring personnel have sufficient training to currently perform necessary functions at an acceptable level.

8.3 Annual Review

All staff within the department are evaluated annually to ensure that they are performing their assigned duties and are working according to the guiding quality documents. Staff performance evaluations are written by the supervising manager and reviewed and approved by the Director. Managers are evaluated by the Director. Staff performance evaluations may include ride-along inspections in order to accurately review the quality of the work performed. Each employee evaluation is performed in accordance with documents provided by Knox County Human Resources. The staff performance evaluation includes an assessment of job duties,

completeness, thoroughness, and timeliness of duties performed. Employee competency checks should be performed annually by the Air Monitoring Program Manager. The AMPM will observe the employee perform various routine tasks and sign off on the check sheet (figure 8.1) that the task was observed. The proficiency check process allows management to assess the staff's understanding of standard procedures and determine future training needs.

Figure 8.1 Employee Competency Check Sheet

Year Employee Competency Check Sheet — Employee Name

PM 2.5

Date: _____	Fill out tracking sheet	Observed/Reviewed by: _____
Date: _____	Filters to magazines	Observed/Reviewed by: _____
Date: _____	PM 2.5 filter exchange	Observed/Reviewed by: _____
Date: _____	Downloading PM 2.5 data	Observed/Reviewed by: _____
Date: _____	Shipping PM 2.5 filters	Observed/Reviewed by: _____
Date: _____	Verification/Calibration	Observed/Reviewed by: _____

Lead

Date: _____	Pb Filter Prep.	Observed/Reviewed by: _____
Date: _____	Pb Filter Exchange	Observed/Reviewed by: _____
Date: _____	Pb Shipping	Observed/Reviewed by: _____
Date: _____	Review of Pb Calculations Sheet	Observed/Reviewed by: _____

Ozone

Date: _____	Precision Check	Observed/Reviewed by: _____
Date: _____	Weekly Check	Observed/Reviewed by: _____
Date: _____	Level II to Level III Comparison	Observed/Reviewed by: _____

PM 10

Date: _____	Filter Exchange	Observed/Reviewed by: _____
Date: _____	Verification/Calibration	Observed/Reviewed by: _____

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8.4 Safety and Additional Training

Air Quality staff have the potential to encounter hazardous situations. To minimize these risks, in accordance with the Knox County Safety Program, Air Quality utilizes a web-based training module. The county safety officer and the health department training coordinator assign modules to staff according to their Job Hazard and Risk Assessments. These trainings include but are not limited to Hearing Conservation, Hazard Communication and Fire Extinguishers. Additional training such as Slips and Falls, Severe Weather, and Laboratory Safety are assigned by the Director. Certifications of completion are retained within the training module and can be accessed by Knox County Risk Management. Staff receive additional training through EPA-APTI, EPA-NETI, Metro 4 and workshops as needed.

9.0 Documentation and Records

The following information describes the document and records procedures for the Ambient Air Quality Monitoring Program. The Reporting Package is the collection of documents and records pertaining to all data required and all other data deemed important including documents and records required to support the concentration data reported to EPA.

9.1 Data Reporting Package Format and Documentation Control

Table 9.1 lists the documents and records that are pertinent to the reporting package. All raw data required for calculations, the submissions to the AQS database, and the QA/QC data shall be collected electronically or on data forms that are included in the field and analytical methods, see Section 11. All hardcopy information shall be filled out in indelible ink. Corrections shall be made by inserting one line through the incorrect entry, initialing and dating the correction, and placing the correct entry alongside the incorrect entry if this can be accomplished legibly or by providing information on a new line if the above is not possible.

Table 9.1 Reporting Package Information

Categories	Record/Document Type	File Location
Management and Organization	Reporting Agency Information	Air Lab/QAPP
	Organizational Structure	Air Lab/ QAPP
	Personnel Training	Air Lab in file
	Quality Management Plan	Air Lab/ electronic
	Grant Allocation	Main Office
	Support Contracts	Main Office
	Records Retention Policy, Revision 0	Air Lab/electronic
Site Information	Network Description	Air Lab/ AMP, QAPP
	Site Files	Air Lab/electronic
	Site Maps	Air Lab/AMP, QAPP
	Site Pictures	Air Lab/ AMP, QAPP
Environmental Data Operations	Quality Assurance Project Plans	Air Lab/ electronic
	Standard Operating Procedures	Air Lab/electronic
	Field and Lab Logs	Air Lab/archive boxes
	Custody Records	Air Lab/ archive boxes
	Maintenance Records	Air Lab/logbooks
	Standard Certifications	Air Lab/file, QA Program
Raw Data	Original Data (Routine and QC)	Air Lab/ electronic & archive boxes
Data Reporting	AQI Reports	Air Lab/ archive boxes
	Data/Summary Reports	Air Lab
	Annual Monitoring Plan (AMP)	Air Lab/electronic
	Journals/Articles/Papers/Presentations	Air Lab
Quality Assurance	Control Charts	Air Lab/electronic
	Data Quality Assessments	Air Lab
	Quality Assurance Reports	Air Lab
	Technical System Audits	Air Lab
	Response/Corrective Action Reports	Air Lab
	Audit Results	Air Lab/electronic & file
Data Management	Data Management Plans/Flowcharts	Air Lab/QAPP, QMP, SOP
	Data Management Systems	Air Lab/QAPP, QMP

9.1.1 Logbooks

Each operator will be responsible for obtaining and maintaining appropriate field and lab logbooks. These logbooks will be bound, uniquely numbered and associated with a specific monitor or operation. The logbooks will be used to record information about the site and laboratory operations, as well as document routine operations. The logs are brought in monthly and scanned into the shared drive.

Completion of data entry forms, both handwritten and electronic, associated with all routine environmental data operations, are required even when the field logbooks contain all appropriate information required for the routine operation being performed.

Field logbooks will be used for each sample site and specific monitor. At sites with only one monitor a single logbook will serve as both the site logbook and the monitor logbook. Each logbook should be hardbound and paginated. Operators must make logbook entries even when printed forms are used, this may include the use of “stickies”, a compact form printed on clear sticky paper to aid in transferring data to the logbook.

9.1.2 Electronic Data Collection

Continuous ambient air data (ozone & PM continuous) are collected electronically via AirVision data management system. Additionally, AirVision data management system collects diagnostic (flow, temperature, pressure, etc) and shelter temperature data. Sequential PM 2.5 samplers record electronic data which is collected via memory stick transfer and stored on the shared drive. In order to reduce the potential for data entry errors, automated systems will be utilized where appropriate. Information on these systems is detailed in Section 19. The electronic data collection in AirVision is backed up nightly on the C: drive of the local AirVision Computer. Quarterly, a manual back up is performed and placed on the shared server.

9.1.3 Quality Bulletins

Quality Bulletins are created by the QA Officer and signed by the Ambient Air Monitoring Program Manager. These documents update or clarify procedures and protocols within a Standard Operating Procedure (SOP) for immediate implementation. Quality Bulletins are printed, signed and distributed to staff, as well as attached to any SOPs they augment. These documents provide an efficient way to implement procedural changes that shall be included in the future SOP revision.

9.1.4 Chain of Custody

Any samples collected for analysis that are packaged and transported to another location are required to be accompanied with a Chain of Custody (COC) form that includes specific information regarding the sample. This form assists in tracking the integrity of the sample through the various stages of transport and receipt. COCs are stored electronically on the shared drive. Section 12 of this document covers CoCs in greater detail. General content of the form includes:

- Submitter – Person submitting the sample to the lab
- Submission Date- Date the sample was transferred to a new entity
- Delivery Method- Mode of transport used to transfer possession of the sample(s)
- Sample specific information- Date, time of sample, site identifiers, field operator

9.2 Data Reporting Package Archiving and Retrieval

Air Quality maintains records in appropriate files that allow for the efficient archival and retrieval of records.. All the information listed in Table 9.1 will be retained for five years from the date of collection in accordance with the Records Retention Policy (See Appendix A). However, if any litigation, claim, negotiation, audit or other action involving the records has been started before the expiration of the five-year period, the records will be retained until completion of the action and resolution of all issues which arise from it, or until the end of the regular five-year period, whichever is later.

10.0 Network Description

The purpose of this section is to:

- Identify the functions associated with Knox County's Ambient Air Quality Monitoring Network
- Outline the network's objectives
- Establish the criteria for sampling network design and monitoring site selection
- Identify the intended frequency

The primary function of the Air Monitoring Program is to verify compliance with the NAAQS. Other purposes include determining trends over time, providing ozone and $PM_{2.5}$ data to the public, and correlating health effects to air quality. Sampling network design and monitoring site selection comply with the following appendices of 40 CFR Part 58:

- Appendix A – Quality Assurance Requirements for state and Local Air Monitoring Stations
- Appendix D – Network Design for State and Local Air Monitoring Stations and National Air Monitoring Stations, and Photochemical Assessment Monitoring Stations
- Appendix E – Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring

10.1 Network Objectives

The Ambient Air Quality Monitoring Network is designed to meet a minimum of six basic monitoring objectives. These basic monitoring objectives are to:

- determine the highest concentrations expected to occur in the area covered by the network,
- determine representative concentrations in areas of high population density,
- determine the impact of significant sources or source categories on ambient pollution levels,
- determine general background concentration levels,
- determine the extent of regional pollutant transport among populated areas and in support of secondary standards, and
- determine the welfare-related impacts in rural and remote areas (such as visibility impairment and effects on vegetation).

Air Quality utilizes the network design criteria specified in 40 CFR Part 58, Appendix D to establish the appropriate network configuration necessary to meet these objectives.

10.1.1 Monitoring Types

Each monitor within Air Quality's Ambient Air Quality Monitoring Network is assigned one of the following monitoring objective designations:

- Population exposure – the monitor is located in an area associated with high population density.
- Background – the monitor is located where manmade pollutant emissions are minimal.
- Transport – the monitor is located to measure pollutants transported from other areas.
- Maximum concentration – the monitor is located where a high concentration of the pollutant is expected (often based on results of receptor models).
- Comparison study – the monitor is located adjacent to other instrumentation measuring the same pollutant to compare different sampling/monitoring methodologies
- Air Quality Index (AQI) – the monitor provides data primarily for reporting to the AQI (previously called the Pollutant Standards Index).

Data collected within the network must be representative of the spatial area under study. The goal in siting a monitoring station is to match the spatial scale represented by the samples obtained with the spatial scale most appropriate for the monitoring objective of the station. For a description of representative measurement scales see Section 7.3.

10.2 Site Selection

The selection of a specific monitoring site includes the following activities:

- developing and understanding the monitoring objective and appropriate data quality objectives,
- identifying the spatial scale most appropriate for the monitoring objective of the site,
- identifying potential locations where the monitoring site could be placed,
- identifying the specific monitoring site, and
- adhering to the site selection criteria specified in 40 CFR Part 58, Appendix D.

Four criteria should be considered when evaluating potential sites. Monitoring sites should be oriented to measure the following (singly or in combination as appropriate for the sampling objective):

- impacts of known pollutant emission categories on air quality,
- population density relative to receptor-dose levels, both short- and long-term,
- impacts of known pollutant emission sources (area and point) on air quality, and
- representative air quality.

Selection according to these criteria requires detailed information concerning the location of sources, geographic variability of ambient pollutant concentrations, meteorological conditions, and population density. Selection of the number, geographic locations, and types of sampling stations are, therefore, a complex process. The sampling site selection process also involves consideration of the following factors:

- **Economics –** The quantity of resources required to accomplish all data collection activities, including instrumentation, installation, maintenance, data retrieval, data analysis.QA and data interpretation, must be established.
- **Security –** In some cases, a preferred location may have associated problems that compromise the security of monitoring equipment (i.e. high risk of theft, vandalism etc.). If such problems cannot be remedied through the use of standard measures such as additional lighting and fencing, then an attempt to locate the site as near to the preferred location as possible shall be made.
- **Logistics –** This process includes procurement, maintenance, and transportation of material and personnel for the monitoring operation. The logistics process requires full knowledge of all aspects of the data collection operation including planning, reconnaissance, training, scheduling, safety, staffing, procuring goods and services, communications and inventory management.
- **Atmospheric Considerations –** These considerations may include spatial and temporal variably of pollutants and their transport. Effects of buildings, terrain, and heat sources or sinks on air trajectories can produce localized anomalies of pollutant concentrations. Meteorology must be considered in determining the geographic location of a site as well as the height, direction, and extensions of sampling probes. Evaluation of a local wind rose is essential to properly locate many monitoring sites (e.g. siting either to detect or avoid emissions from specific sources).
- **Topography –** Evaluation of the local topography based upon land uses maps, U.S. Geological Survey topographic maps from Knoxville Geographic Information Systems (KGIS), and other available resources should be considered. Minor and major topological features that impact both the transport and diffusion of air pollutants should be evaluated.
- **Pollutant Considerations –** The monitoring site location for a specific pollutant may or may not be appropriate for another pollutant. Evaluation of the changes that pollutants undergo temporally and spatially must be considered in order to determine the applicability of each particular site for a specific pollutant.

Interdependence exists between all of the factors listed above. Consequently, an iterative procedure must be employed in order to successfully select appropriate sites that can provide the data necessary to accomplish the project's stated objectives. The network is assessed every 5 years and each site is evaluated on a weighted scale to review its usefulness in meeting the project objective. In situations where the sites do not specifically meet the requirements necessary to obtain the project objectives, reevaluation of the project will occur.

10.3 Monitor Placement

The placement of each monitor is generally determined by the defined monitoring objective. Monitors are thus usually placed according to potential exposure to pollution. Due to the various factors discussed above, tradeoffs are often necessary to locate a site for collection of optimally representative data. Final placement of a particular monitor at a selected site is dependent on physical obstructions and activities in the immediate area. As specified in 40 CFR Part 58, Appendix E, monitors should be placed away from obstructions such as tress and fences in order to avoid their effects on airflow. To present sampling bias, airflow around monitor sampling probes must be representative of the general airflow in the area. In addition, the availability of utilities (i.e. electricity, cable and telephone services) is critical. Site evaluations are done annually as part of the Annual Network Plan, ensuring sites continue to meet siting criteria.

10.4 Probe Siting Criteria for Pollutant Sampler

General probe and monitoring path siting criteria for criteria pollutants shall adhere to the requirements listed in 40 CFR 58, Appendix E and the instructions outlined below. Appendix E is the primary siting reference and should be consulted first when evaluating siting criteria.

10.4.1 Ozone (O_3)

The probe intake is to be located from 3-15 meters above the ground. The probe must be at least 1 meter horizontally or vertically away from any supporting structures. There must be 270° of unrestricted airflow around the sample probe. It should be at least 20 meters away from the dripline of any trees or shrubs, but must be at least 10 meters away due to their ability to alter normal wind flow patterns and provide surfaces for absorption or reactions (the scavenging effect of vegetation is greater for ozone than for other criteria pollutants). Trees and shrubs shall not be located between a nearby source and the sample probe. Samplers monitoring O3 transported over a long distance, such as from an urban city core area, should be sited so that no tress are within 20 meters of the sampler along the predominant summer daytime wind direction. The distance shall be measured from the dripline or outside edge of the crown, the trunk. For monitors to be operated at the same site for several years, it is best to allow some additional space for vegetation growth. In situations where a tree is considered an obstruction, the trees dripline ***must*** be at least 10m from the probe. In order to not be considered an obstruction, the distance between the probe and the tree must be at least twice the height that the obstruction extends above the probe. If a tree is deemed an obstruction, and is greater than 10 meters away, then the obstructed path area must be taken into consideration when reviewing unrestricted airflow degrees.

10.4.2 Particulate Matter (PM)

When monitoring for PM, it is important to select a site or sites where the collected particulate mass is representative of the monitored area. Optimum placement of the sampling inlet for PM is at breathing height level. However, practical factors such as prevention of vandalism, security, and safety precautions must also be considered. Given these considerations, the sampler inlet for micro scale PM monitors must be between 2 and 7 meters above the ground. For middle or larger spatial scales, the inlet must be 2-15 meters above the ground.

The sampler must be located away from obstacles such as buildings, so that the distance between the obstacle and the sampler is at least two times the height that the obstacle protrudes above the sampler. If the sampler is located on a roof or other structure, there must be 2 meters separation from walls, parapets, penthouses, etc. No furnace or incarnation flues should be nearby. Collocated samplers must be at least 1 meter, but not greater than 4 meters, away from each other. Samplers ***should*** be located at least 20 meters from the dripline of the nearest trees but ***must*** be 10 meters from the dripline when it acts as an obstruction.

There must be unrestricted airflow in an arc of at least 270° around the sampler. The predominant wind direction for the season with the greatest pollutant concentration potential must be included in the 270°

unrestricted arc. If the sampler is to measure concentrations from a road or point source, there must be no obstructions between the road or point source and the sampler, even when other spacing from obstruction criteria is met.

There are many factors to be considered in establishing a particulate sampling location. These include: accessibility under all weather conditions, availability of adequate electricity, a paved area or ground cover year-round to minimize windblown dust, and the security of the monitoring personnel and equipment. The sampler must be situated where the operator can reach it safely despite adverse weather conditions. If the sampler is located on a rooftop, care should be taken that the operator's personal safety is not jeopardized by a slippery roof surface. Consideration should also be given to the fact that routine operational procedures such as calibration, maintenance, and filter installation and recovery involve transporting supplies and equipment to and from the monitoring site. The lack of suitable power source can often result in the loss of many samples because of power interruptions or fluctuations. To ensure that adequate power is available, consult the manufacturer's instructional manual for the sampler's minimum voltage and power requirements. The security of the sampler depends mostly on the location. Rooftop sites with locked access and ground level sites with fences are common. In all cases, the security of the operating personnel as well as the sampler should be considered.

10.4.3 Lead (Pb)

Lead monitoring has the same requirements as PM described in section 10.4.2. Knox County has one lead source emitting > 0.5 Ton annually and thus requires ambient monitoring. It is important to select a site or sites where the collected particulate mass is representative of the monitored area. It is especially important for source-oriented monitors to be located downwind of the source. Optimum placement of the sampling inlet for lead in a micro or middle scale site is between 2 and 7 meters above the ground.

10.5 Sampling Frequency

Minimum sampling frequencies are established by EPA and followed accordingly. In instances requiring every third and sixth day sampling, specific days must be sampled in order that the entire nation is sampling on the same day. This intermittent sampling is accomplished in accordance with a national sampling schedule published annually by EPA. See table 10.1 for the sampling schedule and frequency of the Criteria Pollutant Network. The National Sampling Calendar can be found on the AMTIC website https:www3.epa.gov/tnn/amtic/calendar.html

Table 10.1 Criteria Pollutant Sampling Schedule and Frequency

Pollutant	Site	Time Frame	Frequency	Monitor Type
Ozone	East Knox, Spring Hill	Midnight to midnight	24/7	Continuous
PM 2.5	Spring Hill, Rule, Bearden	Midnight to midnight	1 in 3	Filter based
PM 2.5 (Collocated)	Bearden, Air Lab	Midnight to midnight	1 in 6	Filter based
PM 10	Air Lab	Midnight to midnight	24/7	Continuous
Lead	Ameristeel, Burnside	Midnight to midnight	1 in 6	Filter based

The minimum number of samples required for appropriate summary statistics should be sampled. At least 75% of total possible observations must be present before summary statistics are calculated. The exact requirements appear in Table 10.2 below. For filter-based $PM_{2.5}$ monitoring, 40 CFR part 50 App N is followed when make-up samples are needed.

Table 10.2 Requirements for Calculating Summary Statistics

Pollutant	Completeness Requirement (%)	Time Frame
Ozone	75% / 90%	Per hour, 8-hour, and season/ per 3 years
$PM_{2.5}$	75 %	Per quarter
PM_{10}	75 %	Per quarter
Lead	75 %	Per month and average of the 3 constituent monthly means

The emphasis of Knox County's Ambient Air Quality Monitoring Network has been in areas where elevated concentrations are known or suspected. Expanded monitoring (i.e. number of sites and increased sampling frequency) occurs in areas where a previous exceedance of a standard has been recorded.

11.0 Sampling Method Requirements

11.1 Purpose

The purpose of this section is to:

- identify the sampling methods,
- identify the equipment used in sampling, and
- outline procedures for collecting the required environmental samples.

Describe the:

- equipment used in the data collection network,
- necessary support facilities,
- sample reservation requirements,
- implementation requirements,
- required materials, and
- processes for preparing and decontamination sample equipment.

Identify the:

- corrective actions necessary to reestablish network data integrity,
- responsible parties to implement the corrective actions, and
- methods required to verify corrective action effectiveness.

11.2 Monitoring Technology/Methodology

Methods for ambient air monitoring are continuously changing and advancing. There are two general categories of samplers: intermittent samples and continuous samplers. In intermittent (sequential/non-continuous) sampling a physical sample is collected using a monitoring device that passes ambient air through a filter, in a container or by exposing a sample media to a sample stream. Sample media is then removed and analyzed by a laboratory method (addressed in section 12). In continuous (real-time) sampling not physical samples are collected. The analysis is performed within the sampler itself using a unit specific methodology.

The analyzers and monitors, method codes and the related SOPs used in the Knox County Ambient Air Monitoring Network are listed in tables 11.1 and 11.2 below.

Table 11.1 Air Quality Network Analyzers

Pollutant	Analyzer	EPA Reference/ Equivalence	SOP
Ozone	Teledyne (API) 400 series	EQOA-0992-087	Ozone Monitoring with UV Spectrophotometry Standard Operating Procedures, Rev. 0
PM_{10} continuous	Teledyne T640X	EQPM-0516-0239	Teledyne T640X Model Continuous PM10/PM2.5 SOP, Rev 0,
$PM_{2.5}$ continuous	Teledyne T640X	EQPM-0516-0238	Teledyne T640X Model Continuous PM10/PM2.5 SOP, Rev 0,

Table 11.2 Air Quality Network Monitors

Pollutant	Analyzer	EPA Reference/ Equivalence	SOP
PM $_{2.5}$ sequential	R&P 2025 Partisol Plus	RFPS-0498-145	THERMO Model 2025 Sequential Sampler Standard Operating Procedures, Rev. 0 IML Low Volume PM QAPP, Rev. 14
TSP Lead (Sampling method)	Determination of suspended Particulate matter in the Atmosphere, Hi-Vol Method,	40 CFR Part 50, Appendix B	Volumetric-Flow-Control (VFC) High Volume TSP/Pb Monitors Standard Operating Procedures, Rev 0, Support for the EPA national Contract for Lead Analysis QAPP, ERG Rev 0

11.3 Support Facilities

11.3.1 Monitoring Station Design

The monitoring station design must encompass the operational needs of the equipment, provide and environment that supports sample integrity, and allow the operator to safely and easily service and maintain the equipment. Winter weather conditions must be considered during site selection in order to meet the station safety and serviceability requirements

11.3.2 Shelter Criteria

Continuous air monitoring analyzers should be sheltered inside a shelter capable of fulfilling the following requirements:

- The Shelter temperature must meet the most limiting temperature range of monitoring equipment present. Air Quality has monitoring equipment that can operate at a variety of temperature ranges that can be found in the instrument specific SOPs in Appendix C-F.
- The power supply should not vary more than ±10% from 117 Alternation Current Voltage (ACV).
- The shelter must protect the instrumentation from precipitation and excessive dust and dirt, provide third wire grounding as in modern electrical codes, meet federal Occupational Safety and Health Administration (OSHA) regulations, and be cleaned regularly to prevent a buildup of dust.
- The shelter must protect the instrumentation from any environmental stress such as vibration, corrosive chemicals, intense light, or radiation.
- Shelters shall be kept clean and well maintained by the site operator. Shelter cleanliness is evaluated during quarterly audits.

11.3.3 Probe Design

For reactive-gas monitors (O3) the probe manifold material (sample lines and fittings) must be Teflon® or borosilicate (Pyrex®) glass per 40 CFR Part 58, Appendix E. These materials lessen the oxidation of gases as they enter the sampling train. Furthermore, Teflon® or borosilicate (Pyrex®) glass must be used as the probe material for delivering calibration test gas concentrations. Additionally, all ozone monitors must have a sample residence time of less than 20 seconds. Residence time is the amount of time it takes for the sample to travel from the probe inlet to the sample intake. Residence time is calculated by totaling the volume of total sample line as well as the inline filter, and solenoid valve assembly then dividing that total volume by the flow rate of all instruments and pumps used.

Ozone probes are designed such that precisions checks and calibration verifications can be performed through the probe while the analyzer is operating in its normal sampling mode and the test atmosphere must pass through all filters, scrubbers, conditioners and other components used during normal ambient sampling. i.e. in such a way that calibration gas can be introduced and travel the sample train.

The probe protrudes from a water tight box on top of the station. A Teflon or glass funnel is used to shield the ¼" sample line inlet from precipitation. Inside the box, the sample line is attached to a 20 volt solenoid valve. This valve provides a switch and is attached to the ambient sample line which goes through the insulated pipe into the shelter and into the analyzer. The valve is also connected to the calibration gas line which originates from the calibrator, comes through the insulated pipe and into the solenoid valve. The Solenoid valve power supply is controlled by the data logger and a manual override plug. The valve is set to the "open" position when no power is being supplied. This ensures the continuation of ambient monitoring in the event of power failure or having no plug connected.

11.4 Sample Collection

All samples for criteria pollutants will be collected using Federal Reference of Equivalent Methods (Tables 11.1 and 11.2). Procedures set forth in the approved standard operating procedures (Appendix C-F), and the reference/equivalent method approvals will be used. A full list of approved Federal Reference Methods and Federal Equivalent Methods can be found on the AMTIC website https://www.epa.gov/amtic/air-monitoring-methods-criteria-pollutants

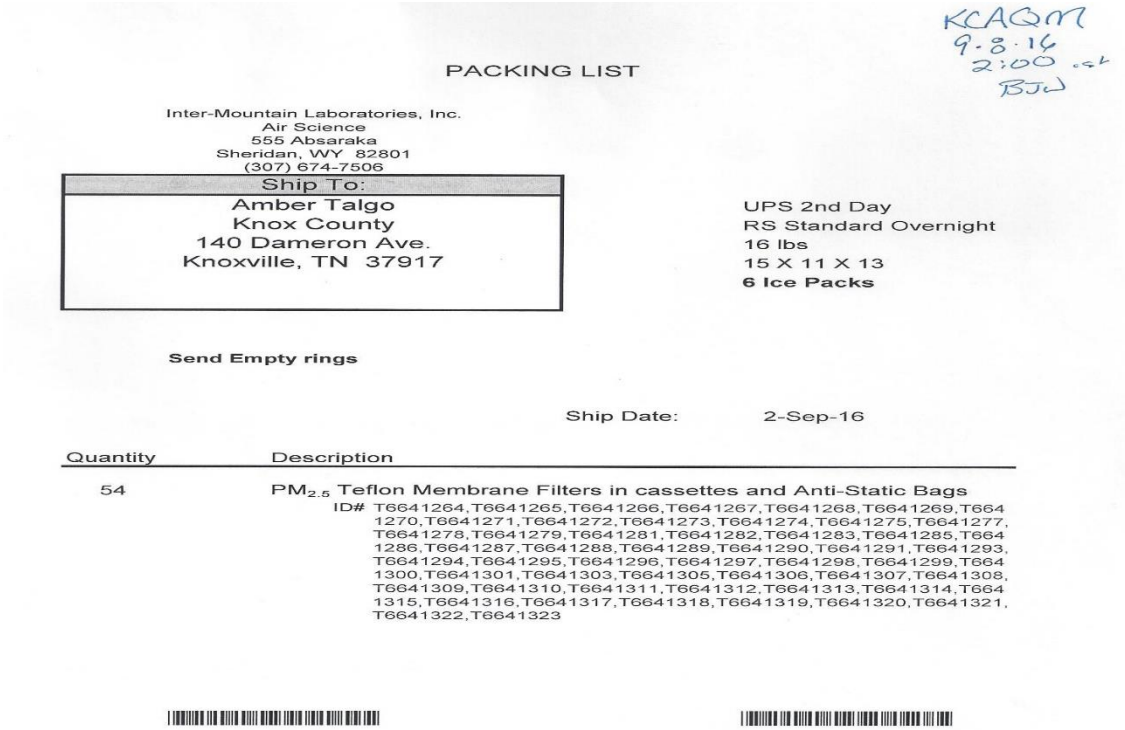
12.0 Sample Handling and Custody Procedures

The physical collection of intermittent samples, sample transport, and sample preservation techniques adhere to the requirements of 40 CFR Part 50, Appendix G and L, the “Quality Assurance Handbook for Air Pollution Measurements Systems, Volume II,” the SOPs for Lead and $PM_{2.5}$, and Ambient Air Specific Methods (See table 11.2) Due to the use of data for comparison to the NAAQS and the requirement for extreme care in sample collection, sample custody procedures must be followed. An example of custody procedures are the Chain of Custody (CoC) Record Form used to ship $PM_{2.5}$ filters to Inter Mountain Laboratories (IML) and Lead filters to Eastern Research Group (ERG) which are used to track the states of filter handling throughout the data collection operation. The CoC shall be stored electronically prior to shipment and maintained at the analytical laboratory for proof of receipt. CoC discrepancies will be resolved via email with the laboratory.

12.1 PM 2.5

Pre-weighed filters are received from IML in a cooler. This cooler will be used for returned shipping. These filters must be used within 30 days of the initial weighing date listed on the individual filter bag. A packing list is included that itemizes the filters shipped (Figure 12.1). The packing list is signed and dated when received. The filters are received pre-loaded into cassettes and individually packed in Ziploc bags. Each filter bag has a label on it with the filter number, the expiration date (date it must be used by), and cassette number. Filters are only handled using powder free anti-static gloves. Once the filters are exposed, they must be retrieved within 177 hours. Once retrieved from the field, they are immediately placed in cold storage. First, they are placed in an ice packed cooler during transport from the monitoring site to Air Quality’s lab. Upon arrival at Air Quality’s lab they are placed into the $PM_{2.5}$ refrigerator set to keep samples between 0-4° C. The maximum and minimum temperatures are recorded in a logbook for the refrigerator. Filters are placed back in their shipping bags, placed in an ice packed cooler and shipped back to IML every two weeks, accompanied by a CoC. (Figure 12.2). IML archives the filters for 1 year in cold storage. The filters are returned to Air Quality after the year and are kept for 4 more years at room temperature.

Figure 12.1 Example IML Packing List



KCAQM
9-8-16
2:00 pm
BJW

PACKING LIST

Inter-Mountain Laboratories, Inc.
Air Science
555 Absaraka
Sheridan, WY 82801
(307) 674-7506

Ship To:
Amber Talgo
Knox County
140 Dameron Ave.
Knoxville, TN 37917

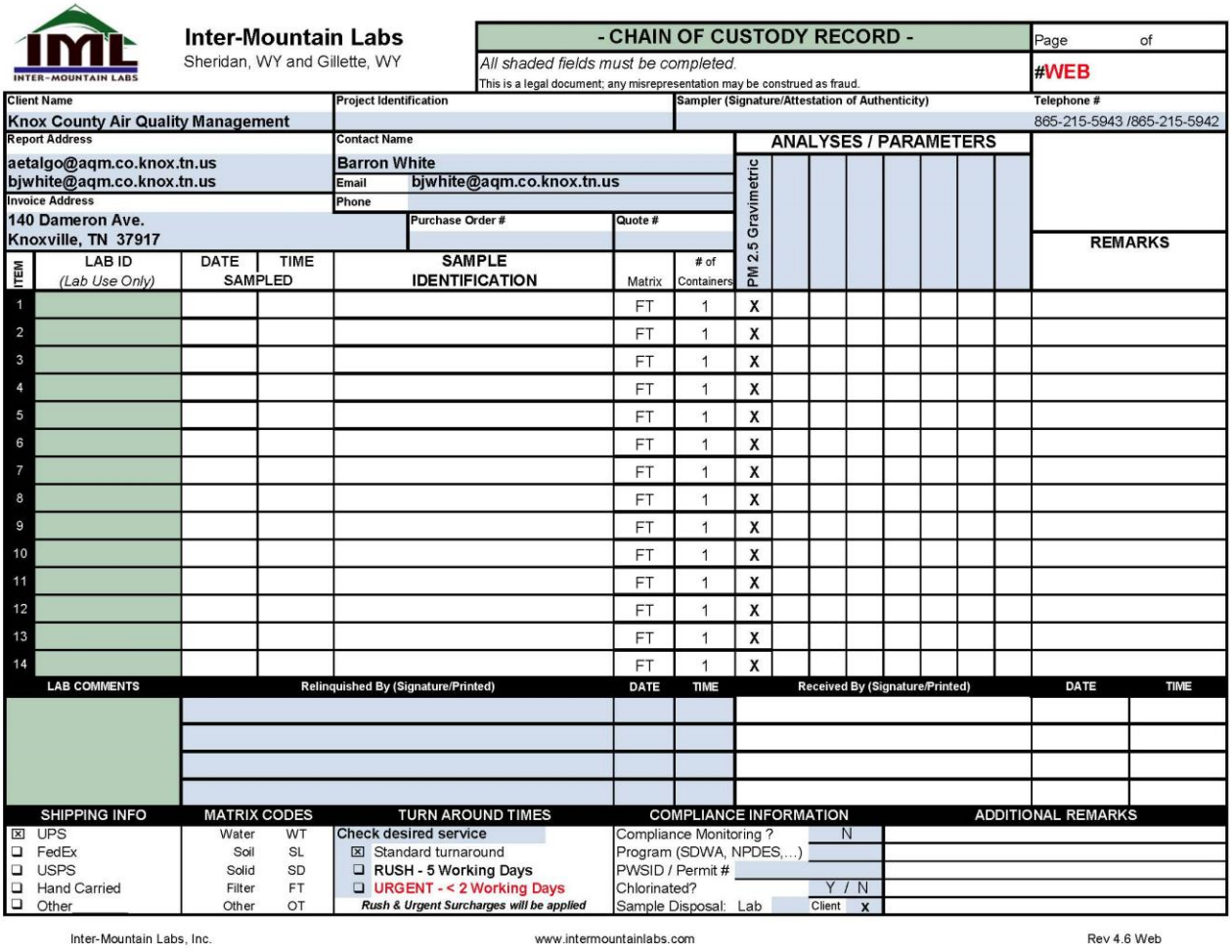
UPS 2nd Day
RS Standard Overnight
16 lbs
15 X 11 X 13
6 Ice Packs

Send Empty rings

Ship Date: 2-Sep-16

Quantity	Description
54	$PM_{2.5}$ Teflon Membrane Filters in cassettes and Anti-Static Bags ID# T6641264,T6641265,T6641266,T6641267,T6641268,T6641269,T6641270,T6641271,T6641272,T6641273,T6641274,T6641275,T6641277,T6641278,T6641279,T6641281,T6641282,T6641283,T6641285,T6641286,T6641287,T6641288,T6641289,T6641290,T6641291,T6641293,T6641294,T6641295,T6641296,T6641297,T6641298,T6641299,T6641300,T6641301,T6641303,T6641305,T6641306,T6641307,T6641308,T6641309,T6641310,T6641311,T6641312,T6641313,T6641314,T6641315,T6641316,T6641317,T6641318,T6641319,T6641320,T6641321,T6641322,T6641323

Figure 12.2 IML Chain of Custody



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Sheridan, WY and Gillette, WY

- CHAIN OF CUSTODY RECORD -

Page of

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 #
Knox County Air Quality Management			865-215-5943 /865-215-5942

Report Address	Contact Name
aetalgo@aqm.co.knox.tn.us bjwhite@aqm.co.knox.tn.us	Barron White Email bjwhite@aqm.co.knox.tn.us
Invoice Address	Phone
140 Dameron Ave. Knoxville, TN 37917	Purchase Order # / Quote #

ANALYSES / PARAMETERS: PM 2.5 Gravimetric

ITEM	LAB ID (Lab Use Only)	DATE SAMPLED	TIME SAMPLED	SAMPLE IDENTIFICATION	Matrix	# of Containers	PM 2.5 Gravimetric	REMARKS
1					FT	1	X	
2					FT	1	X	
3					FT	1	X	
4					FT	1	X	
5					FT	1	X	
6					FT	1	X	
7					FT	1	X	
8					FT	1	X	
9					FT	1	X	
10					FT	1	X	
11					FT	1	X	
12					FT	1	X	
13					FT	1	X	
14					FT	1	X	

LAB COMMENTS	Relinquished By (Signature/Printed)	DATE	TIME	Received By (Signature/Printed)	DATE	TIME

SHIPPING INFO	MATRIX CODES		TURN AROUND TIMES	COMPLIANCE INFORMATION	ADDITIONAL REMARKS
☒ UPS	Water	WT	Check desired service	Compliance Monitoring ? N	
☐ FedEx	Soil	SL	☒ Standard turnaround	Program (SDWA, NPDES,...)	
☐ USPS	Solid	SD	☐ RUSH - 5 Working Days	PWSID / Permit #	
☐ Hand Carried	Filter	FT	☐ URGENT - < 2 Working Days	Chlorinated? Y / N	
☐ Other	Other	OT	*Rush & Urgent Surcharges will be applied*	Sample Disposal: Lab Client x	

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12.2 Lead

Lead filters, unlike PM2.5 filters, do not originate at an outside lab. Air Quality receives the filters new in the box from EPA near the start of each calendar year and ships them to the contract laboratory post-sampling. Lead filters are only handled using powder free anti-static gloves. Once a box of filters is opened, the entire box of filters is inspected, one by one on a light box for pin holes and defects. The box is then labeled as inspected by writing the word “checked”, the initials of the specialist who inspected, and writing the date the inspection was completed on the front of the box. Filters are removed from the box and placed in the filter cassette when ready for use. Each filter is assigned a filter envelope before placement in the field. Filters are collected as soon as practical after the run. They are then brought back to the Air Quality laboratory and removed from the filter cassette, folded in half and placed in a glassine envelope inside the filter envelope. Filter envelopes are stacked and placed in a large envelope for shipment via regular USPS mail, typically a month’s worth at a time, to ERG. A CoC (Fig. 12.3) is filled out containing the required information and shipped with the filters to ERG. ERG keeps the digest of each filter for 6 months. .

Figure 12.3 ERG Chain of Custody

ERG

Chain of Custody TSP/PM10 Lead Analysis (EP-BPA-15-D-0004)

601 Keystone Park Drive, Suite 700, Morrisville, NC 27560

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SITE Knox County call #24

AQS Code 47-093-1017 (1-6)
0023 (7-11), 0027-01 (12-17) 0027-02 (18-23)

Submitter:
Test

Relinquished by: Barron J. White

Date/Time: 7/11/17 12:00pm

Received by:

Date/Time:

#	Filter ID	Date	ANALYSES: TSP (ICPMS)	PM10 (ICPMS)	PM10 (XRF)	Sample Volume (m^3)	Comments	ERG LIMS ID (Lab use only)
1	G7522694	7/6/2017	x					
2	G7522692	7/8/2017	x					
3	G7522687	7/12/2017	x					
4	G7522680	7/18/2017	x					
5	G7522677	7/24/2017	x					
6	G7522674	7/30/2017	x					
7	G7522693	7/6/2017	x					
8	G7522681	7/15/2017	x					
9	G7522683	7/18/2017	x					
10	G7522682	7/24/2017	x					
11	G7522673	7/30/2017	x					
12	G7522696	7/6/2017	x					
13	G7522691	7/8/2017	x					
14	G7522685	7/12/2017	x					
15	G7522678	7/18/2017	x					
16	G7522675	7/24/2017	x					
17	G7522671	7/30/2017	x					
18	G7522695	7/6/2017	x					
19	G7522690	7/8/2017	x					
20	G7522684	7/12/2017	x					
21	G7522679	7/18/2017						
22	G7522676	7/24/2017						
23	G7522672	7/30/2017						
24								
25								

13.0 Analytical Methods Requirements

13.1 Purpose / Background

The analytical method employed for a specific criteria pollutant evaluation is dependent upon the monitoring technology utilized. For the gaseous criteria pollutants like ozone and continuous PM monitors, the analyzers are designed as completely contained monitoring units that do not require additional analytical methods to establish the pollutants' environmental concentrations. The particulate matter criteria pollutants lead and $PM_{2.5}$ do require analytical methods to evaluate the captured sample in order to establish the pollutant concentrations present in the environment.

The FRM employed by Air Quality for $PM_{2.5}$ monitoring follows 40 CFR 50 Appendix L which utilizes a gravimetric analysis. Inter-Mountain Laboratory (IML) has been contracted to conduct the filter analyses. A filter's net weight gain identifies the sample's captured particulate mass. This net weight gain is obtained by subtracting the initial filter weight from the final weight of the exposed filter. Once calculated, the net weight gain can be used with the total filter flow to calculate the concentration for comparison to the daily and annual NAAQS. See IML QAPP and SOP in Appendix G for details. Air Quality uses the National Contract for lead analysis. Air Quality's filters are analyzed via FEM method EQL-0512-202, Inductively Coupled Plasma Mass Spectrometry (ICP-MS). Eastern Research Group (ERG) is the current analytical laboratory for the national contract. Air Quality maintains a copy of ERG's QAPP including the method SOP. The QAPP can also be found on the AMTIC website at https://www3.epa.gov/ttn/amtic/files/ambient/pb/nationalcontractforleadanalysis.pdf. All analytical methods used are previously listed in tables 11.1 and 11.2.

13.2 Preparation of Samples

Most preparation will take place at the contract laboratories. A more detailed description is outlined in the corresponding pollutant SOP.

13.2.1 PM $_{2.5}$

$PM_{2.5}$ 47mm Teflon filters will be received from the US EPA following acceptance testing and forwarded to IML in original packaging. IML returns pre-weighed filters, to Air Quality, pre-installed in filter cassettes and in individual anti-static bags. Pre-weighed filters must be used for sampling within 30 days of the initial weighing. The operator puts filter cassettes in proper filter magazines and installs in the monitor. The filters are retrieved after the sampling run and removed from the magazine. The filters in the cassettes are returned to the anti-static bags and returned to IML for post filter analysis. Post sampled filters are kept between 0 – 4 °C.

13.2.2 Lead

Lead filters, a glass fiber filter, will be received from the US EPA following acceptance testing and prepped for use as needed. Receiving and inspection procedures are described previously in section 12.2. The inspected filters are loaded into a filter cassette and installed in a HI-VOL TSP monitor for sampling. Upon retrieval from the field, the filters are removed from the filter cassette, folded to reduce content loss and placed in a glassine sleeve. The filter and sleeve are then placed in a properly labeled envelope. Filters are batched and shipped monthly to ERG for analysis

13.3 Analysis Method

The analytical instrument used by the contract laboratories is outlined in the contract laboratory specific SOPs and QAPPs (See Appendix G &H).

13.4 Filter Sample Contamination Prevention

Air Quality strives to ensure filters are handled carefully. Filters are handled as minimally as possible. See IML and ERG's SOP and QAPP in Appendix G & H for more information on steps taken by the analyzing lab.

13.4.1 PM 2.5

Filter prep is done on a clean surface, in a non-drafty area that has been wiped down with an anti-static cleaning cloth. Anti-static powder free gloves are worn. Magazine caps are wiped down with an alcohol wipe between uses and stored in a sealable container. The caps are promptly placed on the magazine after filters are installed and again promptly when the magazine is removed from the sampler. Magazines are kept capped during transport and cold storage whenever filters are held within.

13.4.2 Lead

Filter prep is done on a clean surface. The filters are handled using anti-static powder-free gloves. The filter cassettes are cleaned with an alcohol wipe between uses. A dust cover is installed anytime the filters are not installed in the HI-VOL TSP monitor. After sampling and removal from the site, the dust cover is carefully removed as to not displace the particulates collected. Once the filter is removed from the filter cassette, it is folded dirty side in on itself to prevent the loss of particulate and place in a glassine sleeve before being put into the filter envelope.

14.0 Quality Control Requirements

Quality control is the overall system of technical activities that measure 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. In the case of the Ambient Air Quality Monitoring Network, QC activities are used to ensure that measurement uncertainty, as discussed in Section 7, is maintained within acceptance criteria for the attainment of the DQOs. Lists of pertinent QC checks are provided in the standard operating procedures and instrument manuals.

To assure the quality of data from air monitoring measurements, two distinct and important interrelated functions must be performed. One function is the control of the measurement process through broad QA activities, such as establishing policies and procedures to ensure DQOs are met, assigning roles and responsibilities, conducting oversight and reviews, and implementing corrective actions. The other function is the control of the measurement process through the implementation of specific quality control procedures, such as audits, calibrations, checks, replicates, blanks and routine self-assessments.

14.1 Quality Control Procedures

Quality control is achieved through periodic maintenance, performance checks, and control charts. This section details the performance check procedures for calibrations, precision checks, accuracy, and flow rate & performance audits. Tables 14.1-14.4 detail the quality control procedures performed as part of the data quality indicator and the suggested corrective actions if outside the action point. In calculating the percent difference for the objectives, the formula used is

$$\left\{\frac{(\text{monitor value} - \text{reference device value})}{\text{reference device value}}\right\} * 100.$$

Data quality objective (DQO) error limits are promulgated in 40 CFR 58 App A sec 2. Regarding the quality of the measurement system, the objective is to control precision and bias in order to reduce the probability of decision errors. Air Quality refers to the validation templates in the “Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II” as a guide to establish criteria for precision and bias limits that are used to determine the validity of data. Air Quality has also set corrective action limits that in some cases are more strenuous than the validation criteria to identify problems and correct them before the point of invalidating data. These limits are outlined in tables 14.1- to 14.4 and provide the action point at which corrective action should be taken to reduce the loss of data.

Table 14.1 Quality Control and Corrective Action PM_{10} and $PM_{2.5}$ continuous

Data Quality Indicator	Frequency	Action Point	Corrective Action
Parameter Check PM10	Weekly	Shelter Temp. 0-50 °C ±5% of design Flow	Investigate, Check warnings Document actions Perform maintenance/ verification
Flow Verification	Monthly	Main and total , ±3.5% of transfer standard	Investigate, check for damage to bypass line, recalibrate.
Temperature & Pressure Verification	Monthly	±2° C ±10mmHg	Check for leaks, Investigate, Advise Management Perform Cal Verification
Quarterly Internal Flow Audit	Quarterly	±3.5% Main, total Flow	Check for leaks, Investigate, Advise Management. Perform Cal Verification
Leak Check	Monthly	0.0-0.2 µg/m^3	Check all connections, Investigate hoses. Repeat Test. Perform Maintenance

Table 14.2 Quality Control and Corrective Action $PM_{2.5}$

Data Quality Indicator	Frequency	Action Point	Corrective Action
Flow verification	Every 30 days separated by at least 14 days	±3.5% of transfer standard ±4.0% of design value	Leak Check, Document actions Perform multi point Calibration/ Verification
Temperature Verification	Every 30 days separated by at least 14 days	±2°C	Investigate, Check damage/ blockage of fan. Document actions, Recalibrate
Pressure verification	Every 30 days separated by at least 14 days	±10 mmHg	Investigate, Document actions, Recalibrate
Quarterly Internal Audit	Quarterly	±4% of transfer standard ±5 % of design value	Leak Check, Document actions Perform multi point Calibration/ Verification
Laboratory verification	At least Quarterly	Lab QC checks and filter handling verified	Contact Lab
Internal Leak check	Every 30 days separated by at least 14 days	Pass	Investigate, Check cassette ring, seals, O-rings. Replace or lubricate as necessary. Repeat test.
Multipoint Verification	Annually	Same as monthly Flow/ Temperature/ Pressure verifications	Re-calibrate
Collocated Samples	Every 6 days, aggregated quarterly, annually	CV ± 10% for samples > 3.0 µg/m3	Notify Lab Manager Review flow rates of collocated monitors
Calibration Temperature, pressure & Flow	At installation, post-major repair, after failed verification,)	See SOP & Operation Manual	Contact Equipment Manufacturer
Field Blank	10% or 1 per weighing session	< ±30.1 µg change between weighings	Flag data associated with blank filter, investigate possible source of contamination, notify air lab manager

Table 14.3 Quality Control and Corrective Action Ozone

Data Quality Indicator	Frequency	Action Point	Corrective Action
Automated Nightly Zero/Span	Daily	±5% span, ±3 ppb zero	Visit Site to investigate Check warnings Document actions Perform manual calibration verification
Operating Parameters	Weekly	Sample flow 740-860 cc/min, filter and zero air maintenance	Check for leaks, Investigate, Advise Management Perform Cal Verification
Manual Zero/Precision /Span	Bi-weekly	±5% precision &span, 3ppb zero	Check for leaks, repeat test Check parameters Recalibrate
Quarterly Internal Audit	4 times a season	±7% every point & ±3ppb zero	Check for leaks, Investigate, Advise Management Perform Cal Verification
Manual Calibration Verification	Start, mid and End Season	All points <± 2.1% or ≤±1.5 pp difference of best fit straight line whichever is greater and Slope 1 ±.05	Check for leaks, repeat test Check parameters Recalibrate

Table 14.4 Quality Control and Corrective Action Lead

Data Quality Indicator	Frequency	Action Point	Corrective Action
Flow Rate Verification	Monthly	±7%	Check for leaks, Investigate, Advise Management, Replace motor
Multi point Flow Verification	Annually	5 points (over range of 1.1 to 1.7m3/min) ±5% limit of linearity	Check for leaks, Investigate, Advise Management, Replace motor or orifice
Collocated Samples Precision	Every 6 days, aggregated quarterly, annually	CV ± 10% for samples > 0.02µg/m3	Notify Lab Manager, review flow rates of collocated monitors
Quarterly Internal Audit	4 times a season	Flow Rate ±7% audit standard	Check for leaks, Investigate, Advise Management, Replace motor
Flow Rate Bias	Aggregate monthly values, quarterly and annually, by monitor and by network	Absolute bias ± 7%, Signed bias ± 5%,	Notify Lab Manager, Review outliers
Analysis Audits	6 strips/quarter 3 at each concentration	< 10.1% difference (Done by contract laboratory, not conducted by Air Quality.)	Contact contract lab to see if multiple agencies or individual agency is affected

14.1.1 Calibrations

Calibration is the process employed to verify and rectify an instrument's measurements in order to minimize deviation from a standard. This multiphase process begins with certifying a calibration or transfer standard against an authoritative (NIST) standard. The sampling or analytical instrument's measurements are then compared to this calibration/transfer standard. If significant deviations exist between the instrument's measurements and the calibration/transfer standard's measurements, corrective action is implemented to rectify the analytical instrument's measurements. Some equipment, such as the PM2.5 samplers, do not utilize a concentration standard and must rely on the calibration of individual components of the sampling method (flow, temperature, pressure) to ensure data integrity. A calibration verification shall be performed at receipt/ install of monitoring equipment, when major repair occurs, when a calibration verification fails or when it is required to meet manufacturer's recommendations. All Air Quality's instruments must have a calibration verification at least annually to indicate if calibration is necessary. All calibration frequency requirements are listed in the corresponding sampler/pollutant SOP. A calibration should have a verification before and after the calibration takes place to "book-end" data, which indicates that data before and after the calibration was collected within the acceptable limits. A pre-calibration verification is not necessary to do upon installation, but often is helpful to establish out of the box or as found conditions. Calibration procedures for the instruments are found in the SOPs and in the specific instrument's manual. Calibration/ calibration verification ranges and specific references are listed in Table 14.5 below.

Table 14.5 Calibration Ranges

Pollutant	Calibration Range	CFR Reference	SOP Reference
Ozone	Zero, Span (80% of desired upper range limit), 3 point between, should match audit points (except no low level)	40 CFR Part 50 App D Section 4.5	App E Ozone Monitoring with UV Spectrophotometry SOP Section 6.0 (current span of 400 ppb, 110,70,50,35)
PM2.5	3 pts equal distance from ±10% design flow (15.0,16.7,18.4)	40 CFR Part 50 App L Section 9.2	App F Thermo Model 2025 Sequential Sampler SOP Section 3.0
PM10 & PM2.5 Continuous	Single point ± 2% for Main and Bypass flows	40 CFR Part 50 App L Section 9.2	App C Teledyne T640x Model Continuous PM10/PM25 SOP Section 3.0
Lead	Minimum 4 points between 1.1-1.7 std m^3/min	40 CFR Part 50 Appendix B Section 9.3	App D Volumetric-Flow-Control High Volume TSP/Pb Monitors SOP Section 3.4

14.1.2 Precision Checks

Precision as described in 40 CFR part 58 Appendix A, is the measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision estimates for Ozone are determined from the biweekly one-point quality control checks (precision checks); gaseous precision checks are measurements of the analyzer response to a test gas concentration at a level near the national level for ambient air. Ozone operators combine the precision check with the zero and span drift check called ZPS (zero, precision, span). This is performed through the probe following the full sampling path.

Precision estimates for automated and manual PM methods are calculated using the results of collocated samplers. Precision checks will ensure the entire measurement process is within statistical control. Fifteen percent of the $PM_{2.5}$ and lead network sites are outfitted with collocated monitors to actively support precision checks. $PM_{2.5}$ precision, as a relative percent difference, is reported with the weigh data by the contract laboratory and confirmed by the Quality Assurance Officer at least quarterly. For the precision estimate, data pairs are considered valid if both concentrations are greater than or equal to 3 µg/m3 . The precision data is aggregated quarterly, annually, and at the 3-year level, and compared to the requirements in Table 7.2 of this QAPP. Lead precision, as a relative percent difference, is calculated with the concentration data using spreadsheet "PbCalculations, Rev.1" See Section 19.1 of this document for additional information on this form. For the precision estimate, data pairs are considered valid if both concentrations are greater than or equal to 0.02 µg/m3. The precision data is aggregated quarterly, annually, and at the 3-year level, and compared to the requirements in Table 7.5 of this QAPP. Precision requirements for each pollutant are summarized in table 14.6 below and are found in the SOPs, and in the specific instrument's operators manual.

Table 14.6 Precision Detail and Reference

Pollutant	Precision Detail	CFR Reference	SOP Reference
Ozone	1-pt between 5-80ppb (Air Quality uses a point close to the NAAQS)	40 CFR Part 58 Appendix A 3.1.1	App E Ozone Monitoring with UV Spectrophotometry SOP Section 7.2
PM2.5	Collocate runs 1:6, relative % D	40 CFR Part 58 Appendix A 3.2.3	App F Thermo Model 2025 Sequential Sampler SOP Section 8.2.2
Lead	Collocate runs 1:6, relative % D	40 CFR Part 58 Appendix A 3.4.4	App D Volumetric-Flow-Control High Volume TSP/Pb Monitors SOP Section 7.2

14.1.3 Accuracy (Bias Checks)

Accuracy is defined as the degree of agreement between an observed value and an accepted reference value. Accuracy, as described in 40 CFR part 58, Appendix A, is a combination of random error (imprecision), and systematic error (bias) components which are due to sampling and analytical operations. Accuracy is measured by the performance of audits and verifications with known certified standards. The QAO uses the EPAs Data Assessment Statistical Calculator (DASC) tool on the AMTIC site to review bias at both the individual instrument level and at the network level, by entering the flow rate verifications described in section 14.1.4 for particulate pollutant networks and the precision point for the Ozone network. Accuracy or bias requirements for the various types of instrumentation used are found in tables 14.1-14.4.

14.1.4 Flow Rate

For each instrument (except ozone), a flow rate verification is performed monthly. These flow rate checks are made by measuring the instruments normal operating flow rate using a certified flow rate transfer standard. Flow rate ranges and references are listed in table 14.7 below and additional details for implementing flow verifications are found in the appropriate sampling SOP, and the instrument's operations manual.

Table 14.7 Flow Rate Ranges

Pollutant	Flow Rate Range	CFR Reference	SOP Reference
PM2.5	Operating flow rate (≈16.67LPM)	40 CFR Part 58 Appendix A 3.2.1	App F Thermo Model 2025 Sequential Sampler SOP Section 4.3.4
PM 10/25 continuous	Operating Flow rate (Total ≈16.67LPM, Main Flow ≈ 5.00LPM Bypass ≈11.67)	40 CFR Part 58 Appendix A 3.3.1	App C Teledyne T640x Model continuous PM10/PM25 SOP Section 4.0.
Lead	Operating Flow Rate -Based upon Orifice look up table	40 CFR Part 58 Appendix A 3.4.2 40 CFR Part 50 Appendix B 7.4.1	App D Volumetric-Flow-Control High Volume TSP/Pb Monitors SOP Section 3.5

14.1.5 Performance Audits and Semi-Annual Flow Rate Audits

Performance audits are performed in accordance with 40 CFR part 58, Appendix A 3.1.2.1. Air Quality conducts internal audits quarterly. These are done manually by the internal auditor using the auditor's transfer standard and external zero air system. Procedure and acceptance criteria can be found in section 3.3 of Air Quality's SOP for Internal Auditing and System Review, Rev 3 in appendix B of this document. A flow rate audit will be performed every quarter on each instrument in the particulate and lead networks. The flow rate standard used for auditing shall not be the same flow rate standard used for calibration or verification of the instrument. However, both the calibration standard and the audit standard may be referenced to the same primary flow rate or volume standard. Additionally, TDEC conducts audits semi-annually on the ozone, particulate and lead networks. The QAO evaluates process, certification of equipment and the results of the TDEC audits, applying the acceptance criteria established in the QAPP. Audit ranges and references are provided in Table 14.8 below.

Table 14.8 Audit Ranges

Pollutant	Audit Range	CFR Reference	SOP Reference
Ozone	Minimum 3pts, 2-3x Minimum Detection Limit (MDL), <99th percentile, and ≈ NAAQS,	40 CFR Part 58 Appendix A 3.1.2.1	App B SOP for Internal Auditing and System Review, Rev 3. Section 3.2
PM2.5	Operating flow rate (≈16.67LPM)	40 CFR Part 58 Appendix A 3.2.2	App B SOP for Internal Auditing and System Review, Rev 3 Section 4.1.2
PM10/ 25 continuous	Operating Flow rate (Total ≈16.67LPM, Main Flow ≈ 5.00LPM Bypass ≈11.67)	40 CFR Part 58 Appendix A 3.3.3	App C Teledyne T640x Model continuous PM10/PM25 SOP
Lead	Operating Flow Rate -Based upon Orifice look up table	40 CFR Part 58 Appendix A 3.4.3 40 CFR Part 50 Appendix B 7.4.1	App D Volumetric-Flow-Control High Volume TSP/Pb Monitors SOP Section 6.2

14.1.6 Rounding Convention during QC activities

Standardized methods of evaluating data enables a more robust statistical analysis. Air Quality utilizes a rounding convention during equipment verifications whether it be monthly flow, temperature, pressure, humidity verifications or quarterly internal audits. Significant digits are any of the figures 0 through 9 that are used with its place value to denote a numerical quantity, excepting all leading zeros and some trailing zeros. The basis behind the rounding convention is such that measurements are rounded on the more precise instrument to the figures available on the less precise instrument. For example, in ozone, during the biweekly precision check, the monitor is capable of reading to the tenths position ppb but the calibrator is reporting to the nearest whole number ppb. When recording and comparing the percent difference between the data points, the monitor's value shall be rounded to the

nearest whole digit. Another example is the temperature verification of a particulate matter monitor. The monitor measures and reports to the nearest tenth, but the transfer standard is accurate to the 100ths position. The recorded reference value is rounded to the nearest tenth to match the significant digits of the instrument. In the event the operator does not round any digits in the field, the QAO shall apply the rounding convention to QC data when performing statistical analysis or entering into AQS.

When multiple readings are recorded and then averaged before a comparison is done; rounding shall occur on the final averaged result, after all unit conversions. Rounding is not implemented when comparing measurements to best fit lines or linearity tests.

14.2 Control Charts

Control charts will be utilized by Air Quality. They provide a graphical means of determining whether various phases of the measurement process are in statistical control. The control charts will be utilized as an “early warning system” to evaluate trends in precision and bias. Control charts can also be used to discover underlying issues such as pump fatigue and drift that will enable the operator to correct a problem before it results in data loss. The following is a list of regularly used control charts and where they are located:

- Ozone sample flow graphed on AirVision to monitor for pump fatigue, stored within AirVision
- Flow rate and precision control charts created with each monthly lead data validation, stored on shared drive with lead concentration calculations
- Data Review control charts listed in the Quarterly Quality Assurance Report comparing PM 2.5 FRM monitors to the FEM, stored on the shared drive.
- QA Officer’s Data Assessment Statistical Calculator spreadsheets, used to calculate accuracy and bias for the Quarterly QA Report, spreadsheet provided by EPA on AMTIC website, stored on shared drive.

14.3 Sampling/ Measurement System Corrective Action

Corrective action measures in the Ambient Air Quality Monitoring Network will be taken to ensure the data quality objectives are attained. There is the potential for many types of sampling and measurement system corrective actions. Each approved standard operating procedure included in Appendix C-F, details some expected problems and corrective actions needed for a well-run monitoring network. A Corrective Action Report (Figure 14.9) will be filled out for any sampling issue that will impact data. This report will be filled out by the QA Officer or site operator and will be placed on the network drive in “Important Documents” folder

Table 14.9 Corrective Action Report

KNOX COUNTY
HEALTH DEPARTMENT

Corrective Action Report

Issue: *Describe the issue found, what was known preceding discovery, what is believed to occurred etc. If possible, note last passing verification date. Include make, serial number, pollutant for instrument in question and as specific as possible dates and times. IF failed verification be sure to report serial number of standard used for verification.*

Action taken: *Describe all actions performed such as calibrations, maintenance, repair. Include as specific as possible dates and times.*

Post action QC performed: *Note date time and results of verifications post actions. Be sure to note the serial number of verification equipment used.*

Signature _____ Date _____

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14.4 Internal Quality Control and Corrective Actions for Measurement Systems

The contract lab specific QC and corrective action procedures are iterated in laboratory and method specific QAPP and SOPs (See Appendix G&H). The QA Officer reviews the final report sent to Air Quality to ensure the internal QC meets the minimum standards of the analytical lab's SOP. A copy of the 2014 Technical Systems Audit for IML can be found on the AMTIC website https://www3.epa.gov/ttnamti1/files/ambient/qaqc/imllabaudit2014.pdf as well.

15.0 Equipment Testing, Inspection, and Maintenance Requirements

15.1 Purpose/ Background

The purpose of this section is to discuss the procedures used to verify that all instruments and equipment are maintained in sound operating condition and are capable of operating at acceptable performance levels. All instrument inspection and maintenance activities must be documented and filed. See Section 9 for document and record details.

15.2 Testing

All gaseous and particulate matter pollutant monitors used in the Ambient Air Quality Monitoring Network shall be certified to adhere to EPA reference or equivalent method listed in tables 11.1 and 11.2. Therefore, they are assumed to be of sufficient quality for the data collection operation. Records are kept and filed at the Air Lab for all comparisons.

15.2.1 PM

Prior to field installation, new particulate samplers are assembled and tested at the laboratory. The field operators perform external leak checks, temperature, pressure, and flow rate multi-point verification checks. If any of these checks are out of specification the vendor is contacted for initial corrective action. Once installed at the site, the field operators perform an external leak check followed by a 1=point temperature, pressure and flow rate verification. If the sampling instrument meets the acceptance criteria, it is put into operation.

For repaired PM monitors, the monitor should have an external leak check, 1- point Temperature, pressure and flow check before leaving the laboratory. Once installed in the field, the monitor should have the external leak check, 1- point Temperature, pressure and flow check repeated. If the monitor was repaired in the field, a standard flow verification and leak check is required after the repair.

15.2.2 Ozone

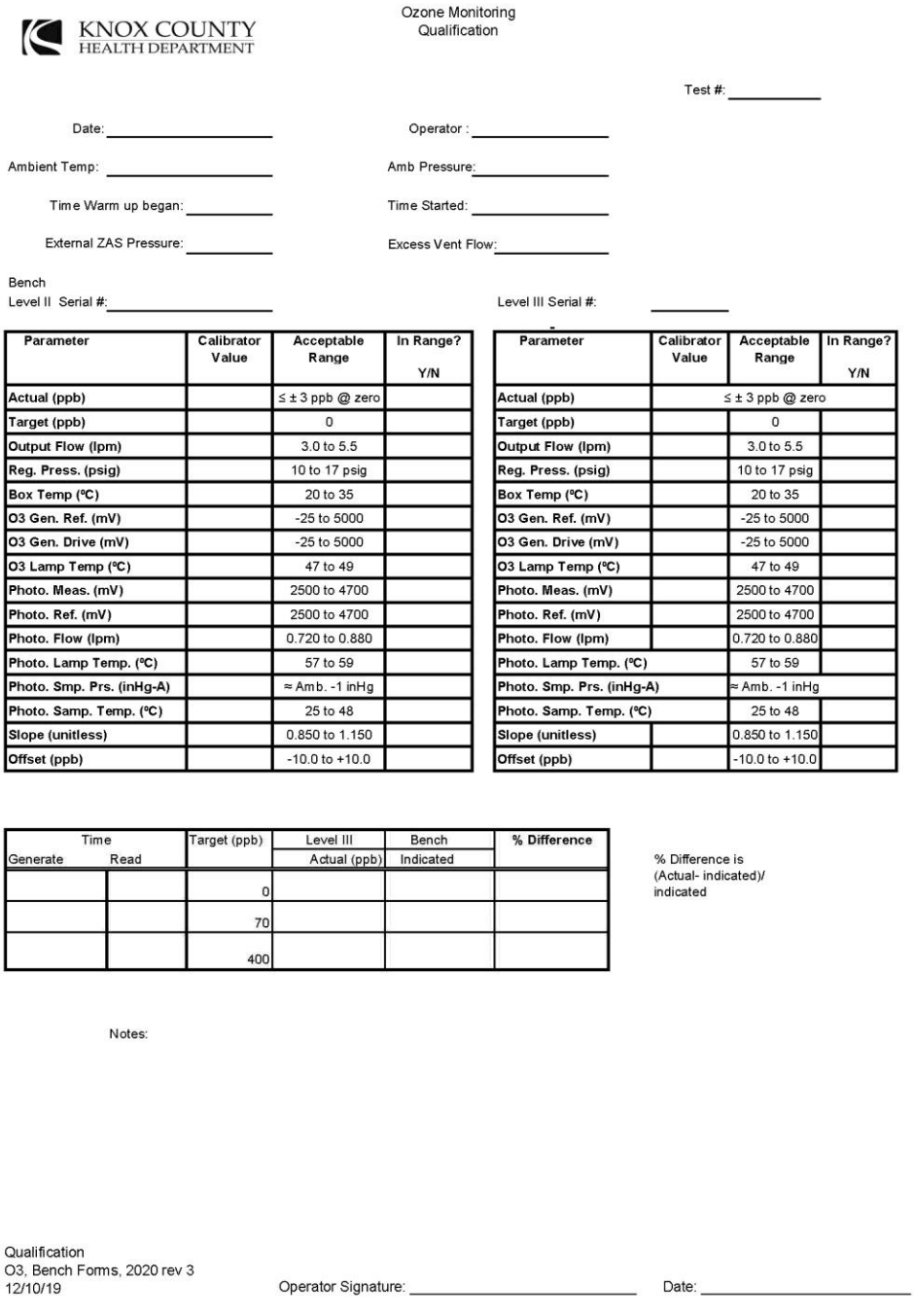
Any new ozone analyzers must pass a multipoint calibration verification and run continuously sampling lab air for 2 days (if time permits). Prior to field installation of any new ozone transfer standard, the equipment shall successfully undergo a qualification process. To qualify the equipment the following comparisons should be completed using a known higher-level transfer standard. See Figure 15.1 for the Qualification Form

- Step 1: Perform the first zero/precision/span (ZPS) check just as a multipoint bench comparison would be performed (See Ozone SOP in Appendix E for multipoint bench comparison procedure) (24 hour warm up, etc) after completion power off instrument, disconnect and cap all lines.
- Step 2: Perform second ZPS after setting up and warming up for less than 24 hours. After completion power down, disconnect & cap lines.
- Step 3: Move instrument into a vehicle and drive around a bit. This is to ensure the instrument is stable and not prone to leaks. Upon return complete warm up and perform third ZPS. After completion power down, disconnect & cap lines.
- Step 4: Repeat step 3 for fourth ZPS comparison, however, do not power down and disconnect when complete.
- Step 5: Perform final ZPS while still warmed up from step 4. This ZPS is performed in the reverse order of 0,70,400 rather than the normal descending scale.

In all qualification testing, keep the read times less than 20 minutes apart, closer to 10 minutes when possible, while still achieving the required stability (<1.0 ppb) to ensure adequate and accurate response time. To pass the qualification, no test should result in greater than 4ppb or 4% difference

(whichever is greater). If a test results in 3ppb/3% different note and discuss with Air Monitoring Program Manager about additional investigation into the variability tested. Analyzers that have been repaired must pass at minimum ZPS before being placed back into service. Repaired transfer standards must have a passing multi-point comparison to the bench before use in the field.

Figure 15.1 Ozone Qualification Form



KNOX COUNTY
HEALTH DEPARTMENT

Ozone Monitoring
Qualification

Test #: _____

Date: _____ Operator : _____

Ambient Temp: _____ Amb Pressure: _____

Time Warm up began: _____ Time Started: _____

External ZAS Pressure: _____ Excess Vent Flow: _____

Bench
Level II Serial #: _____ Level III Serial #: _____

Parameter	Calibrator Value	Acceptable Range	In Range? Y/N
Actual (ppb)		≤ ± 3 ppb @ zero	
Target (ppb)		0	
Output Flow (lpm)		3.0 to 5.5	
Reg. Press. (psig)		10 to 17 psig	
Box Temp (ºC)		20 to 35	
O3 Gen. Ref. (mV)		-25 to 5000	
O3 Gen. Drive (mV)		-25 to 5000	
O3 Lamp Temp (ºC)		47 to 49	
Photo. Meas. (mV)		2500 to 4700	
Photo. Ref. (mV)		2500 to 4700	
Photo. Flow (lpm)		0.720 to 0.880	
Photo. Lamp Temp. (ºC)		57 to 59	
Photo. Smp. Prs. (inHg-A)		≈ Amb. -1 inHg	
Photo. Samp. Temp. (ºC)		25 to 48	
Slope (unitless)		0.850 to 1.150	
Offset (ppb)		-10.0 to +10.0	

Parameter	Calibrator Value	Acceptable Range	In Range? Y/N
Actual (ppb)	≤ ± 3 ppb @ zero		
Target (ppb)		0	
Output Flow (lpm)		3.0 to 5.5	
Reg. Press. (psig)		10 to 17 psig	
Box Temp (ºC)		20 to 35	
O3 Gen. Ref. (mV)		-25 to 5000	
O3 Gen. Drive (mV)		-25 to 5000	
O3 Lamp Temp (ºC)		47 to 49	
Photo. Meas. (mV)		2500 to 4700	
Photo. Ref. (mV)		2500 to 4700	
Photo. Flow (lpm)		0.720 to 0.880	
Photo. Lamp Temp. (ºC)		57 to 59	
Photo. Smp. Prs. (inHg-A)		≈ Amb. -1 inHg	
Photo. Samp. Temp. (ºC)		25 to 48	
Slope (unitless)		0.850 to 1.150	
Offset (ppb)		-10.0 to +10.0	

Time		Target (ppb)	Level III	Bench	% Difference
Generate	Read		Actual (ppb)	Indicated	
		0			
		70			
		400			

% Difference is (Actual- indicated)/ indicated

Notes:

Qualification
O3, Bench Forms, 2020 rev 3
12/10/19

Operator Signature: _____ Date: _____

15.3 Inspection and Maintenance

All equipment needs periodic maintenance and inspection. Air Quality personnel work to ensure monitoring equipment is maintained and working properly. This is achieved through scheduled maintenance, and monthly/ bi-weekly verifications and precision checks. Inspections are subdivided into two sections; conditioning & weighing room and field items.

15.3.1 Inspection & Maintenance in Conditioning/Weighing Room

See IML SOP in Appendix G for specific inspection performed in the weigh laboratory.

15.3.2 Inspections & Maintenance of Field Items

There are several items that require periodic field inspection and maintenance. All are identified in pollutant specific SOPs in Appendix C-F. These include, but are not limited to:

- In-line filter change
- Silica Gel canister color
- Sample lines
- HVAC filters
- Audit filters
- Pump repair/ replacement
- Checking Seals, O-rings and gaskets
- Cleaning inlets
- Changing of tubing

Air Quality also has spare parts and consumables on hand for each monitor used. Air Quality has at least one back-up instrument for $PM_{2.5}$, ozone (analyzer and transfer standard), and lead. Additional back-up reference standards are kept certified.

16.0 Instrument Calibration and Frequency

Specifications and frequency for calibration of field and laboratory standards can be found in the data validation tables listed in Section 7 and the quality control and corrective action tables in Section 14 of this document. Verifications and Calibrations must be conducted with certified traceable reference devices. Traceable is defined in 40 CFR Parts 50 and 58 as meaning that a local standard (i.e., one maintained by a monitoring organization) has been compared and certified, either directly or via not more than one intermediate standard, to a primary standard such as a NIST Standard Reference Material (NIST SRM) or an EPA/NIST-approved Certified Reference Material (CRM). Similarly, traceability is the property of a measurement result whereby the result can be related to a stated reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty. Standard traceability, therefore, is the process of transferring the accuracy or authority of a primary standard to a field-usable standard, resulting in a documented unbroken chain of calibrations/certifications. Most standards used for calibration are purchased and re-certified by vendors with accredited NIST-traceable calibration processes. Devices are re-certified before the current certification expires or repair is needed. Records of these certifications are kept on the shared in the QA database.

The following summarizes the standards used in the network and their recertification process. All certification periods are monitored via report generated by the QA Officer via the QA Database to ensure that equipment or certified materials are sent out for certification and are not used beyond the documented certification expiration dates. Tracking these certifications – those performed in-house, as well as those performed by vendors – is the responsibility of the QA Officer.

Note**:** Calibration/certification of laboratory standards for the $PM_{2.5}$ program is the responsibility of IML and are covered in the IML QAPP and SOPs. Most of the devices must be sent off to a contractor or the manufacturer to be recertified. Some devices, such as volt meters and thermometers, are less expensive to replace than to send off for re-certification. Once the device is returned, the certification paperwork will be evaluated to ensure the proper conditions, as stated on the certification, were met. Certifications are then scanned into the QA database and the hard copy is filed. Each piece of certified equipment will be stored with a copy of the certification or a label made with the certification data.

16.1 Photometers

A standard reference photometer (SRP) is maintained in Region 4 at the Laboratory Services and Applied Sciences Division (LSASD) Lab in Athens. The SRP is the highest-authority ozone standard, equivalent to NIST, and is considered a Level I standard. Two Air Quality Level 2 calibrators (containing photometers) are taken to EPA Region 4 and compared to the SRP on an annual basis. One calibrator (photometer) is defined as the “bench standard,” which means it is stationary in the lab. The other calibrator (photometer) is used as an audit standard, which is used by the internal auditor to conduct performance audits.

All site transfer standards (photometers) — are certified versus the ozone bench standard in the lab, making them a level III standard. These certifications are performed 3 times per ozone season (March 1st-October 31st) pre-season, mid-season and post-season.

16.2 Multi-Parameter Standard

Air Quality uses several types of multi parameter standards within the air monitoring program (e.g., Hi-Vol Cal, and Streamline (SLP)). These devices are multi system standards meaning they not only measure flow, but also ambient temperature and pressure. These devices are certified annually by an accredited organization (currently Chinook Engineering & Mesa Laboratory) that provide a certificate of traceability to NIST standards. It is Air Quality’s policy to stagger the certifications such that a certified device remains available in-house at all times.

The Multi-Parameter Standards are used for a variety of tasks:. The SLPs for calibration, audits and verification of the temperature, pressure and flow of the PM2.5 and PM 10 monitor. The SLPs are also used to verify the thermometers in the shelters bi-annually (Figure 16.2). The Hi-Vol Cals are used for verification and audits of the lead monitors.

16.3 Flow Standard

Air Quality uses two single parameter flow standards. The Defender 510 is used to calibrate the flow of the ozone equipment. It is sent out annually to Mesa Labs to be recertified. The Hi-Vol orifice plate is used for the 4-point calibration verification of the lead monitors. It is sent out annually to Chinook Engineering for re-certification.

16.4 Other Standards

Air Quality has several manometers that are used in the collection and calibration verification of lead and the pressure verification of ozone equipment. These standards are sent to Chinook Engineering annually for certification as well.

The field temperature standards will be maintained and recertified against a NIST-traceable temperature standard on an annual basis or sooner if damaged/malfunctioning, by the manufacturer or qualified vendor. The certification paperwork will be evaluated and maintained at the Air Lab. These standards are used to verify the shelter temperature of the ozone and continuous PM shelters at least 2 times annually. The following form (Figure 16.1) will be used to verify the shelter temperature.

Figure 16.1 Shelter Temperature Verification Form

Knox County Health Department
Every Person, A Healthy Person

Shelter Temperature Verification

Site: _____ **AQS ID:** _____

Date: _____ Ref Device: _____

	Actual (°C)	Reference (°C)	Difference (°C)
Shelter Temp Sensor Display			
Data Logger Display			
Shelter Thermometer (back up)			
Shelter Thermostat			

Operator Intials: _____

Date: _____ Ref Device: _____

	Actual (°C)	Reference (°C)	Difference (°C)
Shelter Temp Sensor Display			
Data Logger Display			
Shelter Thermometer (back up)			
Shelter Thermostat			

Operator Intials: _____

Date: _____ Ref Device: _____

	Actual (°C)	Reference (°C)	Difference (°C)
Shelter Temp Sensor Display			
Data Logger Display			
Shelter Thermometer (back up)			
Shelter Thermostat			

Operator Intials: _____

Shelter Temp Verification
O3, Site Forms rev 1
12/20/17

17.0 Inspection/ Acceptance Requirements for Supplies and Consumables

Spare and consumable parts for most monitoring instrumentation are listed in the manufacturer's operation manuals. Site operators monitor supplies of routine parts and report to the Air Monitoring Program Manager when supplies need to be ordered. The Air Monitoring Program Manager will get a pricing quote from the vendor if necessary and send the quote to the Director for approval. The Director will provide the quote and request to the Office Manager. The Office Manager will work with the Knox County Purchasing Department to complete the order. Air Quality routinely procures supplies directly from the instrument manufacturer or the part vendor. All parts received are inspected for correctness and damage upon receipt.

The following pollutant specific criteria must be followed. The $PM_{2.5}$ and lead filters are certified by EPA prior to distribution to the monitoring agency. They are then inspected for damage upon receipt. Air Quality relies on past practice to know which motors and which brushes perform optimally with the orifices employed in the hi-volume samplers used for the collection of lead. These part numbers are found in the SOP. Finally, the ozone sample lines and fitting in the sample train must be FEP or equivalent.

18.0 Non-Direct Measurements

This section addresses data not obtained by direct measurement from the Air Quality monitoring program. This includes data from outside sources and historical monitoring data. This data should be used in decision making scenarios such as monitor placement and site selection. The information used shall be obtained from reputable sources which include manufacturer's literature, other government agencies and historical data.

Additional information used by Air Quality

- National Weather Service Data
- Sampler Manufacturer's Operational Literature
- Chemical and Physical Properties Data
- Geographic Location Data
- Historical Monitoring Information
- External Monitoring Databases
- US Census Bureau Data
- Total annual emissions data from lead point-source
- Tennessee Department of Transportation traffic count data

19.0 Data Management

Air Quality's Ambient Air Monitoring Program is responsible for the generation of quality ambient air monitoring data and its interpretation. Data available to users must be:

- Accessible to a variety of users,
- Aggregated consistently with their primary use
- Of known quality and reliability

QA/QC of data management begins with raw data and concludes with a defensible reporting of valid data to end users. The following sections will identify the process and procedures that should be followed to collect, record, reduce, transmit, verify, validate, store and retrieve data. These processes and procedures will maintain data integrity and validity throughout.

19.1 Data Collection and Recording

Most of the data collected in Air Quality's network is recorded electronically (with the exception of lead). Electronic data collection is possible through the network's data loggers and modems. This equipment is located at the sites where the data loggers (internal on the $PM_{2.5}$ FRM) record the data history and the modems provide a path to download the data for review and analysis for the continuous monitors. A laptop is used for the transfer of the filter based PM 2.5. Detailed steps can be found in the pollutant specific SOP (See Appendix C-F)

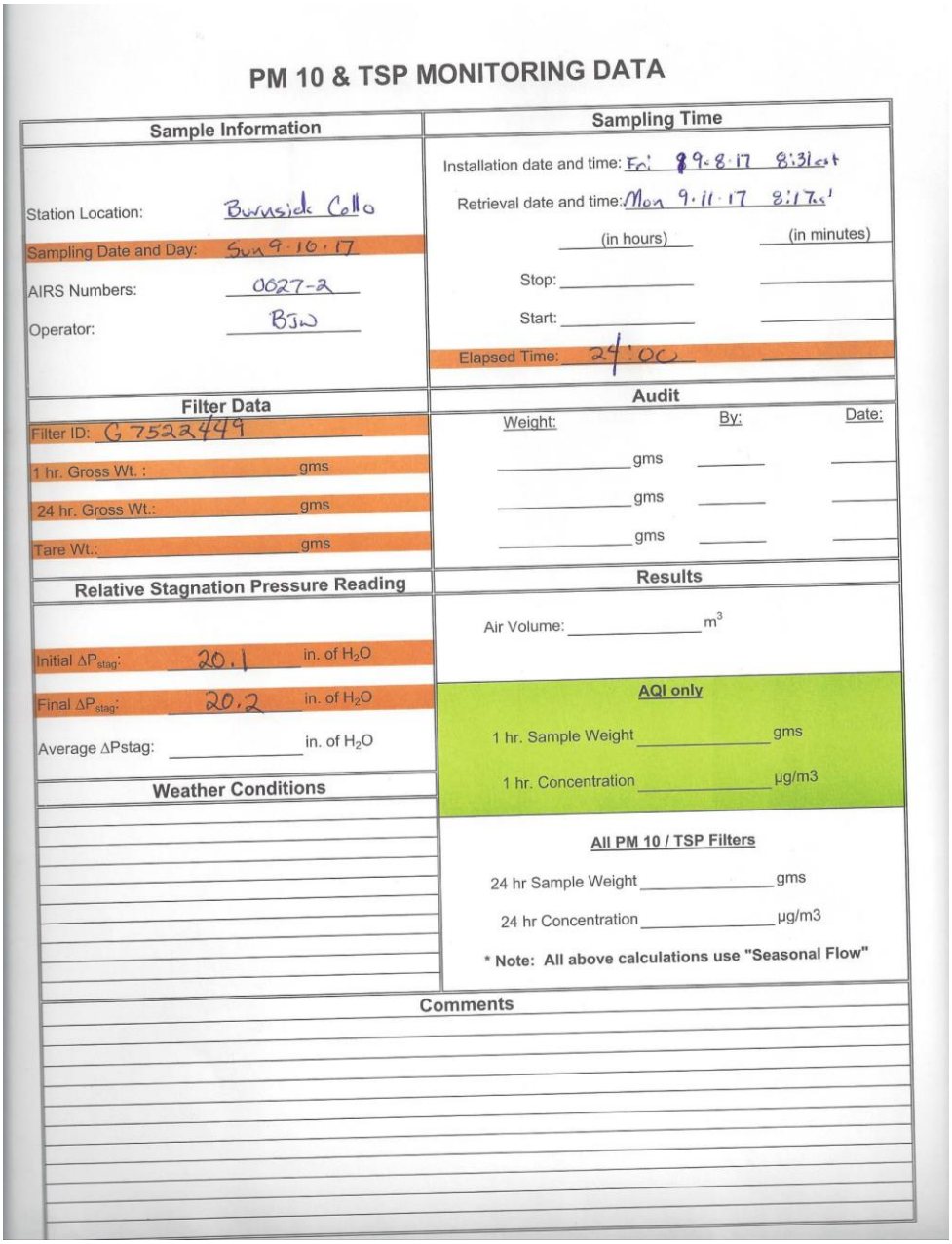
Air Quality's electronic data acquisition system (AirVision) is configured to automatically poll the continuous monitoring stations on an hourly basis. Monitoring personnel can manually poll the stations using AirVision to retrieve current data or determine the status of the systems. Electronic data is collected from the intermittent samplers using RPCOMM software and downloading the data with a laptop. A summary of collection and recording for each pollutant can be found below.

- **Ozone and Continuous PM**: These sites are outfitted with a data logger and high-speed internet connection. The data logger is set up to record each monitor's output, perform specific data manipulations, and format the resulting data in preparation for polling by the data handling software (AirVision).

- **$PM_{2.5}$**: The monitors at these sites have an internal logger that stores data. Specialty software (RPComm) is used with a dial-up modem and analog phone line to download the data to Air Quality's Air Lab. The data are captured and stored as delimited text file on the shared drive. This file is uploaded to MTL software or conditionally formatted Excel form for initial validation. Alternatively, data may be downloaded via laptop using RPComm and transferred to the shared network drive via flash drive.

- **Lead:** Sampler data are recorded on a filter envelop by the operator (Figure 19.1).The filters are shipped with a chain of custody to ERG for analysis. ERG sends Air Quality a report with the total micrograms (µg) of lead on the filter. The operator will combine this data with verified temperature and pressure data, collected from the Teledyne T640X located at the Air Lab site, in a spreadsheet entitled "Pb Calculations, Rev. 1" (See SOP in Appendix D) to produce a concentration of lead mass per volume (ug/m^3) which is then entered into the AQS data base.

Figure 19.1 TSP Filter Envelope



PM 10 & TSP MONITORING DATA

Sample Information		Sampling Time		
Station Location:	Burnside Collo	Installation date and time:	Fri 9.8.17 8:31est	
Sampling Date and Day:	Sun 9.10.17	Retrieval date and time:	Mon 9.11.17 8:17est	
AIRS Numbers:	0027-2		(in hours)	(in minutes)
Operator:	BJW	Stop:		
		Start:		
		Elapsed Time:	24:00	

Filter Data		Audit		
Filter ID:	G 7522449	Weight:	By:	Date:
1 hr. Gross Wt. :	gms	gms		
24 hr. Gross Wt.:	gms	gms		
Tare Wt.:	gms	gms		

Relative Stagnation Pressure Reading		Results	
Initial ΔP_{stag}:	20.1 in. of H_2O	Air Volume:	m^3
Final ΔP_{stag}:	20.2 in. of H_2O	AQI only	
Average ΔPstag:	in. of H_2O	1 hr. Sample Weight	gms
Weather Conditions		1 hr. Concentration	µg/m3
		All PM 10 / TSP Filters	
		24 hr Sample Weight	gms
		24 hr Concentration	µg/m3
		* Note: All above calculations use "Seasonal Flow"	

Comments

19.2 Data Reductions and Transformation

Data collected is categorized by one of two methods; Manual or Continuous.

19.2.1 Manual Method

Manual methods are used for the collection of $PM_{2.5}$ and lead data. For this method category, only the calculations specified in the SOP or CFR can be used. IML is responsible for computing the $PM_{2.5}$ data into concentrations (See IML SOP Appendix G). Air Quality uses an excel spreadsheet (Pb Calculations, Rev.0), found on the shared network drive and in the Lead SOP, which is designed specifically for calculation of lead concentrations. This form has been reviewed by the QA Officer and locked. The original form can only be saved by another name once completed.

Filter samples are physically measured by a laboratory and mathematically reduced into weights. Once the analytical data are combined with the field sampling data, the data are reduced into weights per unit volume.

19.2.2 Continuous Method

Ozone and Continuous PM operate using the continuous method. The inherent accuracy of an instrument is incorporated into the system accuracy when the instrument is calibrated. Each continuous criteria pollutant monitoring instrument has its own internal potentiometers, whether digital or analog, adjusted to accurately reflect the concentration at which the instrument is tested. Each instrument is assumed to be linear within the range of 10-90% of full scale. As long as the background concentrations do not violate this range, the accuracy of the instrument is not questioned. Additionally, ozone calibration verifications review the linearity over the normal operational range.

Data reduction occurs for continuous analyzers, when data collected by the analyzer are averaged to a larger unit of measure. Data are read (scan internal) by the logger every second and reduced to minute data. Minute data are reduced to hourly data by AirVision software. The data are polled (minute) from the logger by AirVision software via a DSL connection. The hourly data are validated and uploaded to AQS.

19.3 Data Transmittal

Data transmittal is accomplished using DSL/telephone line/cable access to the sites' modems, which link to a data logger. Downloading of collected data does not delete the data from the data logger. Data are removed from the data logger continuously by overwriting data on a first-in first-out basis. This configuration requires that the data be extracted from the data logger on a regular basis, thus preventing any loss of data. If communication problems arise, the data shall be retrieved either by going to the site and directly accessing the data logger or retrieving data remotely once the communication problems have been rectified. A site visit is mandatory is the communication problems are not expected to be corrected in time to prevent data from being over written.

Polling of the data loggers at the ozone and PM continuous sites is initiated from the office by AirVision software on an hourly basis. The data (minute & hourly) are received and stored on to the server by AirVision software. Laptop computers running AVTrend software are connected via RS-232 cable to the data logger to record concentrations as a secondary data recording and storage device. AV Trend allows the operator to graph data in real-time which is helpful during QA/QC checks.

Data transmittals required for calculating values associated with the manual method for $PM_{2.5}$ and lead filters are accomplished via email. Once the exposed filters are returned to IML, they are analyzed and the resulting concentration data are e-mailed to Air Quality. The lead filters are analyzed by ERG and the micrograms of lead per filter emailed to Air Quality as a report.

All transmitted raw data sets are stored electronically. These data sets are retained intact by archiving the raw data. Data reduction operations can be performed repeatedly without violating the integrity of the original raw data set.

19.4 Data Validation

Each of the pollutant's analytical instruments, employed to measure ambient concentrations of the criteria pollutants, undergoes periodic audits and calibrations. These procedures are outlined in the appropriate SOPs. Performance audits and calibrations ascertain the accuracy, precision, and repeatability of each instrument in performing its required function.

Data is evaluated for validation as follows:

- Continuous Monitors – A monthly data report is printed and analyzed using the audits, precisions, flow verifications, weekly parameter checks and the logbooks from the time frame represented. The appropriate flags/null data codes are entered into the data. Some flags are automatically generated by AirVision. Each of the auto-generated flags are evaluated for validity and applicability. The data are then put into AQS format and uploaded to AQS.
- $PM_{2.5}$ – The delimited text file is loaded into the MTL program, or conditionally formatted excel form by the user to be evaluated for flags generated from the monitor. The field sheets are reviewed for notes and concerns notated by the operator. Any flags or null data codes are recorded on the field sheets. The data file from the monitor is sent via email to IML. The email also outlines any flags or null data codes needed for the data. Once analyzed, IML will apply and flags or null data codes from the email, and any generated from the lab analysis. The report is sent back to Air Quality with a file ready to load into AQS. The lab report is evaluated for accuracy, audits, flow verifications and logbooks are reviewed, and the file is uploaded to AQS. Once in AQS the data is reviewed to ensure the appropriate null data codes or flags have been applied.
- Lead – ERG will send Air Quality a report with the total microgram of lead on the filter. Air Quality will process this data as discussed in Section 19.1 of this document. The calculated concentrations, copies of filter envelopes, logbooks, flow verifications, and audits are reviewed. Any null data codes or flags are applied and noted on the "Pb Concentrations" spreadsheet. Data are entered manually into AQS. Once data are entered a raw data report is ran and checked by another employee to ensure accurate data entry. The Raw Data Report is initialed by the reviewing employee and filed with the lead filters and supporting paperwork.

19.5 Quarterly Data Submittal

Air Quality shall submit quarterly data, as specified in 40 CFR Part 58, to EPA via the AQS database. This data shall be submitted no later than 90 days following the close of each calendar quarter, as specified in 40 CFR Section 58.16, or the department shall notify EPA Air and Radiation Division (ARD) of the delay. The quarterly data submittal shall contain the following summary data:

- The city name (if applicable), county name, site location/ street address of each monitoring station
- The measurement scale associated with the Parameter Of Occurrence (POC)
- The AQS site code, monitoring method code, and POC
- The results of all valid precision, bias, and accuracy tests performed during the quarter
- All ambient air quality data obtained on O_3, Pb, PM_{10} and $PM_{2.5}$; and all other information specified by the AQS Users Guide (*Volume II, Air Quality Data Coding, and Volume II, Air Quality Data Storage*)
- The location, date, pollution source, and duration of incidents of ambient level exceedances

19.6 Data Storage and Retrieval

The storage and retrieval of the air quality monitoring data shall be possible through Air Quality's archiving system. Logbooks, audit results, field forms and all other hard copies will be stored and labeled by year in archived boxes. Electronic data will be stored on the server. The data shall be stored for a period of five years, unless any litigation, claim, negotiation, audit, or other action involving the records has been started before the expiration of the five-year period. If this happens, the records will be retained until completion of the action and resolution of all issues that arise from it, or until the end of the regular five-year period, whichever is later. This is in accordance with "Knox County Department of Air Quality Management Records Retention Policy, Revision 0" (See Appendix A)

The shared server drive is maintained and backed-up at least daily by the County's Information Technology Department.

20.0 Assessments and Corrective Action

An assessment is the process used to measure the performance of effectiveness of the quality system, the Ambient Air Quality Monitoring Network and its sites, and various measurement phases of the data operation. In order to ensure the adequate performance of the quality system Air Quality will perform or participate in:

- Network Reviews,
- Technical System Audits,
- Performance Evaluations,
- Data Quality Assessments, and
- Annual Employee Competency Assessment.

20.1 Network Review/Assessment

20.1.1 Five Year Network Assessment

A Network assessment is conducted every five years and submitted to EPA Region 4. Air Quality includes this with the Annual Network Plan for the corresponding year. This assessment should determine how well the system is achieving the required monitoring objectives and if modifications need to be made. During this network assessment, the stated objective for each monitoring site will be reconfirmed and the location's spatial scale will be verified. If the site location does not support the stated objectives or the designated spatial scale, changes will be proposed to EPA to rectify the discrepancy.

20.1.2 Annual Network Plan

The Annual Network Plan is compiled yearly by Air Quality and submitted to TDEC by May 1st for review and publication for public comment, then ultimately submitted to EPA. Though Air Quality acts as its own PQAO, TDEC has monitors inside the Knox County's Metropolitan Statistical Area (MSA) and Core Based Statistical Area (CBSA) so the Annual Network Plans are combined to one formal document.

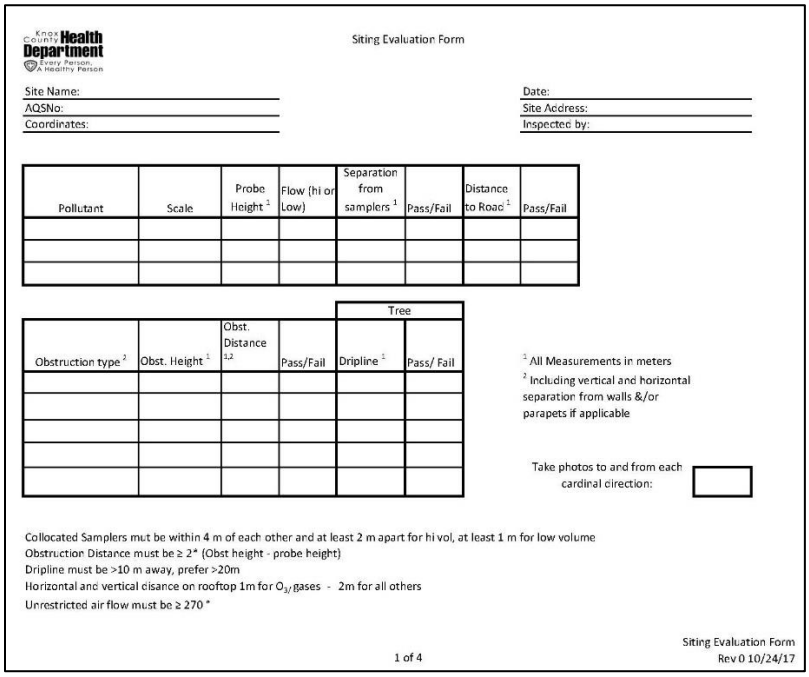
Conformance with network requirements of the Ambient Air Quality Monitoring Network as set forth in 40 CFR Part 58, Appendices D and E are determined through annual network reviews of the ambient air quality monitoring system as required by 40 CFR Section 58.20(d). The network review is used to determine if a particular air monitoring network is collecting adequate, representative, and useful data in pursuit of its air monitoring objectives. Additionally, the network review may identify possible network modifications to enhance the system or correct deficiencies in attaining network objectives. Information compiled for the Annual Network Plan includes:

- Network files (including updated site information and site photographs)
- Proposed network modifications
- Pollutant specific priorities
- Updated site photos and information
- Weather Service data such as wind rose
- AQS reports and data
- Population Data

20.1.3 Annual Site Criteria Evaluation

Each site will be evaluated annually to ensure siting criteria is being met. This data will be included in the Annual Network Plan. Applicable siting criteria for SLAMs are specified in 40 CFR Part 58, Appendix E. The on-site visit will consist of physical measurements and observations to determine compliance with the federal code requirements, such as height above ground level, distance from trees, appropriate ground cover, etc. The "Siting Evaluation Form, Rev,0" (Figures 20.1 and 20.2) shall be used. It can be found on the shared network drive.

Figure 20.1Siting Evaluation Form pgs 1&2



Knox County Health Department
Every Person. A Healthy Person

Siting Evaluation Form

Site Name:
AQSNo:
Coordinates:

Date:
Site Address:
Inspected by:

Pollutant	Scale	Probe Height [1]	Flow (hi or Low)	Separation from samplers [1]	Pass/Fail	Distance to Road [1]	Pass/Fail

				Tree	
Obstruction type [2]	Obst. Height [1]	Obst. Distance [1,2]	Pass/Fail	Dripline [1]	Pass/ Fail

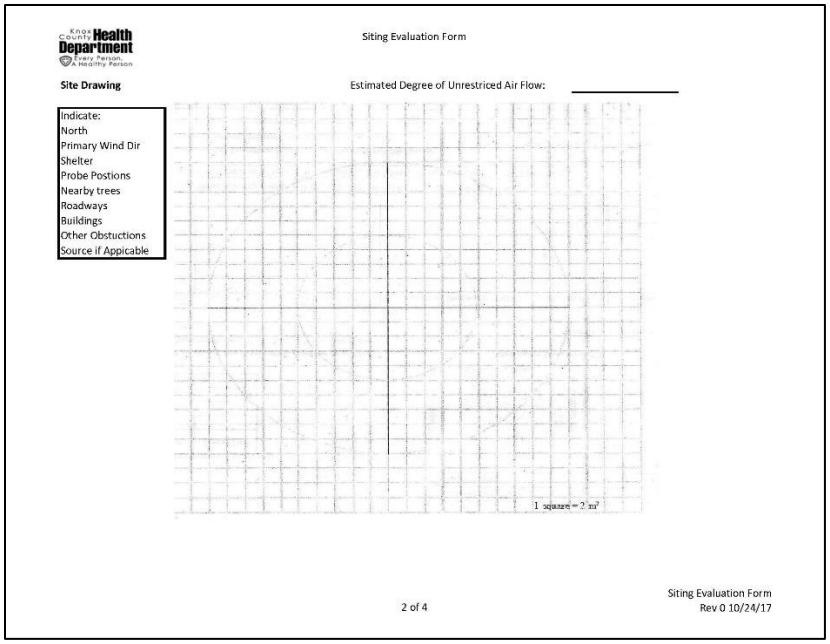
[1] All Measurements in meters
[2] Including vertical and horizontal separation from walls &/or parapets if applicable

Take photos to and from each cardinal direction:

Collocated Samplers mut be within 4 m of each other and at least 2 m apart for hi vol, at least 1 m for low volume
Obstruction Distance must be ≥ 2* (Obst height - probe height)
Dripline must be >10 m away, prefer >20m
Horizontal and vertical disance on rooftop 1m for O_3/gases - 2m for all others
Unrestricted air flow must be ≥ 270 *

1 of 4

Siting Evaluation Form
Rev 0 10/24/17



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Siting Evaluation Form

Site Drawing

Estimated Degree of Unrestriced Air Flow: _____

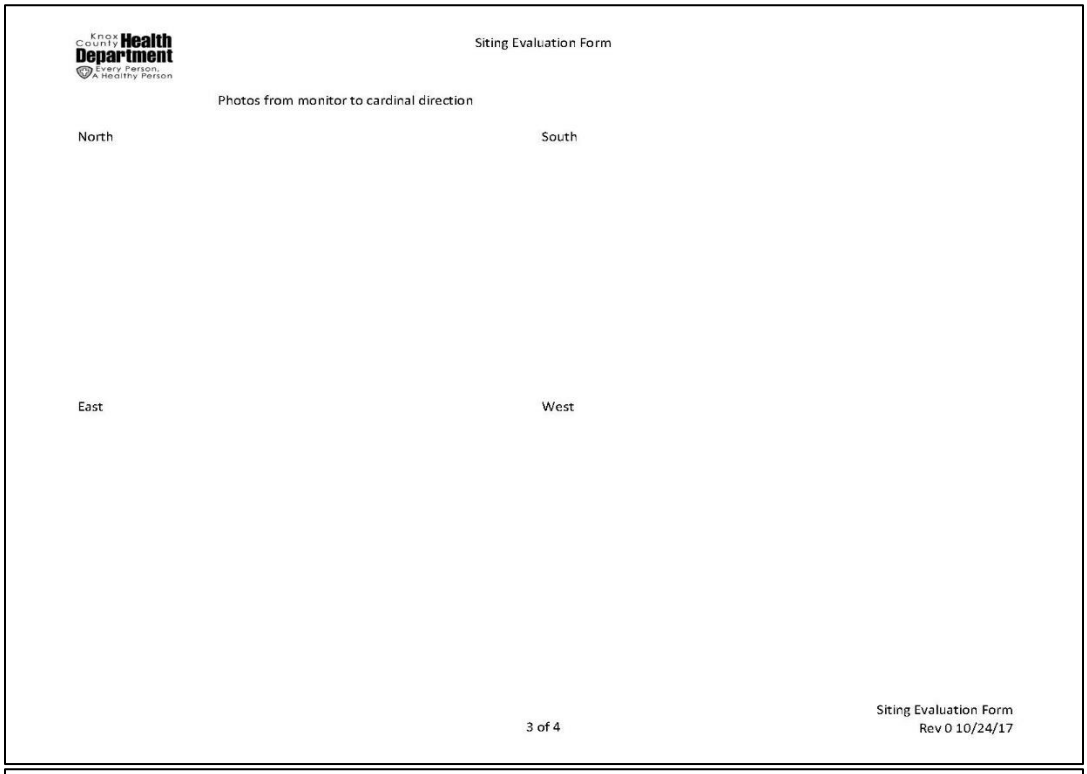
Indicate:
North
Primary Wind Dir
Shelter
Probe Postions
Nearby trees
Roadways
Buildings
Other Obstuctions
Source if Appicable

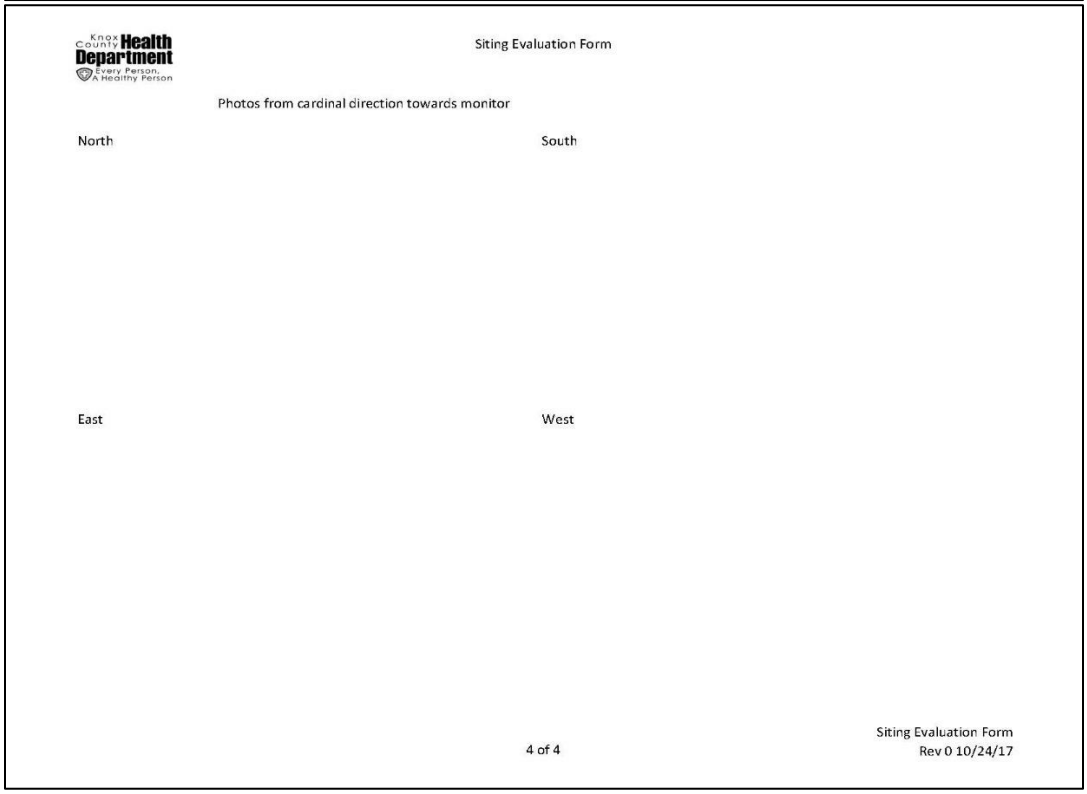
1 square = 2 m²

2 of 4

Siting Evaluation Form
Rev 0 10/24/17

Figure 20.2 Siting Evaluation Form pgs 3 &4





20.1.4 Quarterly Data Validation Checks

Once per quarter, the Air Monitoring Program Manager should choose a data point from each pollutant and trace that data point from origin to AQS submittal, reviewing all QA/QC activities, logbook entries and validation procedures. This will be documented on the Quarterly validation check sheets (Figures 20.3 thru 20.7and filed with the appropriate year in the archive boxes.

Figure 20.3 Quarterly Validation Check Ozone pg1

Run Date:	**Site:**	**Voids/Flags:**	**75% valid data: ☐**
1)Requirement (O_3)	2) Frequency	3) Acceptance Criteria	
CRITICAL CRITERIA-OZONE			
Monitor	NA	Meets requirements listed in FRM/FEM designation	Serial Number: _____
One-point QC Check	Every 14 days	<±7.1% percent difference or <1.5ppb difference whichever is greater	Date Pre-sample: _____ TS#: _____ Cert. Date: _____ Date Post-sample: _____ TS#:_____ Cert. Date: _____
Zero/ Span check	Every 14 days	Zero Drift <±3ppb (24 hr) <±5.1ppb (>24hr-14 day) Span drift <±7.1%	Date Pre-sample: _____ TS#: _____ Cert. Date: _____ Date Post-sample: _____ TS#:_____ Cert. Date: _____
Operational Criteria – Ozone			
Shelter Temperature Range	Daily (hourly values)	15-35 °C hourly avg	Yes / No
Shelter Temperature control	Daily (hourly values)	<2.1°C SD over 24 hours	
Shelter Temperature Device Check	Every 182 days and 2/ calendar year	≤2.1 ° of Standard	Date: _____
Annual Performance Evaluation	Every Site every quarter within period of monitor operation	Percent difference of audit levels 3-10 < ±15.1% Audit levels 1 &2 <±1.5ppb difference or < ±15.1%	Date Pre-sample audit: _____ TS#: ____ Cert. Date: _____
Federal Audits (NPAP)	20% of sites audited in Calendar year	Audit levels 1&2 <±1.5 ppb difference all other levels < ±10.1%	N/A

Figure 20.4 Quarterly Validation Check Ozone pg 2

Operational Criteria - Ozone			
1)Requirement (O_3)	2) Frequency	3) Acceptance Criteria	Information/ Action
Verification/Calibration	Upon receipt/adjustment/repair installation/moving and repair and recalibration of standard of higher level Every 182 day and 2/ calendar year if manual zero/span performed biweekly Every 365 day and 1/ calendar year if continuous zero/span performed daily	All points <± 2.1% or ≤±1.5 ppb difference of best fit straight line whichever is greater and Slop 1 ±.05	Date of Pre-sample cal/cal verification:_____ TS#: _____ Cert. Date:_____
Ozone level 2			
Certification/ recertification to Level 1 SRP	Every 365 days and 1/ calendar year	single point difference <± 3.1%	TS# _____ Date:_____
(if recertified via a transfer standard)	Every 365 days and 1 calendar year	regression slopes = 1.00 ± 0.003 and two intercepts are 0 ± 3 ppb	
Ozone Transfer Standard Level 3 and greater			Level III TS #: _____ Date: _____
Recertification to higher level standard	Beginning and end of O3 season or every 182 days and 2/ calendar year whichever less	New slope - ±0.05 of previous and RSD of six slopes ≤ 3.7% Std. Dev of 6 intercepts ≤ 1.5	New slope ±0.05 of previous ☐ RSD of six slopes ≤ 3.7% ☐ Std. Dev of 6 intercepts ≤ 1.5 ☐
Systematic Criteria - Ozone			
Sample Residence Time Verification	every 365 days and 1/ calendar year	≤ 20 seconds	Sample line approximately 4 Meters
Sample Probe, inlet, sampling train	all sites	Borosilicate glass or Teflon	
Siting	Every 365 days and 1/ calendar year	meets siting criteria or waiver documented	Date: _____

Figure 20.5 Quarterly Validation Check PM $_{2.5}$ pg 1

1)Requirement (PM2.5 LC)	2) Frequency	3) Acceptance Criteria	
Field Activities			
Pre-sampling	all filters	≤ 30 days before sampling	Yes / No
Sample recovery	all filters	≤ 7 days 9 hours from sample end date	Yes / No
Sampling Period	all filters	1380-1500 minutes or if value <1380 and exceedance of NAAQS midnight to midnight local standard time	Yes / No
Sampling Instrument			
Average Flow Rate	every 24 hours of op	average within 5% of 16.67 LPM	Yes / No
Variability in Flow Rate	Every 24 hours of op	CV ≤ 2%	Yes / No
One-Point Flow Rate Verification	Monthly separated by 14 days	< ± 4.1% of transfer standard <± 5.1% of design flow rate	Pre-run verification date: Post run verification date: Temp, Pressure and Flow Pass: ☐ Standards Certified: ☐ Time Verified: ☐ Leak checks passed: ☐
Individual Flow Rates	every 24 hours of op	no flow rate excursions > ± 5% for > 5 min [1]	Yes / No
Filter Temp Sensor	every 24 hours of op	no excursions of > 5° C lasting longer than 30 min [1]	Yes / No

Figure 20.6 Quarterly Validation Check $PM_{2.5}$ pg 2

1)Requirement (PM2.5 LC)	2) Frequency	3) Acceptance Criteria	Information/ Action
Laboratory Activities			
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	Min and Max KCDAQM Storage temps: Min: Max: (0°C - 4°C) ☐ Temp. on IML Receipt: (0°C - 4°C) ☐ IML Storage Temp: (0°C - 4°C) ☐ Days from collection to final weight: _____
Filter Conditioning Environment			
Equilibration	all filters	24 hour minimum	Yes / No
Temp Range	all filters	24-hr mean 20.0-23.0 °C	Yes / No
Temp Control	all filters	<2.1 ° C Std dev over 24 hr.	Yes / No
Humidity Range	all filters	24-hr mean 30.0%-40.0% RH or within ± 5.0% sampling RH but ≥ 20.0 % RH	Yes / No
Humidity Control	all filters	< 5.1% std dev over 24 hr	Yes / No
Pre/post Sampling RH	all filters	difference in 24-hr mean < ± 5.1% RH	Yes / No
Field Activities			
Temperature, Pressure & Multi-point Flow Calibration	on installation, then every 365 days and once a calendar year	< ± 2.1 ° C	Date: Pass: Yes / No Standards Certified: ☐
Temperature, Pressure & Flow Audit	every 180 days and at the time of flow rate audit	< ± 2.1 ° C	Date: Pass: Yes / No Standards Certified: ☐

Figure 20.7 Quarterly Validation Check $PM_{2.5}$ pg 3

1)Requirement (PM2.5 LC))	2) Frequency	3) Acceptance Criteria	Information/ Action
Monitor Maintenance			
Pm $_{2.5}$ separator (VSCC)	every 30 days	cleaned/changed	Date:
Inlet Cleaning	every 30 days	cleaned	Date:
Downtube Cleaning	every 90 days	cleaned	Date:
Filter housing Assembly Cleaning	every 30 days	cleaned	Date:
Circulating Fan Filter Cleaning	every 30 days	cleaned/changed	Date:
Laboratory Activities			
Filter integrity (exposed)	each filter	no visual defects	Noted in IML Report: Yes / No
Field Filter Blank	10 % or 1 per weighing session	< ± 30.1 µg change between weighings	Yes / No
Lab filter Blank	10% or 1 per weighing session	< ± 15.1 µg change between weighings	Yes / No
Balance Check (working standards)	beginning, 10th sample, end	< ± 3.1 µg from certified value	Yes / No
Routine Filter re-weighing	1 per weighing session	< ± 15.1 µg change between weighings	Yes / No
Lap Temp Check	Every 90 days	< ± 2.1 ° C	Yes / No
Lab humidity check	Every 90 days	< ± 2.1 %	Yes / No
Siting	every 365 days and once a calendar year	meets siting criteria or waiver documented	Date:

Figure 20.8 Quarterly Validation Check PM_{10}

Run Date:	**Site:**	**Voids/Flags**	**75% Valid** ☐
1)Requirement (PM_{10} Cont)	2) Frequency	3) Acceptance Criteria	Information/ Action
Sampling Period	all filters	1440 ± 60 mins Midnight to midnight local time	Yes / No
One-Point Flow, Temperature &Pressure Verification	Monthly separated by 14 days	< ± 7.1% of transfer standard	Pre-run verification date: Post run verification date: Temp, Pressure and Flow Pass: ☐ Standards Certified: ☐ Time Verified: ☐ Leak checks passed: ☐
FR multi-point Verification/Calibration	every 365 days and once a calendar year	3-4 cal points within 10.1 % of design	Date: Pass: Yes / No Standards Certified: ☐
Semi-Annual Flow Rate Audit	Quarterly	< ± 10.1% of audit standard	Date: Pass: Yes / No Standards Certified: ☐
Monitor Maintenance			
Inlet/downtube Cleaning	Bi-Annually	Yes / No	Yes / No
Siting	every 365 days and once a calendar year	meets siting criteria or waived documented	Date:

Figure 20.9 Quarterly Validation Check Lead pg 1

Run Day:	**Filter #:**	**AQS:**	
1)Requirement (Pb)	2) Frequency	3) Acceptance Criteria	Information/ Action
Filter Holding Times			
Sample Period	all filters	1440 minutes ± 60 min midnight to midnight local	Yes / No
Sampling Instrument			
Average Flow Rate	every 24 hours of op	1.1-1.70 m3/min (varies with instrument) in actual condition	Yes / No
One-Point Flow Rate Verification	once per calendar month > 14 days apart	<± 7.1% from transfer standard	Pre-run verification date: Post run verification date
Filter			
Pressure Drop Range	all filters	42-54 mmHg	Yes / No
Verification/Calibration			
System Leak Check	during pre-calibration check	Visual and Auditory inspection with faceplate blocked	Yes / No
Multi-point Verification/Calibration	After receipt, or failure of 1 -point check and every 365 days and once a calendar year	5 points over range of 1.1 to 1.7 m3/min <± 5.1% limits of linearity	Date: Pass: Yes / No Standards Certified: □
Precision			
Semiannual flow Rate Audit	every 180 days and twice a calendar year	≤± 7.1% of audit standard	Date: Pass: Yes / No Standards Certified: □
Monitor Maintenance			
Analysis Audits	6 strips/quarter 3 per	< 10.1% (percent difference)	Yes / No
Control Standards	1st, every 10 samples and last sample	Deviation of < 5.1% from value predicted by calibration curve	Yes / No

Figure 20.10 Quarterly Validation Check Lead pg 2

1)Requirement (Pb)	2) Frequency	3) Acceptance Criteria	Information/ Action
Siting	Every 365 days and 1/ calendar year	Meets siting criteria or waiver documented	Date:
Precision/Bias			
Single Analyzer	every 90 days and 4 times a calendar year	CV < 20.1% of samples ≥ 0.02 µg/m3 (cutoff value)	Yes / No
Verification/Calibration Standards and Recertifications - All Standards should have multi-point certifications against NIST Traceable Standards			
Flow Rate Transfer Std	Every 365 days and 1/ calendar year	Resolution 0.02m^3/min ±2% reproducibility	Standards Certified: □
Field Thermometer	Every 365 days and 1/ calendar year	2°C resolution	Standards Certified: □
Field Barometer	Every 365 days and 1/ calendar year	± 5 mmHg resolution	Standards Certified: □
Analytical Standards			
Reagents (HNO_3 and HCL)	all	ACS reagent grade	Yes / No
Pb nitrate Pb $(NO_3)_2$	all	ACS reagent grade (99.0% purity)	Yes / No

20.2 Technical Systems Audits

A Technical Systems Audit (TSA) is a thorough and systematic on-site qualitative audit, where facilities, equipment, personnel, training procedures, protocols, and record keeping are examined for conformance with the QAPP. A TSA of the Ambient Air Quality Monitoring Program will be conducted every three years (subject to regulation changes) by EPA Region 4 quality assurance staff. The TSA will use appropriate federal regulations and this QAPP to determine the adequate operation of the Ambient Air Monitoring Program and its related quality system. The EPA will report its findings to Air Quality's management staff. The report will be reviewed and filed appropriately. The Air Monitoring Program Manager or a duly appointed representative will regularly monitor progress on corrective action(s) and report the corrective action(s) to EPA Region 4.

A TSA team or individual TSA auditor may segregate TSA activities into three categories. The categories may be audited independently or be combined. The TSA categories are:

- Field activities including handling sampling and shipping,
- Laboratory activities, pre-sampling weighing, shipping, receiving, post-sampling weighing, archiving and associated QA/QC activities, and
- Data management activities including collecting flagging, editing, uploading and data security.

Key personnel to be interviewed during the audit are those individuals with responsibilities for planning, field operations, laboratory operations, QA/QC, data management and reporting. Post audit and corrective action response shall be prepared and submitted. The report will include the audit title, identification number, date of report and any other identifying information as well as the summary and conclusions of the audit, deliverables required along with a collaborative timeline to complete corrective action. Corrective actions are discussed in more detail in Section 20.6.

20.3 Performance Evaluations

Air Quality participates in 4 types of performance evaluations; Internal Audits, National Performance Audit Program (NPAP), National Performance Evaluation Program (NPEP), and TDEC Audits.

20.3.1 Internal Audits

Air Quality has independent, certified equipment as well as independent staff to conduct quarterly audits as required in 50 CFR par 58 Appendix A. These audits shall follow the Air Quality SOP "SOP for Internal Auditing and System Review, Rev 3". Any findings will prompt corrective action (Section 20.6).

20.3.2 National Performance Evaluations/Audits

Ambient Air Monitoring Agencies submitting data to AQS for comparison to the NAAQS, according to 40 CFR Part 58, Appendix A, must participate in the NPAP audit program. At least 20% of the PQAO gaseous sites each year and all sites within 6 years must be audited. Air Quality relies on the EPA to contract these audits and consents annually to an appropriate portion of the grant funds to be withheld for the NPAP and PEP audit implementation. EPA contract staff schedule and perform the NPAP audits.

Air Quality participates in the $PM_{2.5}$ and lead Performance Evaluation Program. The PEP program is a quantitative comparison of results between equipment being tested and equipment calibrated by another primary standard.

20.3.3 TDEC Audits

Tennessee Department of Environment and Conservation conducts performance audits on the air Quality's monitoring network in addition to the internal audits conducted by Air Quality. These audits are done when time, resources, and staff are available. TDEC will issue a report to Air Quality along with supporting certification of audit standards. Air Quality will upload all valid TDEC audit data into AQS. Audits deemed to be invalid by Air Quality will be filed with all other TDEC audits with a written

explanation justifying the decision to reject the results. Audits provided without adequate certification and NIST traceability will automatically be deemed invalid.

20.4 Data Quality Assessments

A Data Quality Assessment (DQA) is the statistical analysis of environmental data to determine where the data meet the assumptions that the DQOs and data collection design were developed under, and whether the total error in the data are tolerable. Calculations for measurement uncertainty are carried out by the EPA according to the procedures and equations identified in 40 CFR Part 58, Appendix A, §4. The DQIs are used to assess how well the monitoring data compare to the established DQOs and MQOs. AQS provides statistical software that evaluates the DQIs of precision, bias, and completeness for the monitoring organizations. With that in mind, the PQAOs must report the data for QA/QC checks to AQS. Measurement uncertainty will be estimated for both automated and manual data recording methods.

The statistical estimates of the data quality will be calculated in AQS on the basis of single monitors, as well as aggregated for monitors within the PQAO for a specific pollutant. The precision estimate (calculation) used to assess the precision checks for the gaseous analyzers is found in 40 CFR Part 58, Appendix A, §4.1.2; the bias estimate is found in the §4.1.3. The precision estimate (calculation) for particulates is found in 40 CFR Part 58, Appendix A, §4.2.1; the bias estimate is found in the §4.2.2. Other DQA calculations are also detailed in this section of the CFR.

To complete the DQAs, the QA Officer will generate a series of standard AMP reports from AQS to review and assess the data quality quarterly. For this quarterly assessment the QA officer shall use: the AMP 256 (Data Quality Indicator Report), AMP 480 (Design Value Report), the AMP 430 (Data Completeness Report), EPAs Data Assessment Statistical Calculator (DASC) tool on the AMTIC site, data and audit review. A quarterly Quality Assurance Report is generated by the QA Officer for the review of the Director and the Air Monitoring Program Manager. The Air Monitoring Program Manager also reviews the AMP 600 report from AQS during annual data certification. Air Quality shall submit to the EPA an annual summary report, also known as data certification, of all the ambient air quality monitoring data from all monitoring stations designated as SLAMS, in accordance with 40 CFR Section 58.15. The report will be submitted by May 1 of each year for the data collected from January 1 through December 31 of the previous year. The report must be certified by Air Quality's Program Director to be accurate to the best of his/her knowledge. This certification will be based on the various assessments and reports performed by the organization and accompanied by the annual QA report discussed in Section 21.3 that documents the quality of the ambient air quality data and the effectiveness of the quality system.

Air Quality must submit an Annual Performance Report to EPA Region 4 ARD. This report lists all requirements for the 103 and 105 Grants and Air Quality's completion of those requirements or the corrective action taken if requirements were not met.

20.5 Annual Employee Competency Assessment

Annually the Air Monitoring Program Manager will either observe or review each employee's routine duties and complete the "Employee Competency Form" found in Figure 8.1 of Section 8 of this QAPP. These will be filed in the employees training record.

20.6 Reporting and Resolution of Issues

Any participant in the collection, analysis, audit/assessment, and report generating activities affiliated with the Ambient Air Quality Monitoring Network is responsible for identifying the need for corrective actions. Identifying the need for corrective actions can occur during site visits, audits, data analysis operations, or other monitoring network activities. This shared responsibility, coupled with diligent attention to detail and accuracy, will assure

that the Ambient Air Quality Monitoring Network consistently collects quality data, and that data are reduced, analyzed and presented in an accurate and representative manner.

Any participant that perceives a need for corrective action(s) shall present the situation to the Air Monitoring Program Manager. QA Officer, Environmental Specialist (operators), or the Air Monitoring Program Manager are responsible for implementing corrective actions. A corrective action report template (Figure 14.9) can be found on the shared network drive. Once the corrective action report is completed, it should be presented to the QA Officer to be reviewed and then given to the Air Monitoring Program Manager for approval. The report is then saved on the shared drive in the year subfolder of Corrective Action Reports folder, and included with the validated data packages and used to make validation decisions.

21.0 Reports to Management

This section describes the quality-related reports and communications to management necessary to support SLAMS network operations and the associated data acquisition, validation, assessment, and reporting. Unless otherwise indicated all reports will contain monitoring data for criteria pollutants.

21.1 Frequency, Content, and Distribution of Reports

Table 21.1 Reports Generated

Frequency	Report Name	AQS Report Code	Prepared for	Prepared by
Quarterly	Internal Audit Report	None	Director, AMPM	QA Officer
Quarterly	Quality Assurance Report	AMPs 251, 430 & 256	Director &AMPM	QA Officer
Annually	Certification Evaluation	AMP600	EPA	AMPM & Director

21.2 Quarterly Reports

21.2.1 Quality Assurance Report

QA Officer will generate a series of standard AMP reports from AQS to review and assess the data quality quarterly. For this quarterly assessment the QA officer shall use: the AMP 256 (Data Quality Indicator Report), AMP 480 (Design Value Report), the AMP 430 (Data Completeness Report), EPAs Data Assessment Statistical Calculator (DASC) tool on the AMTIC site, data and audit reports. The quarterly Quality Assurance Report is generated by the QA Officer to provide a concise overview of the completeness, and quality of the data in AQS for the previous quarter. This report is prepared for the review of the Director and the Air Monitoring Program Manager.

21.2.2 Internal/ State Audit Report

An audit report will be generated after each audit and submitted to the Director and Air Monitoring Program Manager. The QA officer will enter the results into AQS. See Internal Audit SOP in Appendix B for more information.

21.3 Annual Reports

Air Quality certifies all criteria pollutant data annually to EPA by May 1st of each year. Part of the annual certification process is generating the AMP 600 Data Certification Report. Once Air Quality reviews the report and AQS recommendations for data certification, concurrence flags are applied and the report is run again and saved. It accompanies the data certification letter sent by the Director to the Regional Administrator for EPA Region 4.

21.4 Response/ Corrective Action Reports

The corrective action report will be generated whenever an issue arises affecting data quality. Reasons to generate a correction action report include, but are not limited to: a failed audit, safety issue, failed monthly verification or bi-weekly precision check. Corrective action shall be recorded on the corrective action report form. The corrective action report is one of the most important, ongoing reports because it documents primary QA activities and provides valuable records of QA activities that can be used in preparing other summary reports. (See Sections 11.6 and 20.6 and figure 14.9)

22.0 Data Validation and Usability

Data verification and validation are described in the "QA Handbook Volume II, Section 17 (2017)" as "*Data review, verification and validation are techniques used to accept, reject, or qualify data in an objective and consistent manner. Verification can be defined as confirmation, through provision of objective evidence, that specified requirements have been fulfilled1. Validation can be defined as confirmation, through provision of objective evidence, that the particular requirements for a specific intended use are fulfilled. So, for example, one could verify that for a monitor all 1-point QC checks were performed every two weeks (specified requirement) as described in standard operating procedures (specified requirement). However, if the checks were outside the QC limits described in the QAPP, the validation process might determine that the data could not be used for NAAQS determinations (intended use)…"*

Criteria for data validation are described in Section 23 of this document. Each of the network's analytical instruments are employed to measure ambient concentrations of specific pollutants. In order to be useful, the data must undergo evaluation to determine the degree to which each data point has met its quality specifications. Air Quality staff, particularly the QA Officer, evaluate the data to establish that data collection is consistent with QAPP and SOP requirements. Then, the Air Monitoring Program Manager, in collaboration with the QA Officer, estimates the potential effect any deviation from the QAPP or SOP requirements may have on the usability of the associated data item, its contribution to the quality of the reduced and analyzed data, and its effect on decisions.

Data review is the in-house examination to ensure that the data has been recorded, transmitted, and processed correctly. It includes completeness checks to determine if there are any deficiencies such as missing data or lost integrity. The data under evaluation should be compared to actual events, as per guidance (*Guidance on Environmental Verification and Validation* (EPA QA/G-8)). In addition, it is expected that some of the QC checks will indicate that the data fail to meet the acceptance criteria. Data identified as suspect, or does not meet the acceptance criteria, shall be flagged with AQS codes prior to upload to AQS. The review of the routine and the associated QC data will be verified and validated on a monthly basis. Continuous data is downloaded to AirVision hourly and examined daily (week days) to ensure the data is acquired according to requirements. Continuous data is later reviewed in batches during the data validation process. Non-continuous data is reviewed and verified by the site operators during collection and retrieval and is reviewed and verified in batches as part of the data reduction and validation process prior to each bi-weekly shipment to IML. Corrective action is taken if errors or anomalies are found. In cases when data does not meet quality goals it may be flagged or invalidated.

Data verification is the process for evaluating the completeness, correctness, and conformance / compliance of the data set against method, procedural and contractual specifications. Verification can be further defined as confirmation, through provision of objective evidence, that specified requirements have been fulfilled. The verification process also involves the inspection and acceptance of the field samples. Site operators verify the gaseous data collected when reviewing and documenting their electronic strip charts throughout the sample collection process, as well as by verifying the status flags applied to data by the site data loggers. Site operators verify intermittent data when downloading/transferring files from PM2.5 samplers, site operators inspect the intermittent samples – pre and post-sampling – to ensure they are intact and undamaged. Data verification also occurs when the QA Officer completes the monthly verification for data submittal to AQS. Any missing data (gaps) are reviewed and accounted for, and unacceptable or questionable data will be flagged during this monthly process. Once the evaluations are complete, the data are reviewed for routine data outliers and conformance to acceptance criteria.

Data validation is a routine process designed to ensure that reported values meet the quality goals of the environmental data operations. Data validation is further defined as examination and provision of objective evidence that the particular requirements for a specific *intended use* are fulfilled. The primary intended use for the Air Quality data set is NAAQS compliance. A progressive, systematic approach to data validation must be

used to ensure and assess the quality of data. Data validation includes the review of the data sets against the individual pollutant MQOs (see Section 7.0 of this QAPP), which is completed by the QA Officer. It also includes the review of data against the Office's QC reports, QA reports, and electronic strip charts, as well as the comparison of the data against basic statistics (such as completeness). Reviewing data long-term (over a monthly or quarterly time-frame) provides information about the structure of the data and may identify patterns, relationships, or potential anomalies. The QA Officer also spot-checks comparisons between the electronic strip charts and data summary reports from the polling CPU data system to ensure data consistency. If a problem/discrepancy is found, further investigations must be done to find the source of the error and then corrected. Invalidated data are replaced with AQS Null Data codes prior to upload to AQS. Deviations from operational procedures or quality assurance requirements that do not result in data invalidation may require that data be qualified with QA qualifier flags prior to upload to AQS. The AMPM spot-checks the data after validation by the QA Officer is completed, prior to AQS upload.

22.1 Sampling Design

The Air Quality Air Monitoring Program's primary function is to verify compliance with the NAAQS. Towards that end, sampling network design and monitoring site selection comply with the following appendices of 40 CFR Part 58:

- 40 CFR Part 58, Appendix A – Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS)
- 40 CFR Part 58, Appendix D – Network Design for State and Local Air Monitoring Stations (SLAMS) and National Air Monitoring Stations (NAMS)
- 40 CFR Part 58, Appendix E – Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring

Additional guidance is provided in "Guidance for Choosing a Sampling Design for Environmental Data Collection" (EPA QA-G-55)

The locations of all the sites have received EPA approval; thus, data from each monitor will be considered spatially representative as long as the sites continue to meet the requirements set forth by 40 CFR 58, Appendix E - Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring. Air Quality will request approval from EPA Region 4 for any new site, prior to establishment. Sometimes deviations are unavoidable. Any deviations from the minimum siting criteria (e.g. shelter location, probe placement, and/or monitor sight path requirements) shall be thoroughly documented in the site's annual site assessment. Measured deviation from the siting criteria will require data to be flagged in the AQS database (i.e., "SX" qualifier) until such time as corrective actions can be implemented (e.g., tree trimming to correct dripline issues), or an approved waiver from EPA Region 4 must be obtained. The impact of any deviations shall be evaluated by the Air Monitoring Program Manager, in consultation with EPA Region 4, prior to the use of the data for calculation of summary statistics.

22.2 Sample Collection Procedures

All criteria pollutant monitors are FRM/FEM designated (Table 11.1). These methodologies are acceptable for regulatory decision making. Data are to be collected with calibrated instrumentation and subject to frequent QC checks as outlined in Section 11 and Section 14 of this QAPP and in detail in Air Quality's pollutant specific SOPs that are reviewed and approved by EPA Region 4. CoC forms shall be filled out when receiving or shipping a sample(s), field forms shall be filled out and initialed to establish a record of where the sample is at all times. Any deviation from the established sample collection procedure must be documented in the appropriate logbook and on the field form. Accurate and complete documentation of any sample collection deviations will assist in subsequent investigations or evaluations. Field staff are evaluated annually using the Employee Competency Sheet (Figure 8.1) to ensure proper procedure is being followed.

22.3 Data Review & Verification

Examine data to ensure accurate recording, transmitting, and processing. Completeness should be checked for deficiencies such as missing data. Any data considered suspect (does not meet all quality objectives found in tables in Section 7.0 and 14.0) should be invalidated or flagged prior to AQS upload. QC data are reviewed by the QA Officer monthly, this includes audit data, flow verification data, precision and calibration data. This ensures that verifications and calibrations are occurring as scheduled.

Continuous data are polled hourly and downloaded to the server via the AirVision Computer. It is reviewed daily to ensure it is being polled properly and appears free of anomalies, such as missing data or logger flag. Non -continuous data are reviewed by the site operator upon retrieval of the filters. Factors such as time and status codes are checked and deficiencies noted as well, to ensure proper conditions were met during sampling. The data reviewer later reviews the sampler data for any flags and complies the data in a text file to be sent with the filters to IML for analysis. More detail can be found in individual pollutant SOPs in Appendix C-F.

22.4 Data Validation

Air Quality utilizes a multilayered validation strategy with different individuals performing validation tasks a process designed to ensure that reported values met the quality goals of the data operation. The operators or field staff perform the first round of validation by applying void codes to the data due to field results (power failure, filter exchange error, QC check, etc). The QA Officer reviews data in monthly and quarterly batches during validation.. This second level review considers additional MQOs, spot checks spreadsheets and calculations. Finally the AMPM reviews the full validation package prior to upload to AQS. Potentially unacceptable data points are routinely identified in the database through electronic application of error flags (AirVision/MTL). Each instrument-specific flag is associated with a unique error. These error flags are routinely reviewed as part of the data validation process. This activity assists in identifying suspect (potentially bad) data points that could invalidate the resulting averaging periods. Void codes are applied to data that is not considered adequate for NAAQS comparison. Qualifier flags are applied to data that may have issues that cause it to not be a perfect representative sample, but still considered adequate for use in NAAQS comparison and statistical use. The use of qualifier flags allows end users of the data provided to evaluate the data point as accurately as possible. A compilation of the typical error flags and void codes is presented in Section 23 of this document table 23.1. A full list of the possible qualifiers (flags and void codes) can be found at https://aqs.epa.gov/aqsweb/documents/codetables/qualifiers.html . While not exhaustive, the list contains some examples of data loss/invalidation and the associated methods of data handling.

- All periods of missing concentration data (e.g. during QC activities, maintenance, and power failures) will be replaced with the appropriate AQS Null Data codes.
- Each hour of pollutant concentration data is composed of at least 45 minutes of valid minute data. Hours of data with less than 45 minutes of valid data collection will require invalidation of the hourly data. The affected data will be replaced with AQS Null Data codes.
- The shelter temperature values will adhere to the proper criterion. All data collected when outside of this temperature range will be invalidated and replaced with AQS Null Data codes.
- The precision point must meet MQO for the 1-point QC checks for ozone. If the point exceeds the allowable criteria, all data will be invalidated back to the point of the last acceptable QC check or a known point of analyzer malfunction. The affected data will be replaced with AQS Null Data codes.
- Precision and Bias Check data uploaded to AQS as QC data must actually quality-assure the applicable concentration data within AQS. Examples include:
    - If concentration data are invalidated due to unacceptable results during a QC check (flow verification or 1-Pt QC), the results of the failed QC Check will be uploaded to AQS.
    - If any equipment used to generate the QC check fails during a check, or generates an unstable or inaccurate test atmosphere, the QC check itself will be invalidated. If the Check itself is deemed invalid. Null code 1C will be uploaded to AQS for the invalid audit readings. No ambient data will be impacted if an investigation determines that the test equipment failed and the analyzer was in control and functioning properly.

- The calculated percent difference during a QA Performance Evaluation must meet the MQO guidance for each pollutant. If the Performance Evaluation exceeds the MQO difference, concentration data will be invalidated back to the last known acceptable QA/QC check or known point of analyzer malfunction. The affected data will be replaced with AQS Null Data codes.
- Performance Evaluation data uploaded to AQS as QA data must quality-assure the applicable concentration data within AQS.

22.5 Exceptional Events

40 CFR 50.14 allows the EPA Administrator to exclude certain data from being used for determinations of exceedances and violations of a NAAQS, so long as a State/Local demonstrates to the Administrator's satisfaction that the exceedance or violation was caused by an “exceptional event.” 40 CFR 50.1 defines an “Exceptional Event” as an event or events, in which:

- The resulting emissions affect air quality in such a way that there exists a clear causal relationship between the specific event(s) and the monitored exceedance(s) or violation(s);
- The event(s) is not reasonably controllable or preventable; and,
- The event(s) is caused by a human activity that is unlikely to recur at a particular location or is a natural event(s).

An Exceptional Event does not include:

- Air pollution relating to source noncompliance;
- Stagnation of air masses or meteorological inversions; and,
- Meteorological events involving high temperatures or lack of precipitation.

Note: Conditions involving high temperatures or a lack of precipitation may promote occurrences of particular types of exceptional events, such as wildfires or high wind events, which do directly cause emissions.

Data impacted by an Exceptional Event is not considered “representative” of air quality for NAAQS comparison purposes, or calculation of certain summary statistics. All concentration data impacted by an Exceptional Event should be flagged with an AQS Information code and linked within AQS to an event description. Exceptional Event codes and descriptions are typically due by July 1 of the following year, but alternative schedules may be established during Federal rulemaking.

It is the responsibility of the Air Monitoring Program Manager to analyze data for potential Exceptional Events and to add the necessary flags and descriptions into AQS by July 1 of the following year (or by applicable regulatory deadlines). A State seeking concurrence must notify and cooperate with the appropriate EPA Regional Office (i.e. EPA Region 4) to prepare a demonstration package for the Administrator. Exceptional Event data in AQS must receive concurrence from the EPA Administrator. Data that does not receive a concurrence is still eligible for NAAQS comparisons, regardless of the application of Request Exclusion flags.

23.0 Verification and Validation Methods

40 CFR Part 58, Appendix A Sec 1.2.3 states:
"*Each PQAO is required to implement a quality system that provides sufficient information to assess the quality of the monitoring data…Failure to conduct or pass a required check or procedure, or a series of required checks or procedures, does not by itself invalidate data for regulatory decision making. Rather PQAOs and the EPA shall use the checks and procedures required in [Part 58, App. A] in combination with other data quality information, reports and similar documentation that demonstrate overall compliance with Part 58. Accordingly, the EPA and PQAOs shall use a "**weight of evidence**" approach when determining the suitability of data for regulatory decisions. Consensus built validation templates or validation criteria already approved in QAPPs should be used as the basis for the weight of evidence approach."*

All verification and validation activities are not a linear process and are performed by continuously by all personnel. The modified validation tables listed in Section 7 (Tables 7.1-7.4) contain measurement quality objectives (MQOs) for each of the NAQQS pollutants measured within the PQAO. There are 3 classifications of criteria within the MQO tables. Each classification has a different significance on the resulting data. These guidelines along with intelligent judgement will be used to implement the weight of evidence approach.

These tables include:

- Critical Criteria- is critical to maintaining the integrity of the sample or group of samples. Data which does not meet all of the critical criteria MQOs shall be invalidated unless there are compelling reason and justification to argue validity. An example would be the sample flow rate, this is designated in the CFR or the designation method and any data produced outside the acceptable range would be invalid. Most critical criteria in the MQOs are designated in the Code of Federal Regulations (CFR). Any validation of data that does not meet all critical criteria must be flagged, documented and approved by the Air Monitoring Program Manager.

- Operational Criteria represents MQOs that as an aggregate create a good quality system. Violation of one of the criterion or a number of criterion may be cause for data invalidation but with weight of evidence approach can often be flagged, yet still reportable valid data. An example of operational criteria violation would be shelter temperature control. A faulty AC unit or thermometer may cause a larger variation in the 24-hour temperature in a shelter. If the shelter temperature remained within the acceptable range of the instrument, and data analysis showed the data as comparable to other locations in the PQAO or regional area, the data would be flagged, but illustrated to still be valid. Data not meeting the operational criteria requires additional quality control or comparison documentation to justify validation.

- Systematic Criteria are those criteria evaluated at the site, network, or PQAO aggregate levels. Often this criterion is the level two validation. The failure to meet the MQOs of systematic criteria does not typically invalidate data on its own. An example may be failure to certify auditing equipment annually, this would not on its own invalidate data, but would question the validity of QA & QC checks performed with the invalid equipment. It shall result in further investigation of the data and documentation of problems found, and corrective action proposed. An additional example would be the clock on PM2.5 filter-based monitor. If it is outside the 1min acceptance criteria, it would not invalidate date, however the data validator would need to review the runtimes for the samples collected while the clock was out of specification to ensure 23-24 hours runtime on the sample day. The QAO and or AMPM shall check the systematic criteria at least quarterly.

23.1 Verification

Verification is the evaluation of the completeness, correctness and conformance of the data set against method, procedural and contractual specifications. Confirmation, through provision of objective evidence, that

specified requirements have been met. Procedures for performing equipment verifications are detailed in the Calibration & Verification sections of Air Quality's pollutant specific SOPs (Appendix C-F). Equipment verifications are performed by site operators. Additional verification activities are listed below:

- All staff (Operator, QA Officer, and Manager) should verify daily that continuous data are being polled and received hourly.
- The site operator should verify that $PM_{2.5}$ data downloads are captured completely at least biweekly.
- The site operator shall inspect non-continuous filter media pre-and post-sampling.
- The QA officer shall ensure continuous data are reviewed for missing data and flags or null codes are added before uploading to AQS
- The QA officer shall review the uploaded files to AQS to ensure all values are captured.
- Any hand-entered data into AQS will be checked for correctness by another employee by generating a raw data report from AQS and checking the entered data. The report should be initial filed accordingly

23.2 Validation

Data validation is the confirmation, through objective evidence, that the requirements are met for the intended use. Air Quality validates with the intended end use of NAAQS comparison as the standard with the weight of evidence approach. Data validation is not a linear process, and data validation is performed at several points internally and externally (contract laboratories). The SOPs for the analytical laboratories describe their data validation procedures in detail. Standardized abbreviations describing the status of individual results are either void (null codes) or status flags. Void/ null codes are assigned to a sample or groups of samples when the critical quality objectives for the intended use are not met. These void codes replace the value in the reporting of the data. Status flags indicate that a sample may have not meet all quality objectives, but the value is still reported. Additionally, flags may be applied for unusual or non-typical events, such as forest fires, construction, or fireworks. These flags provide end users with additional information on a sample.

23.2.1 Validation Levels

Watson et al. (1995) in "*The measurement process; Precision, accuracy and validity.* In *Air Sampling Instruments for Evaluation of Atmospheric Contaminants"* defines a three-level data validation process for environmental measurement studies. Air Quality uses these levels as general guidelines to aid in tracking data validation progress.

Level 0 – This level represents raw data, obtained directly from the instruments. These data are generally unedited and unreviewed, without adjustments. These data are aggregated to obtain averages for the sampling periods. Level 0 data may have been flagged for instrument downtime, QA/QC verification procedures, or other automated flags in the datalogger or AirVision software. These are applied by the site operator or automated by the logger. Uses in data validation for Level 0 data include but is not limited to:

- Verifying completeness of sampling period averages
- Reviewing output of quality control test
- Identifying suspect data, evaluating instrument response
- Providing weight of evidence in support of later validation decisions

Level 1A – Data at this level have passed initial qualitative reviews for accuracy and completeness. This is reviewed at least monthly. This is performed by the field staff or QAO. Level 1A data validation includes:

- Reviewing Temperatures, Pressures, and flow rates of the samplers meet the MQOs
- Reviewing logbooks, filter tracking sheets, reconciling sampler data for dates and maintenance
- The site operator fills out many field forms, verification forms, parameter check forms, and precision check forms (See SOPs in Appendix C-F for complete list). These forms have the acceptance criteria listed on the form. The site operator should verify, while filling out a field form, if acceptance criteria are being met.

- Assigning status flags or void codes due to sampler malfunctions, power outages, QA or QC checks. A compilation of the typical error flags and void codes is presented in tables 23.1-23.4. A full list of the possible qualifiers (flags and void codes) can be found at https://aqs.epa.gov/aqsweb/documents/codetables/qualifiers.html .

Level 1B – Data at this level has passed additional review for quantitative and qualitative consistency. Data are reviewed for all critical and operational requirements of the MQOs in Section 7. This is review occurs monthly for continuous analyzers and quarterly for intermittent samplers by the QAO and AMPM reviews results. Level 1B data is ready for entry into AQS database. Level 1B validation includes:

- Reviewing contract laboratory results, validation, & QC (field, lab blanks, holding temperatures)
- Examining the results of all flow rate checks, precision checks, and internal audit results for the timeframe.
- Reviewing results from audits, verifications, and precision checks and enters them into the QA database as well as AQS. If a failure of a valid audit/verification/precision check occurs, a corrective action report will be initiated. The last valid check will be located and data will be invalidated from the last valid check, until a passing check occurs, unless strong evidence can be supplied for only flagging the data.
- Evaluating individual sample periods for outliers

Level 2 – Data is reviewed at this level at the quarterly, semi-annually, and yearly basis by the QAO or AMPM. Level 2 checks include statistical analysis at the monitor, site and network wide. Level 2 is a method to step back and see the data in larger chunks. Due the rigorous controls in place, it is atypical for level 2 validation to result in data invalidation. Level 2 validation is most useful for seeing long term drift (bias) or instability (precision) of instruments. Level 2 validation includes:

- Statistical analysis of precision, bias, coefficient of variation
- Site by site comparison for outliers
- Review of all certifications
- Data completeness

It should be noted that Level 2 data review is continuous, and as validation is not linear, does not mean it shall only occur after completion of the lower level validations. For example, the certifications and qualifications of equipment are tracked continuously, not just reviewed after all yearly data is attained.

Within each level, data may be found to; meet all objectives of the quality system, be flagged indicating that data does not meet all objectives or that unusual event(s) may reduce the representativeness of the data, or data may be invalidated. Invalidated data is not reported to AQS, only a void code is reported.

Each level of review is documented on the pollutant specific QA tracking sheets a sample is illustrated in figure 23.1 below.

Figure 23.1 QA Tracking Sheet

KNOX COUNTY
HEALTH DEPARTMENT

Month : _____ Site: _____ Monitor SN: _____

Monthly Ozone QA Tracking Sheet	Date	Initials
Level 1 – Completed Daily review of minute data in AirVision		
Level 1 – Logbooks scanning for month complete		
Level 1- All QC checks performed, printed with graphs		
Level 1 – Reviewed monthly hourly report, assigned null codes, created AQS file		
Level 1- Corrective Action Report on File {Initial/ date only if applicable for the month)		
Level 2: Review verifications and audits – verified in database and AQS		
Level 2: Reviewed AirVision reports (shelter T and Sample Flow)		
Level 2: Reconciled logbook entries with QC documentation		
Level 2: Reviewed Flags and null codes by operator, updated AQS file, Void Data Log		
Level 2: Investigate outliers		
Level 2: QC Excel form Calc Check **Date on Form Checked :**_____		
Level 3 : Supervisory Review		
Data Uploaded to AQS		

Analyzer value	Calibrator Value	% D on Form	% D Calc

Level 1 items: Completed by the operator
Level 2 items: Completed by QA personnel
Level 3 items: Completed by Program Manager

23.2.2 Null and Qualifier Codes

The coding charts below (Tables 23.1-23.4) are separated by color; green for often used, yellow for sometimes used, and red for rarely used. These do not represent all possible codes, but rather those consistently applied by Air Quality. Conformity of code application provides a consistent representation of activities. However, data should be coded to provide the best explanation of events, the full list of qualifiers (flags and void codes can be found at https://aqs.epa.gov/aqsweb/documents/codetables/qualifiers.html .

Table 23.2 Standard Null and Qualifier Codes Ozone

Code	Description	Type	Example	Applied to
AN	Machine malfunction	Void	Analyzer photometer readings too high	Individual hourly data
AH	Sample Flow Rate out of Limits	Void	Pump failure (flow not between 720-800cc/min	Either Individual or groups of data
AT	Calibration	Void	Used for Operator initiated Calibration	Individual hourly data
AV	Power Failure	Void	Power outage	Individual hourly data
BA	Maintenance/ Routine Repairs	Void	Changing pump or internal filter	Individual hourly data
BC	Multi-point Calibration	Void	Multi-point calibration	Individual hourly Data
BF	Precision/Zero/Span	Void	When all three points are generated	Individual hourly data
BL	QA Audit	Void	Internal or TDEC audits	Individual hourly data
QV	QC multi-point verification	Void	Multi-point verification with NO calibration	Individual hourly data
1V	Data reviewed and validated	Flag	Failed or missing verification data, but compelling evidence data is good.	Groups of hourly data to last good QC
6	QAPP Issue	Flag	Data collection deviates from the QAPP but reviewed as valid.	Groups of hourly data
AE	Shelter Temperature Outside Limits	Void	AC or heater malfunction, shelter temperature outside range	Groups of hourly data
AS	Poor Quality Assurance Results	Void	Failing QC check, but no obvious machine malfunction	Groups of hourly data to last good QC
AX	Precision Check	Void	Used if only precision point, or precision and zero performed as QC for time offline	Individual hourly data
AY	QC Control Points	Void	Used if only zero and span are performed	Individual hourly data
BK	Site Logger Down	Void	Airvision or logger maintenance or changes resulting in lost data	Individual hourly data
1	Deviation from CFR/ Critical Requirement	Flag	Extreme EPA approved use circumstance	Groups of hourly data
5	Outlier	Flag	Value abnormally high or low, but no evidence to suggest invalid	Individual hourly data
BE	Building/Site Repair	Void	Repairs of shelter that cause data to be loss, rare as repairs scheduled during off season	Individual hourly data
BG	Missing ozone data not likely to exceed standard	Void	Negative values in early morning spring due to condensation	Individual hourly data
EC	Exceeds Critical criteria	Void	Only used if more descriptive code unavailable	Groups of hourly data
SX	Does not meet siting criteria	Flag	Tree growth or obstruction added, temporary must be addressed	Groups of hourly data

Table 23.3 Standard Null and Qualifier Codes $PM_{2.5}$

Code	Description	Type	Example	Usually Applied to
AG	Sample time out of limits	Void	Machine did not run for 23 hours	Single samples
AH	Sample Flow rate/ CV out of limits	Void	Pump failure or variable pump speeds	Single Samples
AN	Machine Malfunction	Void	System malfunction	Single samples
AQ	Collection Error	Void	Filter exchange error	Single Samples
AV	Power Failure	Void	Power down at site	Single Samples
BA	Maintenance/Repairs	Void	Repairs	Single Samples
BJ	Operator Error	Void	Operator fails to set sampler in proper sampling mode	Single Samples
TS	Holding Time or Transport Temp out	Void	Filter shipment arrives >4 degrees C and > than avg T and weighed later than 10 days from sampling, or expired filter sampled	Groups of Samples
X	Filter Temperature Diff	Flag	Filter T > 5 Degrees from Ave T during sampling	Single Samples
1V	Data reviewed and validated	Flag	Failed or missing verification data, but compelling evidence data is good.	Groups of samples to last good QC
6	QAPP Issue	Flag	Data collection deviates from the QAPP but reviewed as valid.	Groups of samples
AK	Filter Leak	Void	Failed leak check	Group of samples to last passing check
AT	Calibration	Void	If calibration performed on run day and exceed 1 hour	Single samples
AZ	QC Audit	Void	If Flow verification performed on run day and exceeds 1 hour	Single Samples
BI	Lost or Damaged in Transit	Void	Filter shipment lost/ delayed to laboratory	Group of samples
BL	QA Audit	Void	Internal or TDEC Audit performed on run day	Single Samples
1	Deviation from CFR/ Critical Requirement	Flag	Extreme EPA approved use circumstance	Groups of samples
5	Outlier	Flag	Value abnormally high or low, but no evidence to suggest invalid	Individual samples
AJ	Filter Damage	Void	Filter is damaged/ torn	Individual samples
AS	Poor Quality Assurance Results	Void	Failing QC check, but no obvious machine malfunction	Groups of samples to last good QC
HT	Holding Time	Flag	Sample retrieved outside of acceptable limits, but determined valid	Individual samples
IT	Wildfire- US	Flag	Informational flag to mark unusually high values due to fire	Single Samples
SX	Does not meet siting criteria	Flag	Tree growth or obstruction added, temporary must be addressed	Groups of Samples

Table 23.4 Standard Null and Qualifier Codes Lead

Code	Description	Type	Example	Usually Applied to
AG	Sample time out of limits	Void	Machine did not run for at least 23 hours	Single samples
AH	Sample Flow rate	Void	Pump failure or exceeds acceptance % difference from design	Single Samples
AN	Machine Malfunction	Void	Filter exchange errors	Single samples
AV	Power Failure	Void	Power down at site	Single Samples
AW	Wildlife Damage	Void	Birds nesting damages filter	Single Samples
BA	Maintenance/Repairs	Void	Repairs	Single Samples
BJ	Operator Error	Void	Operator fails to set sampler in proper sampling mode	Single Samples
6	QAPP Issue	Flag	Data collection deviates from the QAPP but reviewed as valid.	Groups of samples
AZ	QC Audit	Void	Quarterly Collocated Filters shipped to Region 9	Single Samples
BI	Lost or Damaged in Transit	Void	Filter shipment lost/ delayed to laboratory	Group of samples
BL	QA Audit	Void	Internal or TDEC Audit performed on run day	Single Samples
DI	Sample Diluted for Analysis	Flag	Laboratory reports if Dilution method used	Single Samples
1	Deviation from CFR/ Critical Requirement	Flag	Extreme EPA approved use circumstance	Groups of samples
5	Outlier	Flag	Value abnormally high or low, but no evidence to suggest invalid	Individual samples
AJ	Filter Damage	Void	Filter is damaged/ torn not wildlife related	Individual samples
AS	Poor Quality Assurance Results	Void	Failing QC check, but no obvious machine malfunction	Groups of samples to last good QC
SX	Does not meet siting criteria	Flag	Tree growth or obstruction added, temporary must be addressed	Groups of Samples

Figure 23.5 Standard Null and Qualifier Codes PM Continuous

Code	Description	Type	Example	Usually Applied to
AN	Machine Malfunction	Void	system malfunction	Individual Hourly values
AV	Power Failure	Void	Power down at site	Individual Hourly values
AZ	QC Audit	Void	Monthly Flow Verification	Individual Hourly values
BA	Maintenance/Repairs	Void	Repairs	Individual Hourly values
BL	QA Audit	Void	Internal or TDEC Audit performed on run day	Individual Hourly values
AE	Shelter Temperature	Void	AC/Heat failure shelter temperature exceeds criteria	Groups of Hourly Values
AH	Sample Flow rate	Void	Pump failure or exceeds acceptance % difference from design	Groups of Hourly values
AJ	Filter Damage	Void	Filter Loading exceeds acceptance criteria	Groups of Hourly Values
AT	Calibration	Void	Flow calibrations	Individual hourly Values
BK	Site computer/ logger down	Void	Airvision or Logger maintenance resulting in loss of data	Groups of hourly values
1	Deviation from CFR/ Critical Requirement	Flag	Extreme EPA approved use circumstance	Groups of Hourly values
5	Outlier	Flag	Value abnormally high or low, but no evidence to suggest invalid	Individual Hourly values
6	QAPP Issue	Flag	Data collection deviates from the QAPP but reviewed as valid.	Groups of hourly values
AS	Poor Quality Assurance Results	Void	Failing QC check, but no obvious machine malfunction	Hourly values to last good QC
BE	Building Site Repair	Void	Shelter repair requiring loss of data	Individual Hourly Values
SX	Does not meet siting criteria	Flag	Tree growth or obstruction added, temporary must be addressed	Groups of hourly values

23.2.3 Decision Documentation

Records of all invalid samples shall be retained in the QA database. Information shall include a brief summary of why the sample was invalidated, if not obvious from the flag, along with the associated flags. Logbook notes, field data, and QC forms will provide a more detailed information regarding the reason a sample was flagged or invalidated. The official documents remain as the logbooks, or forms. However, a scanned copy of QA and QC forms are saved to the shared drive within the QA database. This provides a back-up for the more fragile paper documents.

Intermittent sampling validation decisions are recorded on the filter tracking sheets for $PM_{2.5}$ and on the Calculations form for lead. Continuous sampling validation decision are recorded on the AirVision monthly report. When a lengthier explanation of validation decisions is needed or when a large group of data are affected, the QAO or the APMP shall prepared a corrective action report that shall document what occurred, how it was resolved, and the validation decisions.

Annually the QAO reviews each of the validation decisions, cross checks data input into AQS with documented data collected, and reviews logbooks, corrective action reports, and QA/QC checks. Upon completion the QAO notifies the AMPM whom then performs an additional review of the data for certification.

24.0 Reconciliation with Data Quality Objectives

EPA established DQOs for each criteria pollutant. These DQOs are used to assess the quality of the data collected. The DQOs are outlined in Section 7.0. A Data Quality Assessment (DQA) is the statistical analysis of environmental data to determine whether the data meet the assumptions that the DQOs and data collection designed were developed under and whether the total error in the data are tolerable. The QA Officer runs statistical reports from AQS quarterly (See Section 21.2.1) to ensure Air Quality is meeting the DQOs. The Air Monitoring Program Manager will generate an AMP 600 report from AQS annually for data certification. This report summarizes bias and precision statistics at the site, national and reporting organization levels. Examination of the AMP 600 report presents statistical calculations performed by AQS. Examinations of these results determine measurement uncertainty, if DQOs are being met or the level at which the DQOs are being violated. The data used in this statistical analysis has been quality assured, coded, qualified and evaluated upon applicable MQOs. Once the AMP 600 has been evaluated, the Air Monitoring Program Manager will apply concurrence flags on all criteria pollutant data, notifying the EPA that the agency certifies the data. The Director will send the AMP 600 report along with a certification letter to the Regional Administrator by May 1st of each year.

Any failure to meet acceptance criteria on the AMP 600 report will initiate corrective action. Corrective action may include tightening MQOs, updating the QAPP or SOPs. The first step in the corrective action plan, is to ensure all site and monitor data are correct in AQS, as this data is imperative to run proper statistical evaluations.

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Appendix A - Record Retentions Policy

Knox County Department of Air Quality Management
Records Retention Policy

KCDAQM Records Schedule 101

Title: Permit Records

Status: Final

Description:

Records pertaining to the permitting of a stationary source of air pollution. These records include permits, permit applications, correspondence, reports and other related documents. These records include information created by the Department, the owners or operators of air contaminant sources, federal agencies, state agencies, local governments and the public.

Retention Instructions:

Item a: New Source Review (NSR) permit application and associated documents establishing a minor source baseline date

- **Permanent**

Item b: All other permit records

- **Disposable**
- Close when source is no longer in operation.
- Destroy 10 years after file closure.

Guidance:

Media neutral – This schedule allows the disposition of the record copy in any media (media neutral). If the record is created in electronic format or digitized (e.g., imaged) and maintained electronically, the electronic records must be retrievable and usable for as long as required by the applicable retention schedule.

Sensitive information – When records are due for destruction according to the retention instructions, records containing sensitive information (e.g., confidential business information) must be shredded or otherwise definitively destroyed to protect confidentiality.

Multiple record schedules – In situations where records are covered by more than one record schedule, retain the record for the longest retention period.

Knox County Department of Air Quality Management Review: 3/3/2017

Knox County Law Department Review: 3/22/2017

TDEC Division of Air Pollution Control Review: 3/22/2017

Public Records Commission Approval: 10/12/2017

Knox County Department of Air Quality Management
Records Retention Policy

KCDAQM Records Schedule 201

Title: Compliance Monitoring Activity Records

Status: Final

Description:

Records pertaining to compliance monitoring activities. These records include correspondence, reports and other related documents. These records include information created by the Department, the owners or operators of air contaminant sources, federal agencies, state agencies, local governments and the public.

Retention Instructions:

Item a: All compliance monitoring activity records

- **Disposable**
- Close when activity is complete.
- Destroy 10 years after file closure.

Guidance:

Media neutral – This schedule allows the disposition of the record copy in any media (media neutral). If the record is created in electronic format or digitized (e.g., imaged) and maintained electronically, the electronic records must be retrievable and usable for as long as required by the applicable retention schedule.

Sensitive information – When records are due for destruction according to the retention instructions, records containing sensitive information (e.g., confidential business information) must be shredded or otherwise definitively destroyed to protect confidentiality.

Multiple record schedules – In situations where records are covered by more than one record schedule, retain the record for the longest retention period.

Knox County Department of Air Quality Management Review: 3/3/2017

Knox County Law Department Review: 3/22/2017

TDEC Division of Air Pollution Control Review: 3/22/2017

Public Records Commission Approval: 10/12/2017

Knox County Department of Air Quality Management
Records Retention Policy

KCDAQM Records Schedule 301

Title: Enforcement Records

Status: Final

Description:

Records pertaining to the enforcement of Knox County Air Quality Management Regulations. These records include compliance monitoring activities records documenting a violation, notice of violations, correspondence, meeting documents, administrative orders, penalty documentation and other related documents. These records include information created by the Department, the owners or operators of air contaminant sources, federal agencies, state agencies, local governments and the public.

Retention Instructions:

Item a: All enforcement records

- **Disposable**
- Close when case is complete.
- Destroy 10 years after file closure.

Guidance:

Media neutral – This schedule allows the disposition of the record copy in any media (media neutral). If the record is created in electronic format or digitized (e.g., imaged) and maintained electronically, the electronic records must be retrievable and usable for as long as required by the applicable retention schedule.

Sensitive information – When records are due for destruction according to the retention instructions, records containing sensitive information (e.g., confidential business information) must be shredded or otherwise definitively destroyed to protect confidentiality.

Multiple record schedules – In situations where records are covered by more than one record schedule, retain the record for the longest retention period.

Knox County Department of Air Quality Management Review: 3/3/2017

Knox County Law Department Review: 3/22/2017

TDEC Division of Air Pollution Control Review: 3/22/2017

Public Records Commission Approval: 10/12/2017

Appendix A: Record Type Guidance

Knox County Department of Air Quality Management
Records Retention Policy

Permit Records

The following records are considered permit records:

- Permit application forms
- MACT, GACT, and NSPS plans including initial notifications; notification of compliance; operation and maintenance plans; and startup, shutdown, and malfunction plans
- Correspondence regarding the permitting of a source including additional information requests; permit application completeness determinations; agreement letters; submitted requested additional information; and exempt source determinations
- Conference minutes regarding the permitting of a source
- Potential to emit calculations
- Department NNSR/PSD determinations (netting analysis, BACT, LAER, etc.)
- Statement of Basis
- Records documenting public notice
- Performance test reports (including notification, observations, and correspondences)
- Construction permits
- Operating permits

Compliance Monitoring Activity Records

The following records are considered compliance monitoring activity records:

- Inspection reports
- Partial and full compliance evaluation reports
- Complaint investigation reports
- Correspondence regarding the compliance monitoring of a source including consumption requests; record/information requests; and submitted requested records/information
- Conference minutes regarding the compliance monitoring of a source
- Actual emission calculations
- Reports/notification required by an operating permit including NO_X/VOC emission statements; emission reports; annual compliance certifications; annual/semi-annual compliance reports; annual/semi-annual monitoring reports; notifications of storage tank inspections; and notifications of malfunction excess emission

Enforcement Records

The following records are considered enforcement records:

- Documents indicating a violation occurred
- Issued Notice of Violations (NOVs)
- Issued Administrative Order and Assessment of Civil Penalty
- Issued Administrative Order and Consent Agreement
- Signed Consent Agreements
- Correspondences regarding an enforcement action including additional information submitted; conference requests; referrals to Knox County Law Director, Knox County District Attorney and/or EPA; Administrative Order appeals; and Air Pollution Control Board final order or determination
- Documents addressing a requirement of an issued Administrative Order
- Conference minutes regarding an enforcement action
- AO appeal hearing minutes

KCDAQM Records Schedule 401

Status: Approved

Title: Monitoring Records

Description:

This schedule covers records of activities related to management of the air monitoring program. These records include but are not limited to site information, QA Project Plans, Standard Operating Procedures, logbooks, exposed filters, quality assurance documents, audits, correspondence and data. These records include information created by the Department, the owners or operators of air contaminant sources, federal agencies, state agencies, local governments and the public.

Retention Instructions:

Chart 1 provides the category of record, record type and retention time. The intent of this schedule is to ensure that for all quality assured data collected all associated documents used in the collection and validation of that data are kept as long as the data is kept.

Category	Record Type	Retention time
Operational	Quality Management Plan	Until approved revision + 5 years
	Quality Assurance Project Plans	Until approved revision + 5 years
	Standard Operating Procedures	Until approved revision + 5 years
	Logbooks	5 years
	Chain of Custody	5 years
	TSP & PM10 Exposed Filters	5 years
	PM2.5 exposed filters	5 years, 1st year cold storage
Site Information	Network Plan	Until approved revision + 5 years
	Site Maps, pictures, characterizations	Until revised + 5 years
Data Management	Raw Data	5 years
	Quality Assured Data	5 years
	AQI Reports	5 years
	Summary reports, presentations	3 years
Quality Assurance	Control and strip charts	5 years
	QA reports, Data Quality Assessments	5 years
	System Audits, Internal Audits	5 years
	Reference Standard Certifications	5 years after last valid certification
	Network reviews	Until approved revision + 5 years

Chart 1

Guidance:

Statute of Limitations – This policy sets asides a minimum of 5 years for all documents. 40 CFR Part 31.42 requires 3 years from the date the grantee submits its final expenditure report. However, if any litigation, claim, negotiation, audit or other action involving the records has been started before the expiration of the 5 year period, the records must be retained until completion of the action and resolution of the issues or until the end of the regular 5 year period whichever is later.

Media neutral – This schedule allows the disposition of the record copy in any media (media neutral). If the record is created in electronic format or digitized (e.g., imaged) and maintained electronically, the electronic records must be retrievable and usable for the time period specified in Chart 1.

Sensitive information – When records are due for destruction according to the retention instructions, records containing sensitive information (e.g., confidential business information) must be shredded or otherwise definitively destroyed to protect confidentiality.

Multiple record schedules – In situations where records are covered by more than one record schedule, retain the record for the longest retention period.

References:

40 CFR 58.16
40 CFR 31.42
EPA Quality Assurance Handbook for Air Pollution Measurement Systems Vol. 2, Jan 2017
EPA Quality Assurance Guidance Document 2.12 Jan 2016

Review:

Knox County Department of Air Quality Management Review: 8/8/2017

Knox County Public Records Board: Approved 10/12/2017

Appendix B – SOP for Internal Audit of Monitors and Technical Systems

Internal Auditing and Systems Review
Standard Operating Procedure

KNOX COUNTY
TENNESSEE
HEALTH DEPARTMENT

Prepared by:
Rebecca Larocque

Knox County Air Quality
140 Dameron Avenue
Knoxville, TN 37917-6413

Identification and Approval

Title: “Internal Auditing and Systems Review Standard Operating Procedure”

The attached “Internal Auditing and Systems Review Standard Operating Procedure” is hereby recommended for approval and commits the Knox County Department of Air Quality, hence forth referred to as Air Quality, to follow the elements described within.

Air Quality

1) Signature: _____ Date: _____
   Brian Rivera, Division Director

2) Signature: _____ Date: _____
   Amber Talgo, Air Monitoring Program Manager

US Environmental Protection Agency, Region 4

3) Signature: _____ Date: _____
   Reviewing US Environmental Protection Agency Region 4 Staff

Revision History

Revision #	Change
1	Inserted Revisions Page, updated the Table of Contents and changed acceptable range for the clock on the Speciation, PM2.5 continuous (Beta 5014i) and the PM 10 continuous (TEOM 1405) changed from ±1 min to ± 5 min based upon the manufacturers recommendations.
2	Updated Ozone Audit levels to reflect latest Guidance using MDL. Made edits suggest by EPA for Document approval including standardizing terms, expanding explanations and correcting 2025 filter arrangement. Added AQS entry and Records Management Sections
3	Removed Beta and TEOM procedures, Added T640/ x procedures. Added Thermometer verification procedures. Added Site Evaluation procedures, Incorporated Quality Bulletins, Lead Auditing, Ozone Auditing, Back Pressure Compensation

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1.0 Introduction

Air Quality operates an ambient air monitoring network across Knox County, Tennessee. This network consists of continuous monitors (ozone and particulate matter) and intermittent samplers (particulate matter and lead). As an EPA funded environmental data collection agency, Air Quality must develop, implement and maintain a quality system. Air Quality is the primary quality assurance organization (PQAO) for the data collected. This Standard Operating Procedure (SOP) describes the procedures followed for assessing compliance with regulatory requirements, ensures the following of technical SOPs and establishes a quality assurance check on data collected. The goal of internal audits within the PQAO is to provide an independent quality control check on the systems. The audits performed shall be used to meet the requirements of semi-annual flow audits for particulate matter and lead, and annual performance audits for ozone.

1.1 Personnel

The procedures by which ambient air quality data are obtained, processed and validated involves the coordination of many staff. It is essential that each individual understand their responsibilities as they relate to the internal auditing procedures in order to assure quality auditing results. The following describes the key personnel involved in the internal auditing procedures and their primary job duties as they relate to internal audits.

- Agency Director
  The Agency Director is the senior administrative official accountable for the quality of all operations performed by air Quality staff. The Director reviews grants, budgets, and allocation of resources, as well as authorizing the purchasing of equipment and issuing of contracts necessary for implementation of monitoring programs. The Director shall provide support to implement changes or upgrades needed as discovered by the internal auditing procedures.

- Air Monitoring Program Manager (AMPM)
  The AMPM supervises the Air Quality monitoring team members. The AMPM is responsible for ensuring the network is operated in accordance with Federal Regulations and follows the Quality Assurance Project Plan for data acquisition, validation, and submittal. The AMPM oversees the implementation of any corrective actions necessary for discovered deficiencies during internal audits.

- Internal Auditor / Quality Assurance Officer
  The Auditor performs the internal audits of the Air Monitoring Program. The Auditor, while supervised by AMPM has direct communication and reports written results to both the Agency Director and the AMPM to ensure audits are as nonbiased as possible in a small local agency. Upon acceptance of the audit findings and documentation by the Agency Director and the AMPM the auditor enters the finding into the Air Quality Systems database (AQS).

- Operator
  The Operator is the primary person responsible for the daily operation of the instruments is accordance with the SOPs. They may assist or observe the Auditor in performing internal audits on samples. They implement any corrective actions needed as a result of deficiencies found by an internal audit.

1.2 Reference Documents

- 40 CFR Part 58 Appendix A and E
- 40 CFR Part 50 Appendix D, G, J, K, and L
- Us Environmental Protection Agency, Quality Assurance Guidance Document 2.11 and 2.12
- US Environmental Protection Agency, Quality Assurance Handbook for Air Pollution Measurements Systems, Volume II, Ambient Air Specific Methods, 2017
- Air Quality, Quality Assurance Project Plan, 2018
- Air Quality, Instrument/ pollutant specific SOPs

2.0 Process Overview

Each audit should follow a similar format. Multiple system audits may take place on the same day which may result in combining steps. This section describes the general process each internal audit will follow.

2.1 Preparing for an audit

Audits shall be performed on each active monitor quarterly. The Auditor should coordinate with the Operator for audit date and time in order to reduce data loss, but also may perform any audit unannounced. Weather should be considered as it can affect the performance of instruments during audits. The Auditor should also take into consideration the national run schedule. All reasonable actions should be taken to avoid interrupting a run and to minimize data collection loss for gaseous and continuous monitor.

The Auditor shall verify the calibrations and traceability of the auditing equipment used on the day of the audit. The serial number of auditing equipment shall be written on the audit from to indicate when and where the equipment is used.

2.2 During the audit

The Auditor should review the logbooks and documentation to verify SOP procedures are followed. The auditor shall perform the internal audit as designated in the appropriate section of this SOP. Forms may be combined for multiple checks on the same day, but in all instances should clearly indicate what monitor, traceable standard used, and location of the audit. In addition to the auditing of the specific monitoring equipment, the following system reviews shall also be completed by the auditor.

A. The Auditor shall observe the site location and physical condition of instruments at all locations. The Auditor should note any changes in siting conditions such as tree growth, new construction or other obstructions that may require further investigation for siting criteria. At least once a year, the Auditor shall complete the site evaluation form (Appendix A figures 12.6 and 12.7). See Section 9.0 of this SOP for full site evaluation procedure.

B. The Auditor shall ensure that logbooks are maintained and that logbooks are present at each site, for each instrument. The site logbooks must record the date, time and initials of the person(s) onsite, brief description of the weather, description of work accomplished, and notes on unusual evens, or anything that may impact monitoring data. Standard abbreviations may be used. The instrument logbook shall contain the verifications, maintenance, repair, and calibration history of the instrument. Each entry shall include date,

time and Operator's initials

C. Equipment calibration verifications and traceability review. The Auditor shall ensure that all audit equipment used has the proper traceability to a NIST or EPA primary standard. Additionally, audit equipment used must not be used in the calibration of the instruments to be audited. The Auditor shall also verify the min max thermometers, as well as site temperature probes and thermostat settings. See Section 10.0 of this SOP for full thermometer verification procedures.

2.3 Following an audit

The Auditor will meet with the AMPM to discuss any immediate concerns found during the audit. The AMPM and the Operator will prepare a plan of action to address any problems found during the audit. The Auditor will compile and Audit Report to be submitted to the Director. The cover letter shall include scope of audit and areas of concern. The report shall also include all calculations, field notes, and results produced during the audit. The Quarterly Audit Workbook should be used to produce the Audit Report. This workbook is an excel document containing the forms and field sheets discussed throughout this SOP. It contains formulas, conditional formatting and directions to increase uniformity of reports.

3.0 Ozone Monitoring Audit

The internal quarterly audit of the ozone analyzers shall meet the requirement for an annual performance evaluation. Table 3.1 below indicates what audit equipment shall be used and it's acceptability range.

Equipment	Recertification	Acceptable Range
Transfer Standard	Annually	0-400, low point capable
Zero Air Pump	N/A	Output pressure 20-30 psi
Zero Air Filtration	Filter/ Charcoal Replaced Annually, Silica As needed	FEP filter, 1 cylinder of charcoal, 2 cylinders of silica gel > 50% blue
Teflon tubing, connections	N/A	Caps should be same material as port
Rotometer	N/A	0-5 LPM
Form: Field Sheet Ozone	N/A	Most recent revision
Clock	N/A	Cell phone updated to towers acceptable
Pen, 5/8" wrench, stickies, Logbooks		

Table 3.1 Ozone Audit Equipment Required

3.1 Preparing for the audit

The audit transfer standard should have all ports on the back panel capped to keep out dust and debris during transport and storage. The audit transfer standard must be allowed to warm up for at least 1 hour but should warm up for 12-24 hours prior to perform the audit. Typically the Auditor should bring the audit standard to the site the day before the planned audit, plug in the standard and turn it on. There should not be any lines set up or uncapped during the warm up process.

3.2 Performing the Audit

A. Connect external zero air source (XZAS), calibration line, exhaust and vent to the audit transfer standard as detailed in figure 3.2 below.

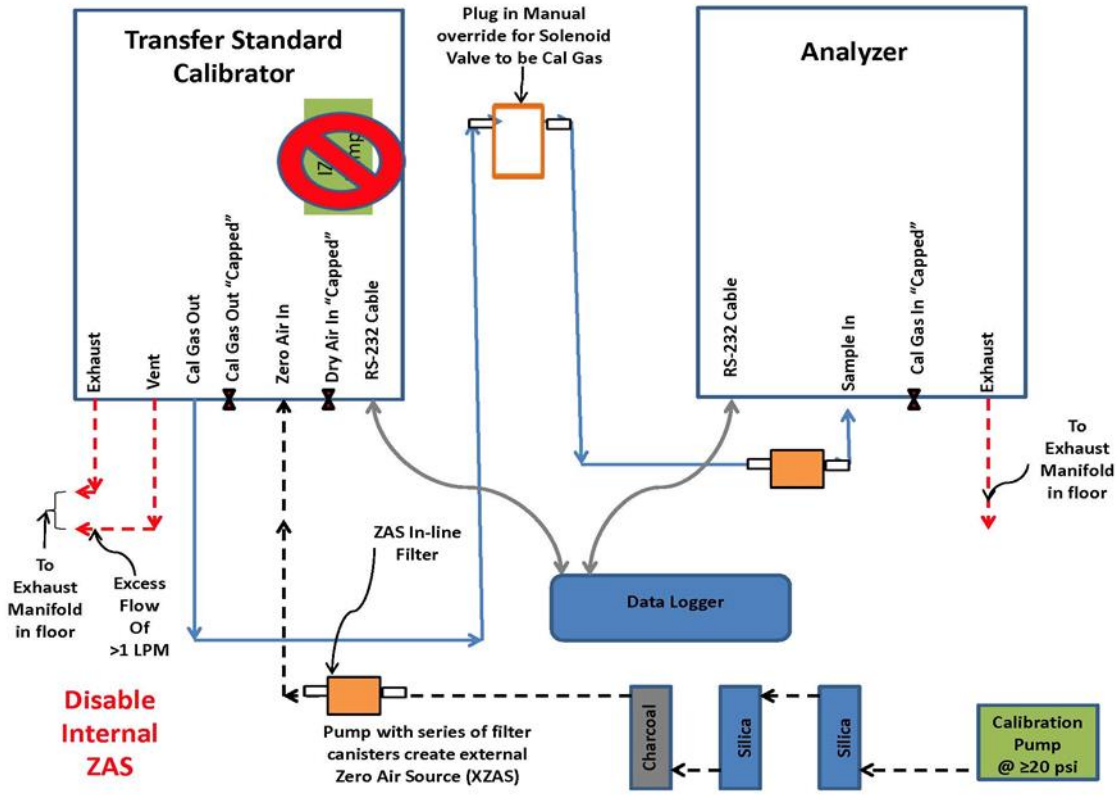


Figure 3.2 Audit Standard Set up

B. Confirm that the internal pump is disabled on the Transfer standard as follows:
   a. Select Setup, then More, then VARS
   b. Enter Password “818”
   c. Use “Next” until “ZA pump ENAB=ON” appears
   d. Select Edit, then “Off” then Enter
   e. Exit to main menu

C. Log in to the ESC8832 and disable the appropriate data logger channels {O3 and O3CAL} as follows:
   a. Press <ESC> key until the login menu screen appears
   b. Use the down arrow key to highlight “Login/Set User Level” and press <Enter>
   c. Hold down the white <Ctrl> key and type the password “nerothecat” then press<Enter>
   d. Use the arrow keys to highlight “configuration Menu” and press <Enter>
   e. Use the arrow keys to scroll down and highlight “Configure Data Channels” and press <Enter>
   f. Use the arrow keys to scroll and select “enable/Mark Channel Online” to enable valid logger channels {or Disable/Mark Channel Offline for disabling logger channels} Then press <Enter>
   g. Use the arrow keys to highlight the desired channel to Enable or Disable per previous choice and press <Enter>
   h. You will be automatically be taken back out to the Channel selection menu where you can choose another channel to mark.
   i. After all desired channels are chosen, press <ESC> to back out to the home Menu.

j. Check the status of channels by following the highlight/enter process to select through the “Real-Time Display Menu” Enabled channels will be in green, and disabled in red in each of the real-time displays.

D. Plug in the manual over ride “M” plug for the solenoid valve to activate the calibration gas line.

E. Turn on the external pump for a minimum 10 minute warm up.

F. Adjust the pump pressure to 20-30psi and record this value on the Field Sheet Ozone (Appendix A Figure 12.1).

G. While warming up the pump, generate 500 on the audit standard to clear lines of any contamination that may have been introduced during the hook up of the instrument. Use this time to also check the Calibrators Reg Pressure is set to near 15 psi and/or the Output Flow is near 5.0 LPM by using the <TST and TST> buttons. And that there is no back pressure by reading the photosPres and watching it as the internal solenoid switches that the difference of the two values is not more than 0.1 inch. Additional information on Back Pressure compensation can be found in section 9.4 of the Ozone SOP.
    a. On the Audit Standard press <TST> button until “ACT=STANDBY” appears on the display
    b. Press the <GEN> button then <AUTO> button.
    c. Toggle the front panel buttons until a concentration of “0500” appears and press the <ENTR> button.
    d. Press <TST> on audit transfer standard until output flow is displayed. This should be set as close to 5.0 LPM as possible. If the flow needs to be adjusted, open the front panel of the audit standard and turn the regulator pressure valve

H. Attach a rotameter to the vent port on the Audit Standard to verify the excess flow rate is >1.0 LPM. Record on the Field Sheet Ozone form.

I. Set up the AV Trend software to display concentrations at 10 second intervals.

J. Challenge the Analyzer with known ozone concentrations
    a. On the Audit Standard press <TST> button until “ACT=STANDBY” appears on the display
    b. Press the <GEN> button then <AUTO> button.
    c. Toggle the front panel buttons until a concentration of “0110” appears and press the <ENTR> button.
    d. After the concentration has run through the system for at least 10 minutes and the Analyzer has stabilized (Indicated by stability ≤ 1ppb) record the Audit Standard concentration, analyzer concentration, stability and times on the Field Sheet Ozone (Appendix A Figure 12.1).
    e. Repeat Steps b-d substituting the “0110” for the concentrations of 70,35,15 and final 0.
    f. While running the final zero, record the parameters from both the analyzer and audit standard by pressing the <TST> button on each to scroll through each of the parameters.
    g. After all points are taken press the <STBY> button on the Transfer Standard.

K. Unplug the “M” manual override plug for the solenoid value and replug in the “A” automated plug. This sets the analyzer to sampling ambient air again.

L. Turn off the external zero air pump and disconnect the Audit Standard and reconnect the site Transfer Standard according to figure 3.3 below.

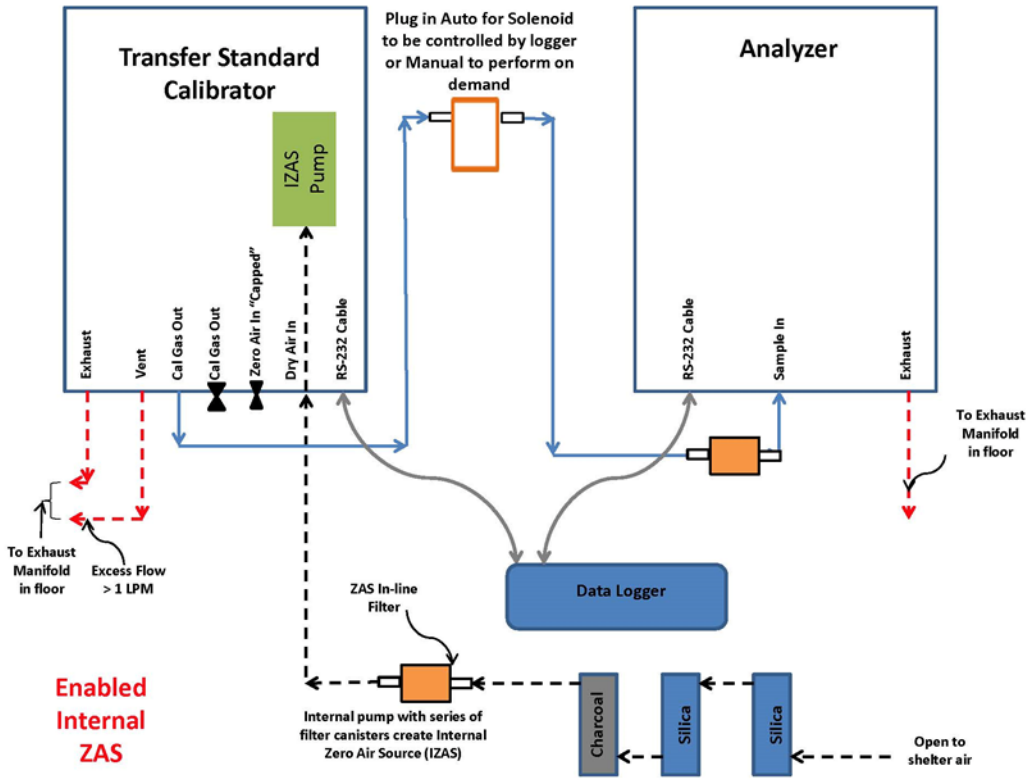


Figure 3.3 Site Transfer Standard set up

M. When the analyzer’s readings and stability indicate it is accurately sampling ambient air enable the data logger. By Repeating step C (a-j) or this section, choosing “Enable/Mark online” in f.

N. Use a sticky (Appendix B figure 13.1), or create a small chart to record the times, concentrations of audit standard and analyzer, and stability in the Analyzer logbook.

O. See section 10.0 of this SOP for procedure in verifying the shelter thermometers.

3.3 Evaluating the results

The audit is considered passing if all the measures in table 3.4 below are met:

Measure	Acceptance Criteria
Analyzer’s Zero Concentration	≤ ±5 ppb
Concentration Points	≤ 15% difference of Audit standard concentration
Slope of linear regression (M)	0.9<M<1.1
Intercept of linear regression (Y)	-5 ppb< Y< 5 ppb
Correlation of linear regression	>0,95

Table 3.4 Audit Acceptance Criteria

If the analyzer's zero concentration or any of the concentration points acceptance criteria are not met, the Auditor shall notify the Operator. The Operator should perform a calibration verification and investigate source of failure. Together with the AMPM, the Operator should prepare a Corrective Action Report. The Auditor shall enter the audit data into the Quarterly Audit Workbook Ozone Tab (Appendix C Figure 14.1). The slope, intercept, and correlation will be verified during this data entry and preparation of the audit report. The Auditor should note the date of the last passing QC activity from the analyzer logbook to add to the final report if deficiency is found. Analyzer concentrations varying by >10% are considered marginal. The Operator should perform a zero / precision/ span check with the site transfer standard and evaluate results with the AMPM and Quality Assurance Officer for further actions.

4.0 PM2.5 Intermittent Sampling Audit

The internal audit of PM2.5 intermittent samplers shall be used to meet the semi-annual flow rate audit Code of Federal Regulations requirement. Table 4.1 below indicates the equipment needed to complete the audit and its acceptability range.

Equipment	Recertification	Acceptable Range / Details
Flow rate Transfer Standard	Annually	±2% of NIST Traceable Std
Field Thermometer	Annually	±0.1°C resolution, ±0.5°C Accuracy
Field Barometer	Annually	±1mmHg resolution, ±5mmHg accuracy
Audit Magazine	N/A	With empty ring and Audit filter
Leak Check Adapter	N/A	
Gloves	N/A	Static free
Magazine Caps	N/A	1 on Audit magazine, 1 for supply magazine
Clock	N/A	Cell phone updated to towers acceptable
Form Field Sheet PM2.5	N/A	Most recent revision
Pen, stickies, logbook	N/A	

Figure 4.1 $PM_{2.5}$ Audit Equipment

4.1 Filter Handling Audit

The Auditor shall observe the filter handling procedures for both clean and exposed filters. The clean filters shall be stored in anti-static bags and handled with anti-static gloves. The work area shall be a clean surface for loading the magazines. The Operator shall enter the filter and cassette number on the field sheets and load the magazines with an organized method to ensure the filters are aligned to run on the correct dates.

The Auditor shall inspect the Air Sample Storage Logbook (refrigerator temperature log book). Each entry should be complete and it shall be up to date. The Auditor shall verify that the maximum temperature recorded from the Min/Max thermometer in the logbook has not exceeded 4°C without documentation. Exceedances shall be noted in the Audit Report.

The Auditor shall observe or question the Operator regarding the filter pick up procedure from the field as well as the shipping procedures. These should match the procedures outlined in the Thermo Model 2025 Sequential Sampler SOP. Particular attention should be paid to the cooling and transport of filters from the field, and the chain of custody/ filter handling/ temperature control during the shipping process.

4.2 Field Audit

The Auditor shall inspect the very sharp cut cyclone (VSCC) and PM_{10} head to ensure that they

are clean and properly maintained. The Auditor shall perform the field audit using Field Sheet PM2.5 (Appendix A figure 12.2), compare results to Table 4.2 below Acceptance Criteria, and enter results in the Audit Workbook for preparation of the Audit Report.

Measure	Acceptance Criteria
Time	± 1 minute
External Leak Check	< 25mmHg
Filter Temperature	±2°C
Ambient Temperature	±2°C
Ambient Pressure	±10mmHg
Flow Rate	±4 % of transfer standard

Figure 4.2 $PM_{2.5}$ Intermittent Acceptance Criteria

A. Check the system time and compare to audit clock. If outside acceptable limit notate and continue with audit. Operator should update clock as soon as possible following a deficiency
    a. Press <F5> Setup
    b. Press <F5> System
    c. Read time Current Time:
    d. Press <ESC> back out to the main screen.

B. Place the monitor in Audit Mode. From the wait or stop mode, press <Run/Stop> once to enter the Audit Mode. The unit will display the Audit Confirmation Screen. Press <F1:Audit> and then <Menu> twice, then select <F1:Audit> Then press <Enter>.

C. Remove storage magazine, cap and set aside.

D. Remove supply magazine and place on storage side.

E. Install audit magazine on the supply side (Audit magazine consists of a filter cassette with filter and an empty ring on top) Filters used for flow rate audits shall not be used for further sampling but may be used in future audits if undamaged.

F. Advance the filter – Press <F4:Filter Advance> to advance the empty ring cassette into the sampling position allowing the filter that was in place to be in the top most position of the supply magazine. Remove and Re-Cap the Supply magazine.

G. Remove the PM10 head by pulling straight up on the sample tube. Check the cleanliness of the PM10 separator.

H. Perform an external leak check.

    a. Install the flow audit adapter on the sample down tube and close the valve on the flow audit adapter.
    b. Press <F5: Leak CHK>, then <F2:Start>, and follow the instructions displayed on the screen.
    c. A pass or fail message will be displayed along with a reading in mmHg. Record this on the Field Sheet PM2.5 form.
    d. If the leak check passed, proceed to step I.
    e. If the leak check failed, check the flow adapter valve to ensure seated on the sample tube. You may check the empty ring was not upside down, or advance the audit filter into place (See step N) Repeat test.
    f. After second failure the audit fails, but continue on to step I to complete the audit.

I. Unlatch and open the sampler's top cover. Pull straight up to remove the VSCC. Check the cleanliness of the VSCC.

J. Turn on streamline pro (or other Audit Standard and allow to equilibrate and zero.) Make sure the temperature is stable and the flow temperature and remote temperature are within 2 degrees of each other. The thermal shield is almost always necessary to ensure the flow temperature does not overheat.

K. Verify the sampler's ambient temperature by measuring the current temperature at the ambient temperature sensor using a calibrated reference thermometer. Place the Remote Temperature Probe within the instrument's radiation shield and allow it to equilibrate. Compare the temperature from the audit standard to the average ambient temperature displayed on the Audit Screen. If the two readings are not within ± 2°C the ambient temperature audit has failed. Record all data on Field sheet PM2.5. Continue to perform the audit, be sure to notify the AMPM and Operator of failure as well as include in the Audit Report.

L. Verify the sampler's filter temperature by inserting the calibrated reference thermometer into the sampling chamber. The empty ring should still be in place, if the audit filter was shifted into place during 4.2 (H)(e) above, the auditor shall, replace the empty ring into the cassette on the supply side and advance the empty ring back into sampling position and allow to equilibrate. Compare the measured temperature (° C) with the filter temperature displayed on the Audit screen. If the two readings are not within ± 2°C the filter temperature audit has failed. Record all data on Field sheet PM2.5. Continue to perform the audit, be sure to notify the AMPM and Operator of failure as well as include in the Audit Report.

M. Verify the sampler's ambient pressure by measuring the current ambient pressure in mmHg with a calibrated reference barometer. Compare this value to the ambient pressure displayed on the Audit screen. If the displayed barometric pressure readings are not within ±10 mmHg from the audit standard's barometric pressure reading, the ambient pressure audit has failed. Record all data on Field sheet PM2.5. Continue to perform the audit, be sure to notify the AMPM and Operator of failure as well as include in the Audit Report.

N. Reinstall the VSCC & lower and re-latch the top cover.

O. Advance Filter – Press <F4: filter Advance> to advance the audit filter cassette into the sampling position. Catch the empty ring as it advances to the storage side and set aside. * If the monitor was not running at the start of the audit, the pump must warm up for 10-15 minutes before completing the flow rate audit.

P. Perform an external leak check.
   a. Install the flow audit adapter on the sample down tube and close the valve on the flow audit adapter.
   b. Press <F5: Leak CHK>, then <F2:Start>, and follow the instructions displayed on the screen.
   c. A pass or fail message will be displayed along with a reading in mmHg. Record this on the Field Sheet PM2.5 form.
   d. If the leak check passed, proceed to step P.
   e. If the leak check failed, check the flow adapter valve to ensure seated on the sample tube, the VSCC and the cover of the instrument. Repeat test.
   f. After second failure the audit fails but continue on to step T to complete the audit.

Q. Place the flow transfer standard on the monitor's inlet. Confirm that 16.7 LPM is the set flow in the Set Flow field. Press <F1:Pump> to initiate the pump for warm up. If necessary wait 10-15 minutes for pump to warm up.

R. Pres <F2:Valve> to open the flow valve, allow time for the audit standard and monitor to stabilize.

S. Record the displayed value on Cur Flow and the audit flow standard. If the percent difference between the sampler flow and the audit standard flow are within ±4% the results are acceptable. If not, the flow audit has failed. Record all results on the Field Sheet PM2.5. Notify the AMPM and Operator of failure as well as include in the Audit Report. Notate the last passing verification indicated in the logbook for the Audit Report.

T. Remove the audit magazine from the supply position and place it on the storage side. Re-install the supply magazine, verifying visually that the previously removed sampling filter cassette is in the top most position. Connect the air hose. Press <F4: Filt Adv> to move the cassette into sampling position. The audit filter will advance to the audit magazine. Remove the audit magazine, place the empty ring back on top and install the original storage magazine (from 4.2.C) back onto the storage side. Cap the audit magazine for use at next location.

U. Utilize the sticky form (Appendix B figure 13.2) to record the audit results into the instrument logbook.

4.3 Evaluation of Results

If a value is outside the acceptance criteria illustrated in table 4.2, the Auditor shall make note of the last successful verification from the instrument logbook. The Operator and AMPM shall be notified and decide on the corrective action. The Auditor shall review the Field Sheet PM2.5 (Appendix A Figure 12.2) for completeness for each sampler. The data shall be entered into the Quarterly Audit Workbook (Appendix C figure 14.2). The Auditor shall prepare a summary of deficiencies including suggested corrective action, or passing but borderline results that may require intervention to avoid future failure.

5.0 PM Continuous Monitoring Audit

Use form field sheet continuous (Appendix A Figure 12.3). Audit equipment to be used and certification acceptability are outlined in Table 5.1 below. Audit equipment shall be separate from equipment used for verifications and calibrations.

Equipment	Recertification	Acceptable Range
Flow rate Transfer Std	1/yr	±2% of NIST Traceable Std
Field Thermometer	1/yr	±0.1°C resolution, ±0.5°C Accuracy
Field Barometer	1/yr	±1mmHg resolution, ±5mmHg accuracy
Leak Check Adapter	N/A	Pm2.5 leak check adapter with HEPA filter
Clock	N/A	Cell phone updated to towers acceptable
Field Sheet PM, misc	N/A	Logbook sticky, pen,

Figure 5.1 Pm Continuous Audit Equipment

5.1 Field Check

The Auditor shall inspect the PM10 inlet to ensure it is clean and properly maintained. The Auditor shall perform the following quality control measures and then ensure the results are within the corresponding limits according to table 5.2.

Measure	Acceptance Criteria
Time	±1 minute
Shelter Temperature	±2°C
Ambient Temperature	±2°C
Ambient Pressure	± 10mmHg
Sample Flow Rate	±4% of transfer standard
Total Flow Rate	±4% of transfer standard

Table 5.2 PM Continuous Acceptance Criteria

The internal components of the T640x are not meant to be under vacuum. DO not close off a leak check adapter or cap the inlet while the pumps are running. The zero test checks for leaks without pulling a vacuum. The theory behind the test is that a HEPA filter is installed on the inlet, if the machine does not reach zero, then a leak must be present allowing particulate matter to reach the optical eye.

A. Time should be compared to audit clock and recorded. If outside limits notate and continue with audit. Operator should update clock after audit is completed.

B. Disable Data Logger: Press <ESC> 1-2 times for menu to appear. Press <Control L> to login in. Press and hold white control key and input the password “nerothecat”. Press enter. Press <Control C> <Enter> for configuration. Press <Control D> <Enter>for Data Channels. Press <Control M> <Enter>for disable/mark channel offline. Select channel and press <Enter> Repeat selection and enter for each channel. Channels to disable are PM2.5 and PM10 STP. <ESC> back to home menu. Press <Control D> <Enter> for Real Time Display followed by<Control L><Enter> for Large Text display. Press <Enter>. Reading should be visible and in red.

C. Remove the inlet head and fit the HEPA filter (attached to a PM2.5 Leak check adapter) on the sample port. Be sure the valve on the leak check adapter is open and the tubing is not kinked, to avoid forming a vacuum.

D. From the Home Screen select > Calibration> Leak Check> Start. See Figure 5.3 below.

Figure 5.3 Leak Check Screen

E. While allowing the leak check to run for at least 5 minutes, Turn on streamline pro (or other transfer standard and allow to equilibrate and zero. Make sure the temperature is stable and the flow and remote temperatures are within 2 degrees each other.

F. Set the streamline pro inside the T640/ T640X shelter and the temperature probe near the shelter temperature thermometer. After stabilized, record Serial Number (SN) for the thermometer, the shelter temperature and the reference temperature on the field sheet.

G. Observe the PM values and record the values after 5 minutes on the Field Sheet SP. 0.0-0.2 is passing, however anything over 0.0 should be noted to the Operator for review.

H. Place the flow transfer standard on the inlet. The remote temperature probe should be placed in the radiation shield of the monitor's ambient temperature probe.

I. From the dashboard record the system ambient pressure in mmHg and from the pressure standard record the reference Amb Pres on the Field Sheet SP. Calculate the difference Monitor – Reference. The values must differ by less than ±10mmHg to pass.

J. From the dashboard record the system ambient temperature in degrees Celsius and from the temperature standard record the remote temperature on the Field Sheet SP. Calculate the difference Monitor- Reference. The values must differ by less than 2 degrees Celsius.

K. From the dashboard (use arrow key to see page 2) record the system total flow in L/min and from the flow standard record the flow. Be sure to use the remote temperature for calculating flow on the transfer standard. Record both values (system and reference) on the Field Sheet SP under Total Flow and calculate the percent difference [(Monitor- Reference)/ Reference] x 100%. Total flow should be within ± 4%

L. Disconnect the bypass flow line from the side of the ASC. Ensure the line is not blocked and only pulling in ambient air. Be especially careful to keep it from setting in water or dirty surface.

M. Cap the Swagelok fitting at the side of the ASC. The Flow standard remains on top of the inlet. From the dashboard (page1) record the system main flow in L/min and from the flow standard record the flow on the Field Sheet SP. Expected main flow is 5L/min. Calculate

the percent difference [(Monitor- Reference)/ Reference] x 100%. Main Flow should be within ±4%.

N. Record the results in the instruments logbook using the sticky form (Appendix B figure 13.3).

O. Allow 10 minutes for the machine to stabilize and then re-enable the logger. Press <ESC> 1-2 times for menu to appear. Press <Control L> to login in. Press and hold white control key and input the password “nerothecat”. Press enter. Press <Control C> <Enter> for configuration. Press <Control D> <Enter>for Data Channels. Press <Control E> <Enter>for Enable/mark channel online. Select channel and press <Enter> Repeat selection and enter for each channel. <ESC> back to home menu. Press <Control D> <Enter> for Real Time Display followed by<Control L><Enter> for Large Text display. Press <Enter>. Reading should be visible and in green.

5.2 Evaluating the results

If a parameter value is outside the acceptance criteria, the Auditor shall make note of the last successful verification from the instrument log book. The Operator shall investigate the cause of the deficiency and follow current SOP procedures for proper corrective action. All information will be forwarded to the AMPM for evaluation

The Auditor shall review field sheet PM SP for completeness for each sampler. The data shall be entered into the Quarterly Audit Workbook on PM Continuous Sheet (See Appendix C Figure 14.3). The Auditor prepares a summary of the audit including any suggested corrective action. The summary should include any borderline results which required Operator intervention to correct onsite but must include any results outside the acceptance criteria.

6.0 Lead Monitoring Audit

Use form field sheet Lead (Appendix A Figure 12.4). Audit equipment to be used and certification acceptability are outlined in Table 6.1 below. Audit equipment shall be separate from equipment used for verifications and calibrations. This SOP details how to use the Hi Vol Cal flow rate transfer standard. In the event that a Hi Vol Cal in unavailable, Auditor shall follow the instructions in the Lead SOP section 3.5.2 for Single point flow rate check 2 manometer procedure, using a certified orifice transfer standard not used for calibrations or QC checks by the Operator.

Equipment	Recertification	Acceptable Range
Flow Rate Standard	1/yr	±2% of NIST Traceable Std
Field Thermometer	1/yr	±0.1°C resolution, ±0.5°C Accuracy
Field Barometer	1/yr	±1mmHg resolution, ±5mmHg accuracy
Field Manometer	1/Yr	±0.1inH2O resolution, ±0.5inH20 accuracy
Orifice Plate	N/A	Clean, seals in place
Clock	N/A	Cell phone updated to towers acceptable
Audit Filter	N/A	No holes or damage, Not used for sampling
Field Sheet Lead, misc	N/A	Logbook sticky, pen,

Table 6.1 Lead Audit Equipment

6.1 Performing the Audit

The Auditor shall perform the following quality control measures and then ensure the results are within the corresponding limits according to table 6.2.

Note: Do not attempt to audit Hi Vol samplers under windy conditions. The audit will be less precise because of pressure variations.

A. Install audit filter ID number facing down without a cassette. (Operator should remove any exposed filter cassettes according to the removal procedure in the Lead SOP)

B. Install the orifice plate on the sampler. Tighten the faceplate nuts evenly on alternate corners to properly align and uniformly seat the gasket. The nuts should be hand-tightened only as too much compression can damage the gasket.

C. Turn on the sampler for 15 minute warm up and verify time.

    a. On the Timer press <ESC> until on the main menu. Compare the timer clock in top right corner with cell phone clock. Record on field sheet Lead.
    b. Then choose Setup – then Diagnostics
    c. Choose Motor and press <Enter> to start the motor

D. Attach a field manometer to the stagnation pressure port. Verify that readings are typical for unexposed filter (17-24). Record this value under leak check on the Field Sheet Lead.

E. Additional auditory leak check should be performed (listen for a leak), using a clipboard to cover the orifice plate and listen for “whistling” sound of leaks. Do not cover the plate for more than 10 seconds at a time, to avoid damaging the motor.

F. Turn on the audit Hi Vol Cal and allow it to zero itself. The audit device will read Qa and Qs as {Under} when ready to use.

G. Install the Hi Vol Cal by screwing it on to the orifice plate. The instrument hood may be used to shield the sun on particularly hot days but keep a close eye on temperature stability. The flow readings, and orifice flow curve are based upon temperature.

H. Record the barometric pressure {BP} and the ambient temperature {Ta} from the Hi Vol Cal, and the stagnation pressure from the manometer onto the Field sheet Lead.

I. Record 10 Qa readings taken at 5 second intervals on the Field Sheet Lead.

J. Turn off the motor from the timer by pressing <Enter> as the Motor screen should still be chosen. You may leave the instrument on this screen, as the Operator will have to run motor before next run cycle. It should be noted that the sampler will not perform a scheduled run if left in diagnostic mode.

K. Turn off audit device and disassemble audit apparatus.

L. Inspect the log book for required maintenance including number of runs between motor changing, and regularly cleaning of surfaces.

M. Back at the office, enter the data into the Quarterly Audit workbook. (Appendix C Figure

14.4) The spreadsheet will calculate percent difference by
   a. Averaging the 10 readings,
   b. Converting from CFM to m3/min, calculating Po/Pa value for the look up table.
   c. Auditor shall use the orifice look up tables to find the corresponding Flow rate (Qm)
   d. Calculating percent difference (Qm – Qa)/Qa *100%

6.2 Evaluating the results

Measure	Acceptance Criteria
Time	±20 minutes
Flow Rate: Orifice Look up Table Flow	±7.1% of transfer standard measured flow
Flow Rate Transfer Standard Measured Flow	±10 of design flow

Table 6.2 Lead Acceptance Criteria

The auditor shall review the Field Sheet PM form for each of the samplers for completeness. Enter the data in the Audit Workbook Lead page (Appendix C 14.4) Percent difference must be ≤ ± 7.1%. If % difference is ≥ ± 6 %, notify the Operator for adjustments. The Auditor prepares a summary of audit findings including any suggested corrective action. The summary should include any borderline results which required Operator intervention to correct onsite, but must include any results outside the acceptance criteria.

7.0 Speciation Audit

Prior to conducting the audit, ensure that it is not a scheduled sample run day. All reasonable actions should be taken to avoid interrupting a run cycle. Use form field sheet SP (Appendix Figure 12.5). Audit equipment to be used and certification acceptability are outlined in Table 7.1 below. Audit standards shall be separate from equipment used for verifications and calibrations.

Equipment	Recertification	Acceptable Range
Flow Rate Standard	1/yr	±2% of NIST Traceable Std
Field Thermometer	1/yr	±0.1°C resolution, ±0.5°C Accuracy
Field Barometer	1/yr	±1mmHg resolution, ±5mmHg accuracy
Clock	N/A	Cell phone updated to towers acceptable
Red (URG) & Yellow (SASS) Caps	N/A	To plug sampling cassettes
Leak Check Adapter	N/A	URG only
Audit Filter Cartridge	N/A	URG only
Field Sheet Lead, misc	N/A	Logbook sticky, pen,

Table 7.1 Speciation Audit Equipment

7.1 URG 3000N Audit

The following procedure details the audit using a Streamline Pro Standard for Flow rate, temperature and barometric pressure. Other certified standards meeting the criteria indicated in Figure 7.1 above may be used, with the audit instrument set up varying, but URG sampler procedures remaining the same as below.

A. Assemble the audit standard
   a. Attach the external temperature probe to the streamline pro.

b. Attach tygon tubing to the flow adapter and secure adapter in the streamline pro.
c. Place flow meter in the thermal shield.
d. Turn on device and allow to equilibrate. DO not place in direct sunlight.

B. Install the Audit Cartridge
   a. Open the Sample Module door and raise the solenoid manifold by pressing the red <↑> up button on the electronic box.
   b. Remove the filter cartridge (if present) from the cassette manifold, place red caps on each of the four filter cassette inlets, then place in plastic bag for safety.
   c. Remove red caps from the Audit cartridge and place the cartridge on the cassette manifold.
   d. Lower the solenoid manifold by pressing and holding the red <↓> down button. Release the button when the solenoid manifold has stopped moving and the filter cassettes are enclosed in the cassette manifold.

C. Place the sampler in AUDIT mode
   a. From the Auto mode press <Enter> then enter code <1123>
   b. Choose <3> for QA, then press <F4> for more.
   c. Choose <F3> for Audit.

D. Perform a leak check
   a. Press <F1> to choose leak check
   b. Remove ambient air inlet cap.
   c. Install leak check adapter with valve open, Press <Enter> when complete.
   d. Ensure pump valve on the pump enclosure box is open. Open is horizontal.
   e. Press the <Enter> key to continue with the leak check. The screen will display that both valves are open and the pump is warming up. The warm up will display a countdown time to ensure proper pump warm up.
   f. Press the <Enter> key to continue to the next screen. This will instruct the auditor to close the leak check adapter at the top of the down tube.
   g. Rotate the lever on the leak check adapter 90° to close. This will begin creating a vacuum in the downtube. The pump will sound a little louder.
   h. Press <Enter> to continue to the next screen. The vacuum will begin to increase. When the pressure reaches around 680mmHg, the time will begin to count down from 15 to 0.
   i. After the countdown reaches zero press <Enter> to continue to the next screen. Promptly rotate the lever on the pump shutoff valve 90° to close the valve.
   j. Press the <Enter> to begin the leak check. The pump will stop automatically. The vacuum will begin to drop. When the vacuum pressure reaches 380mmHg, a timer will count for a maximum of 35 seconds.
   k. After the countdown is complete the results will be displayed and indicate Passed or Failed. The acceptance criterion is a vacuum drop of less than 225 mmHg in 35 seconds. The timer will stop if a leak is large enough for the pressure drop to exceed the passing rate.
   l. Record the pressure drop in mmHg in the URG leak check box of Field Sheet SP (see Appendix A Figure 12.5) and circle the appropriate pass / fail option.
   m. Press the <Enter> key to advance to the next screen. Slowly release the pressure in the sampler by first slowly turning the lever on the leak check adapter. Releasing the vacuum too quickly may rupture or dislodge the audit filter. Then open the pump valve.
   n. Do not remove the flow audit adapter as instructed on the menu, but do save audit results to memory card. Press <Yes> and then after confirmation of save press

<Enter> to return to the Audit Menu.

o. If the leak check passed proceed to 7.1.E below Flow Audit. If leak check failed, reseat the audit filter cartridge and repeat leak check. If leak check fails a second time discontinue audit, notify Operator and AMPM.

E. Flow Audit
   a. Connect the tubing from 7.1.A.b to the leak check adapter still on the downtube after performing the leak test.
   b. From the Audit menu press <F2> for flow rate audit screen. After warning prompts press <Yes> to continue to the flow verification audit.
   c. Verify connection of flow audit adapter, leak check adapter and streamline pro. Press <Enter> the pump will initiate and run for 5 minutes.
   d. After the warm up is complete the display will indicate the samplers flow rate and vacuum. Use the key pad to enter the streamline pro's flow rate value. Enter the reference flow rate on the Field Sheet –SP form.
   e. The sampler will display the sampler's flow rate; enter the sampler's flow rate on the Field Sheet SP form.
   f. Calculate % difference (Sampler – reference) / Reference * 100% and enter on the Field Sheet SP form.
   g. Press <Enter> and then upon prompt <Yes> to save audit results. After confirmation of save, press <Enter> to return to Audit menu.

F. Temperature Audit
   a. At the base of the inlet tee, locate the temperature probe. While holding the probe cable, gently push the temperature probe through the bottom of the sample module. Seating the probe inside away from direct sunlight.
   b. After time for temperature to stabilize. Press <F3> on the Audit menu and enter the streamline pro's remote temperature value in degrees Celsius. The <F1> key toggles negative and positive, and the <F2> key toggles between Celsius and Fahrenheit.
   c. Record the sampler and the streamline audit value on the Field Sheet SP form. Calculate the difference by sampler – reference value. Values should be within ±2°C.
   d. Press<Enter> through the prompts and select <Yes> to save the audit.
   e. Press <Enter> to return to the Audit menu screen.
   f. Failed audit shall be notated and Operator notified, but continue to proceed with the rest of the audit.

G. Barometric Pressure Audit
   a. Select <F4> from the audit menu.
   b. Enter reference the barometric pressure in mmHg using the keypad from the streamline pro.
   c. After entering the reference pressure, the sampler will display both pressures and the difference.
   d. Record the sampler and reference pressure on the Field Sheet SP form. Calculate the difference Sampler – reference. Pressure should be within ± 10mmHg.
   e. Press <Enter> followed by <Yes> to save the Audit results.

H. Return to Auto Mode
   a. Open the Sample Module door and raise the solenoid manifold by pressing the red < ↑> up button the electronic box.
   b. Remove the audit cartridge on the cassette manifold, retrieve the filter cassette and remove the red caps from the bottom of the four filter inlets. Then place the sample filter cassette in the manifold (if present in part B.b. above) Cover the Audit filter

cassette inlets with red caps.
c. Lower the solenoid manifold by pressing and holding the red <↓> down button. Release the button when the solenoid manifold has stopped moving to close the filter cassettes against the cassette manifold.
d. Press <Enter>twice to return to Auto mode screen.

I. Enter the results in the instrument logbook. Utilize the sticky form (Appendix B figure 13.4)

7.2 Met One A Super SASS Audit

The following procedure details the audit using a Streamline Pro Standard for Flow rate, temperature and barometric pressure. Other certified standards meeting the criteria indicated in Figure 7.1 above may be used, with the audit instrument set up varying, but SASS sampler procedures remaining the same as below.

Auditor should perform the checks in the order presented below.

A. Clock Check
   a. From the Main Menu scree, select the Setup Menu and press <F3>.
   b. Record the sampler time as well as the audit clock time on the Field Sheet SP
   c. Press<Exit>twice to exit menu back to the main menu.

B. Leak Check
   a. Lower the weather shield on the sampler from the ready position. (Always good to give it a tap or two to check for wasps before dropping it down.)
   b. From the Main Menu select Calibration Menu, then press <F1> to select system Test.
   c. Press <Pump> button to turn on the pump. When the pump key has been chosen, a warning will come up and ask if you want to continue or cancel the run. Press <Continue> so the pump will start.
   d. Press <Leak> to conduct the leak check test.
   e. Using your finger, tightly seal the inlets of the sharp cut cyclones so that no air can pass up through the nozzles.
   f. Within a few seconds, the flow rate indicated should be 0.0 L/min but it is acceptable up to 0.10 L/min.
   g. Slowly and gently break the seal of your finger from the inlet. Record the leak check for each of the channels in use. (Channels 1 & 2) on the Field Sheet SP.
   h. Note leave the pump running for warm up for the flow check later. Exiting the System Test Screen will stop the pump.

C. Barometric Pressure Audit
   a. On the system Test Menu from the leak check, note the barometric pressure on the upper right hand corner of the screen.
   b. Record the ambient barometric pressure reading and the reference pressure from the Streamline Pro on the Field Sheet SP
   c. Calculate the difference Sampler – reference. Pressure should be within ± 10mmHg.

D. Ambient Temperature Audit
   a. Insert the reference temperature probe into the solar shield of the SASS ambient temperature sensor. Avoid contact with the side and any direct sunlight
   b. Allow the temperature reading to stabilize. Record the SASS ambient temperature

reading from the System Test Screen and the Streamline Pro on the Field Sheet SP.
c. Calculate the difference; Sampler – Reference. Values should be within ±2°C.

E. Flow Audit
a. With the temperature probe still in the solar shield continue to the Flow Audit.
b. Ensure the pump has had at least 5 minutes to warm up. It should still be running from the leak check procedure.
c. Using the tygon tubing, flow rate adapter and streamline pro, attach the tubing to the sharp cut cyclone inlet on the #1 module.
d. Allow the flow to stabilize and record the displayed SASS sample flow rate for channel #1 and the streamline pro reference flow rate on the Field Sheet SP.
e. Without stopping the pump, repeat steps b & c for channel 2.
f. Upon completion of the flow check on each channel, Press <Pump> button to turn off the pump. When the pump key has been chosen, a warning will come up and ask if you want to continue or cancel. Press <Continue> so the pump turns off.
g. Calculate percent difference for each channel. (Sampler – Reference) / Reference *100%. Values >10% fail, values 7-10% are marginal notify Operator.

F. Filter Temperature Audit
a. Remove and cap each of the sampling modules and sharp cut cyclones.
b. Use this time to look at cyclones and ensure that the Operator is keeping them clean.
c. Using the yellow cap with a hole in the center, place the cap in the Channel 1 sampling port and place the temperature probe in the center hole.
d. Allow a couple of minutes for the temperatures to stabilize.
e. Record the filter temperature for channel 1 from the System Test Screen and the reference temperature from the Streamline Pro. Repeat all steps for Channel 2.

G. Completing the Audit
a. Return sampler to main menu by pressing <Exit> until main menu appears.
b. Ensure routine filter canisters are in their original sampling positions.
c. Return the weather shield to the ready position, using the locking pin to secure in place.
d. Return all auditing equipment to their cases.
e. Enter the results in the instrument logbook. Utilize the sticky form (Appendix B figure 13.5)

7.3 Evaluation of Results

The Auditor shall review the Field Sheet – SP for completeness and enter values into the Audit workbook (Appendix C figure 14.5). The Auditor shall review the results of each measure and compare to the acceptance criteria in table 7.2 below. The Auditor prepares a summary of the audit including any suggested corrective action in the audit report. The summary should include any borderline results which require Operator intervention to correct onsite, but must include any results outside the acceptance criteria.

Measure	Acceptance Criteria
Time	±5 min
Leak Test – URG	≤225 mmHg pressure drop
Leak Test – SASS	≤0.10 L/ min

Flow Test	± 10%
Ambient Temperature	± 2°C
Filter Temperature	± 2°C
Barometric Pressure	± 10 mmHg

Table 7.2 Speciation Acceptance Criteria

8.0 Reporting Audit Results

Communicating effectively the results of the audit is essential for maintaining a quality system. Results must be reported to Air Quality Management team members (AMPM and Division Director), and EPA.

8.1 Audit Report to Air Quality Management Team Members

The Audit workbook is used to facilitate this documentation. The Auditor shall use the Field sheets provided in the audit workbook, to record the audit results on the individual sampler and pollutant specific forms. Those forms are conditionally formatted to indicate any failing results in red, and marginal flow results in yellow.

The audit forms and field sheets shall be compiled together with a cover letter (see Appendix C figure 14.6 for sample). This report shall be sent to the Division Director and AMPM. A copy shall be saved on the shared drive under the appropriate year in the Internal Audits subfolder of the Audits folder.

After management has had time to review the document, and address any questions or concerns with the Auditor, the Auditor shall proceed to section 8.2 and enter the results into AQS for reporting to EPA.

8.2 Entering Results into AQS

AQS is the data management system maintained by the US Environmental Protection Agency (EPA). Any Auditor utilizing these instructions must have user name, approved access and basic training provided by EPA on the database usage. AQS is accessed through the internet at https://www.epa.gov/aqs Tables 8.1 and 8.2 below provide a quick reference for codes used within AQS for data entry. Additional codes may be necessary and are defined in the AQS system.

Table 8.1 AQS Site Identifications

Site Name	ID
Ameristeel	0023
Air Lab	1013
Bearden	0028
Burnside	0027
East Knox	0021
Rule	1017
Springhill	**1020**

Table 8.2 AQS Parameter, Method and Unit Codes

Pollutant	Parameter Code	Method Code	Unit code
Lead	14129	201	119 cuft/min
PM 2.5	88101	145 (2025)	118 L/min
PM2.5	88101	238 (T640)	118 L/Min
PM 10	88102	239	118 L/Min
Ozone	44201	087	007 ppm

Note: Be sure to verify that the units for the reference device and the monitoring instrument are the same, and match the unit entry in AQS. Different units will need to be converted to the correct units of measure. It is recommended that you save after each line entered. AQS can have connectivity issues. Frequent saving of data entry reduces the potential data lost.

8.2.1 Entering PM Audits

A. After logging into the AQS application choose the following:
Maintain → QA Assessments → Semi-Annual Flow Rate Audits (see Figure 8.3 for illustrated pathway)

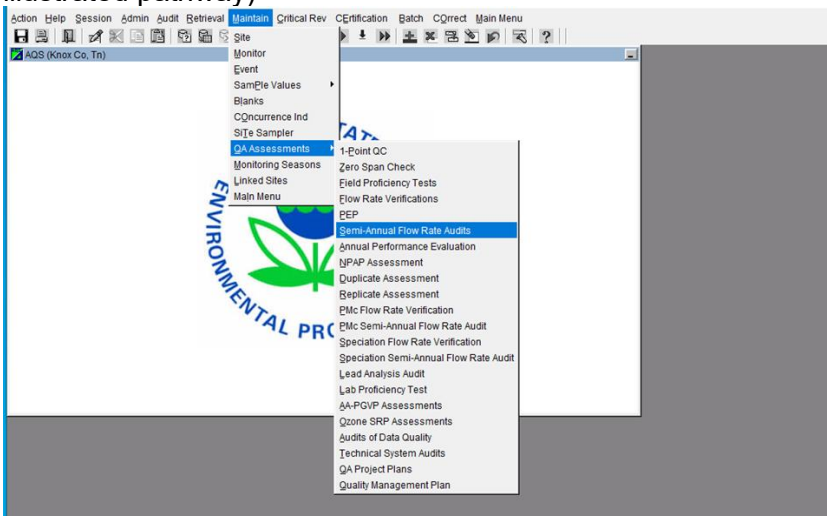


Figure 8.3 AQS PM Audit Pathway

B. Enter data as follows for the flow rate verifications (See Figure 8.4 for sample entries)
State Code: 47
County Code:093
Site ID: See Table 8.1
Parameter Code: See Table 8.2, this is related to the pollutant
POC: 1 for official monitors, 2 for collocated monitors, 3 for PM2.5 continuous, 4 for PM10 Continuous
Performing Agency: 0581
Assessment Date: Date of Audit in format YYYYMMDD
Assessment Number:1 unless repeated on same day, or secondary flow (ie main flow, total flow) then 2,3.
Method Code: See Table 8.2, this is related to the monitoring instrument
Unit: See Table 8.2 for units, this is related to instrument and/ or audit device units of measure.
Assessment Flow Rate: Enter the flow rate provided by the audit flow transfer standard
Monitor flow rate: Enter the flow rate provided by the monitor

Semi-Annual Flow Rate Audits

State Code	County Code	Site ID	Parameter Code	Poc	Perf. Agency Code	Assess. Date	Assess. Number	Monitor Method Code	Unit	Assessment Flow Rate	Monitor Flow Rate
47	093	0023	14129	1	0581	20191106	1	193	119	1.142	1.15
47	093	0027	14129	1	0581	20191106	1	193	119	1.127	1.134
47	093	0027	14129	2	0581	20191106	1	193	119	1.133	1.141
47	093	0028	88101	1	0581	20191106	1	145	118	17.05	16.69
47	093	0028	88101	2	0581	20191106	1	145	118	16.45	16.61
47	093	1013	81102	3	0581	20191107	1	079	118	16.66	16.67
47	093	1013	81102	3	0581	20191107	2	079	118	2.97	3
47	093	1013	81102	4	0581	20191107	1	239	118	16.6	16.6
47	093	1013	81102	4	0581	20191107	2	239	118	4.92	5

Figure 8.4 Sample Semi- Annual Flow Rate Entries

8.2.2 Entering Chemical Speciation Audits

A. After logging into the AQS application choose the following:
Maintain → QA Assessments → Speciation Semi-Annual Flow Rate Audits (see Figure 8.5 for illustrated pathway)

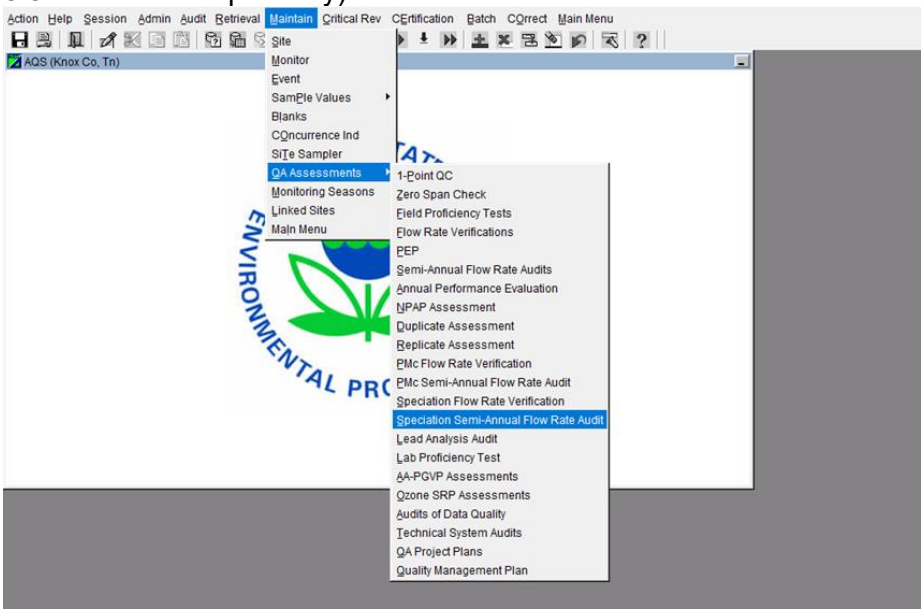


Figure 8.5 AQS Speciation Pathway

B. Enter data as follows for the flow rate verifications (See Figure 8.6 for sample entries)
State Code: 47
County Code:093
Site ID: 1020
Sampler ID: MET-ONE-A (for super SASS) or URG-A
Channel: Specify 1 or 2 for MET-ONE-A, always use 1 for URG-A
Performing Agency: 0581
Assessment Date: Date of Audit in format YYYYMMDD
Assessment Number:1 unless repeated on same day, then 2,3.
Unit: 118 L/min
Assessment Flow Rate: Enter the flow rate provided by the audit flow transfer standard
Monitor flow rate: Enter the flow rate provided by the monitor

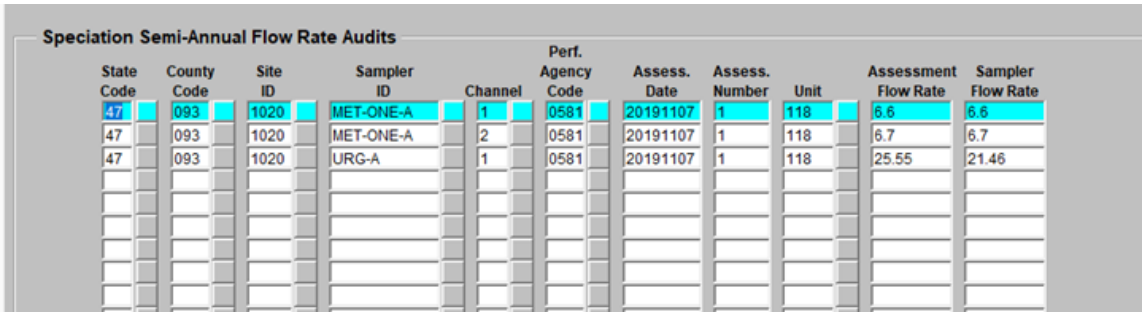


Figure 8.6 AQS Speciation sample entry

8.2.2 Entering Ozone Audits

A. After logging into the AQS application choose the following:
Maintain → QA Assessments → Annual Performance Evaluation (see Figure 8.7 for illustrated pathway)

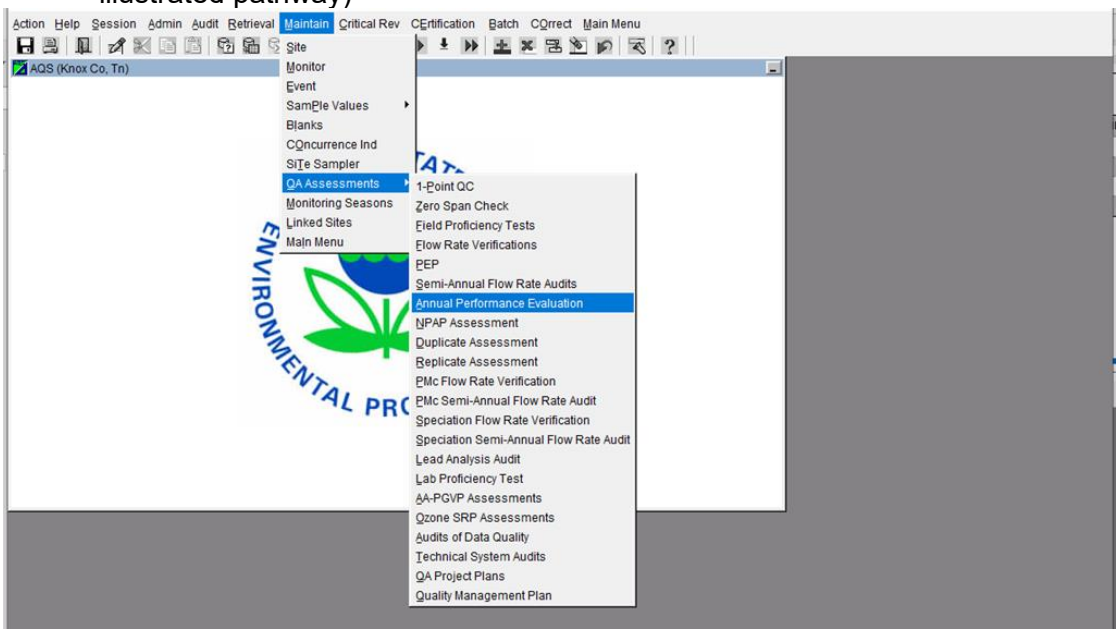


Figure 8.7 AQS Ozone Pathway

B. Enter data as follows for Ozone audits (See Figure 8.9 for sample entries)
State Code: 47
County Code:093
Site ID: See Table 8.1 for Site IDs
Parameter code: 44201
POC: 1 for official monitors
Performing Agency: 0581
Assessment Date: Date of Audit in format YYYYMMDD
Assessment Number:1 unless repeated on same day, then 2,3.
Method Code:087
Unit: 007 ppm
Levels Assessment: Using Table 8.8 below, enter the audit transfer standard value into the correct concentration level. The values indicated in Section 3.2 of this SOP

correspond to Audit Levels, 2, 3, 5, and 6.
Levels Monitor: Using Table 8.8 below enter the monitor's value in the correct concentration level spot. This should correspond to the value attempted and match with the assessment level from above. Repeat the level assessment and level monitor input for each value.

Level	1	2	3	4	5	6	7	8	9	10
Range in ppm	.004-.0059	.006-.019	.020-.039	.040-.069	.070-.089	.090-.119	.120-.139	.140-.169	.170-189	.190-.259

Table 8.8 Assessment Concentration Levels

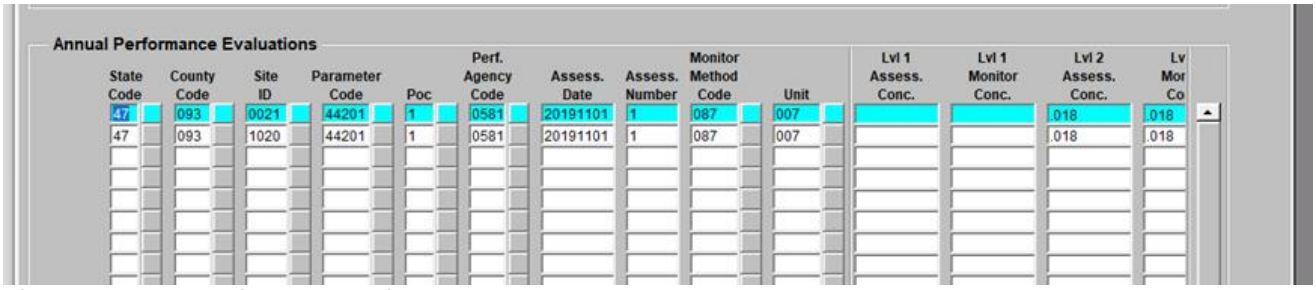


Figure 8.9 AQS Sample Ozone Entries

9.0 Site Evaluations

At least annually, site evaluations must be performed at each of the air monitoring sites. These site evaluations ensure that each monitor at each site continues to meet 40 CFR Part 58 appendix E siting criteria.

9.1 Preparing for the Site Evaluations

Site evaluations must be performed once a year. It is recommended that they be performed once in the first quarter audit typically scheduled in early March, and then again in 4th Quarter audits in early October. Due to weather, length of work day and run scheduling it may be difficult to complete site evaluations on the same day as internal audits. There is no requirement that the evaluations be performed on the same day. Additionally, due to the measuring procedures, it may be necessary for second person to be onsite to provide a larger target for measuring distances using the range finder.

The Auditor shall gather the tools listed in table 9.1 below. Cell phone applications can be used in lieu of compass, GPS unit, and digital camera. Cell phone apps should not be used as measurement tools. Before going to a site, the Auditor should review the instruments onsite, and the assigned scale of the instruments. This can be filled in on the form prior to performing the evaluation. Pages 1 and 2 of the Siting Evaluation Form (Appendix A figures 12.6 and 12.7) should be printed and used in the field for field notes.

Equipment List
Range Finder
Compass
GPS unit (Cell Phone)
100ft measuring tape
Siting Evaluation Form
Digital Camera
Pen
Site Log

Table 9.1 Site Evaluation Equipment

9.2 Performing the Site Evaluation

9.2.1 Horizontal distance Measurements

Using the Siting Evaluation Form (Appendix A figure 12.6), perform the following measurements using the measuring tape and record the results in meters. Note the measuring tape is in feet, conversions to meters is required.

A. Measure the probe height. This measurement is taken from natural ground level to the instruments inlet. The inlet for this measurement is the location that air first enters the sample train. For instruments with PM10 separator heads, this would include the PM10 head and be measured to the first louver on the separator.
B. Measure the distance between sampling instruments. This measurement is taken from the center of the down tubes, for lead instruments the center of the doghouse.
C. Measure the distance to the nearest public road. In many cases this distance is greater than 30 meters. Longer distances to a public road may be calculated using google earth of KGIS.org online measurement tools.
D. Measure the distance to any walls, rooftops, and or parapet obstructions.
E. Measure the distance to tree driplines. This should include the nearest dripline, as well as any trees that meet the obstruction definition detailed in Section 9.2.2
F. Measure the length and width of the platform or shelter. This will be used in the site drawing discussed in section 9.2.3

9.2.2 Vertical height measurements

Using the Siting Evaluation form and the rangefinder, perform the following measurements and record the results in meter. It is important to take into consideration the terrain height. For example a tree might be 10 m tall, but downhill from the instrument, therefore it fall entirely below the monitoring path. Also consider the opposite where a short tree might sit on higher terrain, the bottom of the tree would be extrapolated down to even with the site terrain for more accurate evaluation.

The range finder is used to determine the presence of obstructions and their heights and distances from the monitoring instruments. An Obstruction is an object (tree, building, etc) that may block the free flow of air to the instrument. In order for an object to NOT be considered an obstruction it must be farther away than 2x the difference in heights between the obstruction and the probe. Trees must not only meet the dripline requirements, but must also be evaluated if they provide an obstruction to the air flow.

A. Power on the range finder. Use the mode button to ensure it is set to vertical distance. See figure 9.2 below. This mode takes 2 measurements.
B. Use the view finder till you see the bottom of the object of interest (tree or obstruction).

Press the power button to record the measurement.

C. Use the view finder till you see the top of the object of interest. Press the power button to record the measurement.
D. The unit will display the vertical height on the internal and external screens. Record the value on the Siting Evaluation Form.
E. Select horizontal distance mode. Use the view finder to locate the object of interesting, holding the range finder at square 90 ° angle. Press the power button to record the horizontal distance. This can be used to measure longer distances than the measuring tape can reach, but a horizontal target must be present. A second person can be used to stand in the location and act as a target, but do not shoot the laser at their face or eyes.

Figure 9.2 Rangefinder Display

F. Record the obstruction type, height (h1), and distance. If D > 2 x (h1-probe height) then passes. If fails, deduct this obstructed air flow path in the degrees of air flow observation discussed in section 9.2.3.

9.2.3 Site Drawing and Photographs

The Siting Evaluation Form page two has a grid line area to draw the site and important features. Each grid space equals 2 square meters. The first circle indicates a distance of 10 meters and the second 20 meters.

A. Using the measurements taken in 9.2.1, draw to scale (as close as practical) the site centered on the grid. Include shelter, platforms, location of monitoring equipment
B. Using the compass, on the site drawing indicate with arrow the cardinal direction North.
C. Using the measurements taken in 9.2.1 and 9.2.2 draw nearby trees, roadways, buildings and obstructions.
D. Estimate the degree of unrestricted air flow. Using the compass, estimate the degrees of obstruction produced by each of the obstructions measured in 9.2.2. Add each the degrees obstructed, then subtract total from 360° for the estimated degree of

unrestricted air flow. This value must be ≥270°

E. Take pictures facing out from the monitor to each of the cardinal directions.
F. Take pictures facing in from 15-20 m out facing in towards the site from each of the cardinal directions.

9.3 Reviewing Results

Set up a Siting Evaluation workbook in excel. This includes a Form tab for each site in the monitoring network. Transfer the information gathered in the field, insert photographs taken, and use shapes and drawing tools to create a clear site drawing. Review the results with requirements of 40 CFR part 58 appendix E. Requirements are based upon pollutant, design monitoring scale, and other factors for each parameter.

Include the site evaluation results with the quarterly internal audit. Notify the AMPM immediately of any findings that do not meet the requirements, or trees that are close to exceeding the requirements. Tree growth should be monitored at each internal audit. The Auditor and the Operators should monitor sites each visit even when not performing an official site evaluation, and report any changes to the AMPM.

10.0 Thermometer Verifications

The Internal Auditor shall maintain the verifications of shelter and refrigeration thermometers. Proper filter storage temperatures and instrument operating temperatures are important components of a quality system.

10.1 Ozone Shelter Temperature Verification

Shelter temperature verifications utilizes a streamline pro or other certified temperature standard to check the deviation of the shelter temperature probe, data logger, back up thermometer and HVAC thermostat. The ozone shelter shall be maintained between 15 – 35 °C. This parameter is verified utilizing the shelter temperature probe which reports to the data logger. This probe is not adjustable; therefore the data logger is programed with an adjustment factor to ensure the reported value is accurate. The backup thermometer readings are recorded on the weekly parameter form and provide a secondary verification that the temperatures were not exceeded.

It is best to perform this shelter temperature verifications with the HVAC system not running, or at a minimum insure there is no vent blowing air right at the measurement devices.

Remember the rounding convention. Number recorded shall be rounded to the least precise instrument. So for the backup thermometer which only reports in whole degrees, the reference device values shall be rounded to the nearest whole degree.

10.1.1 Verification Procedures

A. Record the serial number of the shelter back up thermometer and the data logger on the field sheet ozone (Appendix A figure 12.1). The Thermostat and shelter temp sensor do not have serial numbers and this section has been greyed out on the field sheet.
B. Set the reference thermometer probe next to the shelter temperature sensor and allow to equilibrate.

C. Record the values from the shelter temperature sensor display (actual) and the reference thermometer remote T (ref). The shelter temperature sensor may display values in °F. Utilize an online tool to convert to °C.
D. Record the values from the data logger display (actual) and the reference thermometer remote T (ref). The data logger display receives the input from the shelter temperature sensor and applies a conversion and correction factor.
E. Move the reference thermometer remote probe next to the backup thermometer and allow to equilibrate for a couple of minutes.
F. Record the values from the backup thermometer (actual) and the reference thermometer remote T (ref).
G. Move the reference thermometer remote probe next to the shelter HVAC thermostat. Allow to equilibrate for a couple of minutes.
H. Record the values from the thermostat (actual) and the reference thermometer remote T (ref). The Thermostat output is in whole digit °F, utilize an online conversion tool to convert to °C and round to the decimal place recorded by the reference thermometer.
I. Utilize the sticky form (appendix B figure 13.6) to record the results in the SITE logbook.

10.1.2 Evaluating Results

Each temperature device has a different role to play and as such the results are interpreted differently. The Shelter Temperature Sensor does not have an acceptance criteria. These devices are very stable and as such this measurement can be used to compare to previous years and indicate if the probe is failing. The data logger display and back up shelter thermometer should be within ± 2°C of the reference standard. These devices are used to measure compliance with a data quality objective. If either of these are outside the acceptance criteria notify the Operator and AMPM immediately. Finally the shelter thermostat is used by the HVAC system to regulate the shelter temperature. This value deviation is relevant if the shelter is not maintaining the set temperature parameters, or a deviation > ± 2° C may indicate the need to change the batteries or upgrade the thermostat. Notify the Operator to review the HVAC maintenance schedule and response to thermostat setting changes.

10.2 Refrigeration Thermometer Verification

Temperatures are recorded for the PM2.5 filter storage refrigerator. Filters must be stored between 0 and 4 °C after sampling before shipping. The remote thermometers that are used inside the storage refrigerator must be verified against a certified temperature standard at least once a year, but should be verified biannually. This verification should always be performed following a filter shipment, when there are no filters stored in the refrigerator.

10.2.1 Verification Procedures

A. Using Thermometer Verification Form in the Audit workbook (Appendix A figure 12.8) record the date and serial number of the reference device used.
B. Record the refrigeration temperatures in the PM2.5 storage refrigeration logbook.
C. Insert the remote temperature probe into the storage refrigerator, place near the thermometer and pull cord flat to shut the door.
D. Allow ample time for refrigerator and thermometers to reach the set temperature.

Record the thermometer reading (actual) and temperature standard's reading (reference) on the form.

E. Remove all of the thermometers and temperature standard's probe, then place on a counter in the clean room. Allow them to reach room temperature for Temp 2 measurement.
F. After each has stabilized to ambient room temperature, record the thermometer reading (Actual) and the temperature standard's reading (reference on the form under Temp Point 2.
G. Place each of the thermometers and the temperature standard probe into either a freezer or outside in the sun and repeat E for Temp Point 3.
H. After completion of the verification process, return the thermometers to the refrigerator.
I. After the thermometers have had time to return to the refrigeration temperature. Reset the thermometers and record in the PM2.5 storage refrigeration logbook.

10.2.2 Evaluating the Results

Thermometers should be within ±2°C of the certified temperature standard on all 3 temperature points. However for data validation the first temp point or that of the refrigeration set point is the most crucial for data validation. If any points are outside the limits, notify the AMPM, replace the batteries and repeat the verification. If failed a second time thermometers shall be replaced.

11.0 Documentation and Records Management

Recordkeeping is a critical part of the Quality Assurance Program. Careful documentation will salvage data that may otherwise be lost. The sheer repetition of recording data may result in errors. This section highlights specific documentation that must be completed. This shall supplement any documentation elements described in the previous sections.

11.1 Documentation

The Auditor is responsible for fully documenting their actions during an audit, as well as reviewing the documentation performed by the Operator. There are two logbooks which must be updated and maintained properly, the site logbook and the instrument logbook. The Auditor must record in the site logbook the date, time, their initials, weather conditions, and purpose of the visit every time they visit the site. Additionally they shall review that the Operator is also entering this data in the site logbooks. In the sampler logbook the Auditor must record the date, time, their initials, and results of the audit. Operators and the Auditor may use "Stickies" which are results forms printed on clear packing labels to aid in entering data into the instrument logbook. When used, stickies must be printed on clear labels to ensure that no entries are covered in the logbook.

The field sheets which are used to record data in the field, and enter data into the excel Audit workbook must be kept. The Auditor shall include copies of the field sheets in the final audit report, scanned copies are acceptable.

11.2 Records Management

Records management is the professional practice of maintaining the records of an organization throughout their life, from the time they are created to their eventual disposal. This includes identifying, classifying, storing, securing, retrieving, tracking and destroying or permanently preserving records.

11.2.1 Document Integrity

The majority of documents used are formatted through Microsoft Excel. This program performs the calculations, graphs, and formats results. All documents shall be checked annually as part of the records management program for accuracy. The master forms are in read-only format to ensure there are no alterations to the approved formulas and formatting during the year. It is the Auditor's responsibility to ensure they use the most recent approved forms and workbooks when performing an audit.

11.2.2 Records Retention

Audit records are stored in the file cabinet organized by year in the file named Audits. After the current year, the files are maintained according to Air Quality's Record Retention policy. In general, files are maintained at least 5 years onsite.

12.0 Appendix A

KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Air Quality Ozone Field Sheet

Site Name: _____ Date: _____

Generate Time est	Read Time est	Target ppb	Analyzer ppb	Audit STD ppb	Stability ppb
		110			
		70			
		35			
		15			
		0			

Audit std Serial #: _____ Analyzer Serial #: _____

TEST PARAMETER	CALIBRATOR VALUE	ACCEPTABLE RANGE
Output Flow (lpm)		3.0 to 5.5
Reg. Press. (psig)		10 to 17 psig
Box Temp (ºC)		20 to 35
O3 Gen. Ref. (mV)		-25 to 5000
O3 Gen. Drive (mV)		-25 to 5000
O3 Lamp Temp (ºC)		47 to 49
Photo. Meas. (mV)		2500 to 4700
Photo. Ref. (mV)		2500 to 4700
Photo. Flow (lpm)		0.720 to 0.880
Photo. Lamp Temp. (ºC)		57 to 59
Photo. Smp. Prs. (inHg-A)		≈ Amb. -1 inHg
Photo. Samp. Temp. (ºC)		25 to 48
Slope (unitless)		0.850 to 1.150
Offset (ppb)		-10.0 to +10.0

TEST PARAMETER	ANALYZER VALUE	ACCEPTABLE RANGE
Stability (ppb)		< 1 ppb @ zero
O3 Meas. (mV)		2500 to 4800
O3 Ref. (mV)		2500 to 4800
Pressure (inHg-A)		≈ Amb. -2 inHg
Sample Flow (cc/min)		720 to 880
Sample Temp. (°C)		10 to 50
Photo. Lamp Temp. (°		57 to 59
Box Temp (°C)		10 to 50
Slope (unitless)		0.85 to 1.15
Offset (ppb)		-10.0 to +10.0

Excess Flow @ Trans. Stnd. Vent: _____

External ZAS Pressure: _____

	System	Reference	Difference
Logger Time			
Analyzer			

	Serial #	Actual (°C)	Ref (°C)	Diff (°C)
Shelter Temp Sensor Display				
Data Logger Display				
Shelter Thermometer (back up)				
Shelter Thermostat				

Field Sheet Ozone Rev 8
2/27/2020

Figure 12.1 Field sheet Ozone

KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Air Quality PM2.5 Field Sheet

Ref Device: _____
Serial Number: _____ Calibration Date: _____

Site Name: Date:

Sampler ID:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 1 Min.
Filter Temperature				+/- 2° C
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow				+/- 4%

Leak Check ☐ <25mmHG

Site Name: Date:

Sampler ID:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 1 Min.
Filter Temperature				+/- 2° C
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow				+/- 4%

Leak Check ☐ <25mmHG

Site Name: Date:

Sampler ID:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 1 Min.
Filter Temperature				+/- 2° C
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow				+/- 4%

Leak Check ☐ <25mmHG

Site Name: Date:

Sampler ID:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 1 Min.
Filter Temperature				+/- 2° C
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow				+/- 4%

Leak Check ☐ <25mmHG

Site Name: Date:

Sampler ID:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 1 Min.
Filter Temperature				+/- 2° C
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow				+/- 4%

Leak Check ☐ <25mmHG

Field Sheet PM2.5 Rev 8
2/27/2020

Figure 12.2 Field sheet PM 2.5

KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

AIR QUALITY
AUDIT FIELD SHEET

Ref Device: _____
Serial Number: _____ Calibration Date: _____

Site Name: _____ Date: _____

T640x SN:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 5 Min.
Shelter Temperature				+/- 2º C
Ambient Temperature				+/- 2º C
Barometric Pressure				+/- 10mmHg
Total Flow (16.67 l/min)[(Sys-Ref) / ref] * 100				+/- 4%
Main Flow				+/- 4%

Leak Check

SN: _____

Site Name: _____ Date: _____

T640 SN:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 5 Min.
Shelter Temperature				+/- 2º C
Ambient Temperature				+/- 2º C
Barometric Pressure				+/- 10mmHg
Flow				+/- 4%

Leak Check

SN: _____

Site Name: _____ Date: _____

T640 SN:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 5 Min.
Shelter Temperature				+/- 2º C
Ambient Temperature				+/- 2º C
Barometric Pressure				+/- 10mmHg
Flow				+/- 4%

Leak Check

SN: _____

Site Name: _____ Date: _____

T640 SN:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 5 Min.
Shelter Temperature				+/- 2º C
Ambient Temperature				+/- 2º C
Barometric Pressure				+/- 10mmHg
Flow				+/- 4%

Leak Check

SN: _____

Field Sheet Continuous Rev 8
2/27/2020

Figure 12.3 Field Sheet Continuous

KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Lead Audit Field Sheet

TSP/Pb	**Please circle Reference device used for Audit**		
HiVol Cal	DeltaCal	Trical	TetraCal

Serial Number: _____ Calibration Date: _____

Timer: _____
Date: _____ Cell Time: _____

Site: Burnside Official
Orifice: P02875

QaCFM For TSP

Amb Pres: _____
Temp: _____
Stag Pres: _____

Leak Check: _____ {Between 17-24 inH20}

Timer: _____
Date: _____ Cell Time: _____

Site: Burnside Collocated
Orifice: P04302

QaCFM For TSP

Amb Pres: _____
Temp: _____
Stag Pres: _____

Leak Check: _____ {Between 17-24 inH20}

Timer: _____
Date: _____ Cell Time: _____

Site: Ameristeel
Orifice: P04304

QaCFM For TSP

Amb Pres: _____
Temp: _____
Stag Pres: _____

Leak Check: _____ {Between 17-24 inH20}

Timer: _____
Date: _____ Cell Time: _____

Site: _____
Orifice:

QaCFM For TSP

Amb Pres: _____
Temp: _____
Stag Pres: _____

Leak Check: _____ {Between 17

Field Sheet Lead Rev 8
2/27/2020

Figure 12.4 Field sheet Lead

KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Speciation Field Sheet

Please circle Reference device used for Audit

Streamline Pro	TetraCal

Serial Number: _____ Calibration Date: _____

Site Name: _____ Date: _____

URG SN:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 5 Min.
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow (16.67 l/min) [(Sys- Ref) / Ref] * 100				+/- 10 %

Leak Check

Pass/Fail

Site Name: _____ Date: _____

SASS SN:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 5 Min.
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Filter Temp 1				+/- 2° C
Filter Temp 2				+/- 2° C
Sample Flow 1 (sys-ref/ref)*100				+/- 10 %
Sample Flow2[(Sys-ref) / ref] * 100				+/- 10 %

Leak Check

Pass/Fail

Comments:

Field Sheet SP Rev 8
2/27/2020

Figure 12.5 Field Sheet SP

KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Siting Evaluation Form

Site Name:
AQSNo:
Coordinates:

Date:
Site Address:
Inspected by:

Pollutant	Scale	Probe Height [1]	Flow (hi or Low)	Separation from samplers [1]	Pass/Fail	Distance to Road [1]	Pass/Fail

				Tree	
Obstruction type [2]	Obst. Height [1]	Obst. Distance [1,2]	Pass/Fail	Dripline [1]	Pass/ Fail

[1] All Measurements in meters

[2] Including vertical and horizontal separation from walls &/or parapets if applicable

Collocated Samplers must be within 4 m of each other and at least 2 m apart for hi vol, at least 1 m for low volume
Obstruction Distance must be ≥ 2* (Obst height - probe height)
Tree Dripline must be >10 m away, prefer >20m
Horizontal and vertical disance on rooftop 1m for O_3/ gases - 2m for all others
Unrestricted air flow must be ≥ 270 °

Figure 12.6 Site Evaluation pg 1

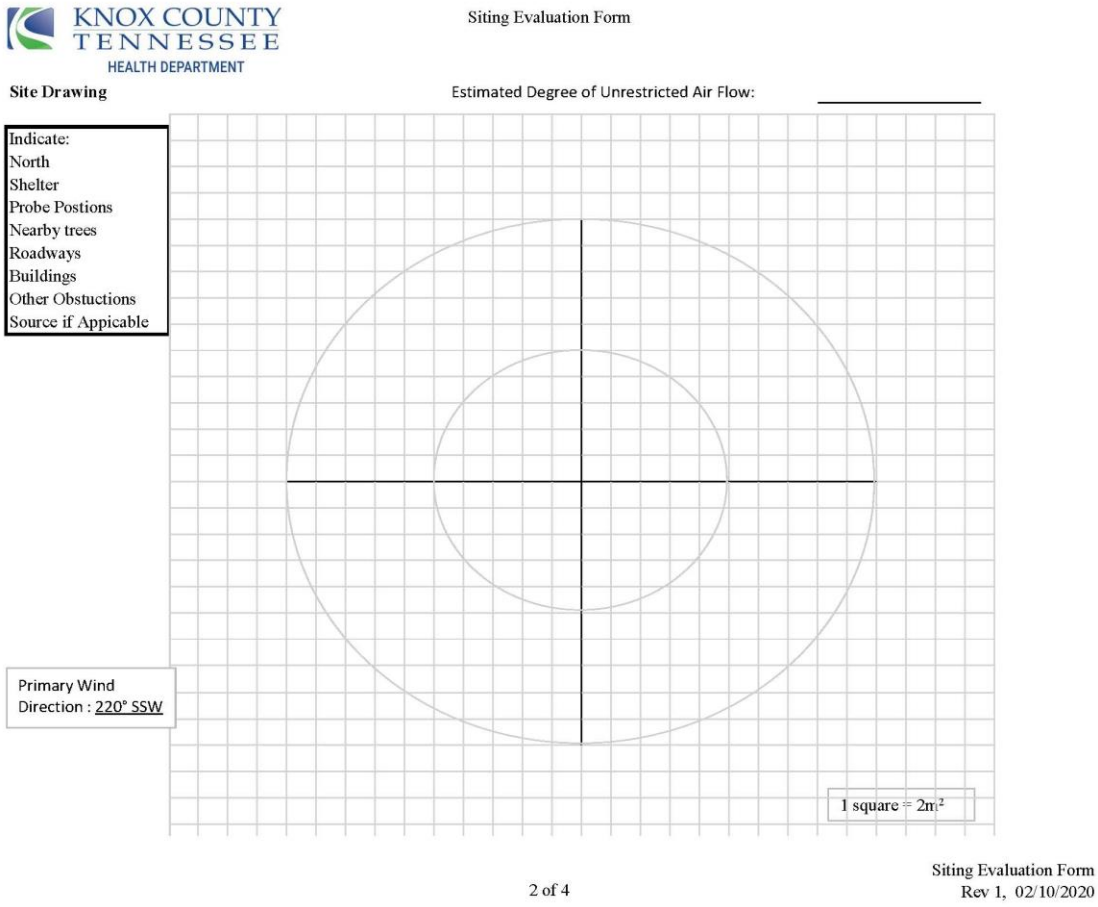


Figure 12.7 Site Evaluation pg 2

KNOX COUNTY TENNESSEE HEALTH DEPARTMENT

Thermometer Verification

Date: _____ Ref Device: _____

Serial Number:	Actual (°C)	Reference (°C)	Difference (°C)
Temp Point 1			
Temp Point 2			
Temp Point 3			

Operator Intials: _____

Date: _____ Ref Device: _____

Serial Number:	Actual (°C)	Reference (°C)	Difference (°C)
Temp Point 1			
Temp Point 2			
Temp Point 3			

Operator Intials: _____

Date: _____ Ref Device: _____

Serial Number	Actual (°C)	Reference (°C)	Difference (°C)
Temp Point 1			
Temp Point 2			
Temp Point 3			

Operator Intials: _____

Thermometer Verification Rev 8
2/27/2020

Figure 12.8 Thermometer Verification

13.0 Appendix B

Operator:		Logger Disable:		(Circle one)		
Date:		Logger Enable:		Pass	Fail	
Generate Time	**Collection Time**	**Target**	**Audit Actual**	**Analyzer Actual**	**Stability**	**Difference**
hr:min	**hr:min**	**ppb**	**ppb**	**ppb**	**ppb**	**%**
		110				
		70				
		35				
		15				
		0				NA

Figure 13.1 Ozone Sticky

Auditor: Date:

Ref ID:	System	Reference	Difference	Criteria
Time (in EST)				+/- 1 Min.
Filter Temp				+/- 2° C
Ambient Temp				+/- 2° C
Baro. Press				+/- 10 mmHg
Sample Flow				+/- 4%

Figure 13.2 PM 2.5 Sticky

Auditor:
Ref ID: Date:

T640X	System	Reference	Difference	Criteria
Time				+/- 1 Min.
Shelter T				+/- 2° C
Amb Temp				+/- 2° C
Amb Press				+/- 10 mmHg
Main Flow				+/- 4%
Total Flow				+/- 4%

Auditor:
Ref ID: Date:

T640	System	Reference	Difference	Acceptance Criteria
Time				+/- 1 Min.
Shelter T				+/- 2° C
Amb Temp				+/- 2° C
Amb Press				+/- 10 mmHg
Total Flow				+/- 4%

Figure 13.3 PM Continuous Sticky

Auditor:
Ref ID: Date:

URG	System	Reference	Difference	Acceptance Criteria
Time				+/- 5 Min.
Amb Temp				+/- 2° C
Baro Press				+/- 10 mmHg
Sample Flow				+/- 10%

Figure 13.4 URG Sticky

Auditor:
Ref ID: Date:

SASS	System	Reference	Difference	Acceptance Criteria
Time				+/- 5 Min.
Amb Temp				+/- 2° C
Baro Press				+/- 10 mmHg
Filt T 1				+/- 2° C
Filt T 2				+/- 2° C
Flow 1				+/- 10%
Flow 2				+/- 10%

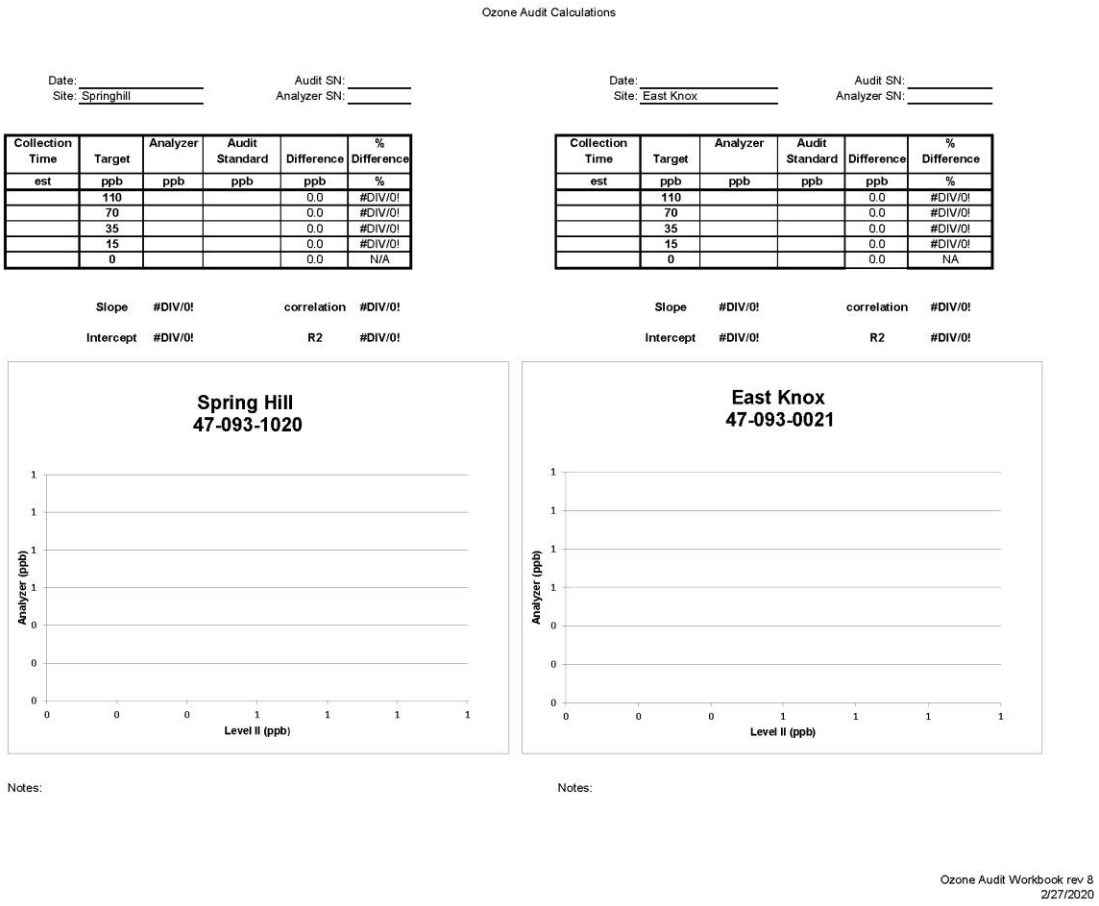
Figure 13.5 SASS Sticky

Date: _____ Operator Intials: _____

Ref Device:	Actual (°C)	Ref (°C)	Dif (°C)
Shelter Temp Sensor Display			
Data Logger Display			
Shelter Thermometer (back up)			
Shelter Thermostat			

Figure 13.6 Shelter Temp Sticky

14.0 Appendix C



Ozone Audit Calculations

Date: _____ Audit SN: _____
Site: Springhill Analyzer SN: _____

Collection Time	Target	Analyzer	Audit Standard	Difference	% Difference
est	ppb	ppb	ppb	ppb	%
	110			0.0	#DIV/0!
	70			0.0	#DIV/0!
	35			0.0	#DIV/0!
	15			0.0	#DIV/0!
	0			0.0	N/A

Slope #DIV/0! correlation #DIV/0!
Intercept #DIV/0! R2 #DIV/0!

Notes:

Date: _____ Audit SN: _____
Site: East Knox Analyzer SN: _____

Collection Time	Target	Analyzer	Audit Standard	Difference	% Difference
est	ppb	ppb	ppb	ppb	%
	110			0.0	#DIV/0!
	70			0.0	#DIV/0!
	35			0.0	#DIV/0!
	15			0.0	#DIV/0!
	0			0.0	NA

Slope #DIV/0! correlation #DIV/0!
Intercept #DIV/0! R2 #DIV/0!

Notes:

Ozone Audit Workbook rev 8
2/27/2020

Figure 14.1 Audit Workbook Ozone

PM 2.5 Audit Calculations

Reference device used for Audit: _____ Serial number : _____
Date of Certification: _____

Date: _____
Site: _____ Monitor Serial number: _____

	units	System Value	Reference Value	Difference (S-R)	%	Acceptance Criteria
Time	hh:mm:ss			#VALUE!		+/- 1 Min.
Filter T	°C			#VALUE!		+/- 2º C
Ambient T	°C			#VALUE!		+/- 2º C
Pressure	mmHg			#VALUE!		+/- 10 mmHg
Flow Rate	lpm			#VALUE!	#VALUE!	+/- 4%

Notes:

Date: _____
Site: _____ Monitor Serial number: _____

	units	System Value	Reference Value	Difference (S-A)	%	Acceptance Criteria
Time	hh:mm:ss			#VALUE!		+/- 1 Min.
Filter T	°C			#VALUE!		+/- 2º C
Ambient T	°C			#VALUE!		+/- 2º C
Pressure	mmHg			#VALUE!		+/- 10 mmHg
Flow Rate	lpm			#VALUE!	#VALUE!	+/- 4%

Notes:

Date: _____
Site: _____ Monitor Serial number: _____

	units	System Value	Reference Value	Difference (S-A)	%	Acceptance Criteria
Time	hh:mm:ss			#VALUE!		+/- 1 Min.
Filter T	°C			#VALUE!		+/- 2º C
Ambient T	°C			#VALUE!		+/- 2º C
Pressure	mmHg			#VALUE!		+/- 10 mmHg
Flow Rate	lpm			#VALUE!	#VALUE!	+/- 4%

Notes:

Date: _____
Site: _____ Monitor Serial number: _____

	units	System Value	Reference Value	Difference (S-A)	%	Acceptance Criteria
Time	hh:mm:ss			#VALUE!		+/- 1 Min.
Filter T	°C			#VALUE!		+/- 2º C
Ambient T	°C			#VALUE!		+/- 2º C
Pressure	mmHg			#VALUE!		+/- 10 mmHg
Flow Rate	lpm			#VALUE!	#VALUE!	+/- 4%

Notes:

Date: _____
Site: _____ Monitor Serial number: _____

	units	System Value	Reference Value	Difference (S-A)	%	Acceptance Criteria
Time	hh:mm:ss			#VALUE!		+/- 1 Min.
Filter T	°C			#VALUE!		+/- 2º C
Ambient T	°C			#VALUE!		+/- 2º C
Pressure	mmHg			#VALUE!		+/- 10 mmHg
Flow Rate	lpm			#VALUE!	#VALUE!	+/- 4%

Notes:

PM 2.5 Audit Workbook rev 8
2/27/2020

Figure 14.2 Audit Workbook PM2.5

PM 2.5 Audit Calculations

Reference device used for Audit: _____ Serial number : _____
Date of Certification: _____

Date: _____
Site: _____ Monitor Serial number: _____

T640 X

	Units	System	Reference	Difference	%	Criteria
Time	hh:mm:ss			0:00:00		+/- 1 Min.
Amb T	°C			0		+/- 2º C
Pressure	mmHg			0		+/- 10mmHg
Total Flow	lpm			0	#DIV/0!	+/- 4 %
MainFlow	lpm			0	#DIV/0!	+/- 4 %

Notes:

Date: _____
Site: _____ Monitor Serial number: _____

	Units	System	Reference	Difference	%	Criteria
Time	hh:mm:ss			0:00:00		+/- 1 Min.
Amb T	°C			0		+/- 2º C
Pressure	mmHg			0		+/- 10mmHg
Flow Rate	lpm			0	#DIV/0!	+/- 4 %

Notes:

Date: _____
Site: _____ Monitor Serial number: _____

	Units	System	Reference	Difference	%	Criteria
Time	hh:mm:ss			0:00:00		+/- 1 Min.
Amb T	°C			0		+/- 2º C
Pressure	mmHg			0		+/- 10mmHg
Flow Rate	lpm			0	#DIV/0!	+/- 4 %

Notes:

Date: _____
Site: _____ Monitor Serial number: _____

	Units	System	Reference	Difference	%	Criteria
Time	hh:mm:ss			0:00:00		+/- 1 Min.
Amb T	°C			0		+/- 2º C
Pressure	mmHg			0		+/- 10mmHg
Flow Rate	lpm			0	#DIV/0!	+/- 4 %

Notes:

Date: _____
Site: _____ Monitor Serial number: _____

	Units	System	Reference	Difference	%	Criteria
Time	hh:mm:ss			0:00:00		+/- 1 Min.
Amb T	°C			0		+/- 2º C
Pressure	mmHg			0		+/- 10mmHg
Flow Rate	lpm			0	#DIV/0!	+/- 4 %

Notes:

PM continuous Audit Workbook rev 8
2/27/2020

Figure 14.3 Workbook Continuous

Lead Audit Calculations

Reference device used for Audit: _____ Serial number : _____
Date of Certification: _____

Date: _____ Bar Press _____ mmHg
Monitor ID: _____ Temp _____ °C
Site: _____

Qa CFM	
	Stag Press: _____ inH20
	Pa: #VALUE! mmHg
	Po/Pa: #VALUE! unitless
	Flow _____
	(from table)
	%D: 0.00%
	{Flow- Qa/Qa}x 100
	% D Design 0.00%
	{Qa - 1.13/1.13}

0.00 CFM
0.000 m^3/min

Date: _____ Bar Press _____ mmHg
Monitor ID: _____ Temp _____ °C
Site: _____

Qa CFM	
	Stag Press: _____ inH20
	Pa: #VALUE! mmHg
	Po/Pa: #VALUE! unitless
	Flow _____
	(from table)
	%D: #VALUE!
	{Flow- Qa/Qa}x 100
	% D Design 0.00%
	{Qa - 1.13/1.13}

0.00 CFM
0.000 m^3/min

Date: _____ Bar Press _____ mmHg
Monitor ID: _____ Temp _____ °C
Site: _____

Qa CFM	
	Stag Press: _____ inH20
	Pa: #VALUE! mmHg
	Po/Pa: #VALUE! unitless
	Flow _____
	(from table)
	%D: #VALUE!
	{Flow- Qa/Qa}x 100
	% D Design 0.00%
	{Qa - 1.13/1.13}

0.00 CFM
0.000 m^3/min

Notes:

Lead Audit Workbook rev 8
2/27/2020

Figure 14.4 Workbook Lead

Speciation Audit Calculations

Reference device used for Audit: _____ Serial number : _____
Date of Certification: _____

Leak Test

	Pass	Fail
URG 3000		
SASS Channel 1		
SASS Channel 2		

Pressure {Ambient}

	System	Reference	Difference
URG 3000N			0.00
SASS			0.00

Flow Rate

	System	Reference	% Difference
URG 3000N			#DIV/0!
SASS channel 1			#DIV/0!
SASS Channel 2			#DIV/0!

Temperature

	System	Reference	Difference
URG 3000N Ambient			0.00
SASS ambient			0.00
SASS filter channel 1			0.00
SASSfilter Channel 2			0.00

Speciation Audit Workbook rev 8
2/27/2020

Figure 14.5 Workbook Speciation

Brian Rivera, P.E.
Division Director
Air Quality
Knox County Health Department
140 Dameron Ave,
Knoxville, TN 37917-6413

November 12, 2019

Re: Fourth Quarter Air Monitoring Audit

Dear Mr. Rivera:

On Nov 1, 2019 – Nov 7, 2019, I conducted the fourth quarter quality assurance performance audit at each site in Air Quality's monitoring network. All of the monitors audited were within the acceptable range for critical criteria except the flow of the URG speciation monitor. One of the Pm2.5 2025 filter based monitors was outside the 1 minute criteria for the clock, and the T640X clock was outside the 5 minute acceptance criteria. The calculations and field notes are included in the Audit Report.

Each physical location was inspected. The site and equipment log books were well kept. The Program Manager was notified of the following logbook finding; the TEOM logbook had a few instances of backfilling. The entries were correctly dated and notated "backfilled" however backfilling should be a very rare event, and justification noted why the entry was not made at the time of occurrence. The siting criteria upon visual inspection was in compliance. Due to time constraints and scheduling, a full siting evaluation with measurements was not completed at this time, but will be completed before the next audit cycle. Data logger thermometer in the Air Lab shelter is still deviating from the goal of ± 2° C from the standard as discussed in the 3rd quarter audit.

The laboratory clean room was inspected. The filter preparation area was clean. The PM2.5 storage temperature log was reviewed. Storage temperatures have exceed 4 °C. The Program Manager and Operator have increased defrosting and temperature checks. No data is affected due to the high ambient temperatures to date.

The data validation and QA review has been completed on most pollutants for 3rd quarter. We are awaiting laboratory results on lead data for September 2019.

If there are any questions regarding this audit, please email Rebecca.Larocque@knoxcounty.org or call 865-215-5941

Rebecca Larocque
Environmental Specialist
Knox County Health Department

Figure 14.6 Sample Cover Letter

Appendix C – T640X SOP

Teledyne T640x Model Continuous PM10/PM2.5
Standard Operating Procedure

KNOX COUNTY
HEALTH DEPARTMENT

Prepared by:
Rebecca Larocque

Knox County Air Quality
140 Dameron Avenue
Knoxville, TN 37917-6413

Identification and Approval

Title: "Teledyne T640x Model Continuous PM10/PM2.5 Standard Operating Procedure"

The attached "Teledyne T640x Model Continuous PM10/PM2.5 Standard Operating Procedure" is hereby recommended for approval and commits the Knox County Department of Air Quality, hence forth referred to as Air Quality, to follow the elements described within.

Air Quality

1) Signature: _____ Date: 12/10/19
Brian Rivera, Division Director

2) Signature: Amber E. Talgo Date: 12/10/19
Amber Talgo, Air Monitoring Program Manager

US Environmental Protection Agency, Region 4

3) Signature: _____ Date: _____
Reviewing US Environmental Protection Agency Region 4 Staff

Revision History

Change Revision#	Page #	Date of Change

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1.0 Introduction

This Standard Operating Procedure (SOP) describes the procedures used by Air Quality to sample PM10, particulate matter with an aerodynamic diameter of 10 micrometers or less) and PM2.5 (particulate matter with an aerodynamic diameter of 2.5 Micrometers or less) using the Teledyne T640x PM Mass Monitor.

The T640x is an approved Federal Equivalent Method (FEM) for PM10 [EQPM-0516-239] and for PM2.5 [EQPM-0516-238] The T640x operates at a total flow of 16.7 liters per minute (L/min). The contents of this SOP are based upon the sample SOP provided by US EPA OAQPS in partnership with Sonoma Technologies, Teledyne, and other air monitoring agencies throughout the US. Many instructions and figures are replicated with only minor changes for this Agency's instrument set up, or document formatting.

1.1 Principles of Operation

The Teledyne Advanced Pollution Instrumentation (TAPI) Model T640 with 640X operation is a real-time, continuous particulate matter (PM) mass monitor that uses scattered light spectrometry for measurement. The T640x measures 2.5, 10 and course PM.

Particle separation in the T640x occurs by drawing a controlled volume of air (16.67 L/min) through a PM10 inlet head. This allows only particles of 10 micrometer in diameter and smaller to pass thru the downtube. The flow is then split into main flow (5.0 L/min) and bypass flow (11.67 L/min). The main flow continues through the Aerosol Sample Conditioner (ASC) and passes thru the optical particle sensor. The optical particle sensor uses the Lorenz-Mie Theory for determining sampled particle size via scattered light at the single particle level. a polychromatic LED light source and 90° white-light scattering spectrometry for measurement. The sample air passes thru a relative humidity and temperature sensor, then a filter, flow meter and exhaust. The bypass flow, diverted prior to the heated downtube, passes thru a filter, flow meter, a flow control valve and then to exhaust. Bypass flow is not used in the measurement process. The purpose of the bypass flow is to achieve the 16.67 L/min flow rate needed for correct participle separation in the PM10 inlet head.

Sample volume is reported in actual conditions by using the instruments ambient temperature and barometric sensor data. However, the T640x has the option for reporting mass adjusted for standard temperature and pressure for PM10

1.2 Method Requirements

The method requirements for the T640x were published in the Federal Register in Volume 81, page 45285 on July 13, 2016 as part of the Federal Equivalent Method (FEM) Designation. These requirements were published as: ‘‘*Teledyne Advanced Pollution Instrumentation Model T640 PM mass monitor with 640X option,”* continuous ambient particulate monitor operated at a volumetric flow rate of 16.67 L/min equipped with the louvered PM10 inlet specified in 40 CFR 50 Appendix L, Figs. L–2 thru L–19, TAPI aerosol sample conditioner (P/N: 081040000), configured for operation with firmware version 1.0.2.126 or later, in accordance with the Teledyne Model T640 Operations Manual.

Additional requirements of operating the method may not be explicitly included as part of the method designation; however, they would apply for consistency with standard monitoring practices and use of the data for comparison to the NAAQS. The sample volume for the method is reported at actual conditions (local conditions or LC) for PM2.5 and at Standard Temperature and Pressure (STP) for PM10. Each of these (i.e., LC and STP) are calculated by using the instruments ambient temperature and barometric sensor data.

1.3 Reference Documents

- 40 CFR Part 58 Appendix A and E
- 40 CFR Part 50 Appendix J, K, and L
- 40 CFR Part 53, FRM/FEM Method List
- US Environmental Protection Agency, Quality Assurance Handbook for Air Pollution Measurements Systems, Volume II, Ambient Air Specific Methods, 2017
- Air Quality, Quality Assurance Project Plan, 2018
- US EPA's OAQPS "Standard Operating Procedure Teledyne Model 640x Real-Time Continuous PM Monitor", Sept. 2018
- Teledyne Advanced Pollution Instrumentation Model T640 PM Mass Monitor User Manual, 2016

2.0 Installation

When installing new equipment, the site specifications, shelter specifications, and monitor specifications must all be considered. Refer to Section 2.0 in the TAPI T640 User Manual (manual) for instructions on both shelter and enclosure installation configurations. This section documents the requirements of acceptance, suggested tools, logger configurations, and pre-sampling calibrations.

2.1 Physical Inspection & Acceptance

Upon receipt of all shipped sampling equipment, parts, and supplies, inspect each for completeness and/or damage. If a shortage or damage is found, immediately notify the Air Monitoring Program Manager (AMPM). The AMPM will work with the Office Manager to contact the shipper and vendor to repair or replace damaged equipment or missing supplies. Remove printed record of final factory performance titled "Final Test and Validation Data Sheet" and give to the Quality Assurance Officer (QAO) or the AMPM for scanning and filing in the quality records file for the instrument.

Follow Section 2.1 of the Teledyne T640 User Manual (manual) for step by step instructions on inspecting the instrument for internal shipping damage. If no damage is present, record the date and time of unboxing and physical inspection in the instrument logbook. Set up the instrument according section 2.3 first inside the KCDAQM air lab clean room. Run the instrument, record parameters (see section 4.2 of this SOP) and view, record, and clear any warnings for at least 24 hours. Stable operation without unexpected warnings along with the passing physical inspection shall deem the monitor acceptable for field installation.

During the acceptance test run, ensure the firmware is up to date, and perform any updates following the manual section 3.2 and 3.2.6.2, prior to installing in the field.

2.2 List of Tools and Equipment

- Rubber tubing
- Allen wrench
- Philips screwdriver
- Swagelok cap for ¼" tubing
- 9/16 wrench
- PM10 inlet head
- Tripod
- Power tools, electric drill, power screwdriver, bits
- Extension cord
- Personal protective equipment
- Optional stability/ shelter moisture control: rubber roof flange, PVC fittings to encase and stabilize the 8' extension for shelter installation

2.3 Siting Requirements

The sampler should be sited to meet the goals of the specific monitoring project. The siting location should be included in the Quality Assurance Project Plan (QAPP) and have been approved in the Annual Network Plan (ANP). Samplers (in the shelter or housing) will be mounted on a safe, suitable monitoring platform according to the following guidelines:

- PM10 samplers must be exposed to unobstructed air flow in all directions
- The sampler inlet must be vertically placed between 2 and 15 meters (m) above ground level.
- If a sampler is collocated with other samplers, the minimum spacing between sampler inlets is as follows:
    - If collocated with other low- volume samplers (flow rate≤ 16.7 L/min), maintain a minimum distance between sampler inlets of 1m.
    - If collocated with other high-volume samplers (flow≥1.13 m^3/min) maintain a minimum distance between sampler inlets of 2m.
- The site security is to be maintained with a locked fence.
- Tree driplines should be at least 20m but must be at least 10m away.
- Follow all requirements established in "40 CFR 58 Appendix E – *Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring"*

2.4 Installation and Assembly

The installation procedures of the T640x are detailed in section 2.0 of the TAPI T640 Manual. All the steps outlined therein must be followed closely according to the type of enclosure or shelter. Upon completing the shelter/ enclosure installation perform all the calibrations listed in section 3.0 of this document for initial calibration.

2.4.1 Indoor/Outdoor Installation

The T640x can be installed in an indoor or outdoor shelter with roof penetration or in an outdoor enclosure. Section 2.4.2 of this SOP provides step by step instructions for ASC connections and installation. If the instrument is being installed in a shelter with roof penetration see Section 2.4.1 of the TAPI Manual for installation instructions. If the instrument is being installed in an outdoor enclosure see section 2.4.2 of the TAPI manual for instructions.

2.4.2 Aerosol Sample Conditioner (ASC) Connections and Installation

The ASC requires an inlet nozzle and an adapter for installation. The black inlet nozzle to the optical sensor is specific to the instrument (not interchangeable to other T640x instruments). Please note, the final assembly differs slightly between the T640x ASC and the T640 option ASC.

1. Insert the black inlet nozzle through the center of the support collar into the top of the optical sensor, seating it tightly so that the nozzle's upper O-ring flange is flush with the upper surface of the sensor body. See figure 2.1.

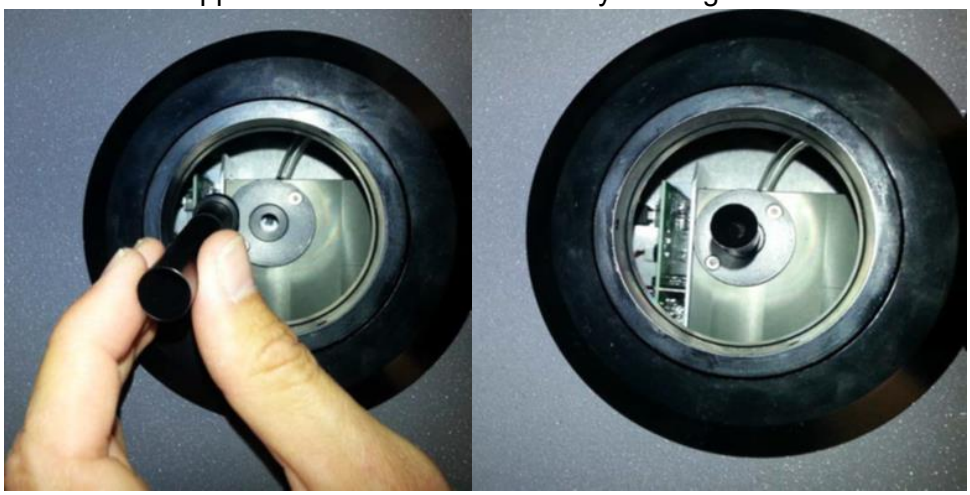


Figure 2.1 ASC Setup: Inlet Nozzle

2. Slide the aluminum adapter over the black inlet nozzle, ensuring its base is flush with the top of the optical sensor. See figure 2.2

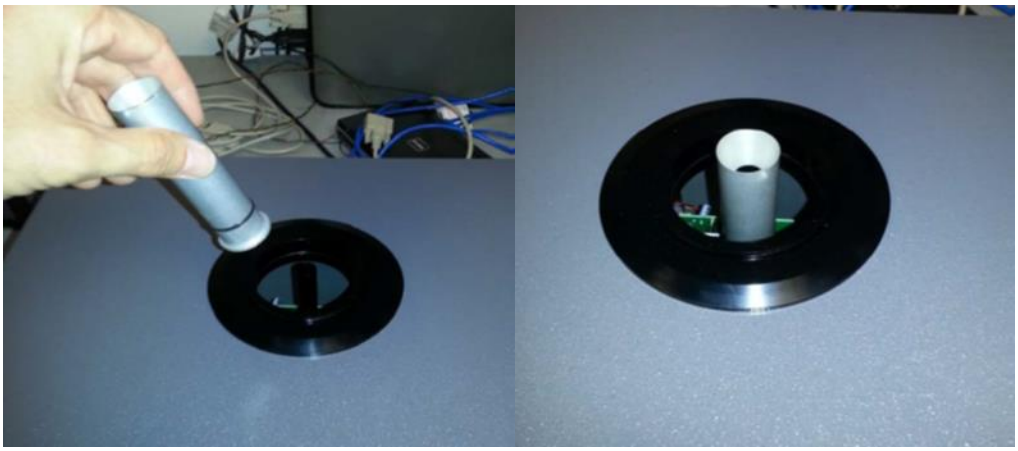


Figure 2.2 Aluminum Adapter

3. Assemble the inlet and the ASC as depicted in figure 2.3, ensuring the parts fit snugly with no gaps. Note that if the instrument is to be installed in a shelter with roof penetration, leave the inlet with the water collector off for the time being.

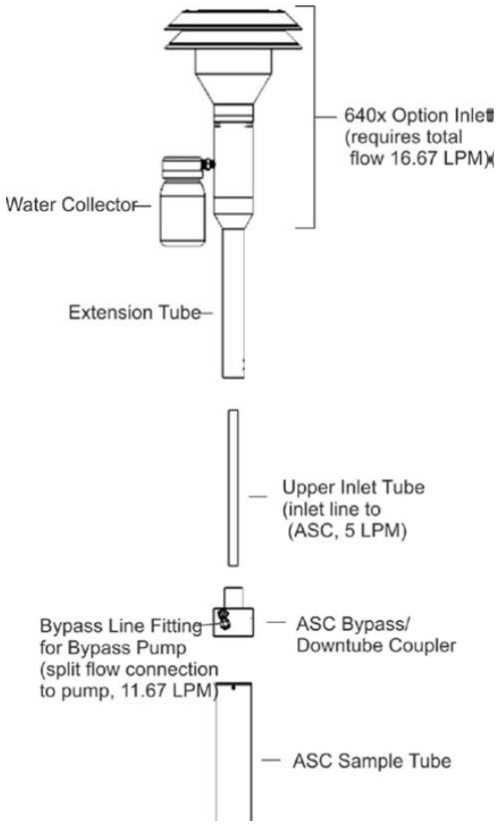


Figure 2.3 T640x Inlet and ASC Assembly

4. Lower the ASC into the support collar, ensuring it is fully seated and fits straight with no gaps. A leak here could produce flow problems that are not visible with leak check. Critical ASC is seated fully.

5. Plug the ASC wiring into the rear panel connector. See Figure 2.4.

2.4.3 Temperature Probe Connection

1. Plug the ambient temperature probe connector into its respective rear panel electrical port. See figure 2.4 below.

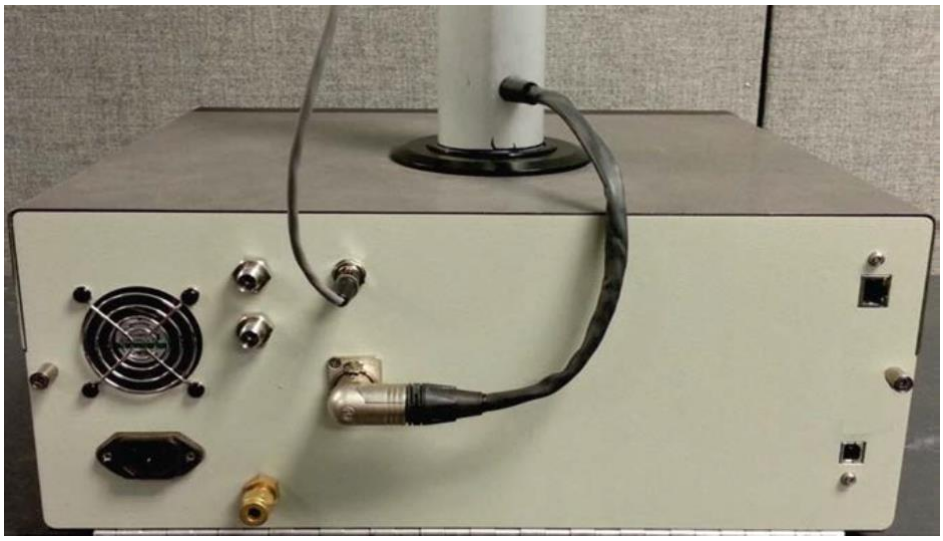


Figure 2.4 Back of Monitor

2. Route the probe outside and insert it into the solar shield, ensuring the solar shield maintains a vertical orientation.

2.4.4 Communications Interface Connections

For internet communication with the instrument, connect an ethernet cable from the analyzer's rear panel Ethernet interface connector to the on-site cable router/modem. The instrument can either be set up to report through a modbus connection to the 8832 data loggers, or can be direct polled by AirVision. Use the T640x manual for instructions on setting up an internal static IP address, and the AirVision manual on configuring AirVision for either data logger or direct polling options.

2.4.5 Outdoor Enclosure Installation

1. Install the enclosure in the location where the instrument is to run.
2. Measure the distances and clearances for the location of the T640x monitor and inlet. Following siting requirements of section 2.3 of this SOP.
3. Slide the ASC up through the port of the doghouse by several inches above where the monitor will sit and tighten the collar over the ASC such that it is held in place. Do not over tighten this will need to be adjusted later.

4. Install the 640x monitor on the shelf of the enclosure, confirming the black inlet nozzle and aluminum adapter are installed on the optical sensor (Step 2.4.2 above)
5. Once the monitor is lined up with the opening on the top of the enclosure, carefully untighten the collar holding the ASC so that it does not fall down on the monitor. Slide it down gently until it inserts into the opening.
6. The ASC should go into the T640x level and plumb, and is in completely when it is felt to bottom out onto the top of the optical sensor. This can be seen by lowing the front panel of the T640x and viewing the ASC touching the top of the optical sensor. Use the small allen wrench to tighten the set screw located on the black collar on the T640x monitor.
7. Plug the ASC connector into the proper fitting on the rear panel of the T640x. See Figure 2.5 below

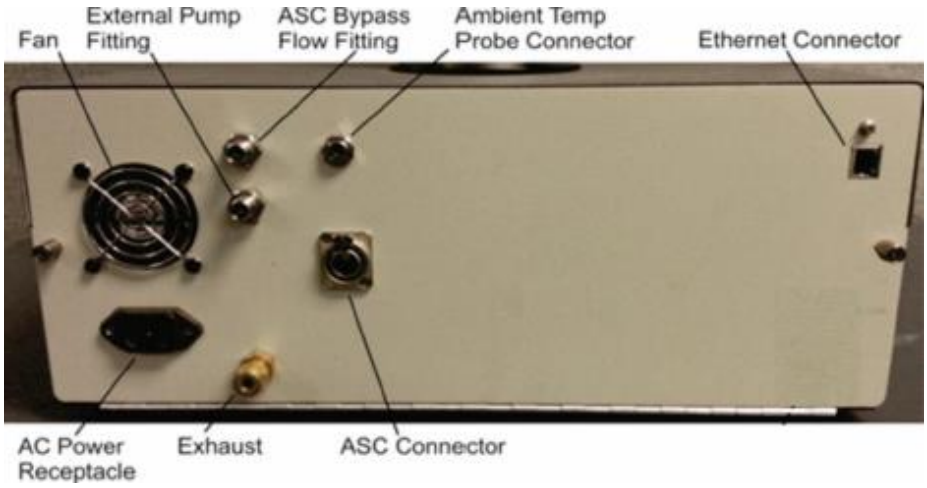


Figure 2.5 Rear Panel

8. Connect the power cable and ambient temperature sensor at the back of the instrument.
9. Using Swagelok ¼ fittings and quick connect fittings on the external DFU install the bypass line from the instrument though to top of ASC.
10. Connect the external pump using ¼ Teflon tubing and Swagelok fittings. And install similar to the exhaust to run outside the enclosure.
11. Power up the instrument and ensure it is running properly. Tighten up the collar around the ASC.
12. Apply clear silicone caulk around the rubber collar and ASC interface to seal it from weather.

3.0 Calibration

Before beginning a calibration, the operator shall complete a pre-calibration verification. Upon completion of a calibration, the operator shall perform a post calibration verification to ensure the calibration was successful. The pre-calibration verification should be recorded in the sampler log on the monthly verification sticky. The post- calibration verification should be

recorded on the calibration sheet in the sampler logbook. There are three checks of the monitor that can be calibrated. The checks that can be calibrated are conducted in the following order, as necessary:

1. Pressure sensor calibration;
2. Sample Flow calibration
3. Bypass flow calibration

The PMT adjustment is discussed separately in section 4.5 of this SOP.

3.1 Calibration Frequency

To ensure accurate measurements of PM concentrations, calibrate the T640 upon installation, annually, after major repairs, and upon failure of monthly verification or audit. Every effort should be made to perform calibrations on days of good stable weather. Annual maintenance calibrations should never be performed during unstable weather including, rain, wind, changing barometric pressure. Any post repair calibrations performed during non-ideal weather situations should be checked with an additional verification as soon as stable weather returns.

3.2 Equipment

- Streamline Pro (SLP) transfer standard or flow audit transfer standard.
- Streamline Pro or aneroid barometer (e.g., a climber's or engineer's altimeter), capable of accurately measuring ambient barometric pressure within ± 1 mm Hg resolution and referenced within ±5 mmHg to a barometer referenced to a NIST standard.
- Streamline Pro or thermometer capable of accurately measuring temperature over the range of - 30°C to +50°C (243 K to 323 K) to the nearest ± 0.1°C and referenced to an NIST thermometer within ± 0.5°C at least annually.
- $9/16^{th}$ wrench, and Swagelok cap
- Flexible rubber tubing to connect bypass line to flow transfer standard adapter.
- Logbook and “sticky” forms

Transfer standards or certified reference device such as the Streamline Pro, thermometers, barometers, or other flow meter must have a recorded certification date to NIST standard within one year. The make, serial number, and certification date shall be recorded in the sampler logbook with the calibration or verification for which the standard was used. Standards used for calibration must not be used for internal auditing for the device.

The calibrations below are recorded in the logbooks by recording the before and after verifications performed and an entry of calibration performed with date, time and operator’s initials. There is not a separate calibration form.

3.3 Pressure Calibration

No direct pneumatic connection to the instrument must be made to perform this calibration. The pressure measurement is for the ambient pressure where the instrument is operated.

A. Disable Data Logger: Press <ESC> 1-2 times for menu to appear. Press <Control L> to login in. Press and hold white control key and input the password “nerothecat”. Press enter. Press <Control C> <Enter> for configuration. Press <Control D> <Enter>for Data Channels. Press <Control M> <Enter>for disable/mark channel offline. Select channel and press <Enter> Repeat selection and enter for each channel. Channels to disable are PM2.5 and PM10 STP. <ESC> back to home menu. Press <Control D> <Enter> for Real Time Display followed by<Control L><Enter> for Large Text display. Press <Enter>. Reading should be visible and in red.
B. Turn on the streamline pro, or other certified pressure standard and allow time to equilibrate (2 minutes).
C. On the T640x monitor navigate to the Pressure Cal menu: >Calibration >Pressure Cal.
D. Compare the “Measured Pressure” on the screen to the certified pressure standard. See Figure 3.1 below for screen shot.

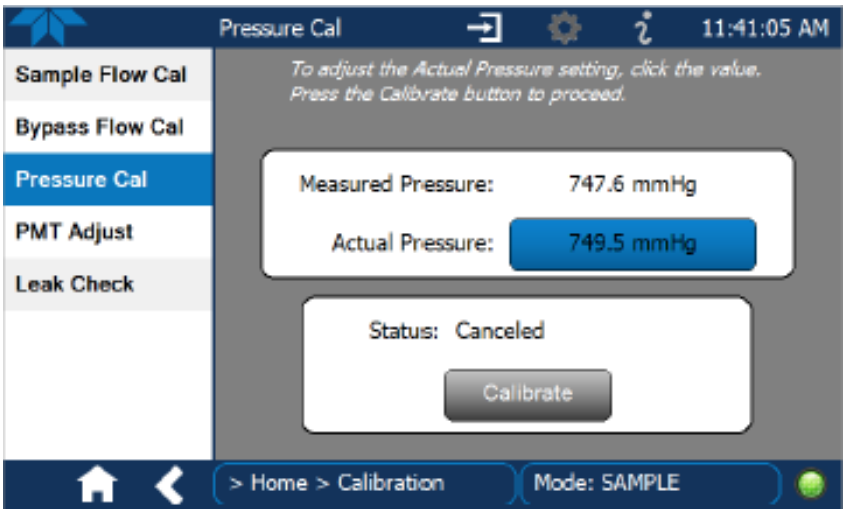


Figure 3.1 Pressure Calibration screen

E. Press the value button in the “Actual Pressure” field, enter the value measured by the pressure standard and press <enter>.
F. Press <Calibrate> button on the screen. The measured pressure value should change to closely match the actual pressure within a few seconds. Post calibration the measured pressure and pressure standard should match within ± 4mmHg. Repeat if necessary.
G. Note the calibration in the logbook, proceed to sample flow calibration.

3.4 Sample Flow Calibration

Following the pressure calibration conduct the sample flow calibration by navigating to the sample flow rate calibration menu. Flow calibrations should be conducted after an initial verification and the pressure calibration.

A. Verify the data logger is disabled (should be disabled from the pressure calibration performed in section 3.3.

B. Disconnect the bypass flow line from the side of the ASC. Ensure the line is not blocked and only pulling in ambient air. Be especially careful to keep it from setting in water or a dirty surface.
C. Cap the Swagelok fitting at the side of the ASC.
D. Turn on streamline pro (or other flow transfer standard and allow to equilibrate and zero. Make sure the temperature is stable and the flow and remote temperatures are within 2 degrees of each other. Verify that the streamline pro is set to use the remote temperature probe for flow calibrations. Remote temperature probe should be placed in the radiation shield of the monitor's ambient temperature probe.
E. Remove the PM10 inlet from the upper inlet tube.
F. Connect the flow standard to the top of the upper inlet tube.
G. Navigate to the sample flow rate calibration menu at > Calibration > Sample Flow Cal. See Figure 3.2 below.

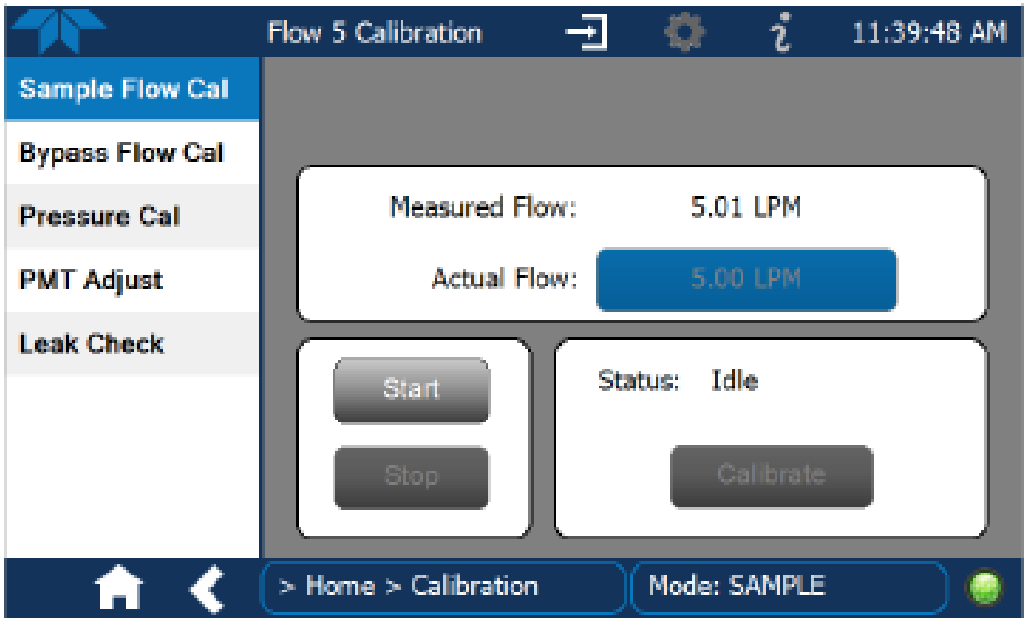


Figure 3.2 Sample Flow Calibration Screen

H. Allow at least one minute for the flow to re-stabilize.
I. Compare the "Measured Flow" on the screen (fig 3.2) to the flow transfer standard.
J. Press the "Start" button (which disables the internal data logging) and then "Actual Flow"
K. Enter the value measured by the flow standard and hit "Enter", then press the "Calibrate" button. The "measured flow" should change to closely match the "Actual Flow" within a few seconds.
L. Continue reading the flow rate standard and ensure the standard is within 2% of the measured flow. If it does not stabilize within 2% repeat the calibration, if stable and within 2% acceptance criteria press "Stop" to end the calibration.
M. Record in the monitor's logbook sample flow rate calibration complete with date, time and operator's initials.

N. Continue to Bypass Flow Calibration. If unnecessary to calibration bypass flow unscrew the Swagelok cap and reconnect the bypass line to the ASC.

3.5 Bypass Flow Calibration

A. Connect the flow transfer standard to the bypass line that was disconnected in the sample flow calibration above in section 3.4. Rubber tubing may be needed to connect the standard to the bypass line. Be careful not to damage the ferrule connection
B. Navigate to the bypass flow cal screen. Starting in the Calibration screen from above, select "Bypass Flow Cal" on the left. See Figure 3.3 below.
C.

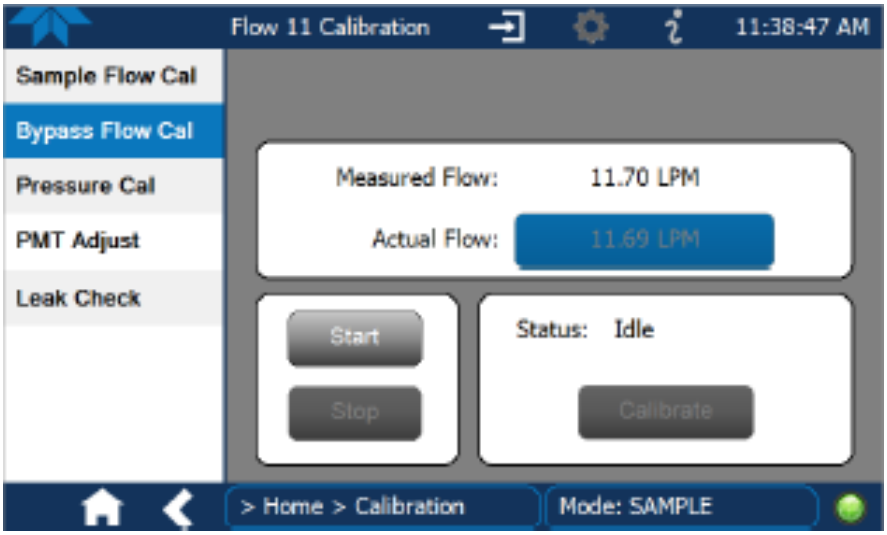


Figure 3.3 Bypass Flow Calibration Screen

D. Wait at least one minute for flow to stabilize.
E. Compare the "Measured Flow" on the screen (fig 3.3) to the flow transfer standard if the flows do not match press the "start" button.
F. Press the "Actual Flow" and enter the bypass flow rate value measured by the flow standard and hit "Enter". Then press the "Calibrate" button. The Measured Flow value should change to closely match the Actual Flow within a few seconds.
G. Continue reading the flow standard and ensure the standard is within 2% of the measured flow or appx (11.47-11.93 L/min). If outside this acceptance criteria repeat the calibration.
H. If reading in F is acceptable, record in the monitor logbook: date, time, and operator's initials.
I. Reattach the bypass line to the ASC.
J. Allow 1 minute for flows to stabilize and proceed to post calibration verification according 4.3 of this SOP.

4.0 Verification

There are five basic verifications performed on the T640x. Three of these verifications could result in calibrations if necessary. There is no adjustment for 2 of the verifications and failure would result in troubleshooting. Verifications should always be performed in the following order: Parameter Check, Leak Check, Pressure, Temperature and Flow check. Then quarterly follow with the PMT Verification.

4.1 Verification Frequency

To ensure accurate measurements of PM concentrations, calibrate the T640x upon installation, then perform verifications according to the frequencies in Table 4.1

Procedure	Frequency
Flow Rate (main & Total)	Monthly
Temperature	Monthly
Pressure	Monthly
Zero Test (Leak Check)	Monthly
PMT verification	Quarterly
Parameter Check	Weekly

Table 4.1 Verification Frequency Chart

4.2 Weekly Parameter Check

A weekly status check is performed to help proactively detect any changes that may affect data validity or monitor performance. Use the Weekly Stickies form (See figure 4.2 below) to record the weekly parameter check. Items reviewed in the weekly parameter check are also part of the Maintenance schedule outlined in section 5.1 of this SOP.

A. Time (DL): if using a data logger this record the difference (data logger time – Clock standard time). If time difference is approaching 2 min, adjust the data logger time to the clock standard time. If direct polling is implemented, and no data logger used, this time should be the difference of the monitor's clock - clock standard time.

B. On the T640x monitor dashboard screen, read and record the parameters on weekly logbook sticky (see figure 4.2 below). Those with acceptance criteria should be investigated if outside the acceptable range.

	Range		
Time (DL)	± 2 min of NIST		
Amb. Press.	mmHg		
Box Temp	0 to 60 ° C		
LED Temp	≤ 70 ° C		
Pump PWM	35 - 80%		
Ambient T	-40° C to 60° C		
Bypass Flow	11.08-12.25		
Sample Flow	4.75-5.25 LPM		
Sample RH	≤ 38%		
Sample T	≥ Amb T		
Valve PWM	35-85%		
Shelter Temp.	0- 50 ^{0}C	Min.	
(Reset)		Max.	
Record any alarms in logbook			

Figure 4.2 Weekly Parameter Check Sticky

C. Review the shelter temperature thermometer and record the minimum and maximum recorded and reset the thermometer upon completion.

D. From the dashboard review any alarms that may be present. Record in the logbook the alarm including the date and time the alarm was present. Clear the alarms, and note if any pop back up, as this would indicate the problem is still ongoing. The greater the detail provided, the better the data validator will be able to establish if the alarm has affected the quality of the data.

4.3 Leak Check (Zero Test)

The internal components of the T640x are not meant to be under vacuum. DO NOT close off a leak check adapter or cap the inlet while the pumps are running. The zero test checks for leaks without pulling a vacuum. The theory behind the test is that a HEPA filter is installed on the inlet, if the machine does not reach zero, then a leak must be present allowing particulate matter to reach the optical eye. Perform the leak test as follows.

A. Disable Data Logger: Press <ESC> 1-2 times for menu to appear. Press <Control L> to login in. Press and hold white control key and input the password “nerothecat”. Press enter. Press <Control C> <Enter> for configuration. Press <Control D> <Enter>for Data Channels. Press <Control M> <Enter>for disable/mark channel offline. Select channel and press <Enter> Repeat selection and enter for each channel. Channels to disable are PM2.5 and PM10 STP. <ESC> back to home menu. Press <Control D> <Enter> for Real Time Display followed by<Control L><Enter> for Large Text display. Press <Enter>. Reading should be visible and in red.

B. Remove the inlet head and fit the HEPA filter (attached to a PM2.5 Leak check adapter) on the sample port. Be sure the valve on the leak check adapter is open and the tubing is not kinked, to avoid forming a vacuum.

C. From the Home Screen select > Calibration> Leak Check> Start. See Figure 4.3 below.

Figure 4.3 Leak Check Screen

D. Observe the PM values and record the values after 5 minutes on the monthly verification sticky See figure 4.4 below. If data does not reach 0.0 but is in the range of 0.1-0.2, accept the data but troubleshoot to identify a leak. IF greater than 0.3, note in logbook data is suspect and troubleshoot.
   a. There may be a leak in the system above the optical sensor, check to make sure each connection is snug and o-rings are intact.
   b. The HEPA filter may be dirty, or leaking. Change and Repeat Test

Date:					Operator:			
Parameter check								
Sample T		≥ Amb T	Sample RH		≤ 38%	Logger Time		±2 mins
Box Temp		0 to 60 ° C	Pump PWM		35 - 80%	Shelter T Min		≥ 0°C
LED Temp		≤ 70 ° C	Valve PWM		35 - 85%	Shelter T Max		≤ 50° C
Verification								
	System Clock	Amb Temp	Amb Pres	Total Flow	Sample Flow	Leak Check		
System:							PM2.5	
Reference:							PM10	
Difference								
Acceptance Criteria	± 1min	≤ ± 2 ºC	≤±10 mmHg	± 4%	± 4%	< 0.2 µg/ L		
Standard Name/Typ			Standard ID:					

Figure 4.4 Monthly Verification Sticky

E. After any adjustments post troubleshooting made (changing filter, tightening connections etc) repeat test (B-D)

F. Once PM Values reach zero for each PM metric, disconnect the HEPA filter. Continue to 4.4 Pressure, Temperature, Flow Verification

4.4 Pressure, Temperature, Flow Verification

The monthly verification should be performed in the following order: pressure, temperature, total flow, and sample flow. The pressure measurements may be made with no direct pneumatic connection to the instrument, however this SOP details the most efficient process to measure all of the required parameters.

A. Logger should be disabled following Leak Check. Turn on streamline pro (or other transfer standard and allow to equilibrate and zero. Make sure the temperature is stable and the flow and remote temperatures are within 2 degrees of each other.

B. Place the streamline pro on the inlet. Remote temperature probe should be placed in the radiation shield of the monitor's ambient temperature probe.

C. From the dashboard record the system ambient pressure in mmHg and from the streamline pro record the reference Amb Pres on the Monthly Verification Sticky (Fig 4.4). Calculate the difference Monitor – Reference. The values must differ by less than ±10mmHg to pass.

D. From the dashboard record the system ambient temperature in degrees Celsius and from the streamline pro record the remote temperature on the Monthly Verification Sticky (Fig 4.4). Calculate the difference Monitor- Reference. The values must differ by less than 2 degrees Celsius.

E. From the dashboard (use arrow key to see page 2) record the system total flow in L/min and from the streamline pro record the flow. Be sure to use the arrow keys to select USE_RT so the flow will calculate using the remote temperature probe (which should still be placed in the radiation shield next to the monitor's ambient temperature probe. Record both values on the Monthly Verification Sticky (Fig 4.4) and calculate the percent difference [(Monitor-Reference)/ Reference] x 100%. Total flow should be within ± 4%.

F. Disconnect the bypass flow line from the side of the ASC. Ensure the line is not blocked and only pulling in ambient air. Be especially careful to keep it from setting in water or on a dirty surface.

G. Cap the Swagelok fitting at the side of the ASC. The streamline pro remains on top of the inlet. From the dashboard (page1) record the system main flow in L/min and from the streamline pro: record the flow on the Monthly Verification Sticky (Fig 4.4). Calculate the

percent difference [(Monitor- Reference)/ Reference] x 100%. Main Flow should be within ±4%.

H. If the total flow and main flow checks meet acceptance criteria, there is no need to verify the bypass flow. Remove the Streamline pro from the inlet, reconnect the bypass line, and replace the PM10 inlet head.

I. Allow 10 minutes for the machine to stabilize and then re-enable the logger. Press <ESC> 1-2 times for menu to appear. Press <Control L> to login in. Press and hold white control key and input the password “nerothecat”. Press enter. Press <Control C> <Enter> for configuration. Press <Control D> <Enter>for Data Channels. Press <Control E> <Enter>for Enable/mark channel online. Select channel and press <Enter> Repeat selection and enter for each channel. <ESC> back to home menu. Press <Control D> <Enter> for Real Time Display followed by<Control L><Enter> for Large Text display. Press <Enter>. Reading should be visible and in green.

J. If the total flow or the main flow checks do not meet acceptance criteria, check the Bypass Flow by connecting the bypass flow line to the streamline pro adapter using a length of rubber tubing. From the dashboard record the system bypass flow in L/min and from the streamline pro record the flow in the logbook. This measurement is for diagnostics of flow problems identified by failing total or sample flow rates.

K. After completing the bypass flow check, continue to trouble shoot problem, and repeat all verification points, and proceed to calibration if necessary. Calibration should be the last option.

***** If the monitor is installed in a shelter where the entire ASC is inside and an extension is used for the inlet, then the bypass flow check would require that you unhook the external temperature probe, and use a shorter probe inside during the bypass flow check only. Verify the shorter temperature probe as well.

4.5 Quarterly Span Dust Check or PMT Calibration

Quarterly a SpanDust™ check of the Particle Sensor should be performed. This check may result in the adjustment or calibration of the optics due to drift caused by contamination. SpanDust™ checks should not be performed in rainy or >90% ambient humidity. The quarterly span dust check should be immediately after a routine monthly verification. They should also be performed after an optics cleaning to ensure accuracy.

A. Disable Logger (according to 3.3.A)
B. Navigate to the PMT Adjust screen: Home> Calibration> PMT Adjust.
C. Press the Start button on the screen to suspend normal data acquisition and start the adjustment process.

D. Remove the PM10 inlet.
E. Prepare the SpanDust™ bottle by uncapping the “air intake” tubing on the cap of the bottle. Leave the other side of the silicone tube attached to the cap.
F. Attach the free side of the silicone tube to a PM2.5 leak check adapter. See photo Figure 4.5 below. Ensure that the silicone tube fits snugly over the leak check adapter. Do not allow the bottle to hang, as it could dislodge the tubing.

Figure 4.5 SpanDust™ connected to leak check adapter

G. Place the leak check adapter on the inlet downtube and give the bottle **a gentle tap** to barely agitate the contents for dust to be pulled into the sensor (DO NOT FLIP THE BOTTLE UPSIDE DOWN).

H. Allow approximately 30 seconds for the peak channel reading on the screen to respond. See figure 4.6 below for sample screen shot.

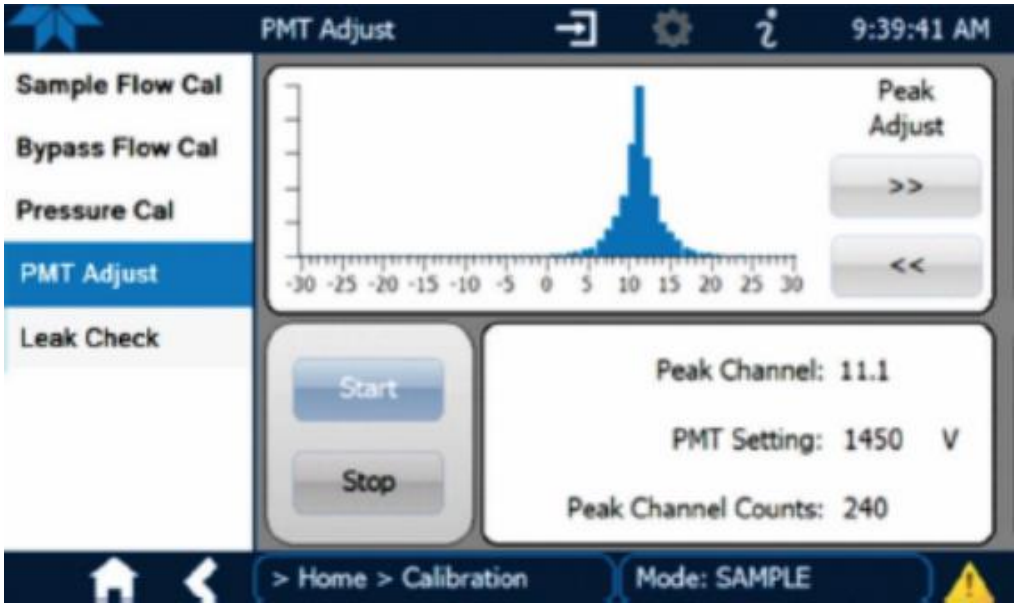


Figure 4.6 PMT Adjust Screen

I. Record the maximum Peak Channel, PMT setting, and Peak Channel Counts in the monitor's logbook using the Quarterly span dust sticky, see Fig 4.7 below.

Quarterly	**Span Dust™** (+/- 0.5 value on Span Dust™ bottle)	Peak Ch. =				Date Performed:
		PMT Setting =				
		Peak Ch. Counts =				
	Inlet	1. Fully Clean PM_{10} Inlet (unscrew top)				
Quarterly	**Span Dust™** (+/- 0.5 value on Span Dust™ bottle)	Peak Ch. =				Date Performed:
		PMT Setting =				
		Peak Ch. Counts =				
	Inlet	1. Fully Clean PM_{10} Inlet (unscrew top)				
Quarterly	**Span Dust™** (+/- 0.5 value on Span Dust™ bottle)	Peak Ch. =				Date Performed:
		PMT Setting =				
		Peak Ch. Counts =				
	Inlet	1. Fully Clean PM_{10} Inlet (unscrew top)				
Quarterly	**Span Dust™** (+/- 0.5 value on Span Dust™ bottle)	Peak Ch. =				Date Performed:
		PMT Setting =				
		Peak Ch. Counts =				
	Inlet	1. Fully Clean PM_{10} Inlet (unscrew top)				
* Record 2nd/3rd span dust readings only if adjustement made						

Figure 4.7 Quarterly SpanDust™ Sticky

J. If the Peak Channel is acceptable, ± 0.5 of SpanDust™ value on bottle, then press Stop, Span Check is complete.
K. If Peak Channel reading does not meet acceptance criteria, then press STOP, remove leak check adapter, and repeat test from F.
L. If after repeat test the Peak Channel reading is not within acceptance criteria, then adjust the PMT Setting by pressing the Peak Adjustment left or right to center the Peak voltage. Tap SpanDust™ bottle when finished adjusting to read again starting back at H above.
M. If a SpanDust™ adjustment is made, schedule an optics cleaning as soon as practical, unless the adjustment was made in the post cleaning check.

5.0 Maintenance

Preventative maintenance is defined as a program of positive actions aimed to prevent failure of monitoring and analytical systems. The overall objective of a routine preventive maintenance program is to increase measurement system reliability and to provide for more complete data acquisitions. This section outlines general maintenance procedures for the APTI T640x. Always check the manufacturer's most recent manual. Records shall be maintained and all maintenance activities must be recorded in the monitor's logbook. Entries will reflect the history of maintenance, part replacements, and quality assurance/control checks performed after maintenance.

5.1 Maintenance Schedule

Preventative maintenance includes inspections, cleaning verifications and calibrations. Table 5.1 below provides a list of actions and their frequencies. Many of these actions are detailed in the Calibrations and Verification sections of this SOP (3.0 and 4.0)

Maintenance or Check Action	Tolerance	Frequency
Check Pump Performance	Pump PWM[1] value < 80% Valve PWM1 value < 85%	Weekly (Parameter check)
Check for leaks with zero filter	Acceptance criteria is 0.0 – 0.3 µg/m3 Action level is 0.1 to 0.3 µg/m3 (for each PM metric)	Monthly
Clean PM10 inlet	NA	Quarterly
Clean PM10 Well	NA	Monthly
Check/Adjust PMT with SpanDust™ (measured peak, limit value displayed on bottle +/- 0.5)	Stated value on Span Dust Bottle +/- 0.5 (e.g., 11.3 with a tolerance of 10.8 to 11.8)	Quarterly. Avoid over performing this procedure. If problems persist, wait 10 minutes and retry.
Ambient Pressure	+/- 10 mm Hg	Monthly
Ambient Temperature	+/- 2 C	Monthly

[1] Pulse width modulation (PWM); proportional, integral, differential (PID) loop

Flow rate verifications (see below)		
Total flow: 16.67 L/min	±4% of standard compared to current reading on T640x. ±5% of design flow	Monthly
Sample flow: 5.0 L/min	+/- 5% of standard compared to current reading on T640x; (e.g., 4.75 – 5.25 L/min if T640x reads 5.00).	Monthly
Bypass Flow: 11.67 L/min	+/- 5% of standard compared to reading on T640x; (e.g., 11.12 – 12.29 L/min if T640x reads 11.7).	As needed if total or sample flow does not meet criteria. Use same tolerance as total and sample flow.
Inspect and clean optical chamber and relative humidity/temperature (RH/T) sensors	NA	Every six months or as needed, e.g., high dust load
Change Disposable Filter Unit (DFU) for 5-L/min sample flow and 11.67 L/min bypass flow (if installed)	NA	Annually or when Pump PVM value approaches 80% or valve PWM approaches 85%
Clean sample tubes	NA	Every six months or each time optics are cleaned

Figure 5.1 Maintenance Schedule

5.2 PM10 Well

Cleaning the PM10 well should occur each month during the monthly verification process. Perform this during the leak check portion of the verification. This will ensure the HEPA filter is keeping the machine clear of contaminants.

A. With the sampling inlet removed from the down tube, unscrew the top of the sampling inlet from the base of the inlet to expose the well.
B. Using a moist lint free towel or wipe (with alcohol) clean the inside base.
C. Using a cotton tipped applicator, clean out each of the three nozzles.
D. Inspect the water trap, remove any insects, spider webs, and moisture. Note anything unusual in the logbook.
E. Allow components to dry, use canned air to blow through the three nozzles to ensure they are dry.
F. Inspect and clean base connection to the downtube with the lint free towel or wipe.
G. Check and if needed replace the o-rings and grease them with vacuum grease.
H. Carefully reassemble the top and base of the sampling inlet, but do not overtighten. Be sure to ensure the o-rings are seated properly and are not damaged when closings.
I. Return to the sampling line when the verification procedure is complete.

5.3 PM 10 Inlet

Cleaning the PM10 Inlet should occur at least quarterly. As part of regular maintenance and after/during a verification procedure. As such data logger should be disabled following procedure in 3.3.A. It is recommended to remove the inlet, and replace with a clean one in the

field during the quarterly maintenance, then proceed to clean the dirty inlet back at the office. However, the procedure below should be followed if cleaning in the field.

A. Power off the internal pump from Setup->Vars->Pump Control. Press edit and then off to turn off the pump.
B. Remove the inlet from the downtube and place the leak check adapter with HEPA filter on the inlet to minimize contaminants entering the monitor.
C. Unscrew the top of the sampling inlet from the base to expose the PM10 well. Clean the PM10well as described in Section 5.2 above.
D. Disassemble the sampling inlet (four screws on the underside of the inlet base plate)
E. Carefully remove dust, debris, and insects from the inside of the inlet and filter screen. When performed at the office use water and mild detergent, when performed in the field alcohol wipes can be used.
F. Dry all components thoroughly with a clean cloth and blow canned air through the nozzles.
G. Check and replace if needed the O-rings located on the outside and inside of the base plate, and grease them.
H. Reassemble the sampling inlet, sliding the baseplate back, lining up the screw holes. The screws should be reinstalled using hand tight pressure.
I. Return inlet onto the downtube.
J. If disabled power up the pumps from Setup->Vars-> Pump Control Menu, Press edit and then auto to turn the pumps back on.
K. Allow a minimum of 10 minutes for the system to return to reliable operation before re-enabling the logger following steps in 4.4.I

5.4 Disposable Filter Units (DFUs)

There are two DFUs on the T640x, one internal filter for the 5.0L/min pump and one external for the 11.67 L/min pump. It is recommended that both be changed at the same time, however not required. Follow maintenance schedule in Table 5.1 for frequency. A verification according to section 4.4 should be completed before changing a filter.

5.4.1 Internal Pump Filter

A. Data logger should remain disabled from the verification procedure.
B. Power off the internal pump from the control menu: Setup-> Vars->Pump ->Edit-> Off-> Done.
C. Pull open the instrument's front panel, using the front panel finger grips. See Figure 5.2 below.

Figure 5.2 Front Panel

D. Write today's date on the new filter with a sharpie.
E. Noting the orientation, remove the old DFU by detaching the pneumatic quick connect fittings and replace with a new DFU matching the orientation. Ensure the quick connect fittings are secure. The orientation **does** matter in correct operation and filter capacity. See figure 5.3 below

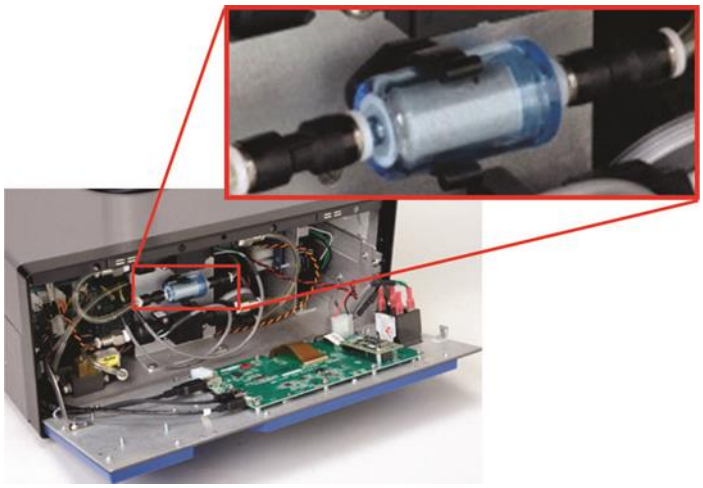


Figure 5.3 Internal DFU

F. Power on the internal pump from the control menu – Setup->Vars->Pump->Edit->Auto-> Done
G. Review the dashboard for the sample flow reading as expected (approx. 5L/min) and the Pump PWM is as expected (35-50%)
H. Proceed to the External Pump filter, if finished with maintenance proceed to complete post maintenance verification according to SOP section 4.0

5.4.2 External Pump Filter

A. Data logger should remain disabled from pre maintenance verification procedure.

B. Unplug or turn off the electrical switch (if available) to the external pump. Preferred to turning off the machine as a whole.
C. Write today's date on the new DFU filter to be installed.
D. Noting its orientation on the bypass line, detach the filter from the quick connect fittings and replace it with a new filter. See Figure 5.4 below.

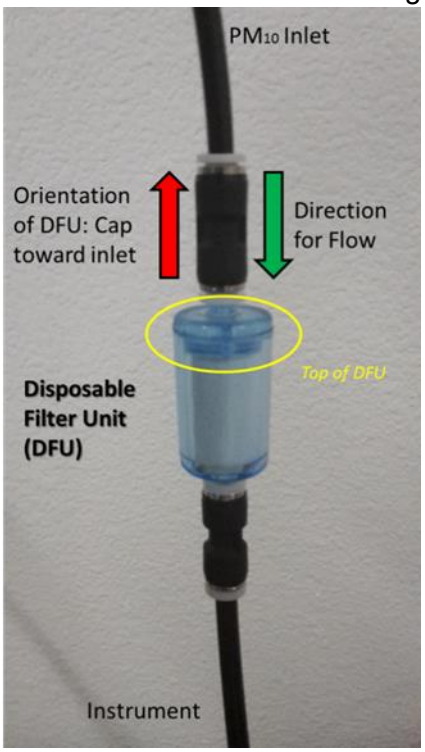


Figure 5.4 External DFU orientation

E. Ensure the filter is seated snugly with no gaps.
F. Plug the external pump's electrical cord back in.
G. Review the Dashboard to ensure the bypass flow is reading as excepted (approx. 11.67) and the Valve PWM has dropped and in range of (35-50%)
H. Proceed to additional maintenance or perform post maintenance verification according to section 4.0 of this SOP.

If a new DFU filter is placed in the instrument and the applicable Pump or Valve PWM does not return to near where it was reading when the filters were previously new then troubleshoot by inspecting all sample lines looking for SpanDust™ or anything else that may be affecting the flow. See Section 6.0 of this SOP for additional troubleshooting guidance.

5.5 Optical Chamber and RH/T Sensor

Cleaning the optical chamber and sample lines connecting to the chamber should be performed at least every 6 months and more frequently if heavy PM load or issues are suspected. IF a SpanDust™ calibration is performed, it is suggested the Optical Chamber cleaning schedule be moved up to as soon as practical. Optical chamber cleaning will be performed in the office laboratory clean room. Carefully consider and plan for time for a pre-removal verification, optics

cleaning at the office, and return to field verification. Every effort should be made to accomplish all tasks within one day. A PMT SpanDust™ verification/calibration will be performed upon reinstallation in the field, so the weather should not be rainy or foggy. Helpful hint: take a few photos with a smart phone or tablet prior to disconnecting any lines inside the monitor, to ensure you get everything hooked back up correctly in the end.

A. Power down the monitor and unplug. (Data logger should still be disabled from verification)
B. Power down the bypass pump.
C. Disconnect the ASC power line and the ambient temperature probe connection from the back of the instrument
D. Disconnect the bypass and vent lines from the back of the instrument.
E. Disconnect the ethernet cable.
F. Remove the PM10 inlet and extension tube.
G. Loosen the set screws where the ASC meets the monitor.
H. Loosen the roof coupler and slide the ASC up carefully lifting it up from the monitor chassis.
I. Tighten the roof coupler to hold the ASC suspended above the monitor. Remove the monitor from the shelter and bring into the office laboratory clean room.
    a. Reattach the extension tube and PM10 inlet (or leak check HEPA filter) to keep dust and debris from entering ASC.
    b. Transporting the monitor -Use the Red rubber cap to cover the inlet during transportation
    c. The aluminum adapter may need to be removed for cap to fit. Be extremely careful with the adapter and the black inlet nozzle that neither gets dented nor damaged.
J. In the office remove and clean the aluminum adapter and black inlet nozzle. (alcohol q-tips) Check o-rings for cracks, breaks and replace as necessary. DO NOT use alcohol on the o-rings as it can speed up the drying and cracking. Set aside to dry.
K. Unscrew the monitor lid and lift lid off the monitor chassis. Take those PHOTOS of the sample lines now!
L. Disconnect sample lines from the ASC support.
M. Remove the ASC support, which straddles the sensors. Four Screws secure the support to the floor of the instrument. A long reaching screw driver is helpful. See figure 5.5 below.

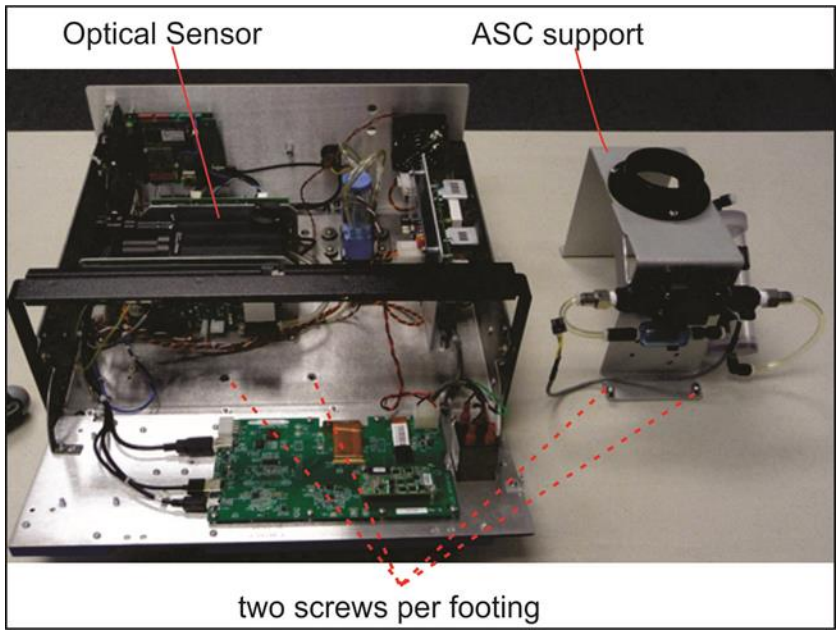


Figure 5.5 Inside the Monitor

N. Locate the optical cell, the cup at the bottom of the optics chamber and its tubing, and the Relative-Humidity and Temperature (RH/T) sensor. See figure 5.6 below.

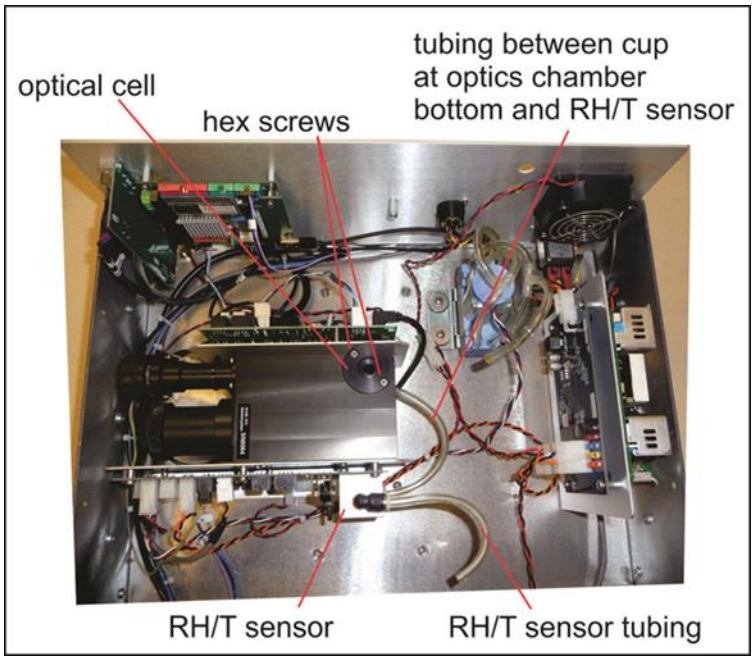


Figure 5.6 Location of Optics Chamber

O. Remove the optical cell from the optics chamber, and remove the cup, including its tubing from the chamber bottom. Detach the RH/T sensor tubing from the DFU. It is

essential that all components are disassembled before blowing air into the chamber. See Figure 5.7 below

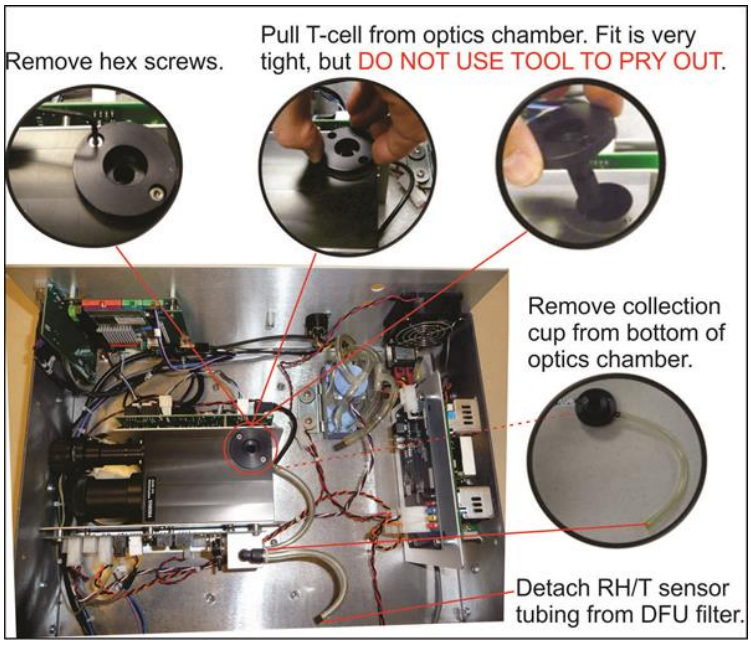


Figure 5.7 Optical Chamber Disassembly

P. Clean the optics chamber's interior surfaces, including windows with a LINT-FREE-CLOTH. See figure 5.8 below.

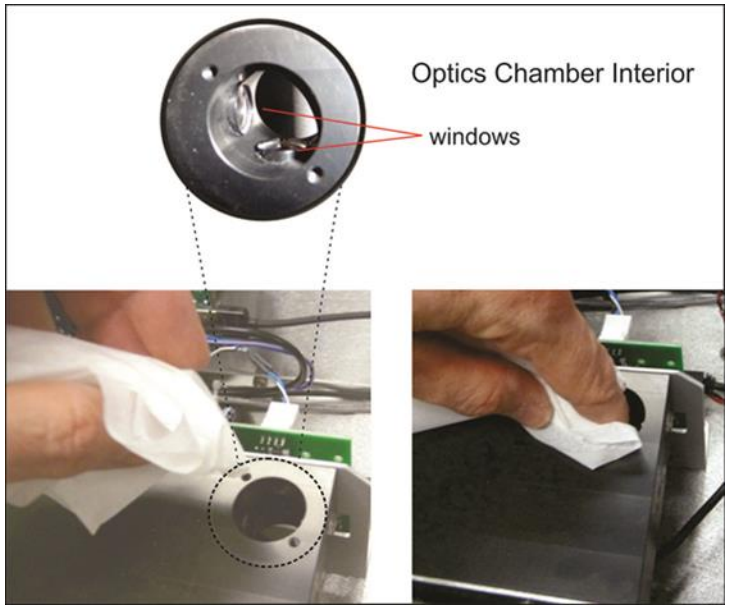


Figure 5.8 Optic Chamber Window

Q. Used canned air (specific for dusting electronics) to blow any dust and debris from the optics chamber, from the cup, the bottom of the optics chamber, and from tubing. See figure 5.9 below.

Figure 5.9 Dusting

R. When cleaning has been completed, reassemble the optics chamber components; reinsert the cup to the bottom of the optics chamber, reconnect the tubing from the cup to the RH/T sensor and from the sensor to the DFU filter. Reinstall the ASC support and reconnect all power and flow lines and black inlet nozzle.
S. Secure the lid of the monitor and Place red rubber cap on the inlet.
T. Return the monitor to the field, Proceed to Sample line cleaning.

5.6 Sample Line

Do not completely block a sample tube while still under vacuum as the sample flows should never be blocked while the unit is operating. This is performed with the unit and pumps off immediately after the optics cleaning.

A. Prepare a length of Teflon tubing with a cut on one end to attach a lint free cloth to.
B. Run the Teflon tubing with the cloth attached through the tubes.
    a. Clean both the extension tube and the upper inlet tube that sit directly above the ASC.
    b. Clean the ASC.
    c. Use canned air (for electronics) to blow out any additional dust and dry the interior of the tubes.
C. Install in shelter and line up. Reinstall the Aluminum adapter over the inlet nozzle.
D. Slowly release roof coupler and slide the ASC back in place on the monitor chassis. Tighten the roof coupler on the shelter and the set screws where the ASC meets the monitor.
E. Reconnect all the pump lines, Ethernet cables, ASC power, Temperature probe connection and electrical plug
F. Power on the bypass pump and monitor.
G. Review the dashboard to ensure all parameters are operating as expected. Clear alarms after reboot has stabilized.
H. Perform a Leak Check (Section 4.3 of this SOP)
I. Perform a PMT sensor check with the SpanDust™ (Section 4.5 of this SOP)

6.0 Operation and Trouble Shooting

6.1 Operation

Upon start up, sampling begins. Allow a minimum of 20 minute warming period for reliable readings. This should provide sufficient time for the LED temperature to stabilize (~10 minutes) and then another 10 minutes for the algorithm to incorporate the data at a stable LED temperature. Track LED temperature from the dashboard to ensure stability of the instrument. This warm up period should be applied to data validation post power outage or other interruption of monitoring process. 10 minutes is sufficient time for stabilization after flow verifications and leak check interruptions.

6.2 Troubleshooting and Service

The T640x has alerts to notify the operator when system components or values are outside operable ranges. To review Alerts, select Alerts from the home screen. Alerts shall be documented in the logbook during the weekly parameter check and before any verification procedures. After documented clear alerts by selecting >Alerts>Select All> Clear Selected. Return to the home screen.

During maintenance and verification procedures it is normal for alerts to pop up. The following alerts do not need to be recorded in the logbook if they occur during the performance of maintenance or verification procedures. However, if after the procedure is complete, the alerts are cleared, and if they reoccur upon returning to the home screen, they should be recorded and investigated.

6.2.1 Fault Diagnosis

The Alerts log is a useful tool in diagnosing problems. Table 6.1 below lists probable alerts that are triggered by faults, a description of event, and troubleshooting suggestions.

Alert	Description	Troubleshooting
System Reset	Warning raised when system is reset	Check for power outage, and electrical connections
Sample Flow High	The sample flow is greater than 5.25L/min	Check pneumatic fittings, re-calibrate flow
Sample Flow Low	The Sample Flow is less than 4.75 L/min	Check pneumatics. Check for blockages. Re-calibrate flow.
Bypass Flow High	The Bypass Flow is greater than 12.25 L/min	Check pneumatic fittings. Re-calibrate flow.
Bypass Flow Low	The Bypass Flow is less than 11.08 L/min	Check pneumatics. Check for blockages. Re-calibrate flow.
Sample RH High	The Sample RH is above the set point	Check if ASC is plugged in. Check control board if ASC control LED is illuminated. Check if water is in the sensor.

Check LED	If the LED temperature is equal to the box temperature, the LED may be OFF	Cycle power. Call Teledyne-API Tech Support.
Check PMT	The PMT HV setting is out of range (800 – 2200)	Check Sensor with SpanDust™. Perform an optical chamber cleaning. Call Teledyne-API Tech Support.
Sample Flow Slope OOR	The Sample Flow Calibration Slope is Out Of Range (OOR)	Check pneumatics for leaks. Re-run flow calibration. Call Teledyne-API Tech Support.
BYPS Flow Slope OOR	The Bypass Flow Calibration Slope is Out Of Range	Check pneumatics for leaks. Re-run flow calibration. Call Teledyne-API Tech Support.
Check Int Pump	Check the internal pump if the PWM is >80	Check pneumatics for blockages. Check pneumatics for leaks. Check flow calibration. Replace pump.
Check Ext Pump	Check the external pump and/or bypass flow control valve if valve PID >85	Check pneumatics for blockages. Check pneumatics for leaks. Check external pump. Check flow calibration. External pump, or bypass flow control valve may need replacing.
Sample Temp Warning	Sample Temperature Warning (>60)	Check ASC (is it latched ON?) Ensure proper climate and ventilation for instrument.
Box Temp Warning	Box Temperature Warning (>60)	Ensure proper climate and ventilation for instrument.
AMB Press Slope OOR	Ambient Pressure Calibration Slope is out of range	Check calibration (make sure units match calibration device). Replace pressure sensor. Call Teledyne-API Tech Support.

Figure 6.1 Alerts and Recommendations

6.2.2 Flow Issues

If a flow auditing device indicates any problems with flow rates, check to ensure the following:

- all connections are seated tightly and evenly (no gaps);
- the inlet is not clogged or blocked;
- the pumps are running and are within their PWM range (35% - 80%);
- internal tubing appears free from SpanDust™ or any other restricting debris;
- the DFUs appear reasonably clean and changed within last 12 months;

- the flow audit device is operating properly (e.g., is the unit appropriately charged).

After making any adjustments and verifying that the flow is still out of limits, run a flow rate calibration and recheck the flow rate. If problems persist, contact Teledyne-API Technical Support for assistance.

6.3 Quality Control and Corrective Actions

Minor problems that affect a few hours or a single day at a site shall be noted in the QA binder during data validation (see Section 8.0). when failed audits, verifications multiple down days for service, or other significant event that affects multiple run days occurs, a “Corrective Action Report” (Appendix Figure 10.1) should be completed by the Operator, Quality Assurance Officer, or Air Monitoring Program Manager. This report shall detail the issue, the actions taken, and quality control procedures performed. Additionally, this report should include the last passing QC check.

Data Quality Indicator	Frequency	Objective	Corrective Action
Flow verification	**Monthly**	**±3.5% of transfer standard ±4.0% of design value**	**Leak Check Document actions Perform Calibration**
Temperature Verification	**Monthly**	**±2°C**	**Investigate Check damage/ blockage of fan. Document actions Recalibrate**
Pressure verification	**Monthly**	**±10 mmHg**	**Investigate Document actions Recalibrate**
Quarterly Internal Audit	**4 times a year**	**±4% of transfer standard ±5 % of design value**	**Leak Check Document actions Perform Calibration**
Leak check	**Monthly**	**Pass**	**Investigate, Check seals, O-rings. Replace or lubricate as necessary. Repeat test.**
Calibration	**At installation, post-major repair, after failed verification,)**	**Operation Manual**	**Contact Equipment Manufacture**

Table 6.2 Quality Control and Corrective Actions

7.0 Audit

The most recently updated SOP {between the monitor’s SOP (this SOP) and the Audit SOP} should be used for auditing procedures and forms. Use form field sheet SP (Appendix Figure 10.2). Audit equipment to be used and certification acceptability are outlined in Table 7.1 below. Audit equipment shall be separate from equipment used for verifications and calibrations.

Equipment	Recertification	Acceptable Range
Flow rate Transfer Std	1/yr	±2% of NIST Traceable Std

Field Thermometer	1/yr	±0.1°C resolution, ±0.5°C Accuracy
Field Barometer	1/yr	±1mmHg resolution, ±5mmHg accuracy
Clock	N/A	Cell phone updated to towers acceptable

Table 7.1 Audit Equipment

7.1 Field Check

The Auditor shall inspect the PM10 inlet to ensure it is clean and properly maintained. The Auditor shall perform or observe the operator complete the following quality control measures and then ensure the results are within the corresponding limits according to table 7.2.

Measure	Acceptance Criteria
Time	±1 minute
Ambient Temperature	±2°C
Ambient Pressure	± 10mmHg
Sample Flow Rate	±4% of transfer standard
Total Flow Rate	±4% of transfer standard

Table 7.2 Audit Acceptance Criteria

The internal components of the T640x are not meant to be under vacuum. DO not close off a leak check adapter or cap the inlet while the pumps are running. The zero test checks for leaks without pulling a vacuum. The theory behind the test is that a HEPA filter is installed on the inlet, if the machine does not reach zero, then a leak must be present allowing particulate matter to reach the optical eye. Perform the leak test as follows

A. Time should be compared to audit clock and recorded. If outside limits notate and continue with audit Operator should update clock after audit is completed.

B. Disable Data Logger: Press <ESC> 1-2 times for menu to appear. Press <Control L> to login in. Press and hold white control key and input the password “nerothecat”. Press enter. Press <Control C> <Enter> for configuration. Press <Control D> <Enter>for Data Channels. Press <Control M> <Enter>for disable/mark channel offline. Select channel and press <Enter> Repeat selection and enter for each channel. Channels to disable are PM2.5 and PM10 STP. <ESC> back to home menu. Press <Control D> <Enter> for Real Time Display followed by<Control L><Enter> for Large Text display. Press <Enter>. Reading should be visible and in red.

C. Remove the inlet head and fit the HEPA filter (attached to a PM2.5 Leak check adapter) on the sample port. Be sure the valve on the leak check adapter is open and the tubing is not kinked, to avoid forming a vacuum.

D. From the Home Screen select > Calibration> Leak Check> Start. See Figure 7.3 below.

Figure 7.3 Leak Check Screen

E. Observe the PM values and record the values after 5 minutes on the Field Sheet SP. 0.0-0.2 is passing, however anything over 0.0 should be noted to the operator for review.

F. Turn on streamline pro (or other transfer standard and allow to equilibrate and zero. Make sure the temperature is stable and the flow and remote temperatures are within 2 degrees each other.

G. Place the flow transfer standard on the inlet (if using the tetraCal be sure the correct venturi for the flow evaluated is installed). The remote temperature probe should be placed in the radiation shield of the monitor's ambient temperature probe.

H. From the dashboard record the system ambient pressure in mmHg and from the pressure standard record the reference Amb Pres on the Field Sheet SP. Calculate the difference Monitor – Reference. The values must differ by less than ±10mmHg to pass.

I. From the dashboard record the system ambient temperature in degrees Celsius and from the temperature standard record the remote temperature on the Field Sheet SP. Calculate the difference Monitor- Reference. The values must differ by less than 2 degrees Celsius.

J. From the dashboard (use arrow key to see page 2) record the system total flow in L/min and from the flow standard record the flow. Be sure to use the remote temperature for calculating flow on the transfer standard and the correct venturi for expected flow rate of 16.7L/min. Record both values (system and reference) on the Field Sheet SP under Total Flow and calculate the percent difference [(Monitor- Reference)/ Reference] x 100%. Total flow should be within ± 4%

K. Disconnect the bypass flow line from the side of the ASC. Ensure the line is not blocked and only pulling in ambient air. Be especially careful to keep it from setting in water or dirty surface.

L. Cap the Swagelok fitting at the side of the ASC. The Flow standard remains on top of the inlet. From the dashboard (page1) record the system main flow in L/min and from the flow

standard record the flow on the Field Sheet SP. Again, if using the TetraCal the venturi may need to be changed for lower flow. Expected main flow is 5L/min. Calculate the percent difference [(Monitor- Reference)/ Reference] x 100%. Main Flow should be within ±4%.

M. Record the results in the instruments logbook using the Audit Stickies.

N. Allow 10 minutes for the machine to stabilize and then re-enable the logger. Press <ESC> 1-2 times for menu to appear. Press <Control L> to login in. Press and hold white control key and input the password “nerothecat”. Press enter. Press <Control C> <Enter> for configuration. Press <Control D> <Enter>for Data Channels. Press <Control E> <Enter>for Enable/mark channel online. Select channel and press <Enter> Repeat selection and enter for each channel. <ESC> back to home menu. Press <Control D> <Enter> for Real Time Display followed by<Control L><Enter> for Large Text display. Press <Enter>. Reading should be visible and in green.

7.2 Evaluating the results

If a parameter value is outside the acceptance criteria, the Auditor shall make note of the last successful verification from the instrument log book. The Operator shall investigate the cause of the deficiency and follow current SOP procedures for proper corrective action. All information will be forwarded to the QAO and AMPM for evaluation

The Auditor shall review field sheet PM SP for completeness for each sampler. The data shall be entered into the Quarterly Audit Workbook on Special Purpose Sheet (See Appendix Figure 10.3). The Auditor prepares a summary of the audit including any suggested corrective action. The summary should include any borderline results which required operator intervention to correct onsite but must include any results outside the acceptance criteria. See Section 6.3 of this SOP for quality control and corrective actions.

8.0 Data Validation and AQS Entry

The validation and AQS entry protocol in this section is a guide for the operator in initial quality assurance. Additional review and statistical analysis is completed by the QAO and those evaluations are not included in the SOP. Please refer to the QAPP and QMP for additional information on verifying data quality. Table 8.1 indicates frequently used null codes in validating data. This is not the exhaustive list and each void set should be evaluated to use the code that best explains the situation.

8.1 Validation Procedures

The T640x collects both PM10 and PM2.5 continuous data. As such each data set must be validated within AirVision, and uploaded to AQS. It would be an unusual event for the null code or qualifier flags to not be identical on both data sets.

A. In the AirVision Program select Reports-> Monthly Report

B. Select the Monthly Data range which is being validated, 1 hour rolling average, backward. In the parameter selection select the site AirLab, parameter name PM10STP or PM2.5 and click Generate Report. Be sure the show null codes and qualifiers is UNCHECKED.
C. The report will have colored squares to indicate null values or flagged data. Print the report.
D. Review each null or flagged hour. Using the EPA's Qualifier codes, assign a code, or notate valid data for each null or flagged hour on the printed sheet.

Commonly used Null Qualifier codes	
AN	Machine Malfunction
AV	Power Failure
BA	Maintenance/ Routine Repairs
AZ	QC Audit/ Monthly Verification
BL	QA Audit/ Internal, TDEC, EPA audits etc

Figure 8.1 Common Null Codes

E. Update the AirVision hourly data with the null code decisions. Click on Data Editors -> Average Data Editor. Again, choose the selected date range, the average internal of 001-hourly average of 60 minutes, the Site name and parameter. Select Retrieve Data.
F. Locate the hour block to receive a null code. Click on the box under AQS Null Code and choose from the drop-down choices. Save Changes.
G. Repeat the report generation as described in steps A and B above. This time the null codes should appear in the colored squares. Print the report with null codes.
H. Each of the printed monthly reports and any written documentation of validation decisions shall be maintained and placed in the QA/QC T640x Binder.
I. Repeat for the second pollutant (PM2.5 88101).

8.2 Entering Data into AQS

AQS is the data management system maintained by the US Environmental Protection Agency (EPA). It is assumed in these instructions that the user has a user name, required access, and basic training provided by EPA. AQS is accessed through the internet. The website name may change. The EPA also uses the ENSC program to upload data into AQS. This program also requires a user name, password and basic training.

A. In AirVision you will create an AQS file for uploading. Click Reports-> AQS Text Report. This shall be completed at least quarterly upon completition of validattion procedures but

can be completed more frequently.

B. Select the pollutant, date range, the average interval of 001 hr average and site AirLab.

C. Click “Save to File” and place on the S:// Drive in a file by year under S:\Air_Quality_Lab\ Air Monitoring 2019 \Pollutants\PM10 & 2.5- continuous. Choosing the Air Monitoring file for the correct year. This can be done using one drive, or a flash drive to move to an S: drive connected computer.

D. The file is then uploaded using the ENSC program. Notify the Quality Assurance Officer and the Air Monitoring Program Manager that the data is exported.

E. Follow the ENSC program upload protocol.

8.3 Entering QA into AQS

Monthly the monitor logbooks shall be brought in from the field and scanned. At this time the QA will be entered into the QA database and AQS. Additional information on this process can be found in the Data Handling SOP. In AQS, both flows (sample, and total) must be entered under each pollutant (88101 and 81102) which leads to 4 entries per flow verification.

A. Enter the QA results into AQS under
   a. Select
   b. Maintain
   c. QA Assessments
   d. Flow Rate Verifications or Semi-Annual Flow Rate Audits

B. Query for the most recent Verification.

C. Enter Data as follows for Flow Rate Verifications
   State Code: 47
   County Code: 093
   Site ID: 1013
   Parameter Code: 88101 for PM2.5 and 81102 for PM10
   POC: 3 for 88101 and 4 for 81102
   Performing Agency: 0581 for Knox County Air Quality Management, 1025 for TDEC
   Assess. Date: Date in format YYYYMMDD
   Assess. Number: 1 for the sample flow entry, 2 for the total flow entry, if more than one in a day continue with 3,4…
   Method Code: 238 for PM2.5 and 239 for PM10
   Unit: 118 for L/min
   Assessment Flow Rate: Enter Flow rate provided by TriCal/ Streamline Pro or Transfer Standard
   Monitor Flow Rate: Enter the Flow rate provided by the monitor. Note: Be sure to verify that all the units are the same and match the unit entry in AQS. Different units will need to be converted to the correct units of measure. It is recommended that you save after each line entered. AQS can have connectivity issues and frequent saves reduces the data lost.

D. After completing AQS entry, notify the Quality Assurance Officer that QA has been updated.

9.0 Documentation and Records

Recordkeeping is a critical part of the QA program. Careful documentation of sampling data will salvage samples that may otherwise be lost. The sheer repetition of recording data may result in errors. This section is to highlight specific documentation that must be completed. This shall supplement any documentation elements described in previous sections.

9.1 Documentation

Maintaining a complete log book, QA/QC documentation, and validation decision data is critical for ensuring a quality data program. Each of these sources of information is addressed in the following section to include what should be recorded and where it should be stored.

9.1.1 Log Books

There are two logbooks which must be updated and maintained properly, the Site Logbook and the Sampler Logbook. Operators must record in the site logbook the date, time, operator name, weather conditions, and purpose of visit every time they visit the site. Operators shall also record any site maintenance, unusual activity or concerns in the Site Logbook. In the Sampler Logbook the operator must record the Date, time, operator name, and machine specific operations such as; parameter checks, QC activities, or maintenance. KCDAQM operators can use “Stickies” or forms printed on CLEAR packing labels to aid in entering data into the sampler log book. When used, stickies must be printed on clear labels to ensure that no entries are covered in the logbook.

9.1.2 QA/QC Documents

Each QA/QC activity such as weekly parameter checks, flow verifications, leak checks, and audits are recorded in the sampler log book, only audits have additional paper documentation outside the sampler logbook. Logbooks shall be digitally scanned monthly to ensure a back of the logbook information is maintained. Any AirVision monthly reports, corrective action reports or quality bulletins shall be maintained in the QA/QC binder.

9.1.3 Validation Documentation

After completion, the data validation (either monthly or quarterly), the operator shall record all validation decisions. This could be as simple as notating a BL on the monthly report with “TDEC Audit”, or could require further explanation especially when using non-typical null data codes or when editing data within the Air Vision software. All validation documentation shall be placed in the QA/QC binder. This binder is organized by month.

9.2 Records Management

Records Management is the professional practice of managing the records of an organization throughout their life cycle, from the time they are created to their eventual disposal. This includes identifying, classifying, storing, securing, retrieving, tracking and destroying or permanently preserving records.

9.2.1 Document Integrity

The majority of documents used are formatted through Microsoft Excel. This program performs the calculations and graphs the results. All documents shall be checked annual as part of the records management program for accuracy. The master forms are in read-only format to help insure there are no alterations to the approved formulas and format during the year.

9.2.2 Records Retention

After the year is complete, and all sampling and QA/QC data are entered and validated in AQS, then the QA/QC T640x binder, any logbooks and documentation for the year are placed in a perma-file box marked with the year and pollutant. The box is stored in the data room. All files are maintained at least 5 years.

10.0 Appendix

Knox County Health Department
Every Person, A Healthy Person

Serial Number: _____

Date: _____

Page: _____ of _____

Corrective Action Report

Issue: *Describe the issue found, what was known preceding discovery, what is believed to have occurred etc. If possible, note last passing verification date. Include make, serial number, pollutant for instrument in question and as specific as possible dates and times. If failed verification be sure to report serial number of standard used for verification.*

Action taken: *Describe all actions performed such as calibrations, maintenance, repair. Include as specific as possible dates and times.*

Post action QC performed: *Note date time and results of verifications post actions. Be sure to note the serial number of verification equipment used.*

_____

Signature

Figure 10.1 Corrective Action Report

Knox County Health Department
Every Person, A Healthy Person

KNOX COUNTY AIR QUALITY MANAGMENT
QUARTERLY AUDIT FIELD SHEET

TEOM and T640x

Please circle Reference device used for Audit

DeltaCal TriCal Tetra Cal Streamline Pro

Serial Number: _____ Calibration Date: _____

Site Name: _____ Date: _____

TEOM	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 5 Min.
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 0.01 atm
Main Flow (3.00 l/min) [(Sys-Ref) / Ref] * 100				+/- 10 %
Bypass Flow (13.67 l/min) [(Sys-Ref) / Ref] * 100				+/- 10 %
Total Flow (16.67 l/min) [(Sys-Ref) / Ref] * 100				+/- 10 %

Site Name: _____ Date: _____

T640	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 5 Min.
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10mmHg
Total Flow (16.67 l/min) [(Sys-Ref) / ref] * 100				+/- 4%
Main Flow				+/- 4%

Leak Check
Pass/Fail

URG and SASS

Please circle Reference device used for Audit

DeltaCal TriCal TetraCal Streamline Pro

Serial Number: _____ Calibration Date: _____

URG Date: _____

Site Name:

Sampler ID:	System	Reference	Difference	Criteria
Time (in EST)				+/- 5 Min.
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow (16.67 l/min) [(Sys- Ref) / Ref] * 100				+/- 10 %

Leak Check
Pass/Fail

Site Name: _____ Date: _____

SASS	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 5 Min.
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Filter Temp 1				+/- 2° C
Filter Temp 2				+/- 2° C
Sample Flow 1 (sys-ref/ref)*100				+/- 10 %
Sample Flow2[(Sys-ref) / ref] * 100				+/- 10 %

Leak Check
Pass/Fail

Field Sheet SP

Figure 10.2 Audit Workbook Field Sheet SP

Knox County Health Department — Every Person. A Healthy Person.

Special Purpose Audit Calculations

Reference device used for Audit: _____ Serial number : _____

Date of Certification: _____

Leak Test

	Pass	Fail
URG 3000		
SASS Channel 1		
SASS Channel 2		

Pressure {Ambient}

	System	Reference	Difference
URG 3000N			0.00
SASS			0.00

Flow Rate

	System	Reference	% Difference
URG 3000N			#DIV/0!
SASS channel 1			#DIV/0!
SASS Channel 2			#DIV/0!

Notes:

Temperature

	System	Reference	Difference
URG 3000N Ambient			0.00
SASS ambient			0.00
SASS filter channel 1			0.00
SASSfilter Channel 2			0.00

TEOM

	Units	System	Reference	Difference	%	Criteria
Time	hh:mm:ss			#VALUE!		+/- 5 Min.
Amb T	°C			0		+/- 2° C
Pressure	atm			0		+/- 0.01 atm
Main Flow	lpm			#VALUE!	#VALUE!	+/- 10 %
Bypass Flow	lpm			#VALUE!	#VALUE!	+/- 10 %
Total Flow	lpm			#VALUE!	#VALUE!	+/- 10 %

T640X

	Units	System	Reference	Difference	%	Criteria
Time	hh:mm:ss			#VALUE!		+/- 5 Min.
Amb T	°C			0		+/- 2° C
Pressure	mmHg	15	10	5		+/- 10mmHg
Total Flow	lpm			0	#DIV/0!	+/- 4 %
MainFlow	lpm			0	#DIV/0!	+/- 4 %

Special Purpose Audit Workbook rev 7
4/18/19

Figure 10.3 Special Purpose Audit Workbook

Appendix D – Hi-Vol Lead SOP

VOLUMETRIC-FLOW-CONTROL (VFC) HIGH VOLUME TSP/Pb MONITORS Standard Operating Procedures

Knox County Health Department
Every Person, A Healthy Person

Prepared By
Rebecca Larocque

Knox County Department of Air Quality Management

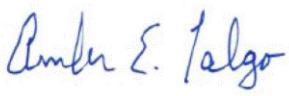
140 Dameron Avenue
Knoxville, TN 37917-6413

SOP Identification and Approval

Title: "Volumetric Flow Control High Volume TSP/Pb Monitors"

The attached "Volumetric Flow Control High Volume TSP/Pb Monitors" is hereby recommended for approval and commits the Knox County Department of Air Quality Management to follow the elements described within.

Knox County Dept. of Air Quality Management



6/19/19

Amber Talgo Date
Air Monitoring Program Manager

EPA, Region IV

Date

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1.0 Introduction

This Standard Operating Procedure (SOP) document describes the field procedures used by the Knox County Department of Air Quality Management (KCDAQM) to sample for lead(Pb) in Total Suspended Particulate (TSP) using volumetric flow controlled high volume TSP monitor. That is particulate matter which is suspended in ambient air and has a mean aerodynamic diameter of up to 25 to 50 micrometers. The mass concentration in ambient air is computed by measuring both the mass of lead collected and the volume of air sampled. The federal reference method for the determination of suspended particulate matter in the atmosphere (High-Volume Method) is presented in 40 CFR Part 50, Appendix B. Sampler siting, operation and quality assurance regulations are presented in 40 CFR Part 58. The operating procedures presented in this SOP are derived from the above cited regulations, guidance presented in equipment manufacturer instructions, and the EPA Quality Assurance Handbook for Air Pollution Measurement Systems Volumes I and II.

Eastern Research Group Laboratory (ERG) analyzes KCDAQM filters using Inductively Coupled Plasma Mass-Spectroscopy (ICP-MS) as approved by 40 CFR 50 Appendix G manual equivalent method EQL0512-202. This provides KCDAQM with the mass of lead collected. The specific procedures used to analyze TSP samples for the presence of lead are documented in a separate SOP prepared by ERG.

1.1 Principles of Operation

An electrical blower motor draws ambient air into the sampler where the suspended particulate matter is collected onto a glass-fiber filter. The air flow through the filter is maintained at a constant volumetric flow rate over a 24-hour period from midnight to midnight. Each high volume (Hi-Vol) sampler consists of:

- An aluminum shelter housing,
- A rectangular gabled roof inlet that directs air flow onto an 8-inch by 10-inch glass-fiber filter,
- A critical flow orifice and motor,
- A filter cassette that holds the filter in place on a support; and
- An elapse timer to document the sampling period.

The blower motor is calibrated with a certified critical orifice flow device (a flow transfer standard) to ensure that the required volumetric flow rate is maintained between 1.1 to 1.7 cubic meters of air per minute (m3 /min). The calibrated elapse time indicator provides an accurate time in minutes for each sample run. Samples are collected according to an EPA 1:6 schedule. Once a month all the collected filter samples are sent to a laboratory for analysis to determine the lead content of particulate matter on each filter. The lead content of the particulate matter on the filter, the flow data, and the sample time collected for each sample are calculated to determine the concentration of lead in the ambient air. The results are expressed as micrograms per cubic meter ($\mu g/m^3$). As revised in 2008, the NAAQS for lead was established at 0.15 $\mu g/m^3$ based on a 3-month rolling average.

1.2 Reference Documents

- *Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High-Volume Method),* 40 CFR 50 Appendix B

- *Reference Method for Determination of Lead in Suspended Particulate Matter Collected from Ambient Air,* 40 CFR 50 Appendix G
- *Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring,* 40 CFR 58 Appendix E
- U.S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurements Systems, Volume II, Ambient Air Specific Methods*
- U.S. Environmental Protection Agency, *Compendium of Methods for the Determination of Inorganic compounds in Ambient Air*, compendium Method IO-2.1
- ASTM D4096, *Application of the High-Volume Sample Method for Collection and Mass Determination of Airborne Particulate Matter*
- *Quality Assurance Project Plan*, Knox County Air Quality Management Ambient Air Monitoring Program, November, 2018

2.0 Installation

This section documents the installation or moving of the VFC Hi Vol monitors in the Knox County Air Quality Management Air Monitoring Program. Hi Vol monitors have not changed very much, however if new equipment is purchased refer to the new equipment manual as a primary reference document and this section as a secondary reference.

2.1 List of Tools, Equipment and Materials

- TSP High Vol Sampler System
- 8” x 10” Glass-fiber filters which meet EPA Method requirements • Filter cartridge (or filter cassette)
- Additional sampler parts and supplies including additional gaskets/seals, spare blower motors, motor housings, wire nuts, etc.
- Miscellaneous hand tools including: hammer, screwdrivers, wrenches, nut drivers, hex key wrenches, wire strippers, voltmeter
- Power tools, electric drill, power screwdriver, bits
- Extension cord
- Personal protective equipment and clothing/footwear

2.2 Physical Inspection

Upon receipt of all shipped sampling equipment, parts and supplies, inspect equipment and accessories for completeness and/or damage. If a shortage or damage is found, immediately notify the lab manager and/or the equipment vendor to repair or replace damaged equipment or missing supplies.

2.3 Siting Requirements

Samplers should be sited to meet the goals of the specific monitoring project. For routine sampling to determine compliance with the Lead NAAQS, the sample location is described in the Quality Assurance Project Plan (QAPP) for KCDAQM Monitoring Program. Samplers will be mounted on a safe, suitable monitoring platform according to the following guidelines:

- TSP samplers must be exposed to unobstructed airflow in all directions.
- The sampler inlet must be vertically placed between 2 and 15 meters (m) above ground level.

- Collocated TSP samplers shall be between 2-4m apart.
- Refer to all guidelines in 40 CFR 58 Appendix E.
- The site security is to be maintained with a locked fence.

2.4 Sampler Installation and Assembly

The location of the Hi-Vol sampler will have already been identified as part of the monitoring site selection and is discussed in the Annual Network Monitoring Plan and QAPP. The sampler will be located on a raised wooden platform or scaffolding. Position the shelter housings to meet the siting requirements as stated above and secure the shelter housing directly to the roof desk or platform surface. Make sure the shelter housing and the platform are sufficiently secured to withstand wind conditions.

Hi-Vol systems may be operated in several different configurations. The following describes component function and configuration used in KCDAQM Hi-Vol Sampler systems.

Timer: (TischTE-303 Digital Timer) The timer controls electrical power to the motor, controlling when the motor turns off and on. This timer also acts as an elapse timer for the sample runs and records the elapsed run time for each scheduled sample run. See Figure 2.1 for illustration of the timer. For additional information view the Tisch TE-303 manual located on the server L:\Air Lab Data\Ambient Air Monitoring\Pollutants\TSP Monitoring\TSP Documents\Manuals

Figure 2.1 Tisch Timer

Blower-Motor & Housing: The blower-motor is the driving force of the sampling system. The blowermotor is a combination of an electrical motor attached to a circular impeller that draws air flow into the sampler inlet, collecting any suspended particulate matter onto the glass-fiber filter in the filter cassette. The air flow continues down through the VFC orifice to the blower-motor housing. The air flow passes through the blow-motor and out the hole in the bottom of the blower-motor housing. The VFC orifice has a pressure tap at the top of the assembly, right below the filter holder, to determine

differential pressure which is a measurement used in the calibration verification process and to calculate volumetric air flow. See figure 2.2 below.

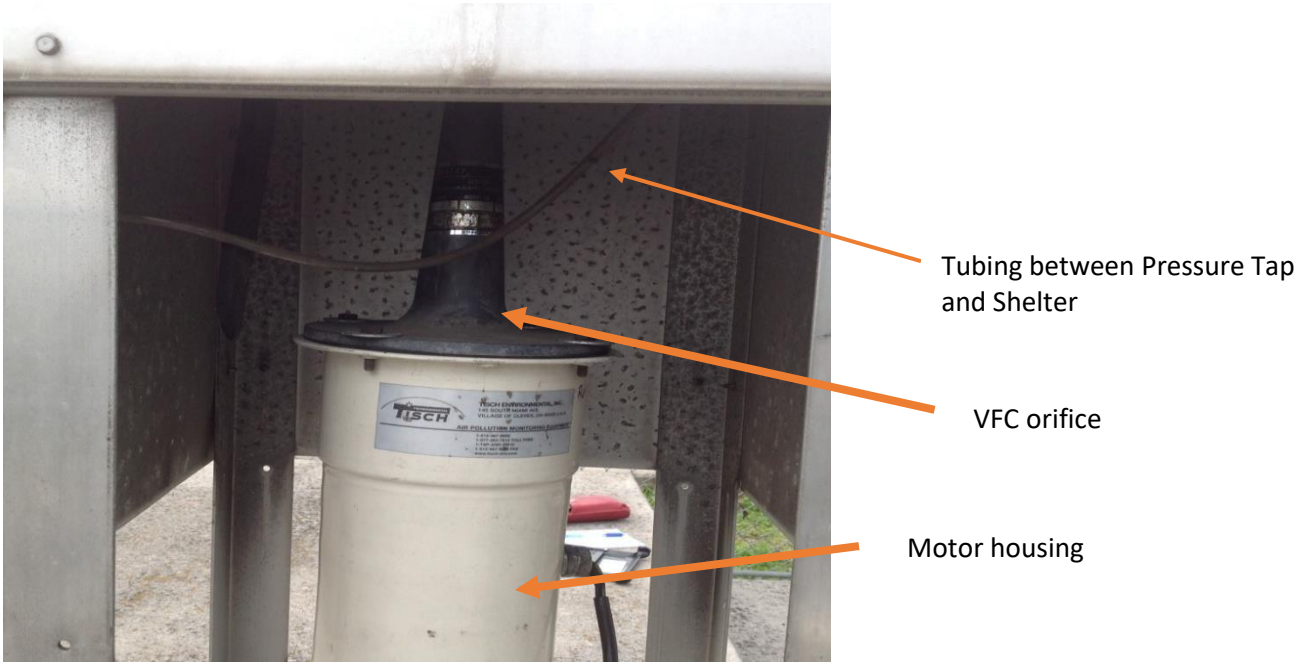


Figure 2.2 Motor and Orifice

Volumetric-Flow-Control (VFC) System: A choked-flow venturi is operated such that the air attains sonic velocity in the throat of the device. The flow rate is unaffected by downstream conditions such as motor speed or exit pressure and is a predictable function of upstream conditions, such as the stagnation pressure ratio and temperature. Thus, the volumetric flow is controlled without any moving parts or electronic components. See Figure 2.2 for view of venturi orifice installed. In this type of flow control system, no means of adjusting the controlled flow rate is provided.

3.0 Calibration and Verification Procedures

Calibration is defined as the relationship between an instrumental output and the input of a known reference standard. TSP/Pb concentration standards are not available for determining calibration relationships therefore, individual components of the sampling method must be calibrated to ensure the integrity of reported data. To establish ambient Pb concentrations, three independent determinations are made: flow rate, sampling time, and Pb mass. KCDAQM verifies the flow rate and timer, and ships known mass test strips to verify the Pb mass with the laboratory.

A certified testing laboratory provides the certification of KCDAQM's orifice transfer standard for multipoint calibration verification, see example document Appendix B.1. The orifice transfer standard is certified yearly or more frequently if needed. Monometers used in verifications and calibration verifications are certified annually with Chinook Engineering (or comparable vendor). The flow rate verifications are done using a BGI Hi-Vol Cal which is certified annually with BGI (or another comparable vendor).

Forms described in this section are illustrated in Appendix C and located at *L:\Air Lab Data\Ambient Air Monitoring\Pollutants\TSP Monitoring\TSP Documents\ Forms.* All completed calibration/ verification forms shall be saved in the folder located *L:\Air Lab Data\Ambient Air Monitoring\Pollutants\TSP*

Monitoring\(year) TSP, Lead\TSP Pb QAQC. Additionally, the operator shall print out completed forms and place in the corresponding QA/QC folder labeled by site and pollutant. The printed form is used in the final data package preparation and is the official record.

3.1 Calibration Verification Frequency

To ensure accurate measurement of Pb concentrations, verify calibration of Hi-Vol TSP samplers upon installation and as follows:

- At least annually,
- After relocation of the sampler to a different site
- After changing the orifice, or
- If the results of a monthly field flow-check exceed quality control limits (>±7percent from the sampler's indicated flow rate)

3.2 Calibration Verification Equipment

- Orifice transfer standard with proper calibration traceable to NIST.
- A traceable manometer, with a 0- to 1,000-mm (0- to 36-in.) range and minimum scale divisions of 2 mm (0.1 in.) or other pressure measurement device for measurement of the sampler stagnation pressure (Stag Manometer).
- A thermometer capable of accurately measuring ambient air temperatures over a range of 0 to 50 ^{o}C to the nearest 0.1 ^{o}C. This thermometer should be traceable with an accuracy of 0.1 ^{o}C to a NIST-certified thermometer or an ASTM thermometer.
- A portable, aneroid barometer capable of accurately measuring ambient barometric pressure over the range of 500 to 800 mm Hg (66 to 106 kPa) to the nearest millimeter of mercury and referenced within ±5 mm Hg to a barometer of known accuracy at least annually.
- TSP MultiPt field sheet (see Appendix C.1) or notebook and the station logbook.
- A clean filter.
- Duct tape (2 in or 51mm wide)

Note: The calibration of some VFC samplers may be affected by changes in line voltage, particularly if the line voltage is below normal (normal is about 115 VAC). For this reason, VFC samplers should always be calibrated at the monitoring site. Also, be sure that replacement blower motors are of the correct type (Grainger Item# 4M903, Mfr. Model # 115923 with replacement brushes Mfr. No. 833392-61).

Caution: Do not attempt to perform calibration verifications of the sampler under windy conditions. Short-term velocity fluctuations will produce variable pressure readings by the orifice transfer standard's manometer. The calibration will be less precise because of the pressure variations.

3.3 Leak Test

A. Install a filter (not in a cassette), followed by the orifice plate and lastly the orifice transfer

standard. Be sure that the orifice plate is centered over the filter so the gaskets will properly seal and tighten the screws. Tighten the orifice plate nuts evenly on alternate corners to properly align and uniformly seat the gaskets. The nuts should be hand-tightened only; too much compression can damage the sealing gasket. Figure 3.1 illustrates the filter and orifice plate configuration.

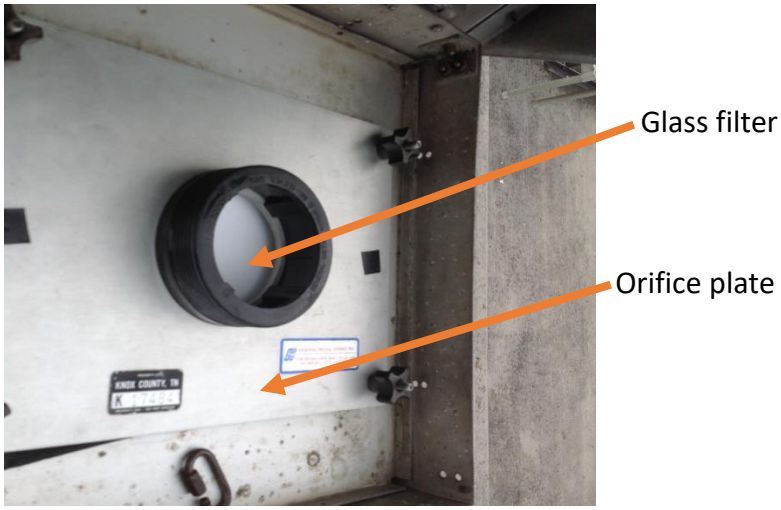


Figure 3.1 Orifice Plate Set up

B. Turn on the blower motor by using the timer:

SETUP
└DIAGNOSTICS
└MOTOR and press “ENT”.

Allow 5 minutes for warm up of the blower motor.

C. Using duct tape, prepare a patch to place over the inlet on the flow orifice. Place the duct tape patch over the inlet of the orifice transfer standard and make sure the inlet is completely sealed preventing any air flow through the orifice transfer standard and into the blower motor. Alternately an operator can use a clipboard to first cover the orifice plate and listen for leaks, then if using orifice transfer standard, block the holes with tape or fingers.

D. Place your thumb over the pressure tap where the manometer tubing attaches to the orifice transfer standard. With the other hand, check the bottom of the blower-motor housing to check for any air flow passing out of the vent hole in the bottom of the housing.

E. Gently rock the orifice transfer standard and listen for a whistling sound that would indicate a leak in the system. Leaks are usually caused either by a damaged or missing gasket between the orifice transfer standard and the orifice plate or by cross threading of the orifice transfer standard on the orifice plate.

F. If there is a slight leak, tighten the screw knobs which hold the orifice transfer standard onto the filter support screen and repeat the leak test. If there is still a leak, check the entire system

(motor, tubing, connection tightness, and gaskets) to find the leak. Repeat this procedure until the leak test is successful.

G. All leaks must be eliminated before proceeding with the calibration. When the system is determined to be leak-free, turn off the sampler and unblock the orifice.

Caution: Running the sampler for too long (30 seconds – 1 minute) with the air flow blocked can increase the chance that the blower motor will overheat due to the lack of cooling air. Overheating can shorten the lifetime of the blower motor and can also result with damage to the motor's electrical insulation, which could cause a fire or electric shock to the user.

3.4 Calibration Verification

Since the venturi orifice is a fixed device, there is no calibration of the sampler flow rate. Instead, the operator performs a flow check to verify the sampler is operating properly. A multipoint calibration verification should be done with a variety of stagnation pressures bracketing the normal operating stagnation pressure by approximately 5 inches of pressure (if normal stagnation pressure is 19 inches check five points from 14-24 inches of pressure). If the results of the check are poor (>±5% of best fit line), it is an indication of a problem with sampler such as a leak, or a weak motor not pulling enough vacuum. The calibration procedure relates known flow rates (Q_a, as determined by the orifice transfer standard) to the ratio of the stagnation pressure to the ambient barometric pressure.

A. Perform Leak Check described in 3.3 before proceeding with calibration verification.

B. Select the first calibration flow rate by adjusting the variable resistance valve (on top of calibration orifice). At least four flow rates are required to define the calibration relationship. At least three flow rates should be within the acceptable flow-rate range (i.e., 1.02 to 1.24 m^3/min.

D. Turn on the sampler by using the timer:

SETUP
└DIAGNOSTICS
└MOTOR and press "ENT".

Allow the sampler to warm up to operating temperature (15 min).

E. Inspect the connecting tubing of the manometers for crimps or cracks. Connect the trans. manometer to the transfer standard orifice and the stag. manometer to the stagnation pressure port on the side of the monitor housing. Make sure the tubing fits snugly on the pressure ports and on the manometers. See Figures 3.1 and 3.2 for location of the pressure ports.

Figure 3.1 Orifice Transfer Standard

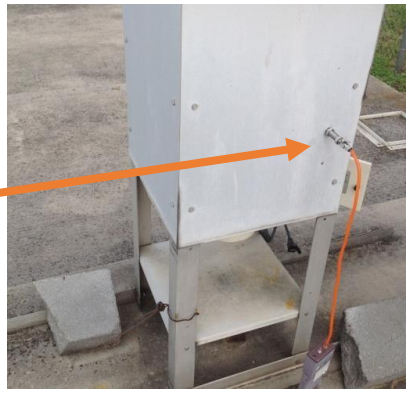


Figure 3.2 Stagnation Port

F. Read and record the following parameters on the MultiPt Field Sheet (see appendix C.1) or note pad.

- Date, location, and operator
- Serial numbers of sampler and all reference devices used
- Ambient barometric pressure (Pa) in mm Hg
- Ambient temperature (Ta) in ^{o}C
- Orifice S/N and calibration relationship.

Regardless of recording document, the field documentation shall be attached to the spreadsheet calculations. (Appendix C.2)

Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield. But do not close all the way, use a block to provide at least 2 in. of clearance at the bottom for air flow and for the manometer tubing.

G. Record the orifice transfer standard's manometer (trans manometer) reading, in inches of H2O in the column labeled "ΔH2O, and the corresponding sampler relative stagnation pressure (stag manometer) reading under the column labeled" P_{stg}", on the data sheet.

H. Adjust the variable orifice value to obtain each of the other calibration flow rates (and repeat Step G for each. A multipoint check should be done with a variety of stagnation pressures bracketing the normal operating stagnation pressure by approximately 5 inches of pressure (if normal stagnation pressure is 19 inches check five points from 14-24 inches of pressure).

I. Running additional calibration points at differing flow rates or repeating the calibration points at the same flow rates is encouraged to improve the precision of the calibration.

J. Turn off the sampler by pressing "ENTER" on the timer. Remove the orifice transfer standard and orifice plate.

K. If a sample day follows: Proceed to Section 4.3.1.

L. Input the data collected on form TSP Multi Point Calibration Verification. See Appendix C.2

M. Use the Po/Pa values and ambient temperature to find the sample flow from the orifice specific look up tables and enter into the spreadsheet.

N. This spreadsheet calculates the flow based on the pressure difference, compares the calculated flow to look up table flow curve and assess the linearity of the best fit line.

O. All points should be within ± 5% of the best fit line. The spreadsheet is formatted to indicate a failure by turning the box red.

Pass/Fail is determined by the spreadsheet calculations on the form (Appendix C.2) which are not completed in the field. The Operator shall complete the spreadsheet as soon as possible so that adjustments or repeating of the test may occur before the next scheduled run day. If the agreement is not ≤ ± 5% of the best fit line, recheck the entered orifice transfer standard slope and intercept and recheck the calibration procedure. Look for leaks, manometer reading errors, incorrect temperature or pressure data, or miscalculations. Also check for abnormally low line voltage at the site (it should be at least 110 VAC), for the correct blower motor, and for the presence of a gasket between the motor and the choked-flow venturi. A factory calibration is not likely to be substantially incorrect, and any discrepancy of more than a few percent is probably due to some problem with the sampler or with the calibration procedure.

3.5 Monthly Flow Verification

The department uses the BGI instrument for monthly flow verifications. However, this SOP also includes the instructions if using the orifice transfer standard and two manometers. Flow verifications are acceptable when the measured flow is within ±10% of the design flow and ±7 % of the actual flow (from orifice look up tables).

3.5.1 BGI Monthly Flow Verification

The BGI Volumetric Flow Calibrator measures and can calculate Qa within the device. Form illustrated in Appendix C.3

A. Collect the flowing equipment and transport it to the monitoring station:
- An electronic, oil, or water manometer, with a 0-400mm (0 to 16 in.) range and minimum scale divisions of 2mm (0.1in.),
- A glass filter,
- An orifice plate,
- Cell phone with tower updated clock,
- The site logbook (LB).
- NIST traceable BGI Hi Vol Cal and
- Pen/Paper.

B. Open the Timer and confirm the clock is within 30 minutes of the cellphone time. If the timer is off by more than 20 minutes, adjust the time by selecting: F3 SETUP
└CONFIGURE
└TIME
Set time to the nearest minute eastern standard time and press "ENT". Use the "ESC" key after saving to back out of the configure menu.

C. Turn on the pump and perform a 15minute warm up of the motor before proceeding with the verification. Turn on the sampler by using the timer: SETUP
└DIAGNOSTICS
└MOTOR and press "ENT".

D. Turn on the manometer to zero prior to installation on the stagnation pressure port. Once zeroed and installed on port, with no filter reading should remain zero.

E. Install glass filter and orifice plate on to the sampler, as indicated in the Leak Check Section 3.3 according to Figure 3.1. Tighten nuts alternately and make sure gaskets and seals secure.

F. Cover the orifice whole with hands, clip board, or another item larger than the orifice. Listen for leaks.

G. Check the manometer in the stagnation port. It should read between 17-24 inches of H2O. This is secondary check for leaks, abnormally low values may indicate a leak.

H. Turn on the BGI high Vol Calibrator. Allow instrument time to self-calibrate

I. Install calibrator on the orifice plate. See Figure 3.5 Read and record pressure and temperature.

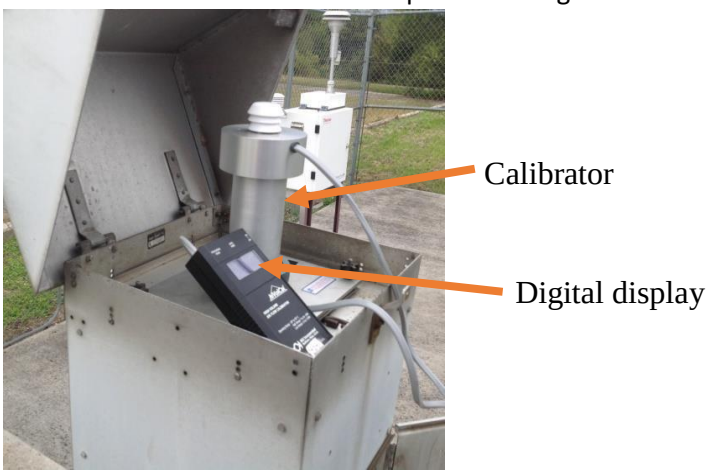


Figure 3.3 BGI Hi Vol Cal Setup

J. Take 10 readings 5 seconds apart of Qa – record all 10 values.

K. Record Stagnation pressure from manometer.

L. Turn off motor by using the timer: SETUP
└DIAGNOSTICS
└MOTOR and press "ENTER".

M. Remove hi-vol cal, orifice plate, and glass filter.

N. Upon returning to the office use Form TSP BGI Flow Verification {Appendix C.3}. It will calculate percent difference from design flow by a-c and from actual flow by d-e below:

a. Averaging the 10 readings,
b. Converting from CFM to m^3/min, and calculating
c. {(1.13-Qa)/Qa} *100%.
d. Use the Po/Pa value calculated on the form and the look up table corresponding to the Sampler's orifice to enter the actual flow value.
e. The excel sheet will calculate % difference from actual flow using the look up table {(flow – Qa)/Qa} *100%.

3.5.2 QC Single-point Flow Rate Check 2 Manometer Procedure—VFC Sampler

When the BGI HiVolCal is unavailable due to recertification or repair, this procedure is followed to verify the flow rate using the pressure differentials and the orifice calibration cure. The indicated flow rate (Q_a samp) for VFC samplers is calculated by determining (1) the relative stagnation pressure (P_{stg}), (2) the ambient temperature (Ta), and (3) the barometric pressure (Pa) during the flow check. These values are then applied to the sampler's calibration relationship.

A. Collect the following equipment and transport it to the monitoring station:

- The orifice transfer standard,
- An electronic, oil, or water manometer, with a 0- to 400-mm (0- to 16-in.) range and minimum scale divisions of 2 mm (0.1 in.),
- A second electronic, oil, or water manometer, with a 0- to 1,000-mm (0- to 36-in.) range and minimum scale divisions of 2 mm (0.1 in.).
- A thermometer capable of accurately measuring ambient air temperatures over a range of 0 to 50 ^oC to the nearest 0.1 ^oC. This thermometer should be traceable with an accuracy of 0.1 ^oC to a NIST-certified thermometer or an ASTM thermometer,
- A portable barometer capable of accurately measuring ambient barometric pressure over the range of 500 to 800 mm Hg (66 to 106 kPa) to the nearest millimeter Hg and referenced within 5 mm Hg of a barometer of known accuracy at least annually,
- A clean flow-check filter,
- TSP Single-point Flow Verification Spread Sheet Appendix C.4 or paper, • Pen and

- The site logbook (LB).

B. Set up the flow-check system with a filter in line (i.e., between the orifice transfer standard and the motor). The orifice transfer standard shall be installed with the adjustable resistance valve fully open. Do not use the flow check filter for subsequent sampling. Perform a Leak Check following Section 3.3.

C. Turn on the sampler by using the menus on the timer:
Press "ESC" until at the main menu
└SETUP
└DIAGNOSTICS
└MOTOR and press "ENTER".
Allow the sampler to warm up to operating temperature (15 min).

D. Enter the following parameters in the site log book and TSP Single-point Flow Verification Spread Sheet (Appendix C.4) or paper:
- Sampler identification, date, and time
- Sampler S/N and model
- Ambient temperature (Ta), ^{o}C
- Ambient barometric pressure (Pa), mm Hg
- Unusual weather conditions
- Orifice transfer standard serial number
- Operator's initials.

E. Inspect the manometers for crimps or cracks in the connecting tubing. Turn on and allow to zero. If using an oil or water manometer, open the valves and blow gently through the tubing, watching for the free flow of the fluid. Adjust the manometers' sliding scales so that the zero lines are at the bottom of the meniscuses. Ensure that one side of each manometer is open to atmospheric pressure.

F. Connect the trans manometer to the orifice transfer standard, and the stag manometer to the sampler stagnation pressure port located on the side of the sampler base. Be sure that the connecting tubing snugly fits the pressure ports and the manometers. See Figures 3.1 and 3.2 in Section 3.4.

G. Read the pressure drop as indicated by the trans manometer (ΔH_2O), and stagnation pressure drop (Pstg) as indicated by the stag manometer. Enter both values in the logbook and the TSP Single-point Flow Rate Verification Spread Sheet (Appendix C.4). The orifice resistance plate valve must be fully open for these measurements.

H. Remove the orifice transfer standard, orifice plate and filter. Install a filter in a regular cassette, install the filter cassette (tightening snugly). Record the stagnation pressure.

Note: Measurements will automatically be converted from in H_2O to mm Hg by the spreadsheet using Equation (1). See appendix A for equations use.

I. Turn off sampler using the timer.
Press "ESC" until at the main menu
└SETUP
└DIAGNOSTICS
└MOTOR and press "ENTER".

J. If sample day follows proceed to Section 4.3.1.

K. The spread sheet will automatically calculate the Qstd orif (calc) flow rate.

4.0 TSP Sample Procedures

4.1 List of Tools, Equipment, and Materials

- EPA sampling calendar,
- Clean, unexposed, EPA approved glass-fiber filters,
- Disposable gloves for handling the filters (powder free),
- Logbook and ink pens,
- Filter cassette, and
- Data envelopes & wax covers.

4.2 Preparing Filters

Find an available work area and surface which is clean, as dust free as possible that can be designated for sample setup and recovery. The following are the procedures to set up the filter cassette preparation for installing the sample filters on-site.

A. Review the sampling calendar to confirm the run date.

B. Before setting up the samples, clean and dry the working surface on any dust or other contaminants.

C. Open a filter cassette by removing the protective cover and unscrewing the thumbscrew which secure the top of the frame to the screen support. Figure 4.1 below

Figure 4.1 Filter Cassette

D. After cleaning hands and putting on a clean pair of disposable gloves, select a clean sample filter from the box. Each filter will be marked with an identification (ID) number located on the back of the filter in the upper right corner. Inspect the filter for pinholes, tears, abrasions, loose material, discoloration, and other non-uniformity. Discard any defective or damaged filters and make note of the discarded filter ID number in the logbook. Filters may be inspected and recorded in batches**.**

E. Record the filter number and scheduled run date on the data envelope. See Appendix B.2 for sample data envelope.

F. Place the filter into an open filter cassette centering the filter over the support screen. Ensure that the filter ID number is facing down. The upward or exposed side of the filter has a textured surface. Close the filter cassette by replacing the top of the frame over the filter ensuring that the gasket material covers the outer edges of the filter. Tighten the thumbscrews until the gasket material seal down firmly on the filter and bottom support. (Note: The seal should be firm but do not over-tighten the thumbscrews, this may damage the filter material.) Replace the cassette cover. The cassette cover must be in place to protect the filter during transport to and from the sample site.

4.3 On-site Installation and Removal of a Sample Run

Upon arrival at the monitoring site unlock the security fencing and inspect the equipment. Note any items needing maintenance or any irregularities that may affect data quality in the comment section on the data envelope. Filters should be installed and removed as close as possible to the run date. Best option is installed the day before the midnight start time and retrieved the day after the 23:59 end time.

4.3.1 Filter Installation

A. Open the main door to sampler; open the door to the 6-day timer

B. Record current date and time on the data envelope under “Installation date and time” (Note: All Sampling time is in Eastern Standard Time year-round. The samplers run time will not be adjusted for daylight savings time.)

C. Compare the current time to the time on the 6-day timer, if the time on the 6-day timer is off more than 20 minutes off, adjustment of the 6-day timer to the current time shall be required. Adjustment of the timer is recommended if over 5 minutes off. Make a note on the log sheet of any adjustment.

D. Unlatch and open the top of the sampler.

E. Inspect the filter screen and clean with alcohol cloth if dirty.

F. Install the new filter cassette: slide studs into notches and tighten thumb screws. Tighten the nuts evenly until they are snug. Do not over-tighten.

G. Be sure to remove the metal cover from the new filter cassette and use it to cover the used filter cassette.

H. Turn on the sampler by using the timer:
SETUP
└DIAGNOSTICS
└MOTOR and press “ENT”.

I. After 3 minutes to allow motor to warm up and flow to stabilize. While the sampler is warming up, record the following parameters on the Data Envelope (Appendix B.2):
- Site location
- Sample date
- Filter ID number
- Sampler model and S/N
- Operator’s initials.
-

J. Bring a manometer to the side of the sampler. This manometer should have a range of 0 to 1,000 mm (0 to 36 in.) and a minimum scale division of 2 mm (0.1 in.). Inspect the manometer for crimps or cracks in its connecting tubing.

K. Connect the manometer tube to the vacuum tap on the stagnation pressure port located on the side of the sampler base.

L. Measure the initial relative stagnation pressure (ΔPstg) and record this reading on Data envelope

M. Turn off the sampler by using the timer:
SETUP
└DIAGNOSTICS
└MOTOR and press “ENT”.

N. "ESC" out to the main screen. The sampler WILL not RUN if left in setup mode. Verify that the countdown clock is set to initiate sample run at the correct time.

O. Close and latch the sampler. Secure all monitors' site doors and locks.

4.3.2 Filter Removal

A. Open the main door to sampler; open the door to the 6-day timer

B. Record current time on the log sheet in the comment section. (Note: All Sampling time is in Eastern Standard Time year-round. The samplers run time will not be adjusted for daylight savings time.)

C. Check the data and Record the runtime on the data envelope and note any problems in the log book by:

a. Starting from the Main Screen, press the "F2" key to access the "DATA" menu.
b. Select "VIEW PAST SAMPLE" and press the "ENT" key.
c. A list showing the timer start dates and times will be displayed. Select the desired date and time and press the "ENT" key.
d. The first page of the sample data for this timer event will be displayed. The arrow keys can be used to switch to the different pages. Press the "ESC" key to exit and return to the list of available dates and times. Continue press the "ESC" key to return to the Main Screen.

D. Turn on the sampler by using the timer:

SETUP
└DIAGNOSTICS
└MOTOR and press "ENT".

E. After 3 minutes to allow motor to warm up and flow to stabilize.

F. Bring a manometer to the side of the sampler. This manometer should have a range of 0 to 1,000 mm (0 to 36 in.) and a minimum scale division of 2 mm (0.1 in.). Inspect the manometer for crimps or cracks in its connecting tubing.

G. Connect the manometer tube to the vacuum tap on the stagnation pressure port located on the side of the sampler base.

H. Measure the final relative stagnation pressure (Pstg) and record this reading on data envelope

I. Turn off the sampler by using the timer:

SETUP
└DIAGNOSTICS

└MOTOR and press “ENT”.

Note: You may leave the timer in diagnostic mode after filter removal for a few less steps in the installation of filters process. However never leave in diagnostic mode after installing a new filter, it will not perform the scheduled run.

J. Remove the old filter cassette by unscrewing the thumb screws until the studs can be pushed out of the notches and away from the cassette. Reinstall cover on the filter cassette before transport.

K. Close and latch the sampler. Secure all monitors’ site doors and locks.

L. Observe conditions around the monitoring site; note any activities that may affect filter particle loading (paving, mowing, fire) and record this information on the Data Envelope.

4.4 Post Sampling Filter Handling

A. Upon return to work area and before uncovering the samples ensure the area is still clean and dust free.

B. Gather the necessary materials to recover and store the samples: clean disposable gloves, protective sheets and data envelopes.

C. After cleaning hands and putting on clean disposable gloves remove the protective cassette cover, open the filter cassette by removing the thumb screws, and remove the top of the cassette. Care must be taken to not lose any particulate matter from the filter.

D. Remove the filter from the cassette and carefully fold the filter so the exposed (dirty) side is folded inward onto itself to prevent loss of particulate matter. Fold longways see figure 4.2. The edges of the filter should be even. (Take care not to bend or tear the corners.) The field ID number will be on the outside of the folded filter.

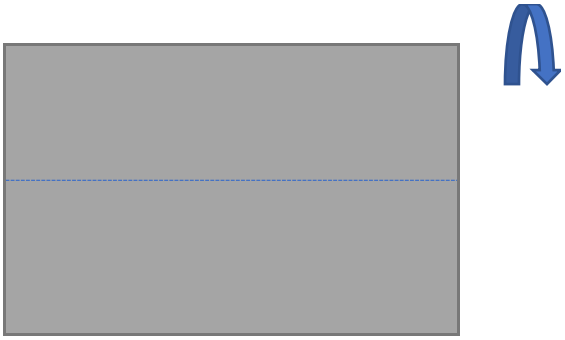


Figure 4.2 Filter Folding

E. Make sure the filter ID number on the data envelope matches the number on the filter.

F. Place the filter into the protective wax insert.

G. Insert the filter and protective insert into the envelope.

H. Close the envelope by tucking the flap inside the lip of the envelope. (Do not lick and seal the adhesive.)

I. Store the sample envelope in a secure dust free place until ready for shipment to the certified laboratory.

4.5 Shipping Procedures

After recovering the final samples of the month, the site operator will prepare and ship all the filters collected for the month to Eastern Research Group for processing and laboratory analysis.

A. Double check the Data Envelope (Appendix B.2). Determine whether all data needed to verify sample validity and to calculate Pb mass concentration are provided (stagnation pressures, elapsed time). Void the sample if data are missing and unobtainable or if a sampler malfunction is evident. Void Samples are not sent for analysis.

B. Make copies of the Data Envelopes. These are saved as records until the samples are returned.

C. Complete the Chain of Custody Sheet (Appendix B.3), keep a copy and place a copy in the shipping envelope with the filters.

D. The filter samples and chain of custody will be shipped through the US Postal Service. Place them in Large white envelope.

E. Mail the filter samples to:
Eastern Research Group, Inc
601 Keystone Park Dr.
SUITE 700
Morrisville, NC 27560

F. Once a quarter, the final run of the month at the collocated site, is not shipped to ERG. This filter (in the data envelope) is given to the Air Monitoring Program Manager for entry into the Region 9 system and shipped to USEPA region 9 Laboratory in Richmond California for processing.

5.0 Maintenance

Preventive maintenance is defined as a program of positive actions aimed to prevent failure of monitoring and analytical systems. The overall objective of a routine preventive maintenance program is to increase measurement system reliability and to provide for more complete data acquisition.

This section outlines general maintenance procedures for HV TSP/Pb samplers. For more complete information on a particular sampler, or on laboratory equipment maintenance, refer to the manufacturer's instruction manual for the individual instrument.
Records shall be maintained for the maintenance of each HV TSP/Pb sampler. Files will reflect the history of maintenance, part replacements, and QA/QC.

5.1 Recommended Maintenance Schedules

Although no maintenance schedule is explicitly prescribed in the Reference Method (40 CFR Part 50, Appendix B), field testing of hi-vol samplers determined the following maintenance frequencies.

A. Power supply cords: checked for crimps or cracks on sample recovery days.

B. A filter screen and the throat of the choked-flow venturi: inspected on sample recovery days for any impacted deposits.

C. Filter cassette gaskets: checked each time a filter is installed. A worn cassette gasket is characterized on exposed filters by a gradual blending of the boundary between the collected particulates and the filter border.

D. Fittings, inspected periodically for cross-threading and tightness.

E. Blower motor brushes: changed every 25 runs of 600 hours. A Flow Verification as described in Section 3.5.1 shall be conducted before the motor is removed, and upon installation of the new motor. **New brushes must be conditioned on a motor for 30 min at 50 percent of the normal line voltage before installation on monitors.**

F. Motor and housing gaskets: checked when motor brushes changed.

5.2 Refurbishment of HV TSP Samplers

If operated in the field for extended periods, HV TSP samplers may require major repairs or complete refurbishment. If so, refer to the manufacturer's instrument manual before work is undertaken. A sampler that has undergone major repairs or refurbishment must be leak-checked and have the calibration verified prior to sample collection.

6.0 Lead Monitoring Audit

Please also consult the most recent KCDAQM Audit SOP. The most recently updated SOP shall be used for auditing procedures and forms.

6.1 Tools and Equipment

Equipment	Recertification	Acceptable Range
Orifice Flow rate Transfer Std	1/yr	±2% of NIST Traceable Std
Field Thermometer	1/yr	±0.1°C resolution, ±0.5°C Accuracy
Field Barometer	1/yr	±1mmHg resolution, ±5mmHg accuracy
Clock	N/A	Cell phone updated to towers acceptable
Orifice plate	N/A	Clean , seals in place
Audit filter	N/A	Clean fiberglass filter
Field Sheet Lead	N/A	Most up to date revision of Audit Workbook

6.2 Field Check

A. Do not attempt to audit Hi Vol TSP samplers under windy conditions. The audit will be less precise because of pressure variations. Operator must remove any exposed filter cassette in the sampler.

B. Install audit filter, ID number facing down, without a cassette. Install the orifice plate in the sampler. Tighten the faceplate nuts evenly on alternate corners to properly align and uniformly seat the gaskets. The nuts should be hand-tightened only: too much compression can damage the sealing gaskets.

C. Turn on sampler for 15-minute warm up. Turn on the sampler by using the timer:

Setup
└Diagnostics
└MOTOR and press "ENT".

D. Upon 15-minute warm up completion, turn on the field manometer and allow to zero itself. Then attach manometer to the stagnation pressure port. Operator should verify that readings are typical for unexposed filter. Expected values indicate system is set up properly with no leaks.

E. Turn on Hi Vol Cal and all it to zero itself. The calibrator will read Qa and Qs as {Under} when ready to use.

F. Record the BP (barometric pressure) and Ta (ambient temperature) readings from the audit

HiVol Cal on the Field Sheet Lead (Appendix B.4) G. Install the Hi Vol Cal to the orifice plate.

H. Record 10 Qa readings at 5 second intervals. Use manometer to take a stagnation pressure reading during the audit. Record on Field Sheet Lead

I. The Quarterly Audit Workbook spreadsheet (Appendix C.5) will calculate percent difference by

a. Averaging the 10 readings,
b. Converting from CFM to m3/min (Q_a), calculating Po/Pa value.
c. Auditor shall use the orifice look up tables to find the corresponding Flow rate (Q_m)
d. $(Q_m - Q_a)/Q_a$ *100%

J. Operator shall disassemble audit apparatus and reset sampler to be ready for next run cycle.

K. Auditor shall inspect log book for required maintenance.

6.3 Evaluating the Results

The auditor shall review the Field Sheet Lead form for each of the samplers for completeness and enter the data into the Audit Workbook page "LEAD" (Appendix C.5) Percent difference must be ≤ ± 7.1%. If % difference is ≥ ± 6 % notify operator for adjustments. The Auditor prepares a summary of audit findings including any suggested corrective action. The summary should include any borderline results which required operator intervention to correct onsite but must include any results outside the acceptance criteria.

7.0 Data Validation

The following steps apply to the validation of single TSP concentrations based upon the field and laboratory data. Additional validation techniques (i.e. statistical techniques) may be specified in the Quality Assurance Project Plan (QAPP). Invalidated data should be flagged for quality assurance review and an explanation should be noted in the free-form notes section on the field data sheet. This section represents an overview of the general techniques used by the Quality Assurance Officer or other

Environmental Specialist to transform and validate the data. The Site Operator should not be responsible for transformation, validation, or entry. However, the operator should have an understanding of the process to help avoid data invalidation through error and ease information flow. Additionally, videos of the process are available on the shared drive How To Videos.

7.1 Validation requirements

A. Verify that the run data from the hi-vol are within the following limits: Average volumetric flow rate = 1.1 to 1.7 m3 /min.
Total sample duration = 24 hours (1440 minute) ± 60 minutes

B. Verify that the sample dates are correct and that all QC procedures for calibration and monthly flow rate checks were completed and validated.

C. Verify that the sample was not damaged during shipping and was transferred to the Lab with appropriate chain of custody.

D. Verify that the sample analysis met all analytical method QA/QC and was not invalidated by lab personnel.

The initial data review and validation is performed by an Environmental Specialist, Quality Assurance Officer. The data will then receive a second-level review and validation by the Air Monitoring Manager.

7.2 Calculating Concentration and Data Validation

A. Open a new copy of the excel form Pb Concentration Calculations located on the shared drive under *L:\Air Lab Data\Ambient Air Monitoring\Pollutants\TSP Monitoring\Forms.* See Appendix C.6 for sample document.

B. Save as Pb Conc Cals, *Month, Year* in corresponding years data folder located on the shared drive. L:\Air Lab Data\Ambient Air Monitoring\Pollutants\TSP Monitoring\2017 TSP Lead\TSP Pb 2017 data { 2017 updates to the corresponding year}

C. Obtain all envelopes of analyzed filters for each site that month or copies. On the newly created Pb Calculations sheet, enter information from sample envelopes for each sample: Date, Filter #, P-Initial, P-Final, time, and if any Voids or Comments. Note in order for the charts to accurately depict the data. All dates that a sample was run at any site must be represented in all sites data. For example, if a make-up run was scheduled at Rule for 7/20/2017. Then the other three sites would have the date 7/20/17 under the date section, but no additional information or a N/A under filter number.

D. The meteorological (Met) data for the run days can be gathered by either the closest 2025 sampler running on all the sample days or from one of the continuous monitors at the Air Lab Site.

a. For retrieving Met data from a 2025
    i. Open Microsoft Excel
    ii. Click File-> open-> L-drive-> air lab data-> ambient air monitoring-> pollutants-> PM 25 monitoring-> PM 25 datawands > the appropriate year-> open the month after the one you need-> rule-> select "all files" from drop down box at bottom of window-> click on earliest file saved-> delimited-> next-> comma-> finish
    iii. Highlight actual start date, amb temp ave, and press ave iv. Delete un-highlighted columns
    v. To save file:
        1. Click file-> save as-> name it (Met data month) -> use drop down box labeled "save as file type" and choose "excel 97-2003 template"
        2. Save file on L drive-> air lab data-> ambient air monitoring-> pollutant-> Met for PM 10 & TSP

b. For retrieving Met data from continuous monitor using Air Vision (preferred method)
    i. In the AirVision Program select Reports-> Monthly Report
    ii. Select the Monthly Data range which is being validated, 1 hour rolling average, backward. In the parameter selection select the AIRLAB site; parameter (AmbTemp and BP). Use the control click method to highlight more than one parameter
    iii. The report will have colored squares to indicate null values or flagged data. Print the report.

E. Enter the average ambient temperature under (Ta) and average ambient pressure (Pa) on the Pb Calculations sheet for each of the lead sample run days. Include where the Met data was obtained in the documentation folder.

F. Print Lab Analysis Report and Transfer result column data from the Analysis Report to "Pb mass" column of Pb Calculations sheet. See Appendix B.6 for sample lab analysis excel report. G. Click "Save" to adjust calculations with recently entered data.

Use the orifice (labeled with site name) specific look-up table using the T_a and P_o/P_a values, to obtain Q_m Enter Qm into the corresponding cells on the Pb Calcualtions sheet.

H. The PB Charts page should automatically pull from the Calculations page. Review the chart and make adjustments if not correctly pull data from the Calculations sheet. Print the Pb Charts upon completion. See Appendix C.8 for sample Charts page.

I. Review the QC%D column on the Pb Calculations page. Verify that all values are less than 10%. The sheet is formatted to turn red if the data is outside of tolerance.

J. Review the logbooks (or scans) and the monthly flow verifications and audits that bracket the data under review.

K. Under Flags/ Void codes column on the Pb Calulcations page apply any necessary flags or codes based upon review.

L. Save. And print the Pb Calculations page.

M. Pb Data Validation complete and ready for AQS entry, click “Save”

N. Prepare the monthly data folder. This should be a manila folder with prongs.

O. Insert the copies of the envelopes, and the met data on the right-side prongs. Insert the Lab report, and Pb Calculations and Pc Charts on the left side. The returned filters are inserted in the middle and package is rubber banded together

7.3 Entering Data into AQS

AQS is the data management system maintained by the US Environmental Protection Agency (EPA). Data is entered in the AQS following data validation and review by the Quality Assurance Officer. Instructions on data entry into AQS, data validation techniques, and use of the QA database can be found in the Data Handling SOP.

8.0 Documentation and Records Management

Recordkeeping is a critical part of the QA program. Careful documentation of sampling data will salvage samples that may otherwise be lost. The sheer repetition of recording data may result in errors; however, this cross-referencing between Data Envelopes (DEs), logbooks (LBs), and calculation spread sheets (SS) will allow the operator to pinpoint discrepancies that may result in a sample’s invalidation. This section is to highlight specific documentation that must be completed. This shall supplement any documentation elements described in previous sections.

Additionally, the QC checks (verifications), audits and void filter data are all entered into the Access file- AQM QA database by the Quality Assurance Officer

8.1 Documentation

The designation in parentheses indicates where the data must be inscribed or is calculated.

8.1.1 Sample Set up

1. Site designation, location, sampler model and sampler serial number (LB). This information must be recorded in the front of the logbook only at the commencement of monitoring unless a new sampler has been deployed.
2. Filter ID number (DE).
3. Installation Date & Time (DE, LB)
4. Sample date (DE) .
5. Initial ΔPstg (DE).

6. Unusual conditions that may affect the results (e.g., subjective evaluation of pollution that day, construction activity, weather conditions) (DE) (LB).
7. Operator's initials (DE) (LB).

8.1.2 Sample Retrieval

1. Retrieval Date & Time (DE, LB) 2.
Elapsed time of the sample run (DE).
3. Final ΔPstg (DE).
4. Existing conditions that may affect the results (DE) (LB).
5. Explanations for voided or questionable samples (DE) (LB).
6. Operator's initials (DE) (LB).
7. The calculated average flow rate (Q a) in m^3/min (SS).
8. The percentage difference between the actual and design flow rates (SS).
9. Average ambient temperature and barometric pressure on the sample run day (SS).

8.1.3 Chain of Custody

The chain of custody must be filled out completely. Each data envelope mailed shall be accounted for. The chain of custody shall include

A. The site name, Blank, or EPA test strip.
B. The site (station) ID.
C. The Sample run date D. Sample filter number
E. Operator's initials.

8.2 Records Management

Records Management is the professional practice of managing the records of an organization throughout their life cycle, from the time they are created to their eventual disposal. This includes identifying, classifying, storing, securing, retrieving, tracking and destroying or permanently preserving records.

8.2.1 Document Integrity

The majority of documents used in the TSP/Pb program are formatted through Microsoft Excel. This program performs the calculations and graphs the results. All documents shall be checked annually by the Quality Assurance Officer as part of the records management program for accuracy. The master

forms are in read-only format to help insure there are no alterations to the approved formulas and format during the year.

Document integrity is managed in hand written logs by using the one-line strike thru on incorrect data and skipped lines or blanks X'ed out to ensure backfilling does not occur.

8.2.2 Records Retention

Upon completion up the data validation from section 7.2 the filters in their data envelopes are bundled by month with the printed QC/ data sheets, met data and lab report. These bundles are stored in a filing cabinet until the conclusion of the sampling year. After the year is complete, and all sampling / QA/QC data are entered and validated in AQS, the monthly bundles are combined with any other logbooks and documentation for the year and placed in a permafile box marked with the year and pollutant. The box is stored in the data room. All TSP/Lead files are maintained at least 5 years in accordance to the KCDAQM Records Retention Policy.

9.0 Data Completeness

KCDAQM is required to have at least 75% data completeness annually. In the event KCDAQM, may not meet this requirement the Tennessee Division of Air Pollution Control and U.S. EPA- Region 4 APTMD will be contacted as soon as possible to assist. The Operator shall endeavor to schedule make up runs when a power outage, animal destruction, or other site related void filter occurs. Make up runs shall be within 7 days and should be before the next scheduled run day.

Appendix A

Equation 1: Pressure conversion

mm Hg = 1.868 X (in. H2O)

Equation 2: Flow rate Transfer Standard

$$Qstd\ orif\ (calc) = \frac{\sqrt{[(\Delta H_2O)(Ta\ /Pa)]} - b}{m}$$

Where: Qstd orif (calc) = actual volumetric flow rate as indicated by the orifice transfer standard, m3/min

ΔH_2O = pressure drop across the orifice, mm (or in.) H2O

Ta = ambient temperature during use, K (K = ^oC + 273) Pa =

ambient barometric pressure during use, mm Hg (or kPa) b =

intercept of the orifice transfer standard's calibration relationship. (TDEC) m

= slope of the orifice transfer standard's calibration relationship. (TDEC)

Equation 3: Absolute Stagnation pressure

$$Po = Pa - Pstg$$

Where:
Pa = ambient barometric pressure, mm Hg.
Pstg = stagnation pressure drop, mm Hg.

Equation 4: absolute average stagnation pressure ratio:

$$P_o/P_a$$

Where:
Po = average absolute stagnation pressure, mm Hg.
Pa = average ambient barometric pressure for the run day (not the retrieval day), mm Hg.

Equation 5: % Difference

$$= \frac{Q_a \text{ samp} - Q_a \text{ orif}}{Q_a \text{ orif}}$$

% Difference is always calculated as (actual – indicated) / indicated so what is being compared may change where a value is being placed.

Equation 6: Average Relative Stagnation pressure

$$P_{stag} \text{ avg} = (P_{stag} \text{ initial} + P_{stag} \text{ final}) / 2$$ Where:

Pav = average ambient barometric pressure for the run day (not the retrieval day), in H_2O.
P_{stag} avg = average stagnation pressure drop, in H_2O.

Equation 7: Total Standard Volume of air sampled

$$V = Qa \ X \ (t)$$

Where:
V = total standardized volume of air sampled, m^3.
Q a = average sampler flow rate (ambient T & P using look up tables), m^3/min.
t = total elapsed sampling time, min.

Equation 8: Pb mass concentration in µg/ m^3.

$$Pb \text{ Conc} = \frac{(10^6) \ X \ (Pb \text{ mass})}{V}$$

Where:
Pb Conc = Pb mass concentration, µg/ m^3.
10^6 = conversion factor, µg/g.

Pb mass = mass of Pb (as determined by atomic absorption at contract laboratory), µg. V= total standard sample volume, m^3.

Equation 9: Design Flow Rate % difference

$$= [100] \ X \ \frac{Qm \text{ (corrected sampler)} - 1.13}{1.13}$$

Appendix B

Appendix B.1 Certifications

IML Air Science
a division of Inter-Mountain Laboratories, Inc.
555 Absaraka, Sheridan, WY 82801
(307) 674-7506

CERTIFICATE OF CALIBRATION

Orifice Transfer Standard: 3614

Knox County Health Department: Knoxville, TN

Calibrated on 10/30/2017 with NIST traceable Dresser Roots Meter SN: 0629846

Calibration Expires: 10/30/2018

The reference flow rate through the orifice can be computed via:

$$Q_r = A(\Delta P_0)^B$$

where: A = **0.599** B = **0.519** Correlation Coefficient (r): 0.9996

ΔP_o = pressure drop across orifice (in H_2O)

Q_r = reference flow rate (cubic meters per minute)

The actual flow rate through the orifice can be computed via:

$$Q_a = \frac{\sqrt{(\Delta P_0)(\frac{T_a}{P_a})} - b}{m}$$

where: m = **0.986** b = **0.042** Correlation Coefficient (r): 0.9996

ΔP_o = pressure drop across orifice (in H_2O)

T_a = ambient temperature (Kelvin)

P_a = ambient pressure (mm Hg)

Q_a = actual flow rate (cubic meters per minute)

The flow rate through the orifice corrected to standard conditions can be computed via:

$$Q_{std} = \frac{\sqrt{(\Delta P_0)(\frac{P_a}{T_a})(\frac{298}{760})} - b}{m}$$

where: m = **1.574** b = **0.059** Correlation Coefficient (r): 0.9996

ΔP_o = pressure drop across orifice (in H_2O)

T_a = ambient temperature (Kelvin)

P_a = ambient pressure (mm Hg)

Q_{std} = flow rate at standard conditions (cubic meters per minute)

Kristy Kola — Reviewed

10/30/2017 — Date

IML Air Science
a division of Inter-Mountain Laboratories, Inc.
555 Absaraka, Sheridan, WY 82801
(307) 674-7506

CERTIFICATE OF CALIBRATION QUALITY ASSURANCE

Orifice Transfer Standard: 3614

Knox County Health Department: Knoxville, TN

Calibrated on 10/30/2017 with NIST traceable Dresser Roots Meter SN: 0629846

The following table compares actual flow rates measured by the NIST traceable roots meter standard verses actual flow rates calculated from the new calibration equation. Quality Assurance guidelines require the difference at each point to be within ±2%. A minimum of three measurement points are required within the flow rate interval of 1.019 to 1.246 m^3/min for PM10 samplers and 1.1 to 1.7 m^3/min for TSP samplers. Since IML has not performed a previous calibration for this orifice, the actual roots meter measured flow rates are not compared to the actual flow rates calculated from an as found calibration equation.

Roots Meter Standard Q_a (m^3/min)	New Calibration Equation Q_a (m^3/min)	% Diff	As Found Calibration Equation Q_a (m^3/min)	% Diff
0.865	0.865	-0.07%	N/A	N/A
0.953	0.960	0.73%	N/A	N/A
1.056	1.048	-0.82%	N/A	N/A
1.125	1.129	0.31%	N/A	N/A
1.240	1.241	0.01%	N/A	N/A
1.400	1.392	-0.55%	N/A	N/A
1.522	1.529	0.42%	N/A	N/A

Laboratory Raw Data and Linear Regression Points

Technician: Ken Trentacosti

Laboratory Temperature (°C): 22.70

Laboratory Pressure ("Hg): 26.20

POINT	ΔVolume m^3	Time Minutes	ΔP Roots mmHg	ΔP OTS "H2O
1	3.40	3.90	4.67	1.80
2	3.40	3.53	5.79	2.20
3	3.40	3.18	6.91	2.60
4	3.40	2.98	8.13	3.00
5	3.40	2.70	9.71	3.60
6	3.40	2.38	12.24	4.50
7	3.40	2.18	14.57	5.40

Pertinent Actual Flow Parameters					Pertinent Standard Flow Parameters				
POINT	Va m^3	Qa m^3/min	Dependent Variable		POINT	Vstd m^3	Qstd m^3/min	Dependent Variable	
1	3.375	0.865	0.895		1	2.976	0.763	1.260	
2	3.369	0.953	0.989		2	2.971	0.841	1.393	
3	3.363	1.056	1.075		3	2.966	0.932	1.514	
4	3.357	1.125	1.155		4	2.961	0.992	1.627	
5	3.349	1.240	1.265		5	2.954	1.094	1.782	
6	3.336	1.400	1.414		6	2.942	1.235	1.992	
7	3.324	1.522	1.549		7	2.932	1.343	2.182	

REFERENCES: 40 CFR 50, Appendix B, Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High Volume Method). 40 CFR 50, Appendix J, Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere and Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II: Ambient Air Specific Methods. (EPA 600/4-77/027a, June 1992). Sections 2.2.2.5 and 2.11.2.2.1.

Appendix B.2 Data Envelope

PM 10 & TSP MONITORING DATA

Sample Information

Station Location: Burnside Cello

Sampling Date and Day: Sun 9.10.17

AIRS Numbers: 0027-2

Operator: BJW

Sampling Time

Installation date and time: Fri 9.8.17 8:31est

Retrieval date and time: Mon 9.11.17 8:17est

	(in hours)	(in minutes)
Stop:		
Start:		
Elapsed Time:	24:00	

Filter Data

Filter ID: G 7522449

1 hr. Gross Wt. : _____ gms

24 hr. Gross Wt.: _____ gms

Tare Wt.: _____ gms

Audit

Weight:	By:	Date:
_____ gms		
_____ gms		
_____ gms		

Relative Stagnation Pressure Reading

Initial ΔP_{stag}: 20.1 in. of H_2O

Final ΔP_{stag}: 20.2 in. of H_2O

Average ΔPstag: _____ in. of H_2O

Results

Air Volume: _____ m^3

AQI only

1 hr. Sample Weight _____ gms

1 hr. Concentration _____ µg/m3

Weather Conditions

All PM 10 / TSP Filters

24 hr Sample Weight _____ gms

24 hr Concentration _____ µg/m3

*** Note: All above calculations use "Seasonal Flow"**

Comments

Appendix B.3 Chain of Custody

ERG

Chain of Custody TSP/PM10 Lead Analysis (EP-BPA-15-D-0004)

601 Keystone Park Drive, Suite 700, Morrisville, NC 27560

Page ___1___ of ___1___

SITE Knox County call #24

AQS Code 47-093-1017 (1-6)
0023 (7-11), 0027-01 (12-17) 0027-02 (18-23)

Submitter:
Test

Relinquished by: Barron J. White

Date/Time: 7/11/17 12:00pm

Received by:

Date/Time:

#	Filter ID	Date	ANALYSES: TSP (ICPMS)	PM10 (ICPMS)	PM10 (XRF)	Sample Volume (m^3)	Comments	ERG LIMS ID (Lab use only)
1	G7522694	7/6/2017	x					
2	G7522692	7/8/2017	x					
3	G7522687	7/12/2017	x					
4	G7522680	7/18/2017	x					
5	G7522677	7/24/2017	x					
6	G7522674	7/30/2017	x					
7	G7522693	7/6/2017	x					
8	G7522681	7/15/2017	x					
9	G7522683	7/18/2017	x					
10	G7522682	7/24/2017	x					
11	G7522673	7/30/2017	x					
12	G7522696	7/6/2017	x					
13	G7522691	7/8/2017	x					
14	G7522685	7/12/2017	x					
15	G7522678	7/18/2017	x					
16	G7522675	7/24/2017	x					
17	G7522671	7/30/2017	x					
18	G7522695	7/6/2017	x					
19	G7522690	7/8/2017	x					
20	G7522684	7/12/2017	x					
21	G7522679	7/18/2017						
22	G7522676	7/24/2017						
23	G7522672	7/30/2017						
24								
25								

Appendix B.4 Audit Field Sheet

Knox County Health Department
Every Person, A Healthy Person

KNOX COUNTY AIR QUALITY MANAGMENT
QUARTERLY AUDIT FIELD SHEET

TSP/Pb	**Please circle Reference device used for Audit**		
HiVol Cal	DeltaCal	Trical	TetraCal

Serial Number: _____ Calibration Date: _____

Date: _____ Date: _____

Site: _____ Site: _____
Orifice: _____ Orifice: _____

QaCFM For TSP | Amb Pres: _____ | Temp: _____ | Stag Pres: _____

QaCFM For TSP | Amb Pres: _____ | Temp: _____ | Stag Pres: _____

Leak Check: _____ {Between 17-24 inH20} Leak Check: _____ {Between 17-24 inH20}

Date: _____ Date: _____

Site: _____ Site: _____
Orifice: _____ Orifice: _____

QaCFM For TSP | Amb Pres: _____ | Temp: _____ | Stag Pres: _____

QaCFM For TSP | Amb Pres: _____ | Temp: _____ | Stag Pres: _____

Leak Check: _____ {Between 17-24 inH20} Leak Check: _____ {Between 17-24 inH20}

Field Sheet Lead

Appendix C

1 – Multi- point Field Sheet

Knox County Health Department
Every Person. A Healthy Person

Lead Multi-Point
Field Sheet

Site: _____ **Date:** _____
Time: _____

Technician: _____

Monitor: _____ Manometers: _____

Orifice: _____ Slope: _____ Intercept: _____

Ta = _____ °C = Temp Standard: _____
Pressure Standard: _____

Pa = _____ mm Hg

	obs	obs
Flow	ΔH2O (in H20)	Pstag (in H20)
1		
2		
3		
4		
5		

Notes:

A multipoint check should be done with a variety of stagnation pressures bracketing the normal operating stagnation pressure by approximately 5 inches of pressure (if normal stagnation pressure is 19 inches check five points from 14-24 inches of pressure).

Appendix C.2 Multi point Verification Calculations

King County Health Department
Every Person. A Healthy Person.

TSP Calibration Verification

Site:

Date:
Time:

Technician:

Manometers:

Monitor:

slope, m
intercept, b

From TS Orifice Calibration

Orifice:

Ta = °C = #VALUE! K

Temperature and Pressure observed using:

Pa = mm Hg = #VALUE! in Hg

Flow	obs ΔH2O (in H20)	obs Pstag (in H20)	conv Pstag (mm Hg)	calc Po = Pa-PStag (mm Hg)	calc Po/Pa (unitless)	calc Qorf m3/min
1			0.0	#VALUE!	#VALUE!	#VALUE!
2			0.0	#VALUE!	#VALUE!	#VALUE!
3			0.0	#VALUE!	#VALUE!	#VALUE!
4			0.0	#VALUE!	#VALUE!	#VALUE!
5			0.0	#VALUE!	#VALUE!	#VALUE!

Qorf = (y-b)/m

y = √[ΔH2O X (Pstd/Tstd) Ta/Pa]

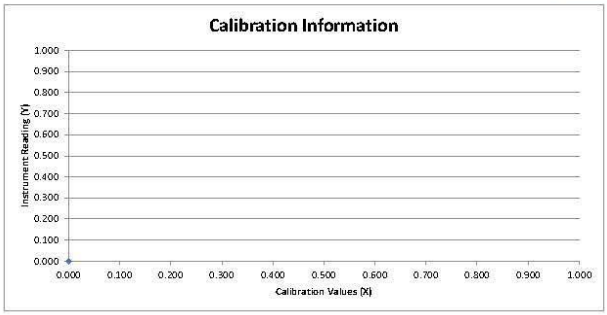
Qorf = (√[ΔH2O X (Pstd/Tstd) Ta/Pa]) - b / m

(Constant) Qorf	(Chart) Po/Pa	(Look up table) Qsamp	Qsamp in STP
#VALUE!	#VALUE!		#VALUE!
#VALUE!	#VALUE!		#VALUE!
#VALUE!	#VALUE!		#VALUE!
#VALUE!	#VALUE!		#VALUE!
#VALUE!	#VALUE!		#VALUE!

	1	2	3	4	5
Qorf (X)	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!
Qsamp (Y)	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!

Best Fit Concentration	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!
Percent Difference (Best fit Conc vs. Y)	#VALUE!	#VALUE!	#VALUE!	#VALUE!	#VALUE!

r	slope (m_1)	intcpt (I_1)	lin reg
#VALUE!	#VALUE!	#VALUE!	#VALUE!



3 - BGI Flow Verification

Knox County Health Department
Every Person. A Healthy Person.

Flow Verification Sheet

Site Name		15 minute Warm up	Yes	No
Date		Check for Leak	Yes	No
Time		Timer w/in 30min	YES	No
Operator		Site AQS Number		

Reason for Verification

Calibration device: BGI Hi-vol calibrator — Monthly — Quartely Audit — Motor Change: Final / Initail
ID #: 95

Bar Press		mmHg	Stagnation pressure		inH20
Temp		°C	convert Pa	0	mmhg
			Po/Pa	#DIV/0!	unitless

Qa CFM For TSP

0.00 CFM
0.000 m^3/min

Qa percent of difference from design
{1.13-Qa/Qa}x 100
#DIV/0! % of difference

Note: Design flow rate = 1.13 m^3/min

Is this value ≤ 10%
Yes No

Secondary QC	flow from look up table:	
% Diff Actual		
	%D is(Flow - Qa)/Qa *100%	#VALUE!
Action point 6 %, Failed QC >±7.1%		
Action point Timer : >20 min		

4 1 Point Flow Verification

Knox County Health Department

TSP 1-point Flow Verification

Site: _____ **Operator's Orifice** _____ Audit Stnd: _____ **Date:** _____
manometers: _____

Technician: _____

Monitor:
Model: Andersen/GMW Hi-Vol
VFC SN: _____

slope (m) _____ (TS Orifice calibration curve)
Intercept (b) _____

Sec 2.7.2, Step 4

	obs		conv	
T_a =	_____	°C =	#VALUE!	° K
P_a =	_____	mm Hg =	#VALUE!	in Hg

P_{std}/T_{std} = 0.1004

T_{std}/P_{std} = 9.959893

Pass Leak Test? Yes No

ΔH_2O = _____ in H_2O

ΔP_{stag} = _____ in H_2O = #VALUE! mmHg

With Orifice and Filter

Sec 2.7.2, Step 7

With Orifice and Filter

Q_{std} orif (calc)= #VALUE! m^3/min = #VALUE! m3/min cfm

$Q_a = (y-b)/m$

$y = \sqrt{[\Delta H_2O \times (P_{std}/T_{std})\ T_a/P_a]}$

$Q_a = \dfrac{\sqrt{[\Delta H_2O \times (P_{std}/T_{std})\ T_a/P_a]} - b}{m}$

P_a/P_a = #VALUE!

Q_a orf (tab)= _____ m^3/min

$P_a/P_a = 1-(\Delta P_{stag}/P_a)$ =
Absolute Stagnation Pressure Ratio

Q_{std} orif (tab)= Q_{std} (P_a/T_a) (T_{std}/P_{std}) = Q_{std} (T_a/P_a)(9.9599)

Q_{std} orif (tab)= #VALUE! m^3/min

Use the Absolute Stagantion Pressure Ratio (P_a/P_a)
and the temperature (T_a) to find the actual flowrate (Q_a)in the
monitor's look up table.

% difference between orifice flow rate and sampler flow rate
With Orifice and Filter

QC % difference = 100 X $\dfrac{Q_{std}\text{ orf (tab)} - Q_{std}\text{ orif (calc)}}{Q_{std}\text{ orif (calc)}}$ = #VALUE! %

Is this value ≤ 7.1% ? Yes No

if yes, continue
If no, investigate

Without orifice. Filter in a cassette only

$P_a/P_a = 1-(\Delta P_{stag}/P_a)$ = Absolute Stagnation Pressure Ratio

ΔP_{stag} = _____ in H_2O = #VALUE! mmHg **Without Orifice. Filter in cassette only**

P_a/P_a = #VALUE!

Q_a samp = _____ m^3/min

Use the Absolute Stagantion Pressure Ratio (P_a/P_a)
and the temperature (T_a) to find the actual flowrate (Q_a)in the
monitor's look up table.

Qstd = Qa * (Pa/Ta) * Tstd/Pstd = Qa (Pa/Ta) * 9.9599

Qstd = #VALUE! m3/min #VALUE! cfm

Q_a correct
Without Orifice. Filter Only

Q_a correct = $\dfrac{Q_a\text{ samp} \times (100 - \%\text{ diff})}{100}$ = #VALUE! m^3/min

% difference between Q_a correct and Q_a design

% difference = 100 X $\dfrac{Q_a\text{ correct} - 1.130}{1.130}$ = #VALUE! %

Is this ≤ 10%? Yes No

if yes, flow check has been passed
if no, investigate

Note: Design flow rate = 1.13 m^3/min

Note: If % diff is ≥ 6 % notifiy site operator.

Reference Method 2.2 "Determination of Suspended Particulates in the Atmosphere (High-Volume Method)

5 Audit Workbook Calculations page

Knox County Health Department

Lead Audit Calculations

Reference device used for Audit: _____ Serial number : _____
Date of Certification: _____

Date: _____ Bar Press _____ mmHg
Monitor ID: _____ Temp _____ °C
Site: _____

Qa CFM		
	Stag Press: _____	inH20
	Pa: #VALUE!	mmHg
	Po/Pa: #VALUE!	unitless
	Flow _____	
	(from table)	
	%D: #VALUE!	
	{Flow- Qa/Qa}x 100	

0.00 CFM
0.000 m^3/min

Date: _____ Bar Press _____ mmHg
Monitor ID: _____ Temp _____ °C
Site: _____

Qa CFM		
	Stag Press: _____	inH20
	Pa: #VALUE!	mmHg
	Po/Pa: #VALUE!	unitless
	Flow _____	
	(from table)	
	%D: #VALUE!	
	{Flow- Qa/Qa}x 100	

0.00 CFM
0.000 m^3/min

Date: _____ Bar Press _____ mmHg
Monitor ID: _____ Temp _____ °C
Site: _____

Qa CFM		
	Stag Press: _____	inH20
	Pa: #VALUE!	mmHg
	Po/Pa: #VALUE!	unitless
	Flow _____	
	(from table)	
	%D: #VALUE!	
	{Flow- Qa/Qa}x 100	

0.00 CFM
0.000 m^3/min

Date: _____ Bar Press _____ mmHg
Monitor ID: _____ Temp _____ °C
Site: _____

Qa CFM		
	Stag Press: _____	inH20
	Pa: #VALUE!	mmHg
	Po/Pa: #VALUE!	unitless
	Flow _____	
	(from table)	
	%D: #VALUE!	
	{Flow- Qa/Qa}x 100	

0.00 CFM
0.000 m^3/min

6 Concentration Calculations and Charts

Knox County Health Department
Every Person, A Healthy Person

Pb Concentration Calculations 9/18/2017

Ameristeel

Date	Filter #	Pbmass	$P_{initial}$	P_{final}	time	T_a	P_a	P_{avg}	P_a/P_s	Qm	Pb Conc	QC%D	Flags/Void Code	Comments
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		

Burnside

Date	Filter #	Pbmass	$P_{initial}$	P_{final}	time	T_a	P_a	P_{avg}	P_a/P_s	Qm	Pb Conc	QC%D	Flags/Void Code	Comments
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		

Burnside Collocated

Date	Filter #	Pbmass	$P_{initial}$	P_{final}	time	T_a	P_a	P_{avg}	P_a/P_s	Qm	Pb Conc	QC%D	Flags/Void Code	Comments
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		

Rule

Date	Filter #	Pbmass	$P_{initial}$	P_{final}	time	T_a	P_a	P_{avg}	P_a/P_s	Qm	Pb Conc	QC%D	Flags/Void Code	Comments
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		
								0.0	#DIV/0!		#VALUE!	#VALUE!		

Precision

Date	Burnside	Burnside Collocate	% Diff
1/0/1900	#VALUE!	#VALUE!	#VALUE!
1/0/1900	#VALUE!	#VALUE!	#VALUE!
1/0/1900	#VALUE!	#VALUE!	#VALUE!
1/0/1900	#VALUE!	#VALUE!	#VALUE!
1/0/1900	#VALUE!	#VALUE!	#VALUE!
1/0/1900	#VALUE!	#VALUE!	#VALUE!
1/0/1900	#VALUE!	#VALUE!	#VALUE!

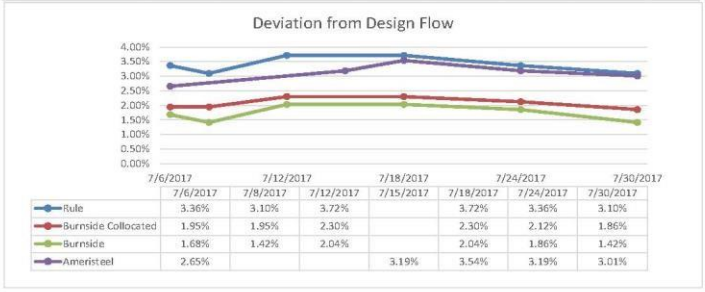
* only concentrations >.02 used to calculate precision

Pb Calculations rev 0
9/13/2017

Knox County Health Department
Every Person, A Healthy Person

July Pb Concentration Calculations Charts 9/18/2017

	7/6/2017	7/8/2017	7/12/2017	7/15/2017	7/18/2017	7/24/2017	7/30/2017
Rule	0.004	0.004	0.025		0.015	0.01	0.001
Burnside	0.023	0.047	0.053		0.025	0.013	0.002
Burnside Collocated	0.022	0.046	0.048		0.027	0.013	0.002
Ameristeel	0.035			0.043	0.029	0.028	0.008



	7/6/2017	7/8/2017	7/12/2017	7/15/2017	7/18/2017	7/24/2017	7/30/2017
Rule	3.36%	3.10%	3.72%		3.72%	3.36%	3.10%
Burnside Collocated	1.95%	1.95%	2.30%		2.30%	2.12%	1.86%
Burnside	1.68%	1.42%	2.04%		2.04%	1.86%	1.42%
Ameristeel	2.65%			3.19%	3.54%	3.19%	3.01%

Pb Charts rev 0
9/13/2017

Appendix E – Ozone SOP

Ozone Monitoring with UV Spectrophotometry
Standard Operating Procedure

KNOX COUNTY
TENNESSEE
HEALTH DEPARTMENT

Prepared by:
Rebecca Larocque
Knox County Air Quality
140 Dameron Avenue
Knoxville, TN 37917-6413

1.0 Identification and Approval

Title: “Ozone Monitoring with UV Spectrophotometry Standard Operating Procedure”

The attached “Ozone Monitoring with UV Spectrophotometry Standard Operating Procedure” is hereby recommended for approval and commits the Knox County Department of Air Quality, hence forth referred to as Air Quality, to follow the elements described within.

Air Quality

1) Signature: _____ Date: _____
   Brian Rivera, Director

2) Signature: _____ Date: _____
   Amber Talgo, Air Monitoring Program Manager

US Environmental Protection Agency, Region 4

3) Signature: _____ Date: _____
   Reviewing US Environmental Protection Agency Region 4 Staff

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2.2 Change Tracking

Change	Page #	Date of Change	Revision#

3.0 Introduction

3.1 Purpose

This document shall establish standard operating procedures (SOP) for the collection, review, processing, and reporting of ozone (O_3) ambient air quality monitoring data collected by the Knox County Department of Air Quality (Air Quality). This SOP is intended for individuals responsible for the ozone monitoring network and shall include the setup, operation, quality control and quality assurance procedures, and maintenance of ozone monitors. The U.S. Environmental Protection Agency (EPA) has determined ground level ozone is a health and environmental concern. For this reason, National Ambient Air Quality Standards (NAAQS) have been established for ozone. Hence, the air monitoring network for ground level ozone was established in Knox County, Tennessee. The monitoring season for East Tennessee is established by USEPA as March through October of each year.

3.2 Principles of Operation

The ozone analyzer operates on the principles of ultraviolet (UV) absorption by ozone. A sample of ambient air is drawn into the instrument and illuminated at one end of the optical bench by a UV lamp. A photodetector, located at the opposite end of the sample cell, measures the reduction of intensity of the light. To calculate the amount of UV absorbed by ozone, the measurement cycle is used to alternately obtain the reference and measurement UV intensity values by sending ozone-free air and sample air to the optical bench respectively. The cycle repeats every 6 seconds. The ozone free air is produced by using a dry air system consisting of silica gel and activated carbon external to the monitor, then a manganese dioxide scrubber within the analyzer. A microprocessor combines the reference and measurement UV signals, ambient temperature, pressure and the calibration factors to calculate the final ozone concentrations using the Beer-Lambert law.

3.3 Reference Documents

- *Measurement Principle and Calibration Procedure for the Measurement of Ozone in the Atmosphere),* 40 CFR 50 Appendix D
- *Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring,* 40 CFR 58 Appendix E
- U.S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurements Systems, Volume II, Ambient Air Specific Methods*
- Transfer Standards for the Calibration of Ambient Air Monitoring Analyzers for Ozone, Technical Assistance Document, Research Triangle Park, NC. 2013.
- Teledyne API *Model 400 E Technical Manual*, 23 May 2011
- Teledyne API *T703/ U Photometric O3 Calibrator User's Manual*, 3 Aug 2016
- Environmental Systems Corp, ESC 8800 series Data Logger Operation Manual
- *Quality Assurance Project Plan for Knox County Air Quality Management Ambient Air Monitoring Program, revision 0, November 30,2018*

3.4 Health and Safety

Prolonged or substantial exposure to ozone can lead to irritation of the respiratory system, reduced lung function and long-term damage to lung tissues. The following basic precautions should be taken while working with ozone instrumentation and equipment:

1. Properly exhaust any gas output from the ozone analyzer and any excess gas generated from the calibration system to the outside of the monitoring shelter.
2. Assure that all electrical power connections for the monitoring instrumentation and auxiliary equipment are properly grounded with a 3-wire plug.

3. All exterior power circuits must include a ground-fault interrupter.
4. When working on, troubleshooting, or repairing any electrical instrumentation or equipment the technician should remove any jewelry (rings, necklaces or chains) or other personal items which could conduct electricity and result in electrical shock or damage to equipment.
5. Before beginning any repair of electrical instrumentation or equipment (unless otherwise indicated by the equipment service manual), the power shall be turned off and the power cord disconnected.
6. When working on electronic instrumentation and components, use the provided antistatic wristband. Properly grounded, these devices will reduce the risk of a static electric discharge which could result in an electrical shock and damage to sensitive electronic components.

The instruments should be placed on a stable bench surface or in an instrument rack with at least 4 inches clearance in the back and 1-inch clearance for the sides, above and below. They must not be placed against heaters or air conditioners. Other routine safety practices shall be observed for the monitoring shelter: the shelter shall be organized and free of clutter for ease of entry and exit; the electrical service panel shall have unencumbered access; and all walking surfaces, platforms, and ladders shall be maintained to avoid slip and fall hazards.

4.0 Equipment and Software

This list of equipment and software provides an overview of how the ozone program is designed. Additional equipment may be mentioned further in the SOP. The operation of computer software is subject to change and the most recent manual for each piece of equipment and software is key for the proper operation.

Table 4.1 below provides a summary of diagnostic tools, parts and supplies necessary to operate and maintain an ozone monitoring site.

Equipment	Replacement/ Purchase Schedule
Ozone Analyzers (Teledyne 400E)	8-10 years lifespan
Ozone Transfer Standards (Teledyne 703 /U)	8-10 years lifespan
Flexible FEP tubing (1/4”) and (1/8”)	Yearly with some spare available
FEP, glass coated stainless steel, brass fittings (1/4”)	As needed, FEP more often than metals
Sampling funnel	Once, washing annual, replace as needed
NIST-traceable flow meter	Certified annually
Various hand tools (5/16th wrench, screwdriver, etc)	once
External PTFE particulate filter (47mm diameter)	Replaced biweekly
Charcoal, Purafil, silica gel	Yearly or as needed

Table 4-1 Equipment List

4.1 Calibration Standards Hierarchy

Due to ozone’s strong reactivity and instability, concentrated ozone cannot be practically stored and transported in compress cylinders like other gases. In order to generate accurate ozone concentrations when calibrating or evaluating the analyzers onsite, USEPA requires that precise ozone calibrators, called transfer standards, must be certified for traceability to other standards with higher authority and accuracy as summarized in figure 4.2 below.

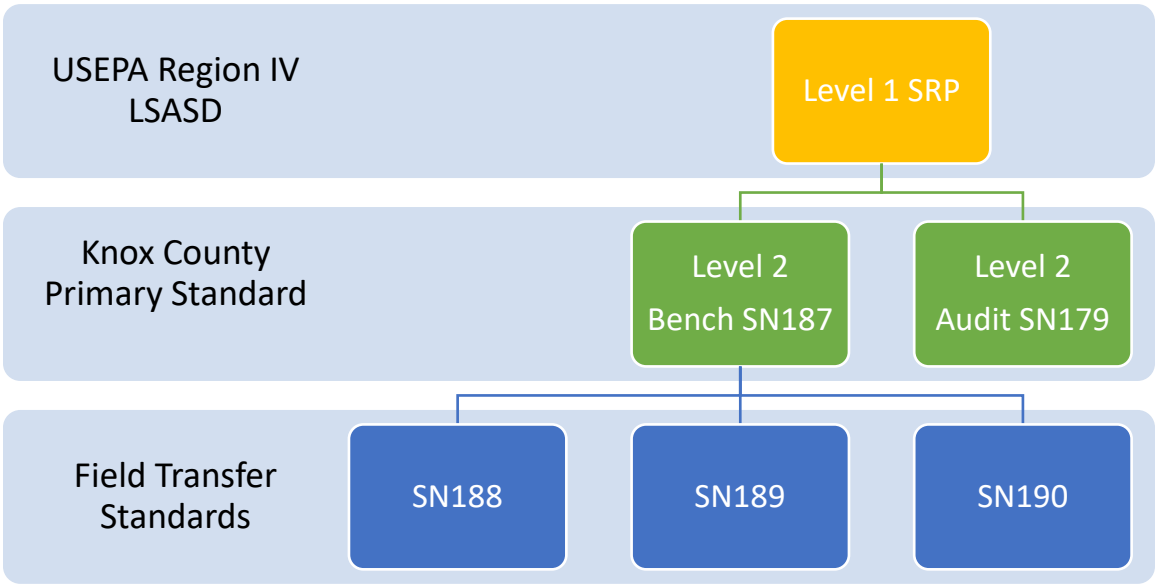


Figure 4-2 Ozone Standard Hierarchy Chart

- Level 1: Standard Reference Photometer (SRP) The EPA regional SRP is located in Athens, Georgia and maintained by EPA's Laboratory Services and Applied Sciences Division (LSASD)
- Level 2 Bench Standard – SN187 is a Teledyne calibrator703. To operate as a bench standard the measurement flow is plumbed into the photo in port, bypassing the machine's internal generation system. The Bench Standard is compared to the Level 1 SRP annually. See the M703E Calibrator Operator's Manual Section 6.11 Operating the M703E Calibrator as an O3 photometer for additional information on set up of the bench
- Level 2 Audit Standard – SN179 is a Teledyne 703U designed to produce low levels of ozone for auditing at 15ppm. The Audit Standard is compared to the Level 1 SRP annually. The Audit Standard must not be used to calibrate other ozone monitoring equipment.
- Level 3 – Field Transfer Standards – SN188,189,190 are Teledyne 700 Series Photometric Calibrators. Field Transfer standards should be calibrated to the Bench standard at the Beginning, Middle and End of Ozone season, but must be compared at least every 182 days.

4.2 Analyzers

Air Quality operates 2 field analyzers at the sites of East Knox Elementary and Springhill Elementary. The analyzers are Teledyne 400 Series Ozone Analyzers. SN4005, SN4006, and SN2013 is maintained as a backup instrument.

4.3 Data Collection and Storage

Air Quality operates Agilaire's AirVision software for data collection, storage, and management. The AirVision computer is a dedicated PC located at the Air Lab used only for running the AirVision Software. At the monitoring location, an ESC model 8832 Data System Controller, commonly called "Data Logger" is used to collect continuous data from the analyzers and transmit it via internet connection to AirVision. The 8832 Data logger also initiates automated QC procedures.

5.0 Installation Procedure

The air monitoring station contains several instruments linked together to form a system that will analyze, record, store and verify ambient ozone data. Once the structure to house the equipment is ready; the operator must obtain and install the instruments and supplies that will be required. Each site requires;

- An ozone analyzer
- A certified field transfer standard
- Probe set up – funnel, Solenoid valve, power supply, various Teflon fittings
- A laptop computer with AV Trend installed
- A data logger
- FEP Teflon tubing for sampling lines ¼ " outside diameter
- Charcoal and Silica canisters for zero air system
- An electronic temperature sensor connected to the data logger, and an independent min/max thermometer
- A logbook for each piece of monitoring equipment {analyzer and transfer standard}
- A Station site logbook.

5.1 Siting and Shelters

5.1.1 Siting Criteria

Siting requirements for ozone monitoring probes are summarized in Table 5.1 below. Siting a new monitor will not be the responsibility of an operator, but will be established by the Air Monitoring Program Manager in the Annual Network Plan and Quality Assurance Project Plan prior to installation. However, it is important for an operator to be familiar with the siting criteria to ensure that changes onsite do not violate the established parameters. These requirements are established in 40 CFR Part 58 Appendix E.

Parameter	Category	Siting Requirement
Inlet height	General	2-15 m above ground level
Inlet Radius Clearance	General	≥1 m vertically and horizontally away from supporting structure
	Near Obstructions (walls buildings)	≥2X height of the obstruction extended above the probe
	Near Trees above the probe	≥10 m from the drip line
	Arc of air flow	Unrestricted 270° arc that includes prevailing wind direction of highest concentrations
Distance from roadways	≤ 1,000 vehicles per day	≥10 m from nearest traffic lane
	1,000- 10,000 vehicles per day	≥20 m from nearest traffic lane
	>10,000 vehicles per day	Refer to 40 CFR Part 58 App E Table E-1
Distance from sources	General	As far as possible

Table 5-1 Summary of Siting Criteria

5.1.2 Shelter Requirements

Ozone analyzers and transfer standards must be installed in clean, dry, temperature-controlled shelters with reliable 110-120 power. Shelters must be secure, lockable, and have exterior fencing. Shelters must be equipped with adequate HVAC systems to maintain room temperatures that do not vary more than ± 2°C per hour. Analyzers may be operated in shelter temperatures from 5- 40 °C according to the

manufacturer's specifications, however every effort should be made to maintain within 15-35 °C. Additionally, the instruments must not be located against heaters or air conditions as it may affect performance.

5.1.3 Site Maintenance

Record all site maintenance procedures in the SITE log. A start of season and mid-season check list form is also used to track Items A-C. (Appendix A Figure 12-1)

A. Start of season and regularly thereafter: dust, sweep or vacuum all surface areas inside sample shelter
B. Start and Mid-Season: Change shelter's HVAC system filters
C. Start and Mid-Season: Change Analyzers internal particulate filter – See API T400 Manual Section 10.3.1
D. Start of Season and regularly thereafter: Replace IZAS canisters
    a. Charcoal new annually
    b. Silica new or regenerated replaced as indicating color approaches 25% blue.
    c. Change the particulate filter annually. After changing charcoal, use pump to blow through open system for 2 minutes, then change the particulate filter.

5.1.4 Zero Air System Set up

The Zero Air System (ZAS) consists of one canister of activated charcoal, 2 canisters of silica gel, one inline Teflon particulate filter. The external zero air system (XZAS) used for calibrations, multipoint verifications, and audits also requires a pump capable of maintaining 30 psi {required set output during checks is minimum 20psi}.

A. Connect the canisters together with short pieces of Teflon tubing and quick connect fittings. The air moves from bottom to top, so the first silica canister has a short piece of tubing as intake (or connected to the pump for XZAS). The top of the first canister is then connected to the bottom of the second silica, the top of the second silica is connected to the bottom of the charcoal.

B. Connect the top port of the charcoal canister to Teflon particulate filter using quick connect fitting on canisters, a short piece of ¼" FEP tubing and Teflon fittings on the particulate filter housing.

C. Then connect with FEP tubing the particulate filter housing to the Dry Air In port on the back of the Transfer Standard. If using with a pump for XZAS then connect to the "Zero Air In" port. Connection to the instrument is with a stainless-steel fitting.

D. Remember XZAS uses external pump, so the internal pump must be disabled. See Section 5.5.B for disabling internal pump for Calibrations and multipoint verifications.

5.2 Probe Configuration

Figure 5.2 below illustrates the probe design, one side illustrating the lines, the other side illustrating the insulated lines with inline filter on the sample line. The probe protrudes from a water tight box on top of the station. A Teflon or glass funnel is used to shield the ¼" sample line inlet from precipitation. Inside the box, the sample line is attached to a 20 volt solenoid valve. This valve provides a switch and is attached to the ambient sample line which goes through the insulated PVC pipe into the shelter and into the analyzer. The valve is also connected to the calibration gas line which originates from the calibrator, comes through the insulated PVC pipe and into the solenoid valve. Additionally, the electrical wires which operate the solenoid valve are run through the PVC pipe and into the shelter. See Figure 5.3 for wiring. The solenoid valve power supply is controlled by the data logger and a manual override plug. The valve is set to the "open" position

when no power is being supplied. This ensures the continuation of ambient monitoring in the event of power failure or having no plug connected.

It is most efficient to set up the probe assemble first, then using fish tape, run the sample line, calibration gas line, solenoid power cord, and insulation through the PVC extension, leaving excess on both ends, and marking the lines so there is no confusion between sample and calibration gas line. Then once instruments are in place, cut and attach fittings and connect to instruments.

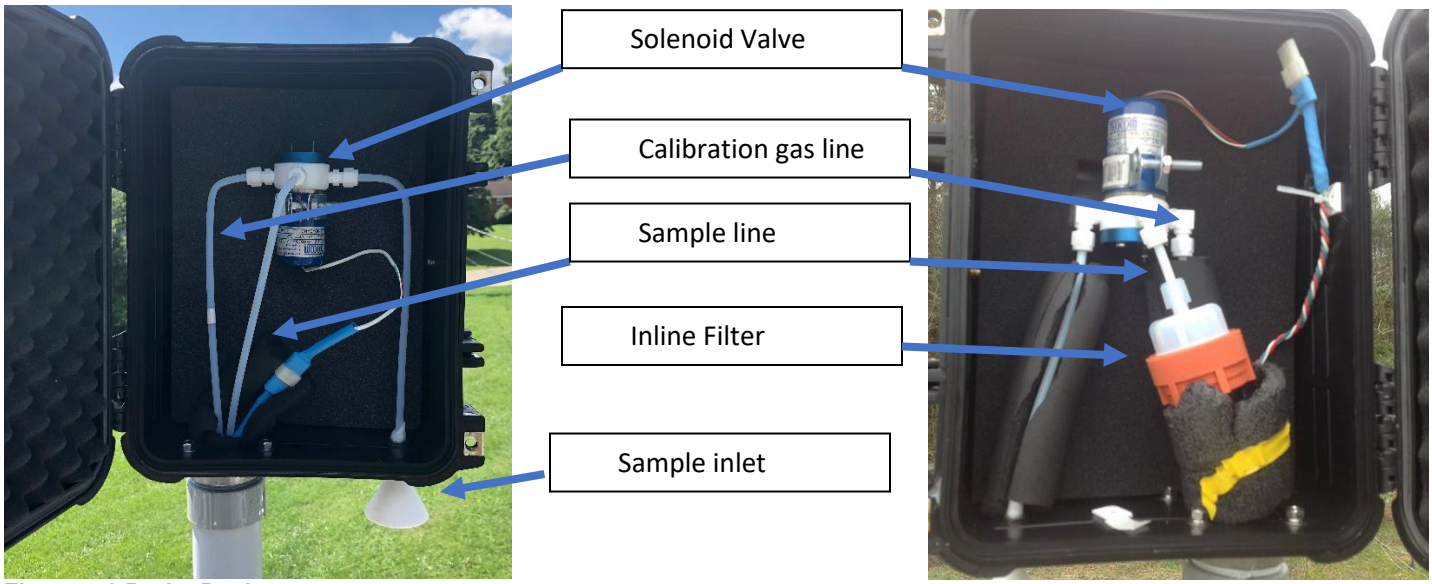


Figure 5-2 Probe Design

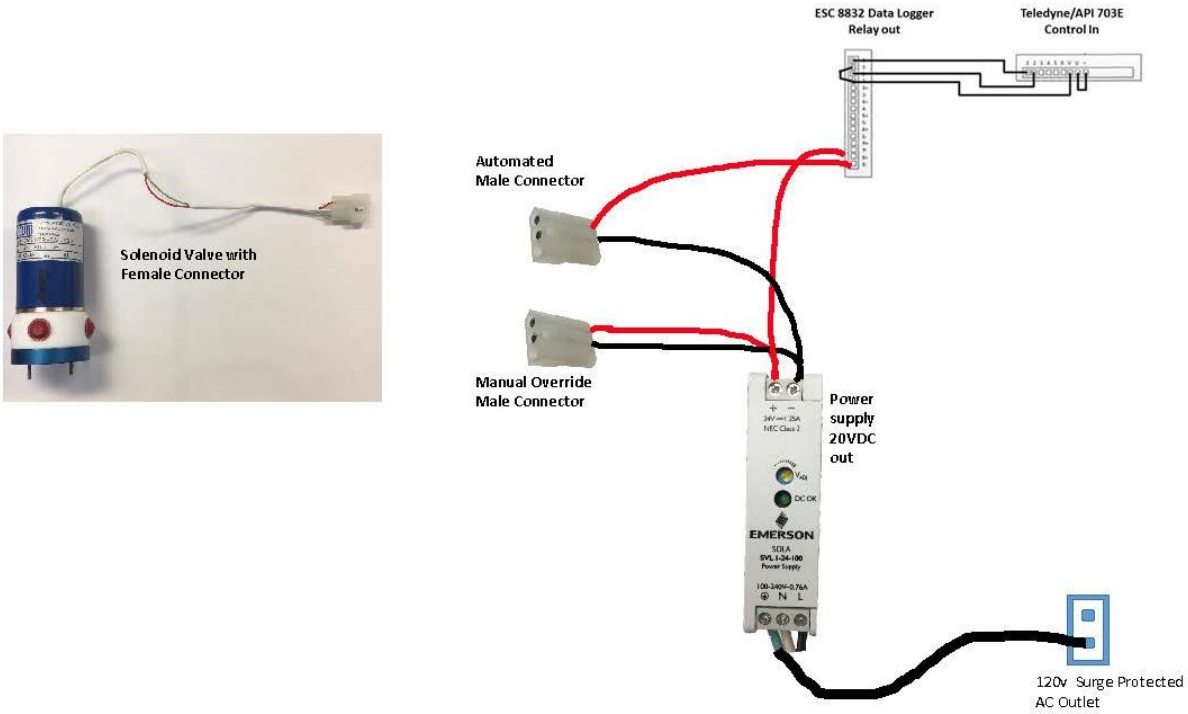


Figure 5-3 Solenoid valve wiring

5.3 Instrument Set up

5.3.1 Analyzer

A. Perform an initial inspection after transporting the instrument. Look for any damage, loose electrical connections or loose pneumatic fittings.
B. Connect the Teflon tubing from the ambient sample line that was run from the solenoid valve to an inline particulate filter housing and connect using Teflon ferrules and fittings. The inline particulate filter housing must be placed on the sample line after the solenoid value. It may be placed just after the solenoid valve inside the probe housing, or closer to the instrument inside the shelter.
C. Connect the inline particulate filter housing to the sample inlet on the rear panel of the analyzer using a cut to fit piece of Teflon tubing and Teflon fittings.
D. Connect one end of a ¼" tubing to the exhaust port on the rear panel using brass fittings. The other end of the tubing is vented to the outside via the exhaust manifold on the floor of the shelter.
E. Ensure the power switch is in the off position and plug the power cord into the back of the instrument and then into a surge protected outlet, preferably an un-interrupted power supply (UPS) back up.
F. Connect the Analyzer to the data logger using the RS-232 null modem cable to serial com port 0 on the back of the ESC 8832 data logger.
G. Power on the instrument.

5.3.2 Transfer Standard (Calibrator)

A. Perform an initial inspection after transporting the instrument. Look for any damage, loose electrical connections or loose pneumatic fittings. See Figure 5.4 for schematic.
B. Connect a length of tubing (1/4" Teflon) from the Transfer Standards Cal Gas 1 port to the solenoid valve in the probe box. Ensure, if present, the Cal Gas 2 port is capped.
C. Connect one end of a length of **3/8" outer diameter tubing** to the vent port on the rear panel of the transfer standard (using Parker part #4TUR6-361). The other end of the tubing is connected via push connect fitting to the exhaust manifold.
D. Connect one end of a length of ¼" tubing to the exhaust port on the rear panel of the Transfer standard. The other end is vented to the exhaust manifold via push connect fitting.
E. Connect one end of a length of ¼" Teflon tubing into the zero air in port via stainless steel compression fitting, then connect the other end to the inline particulate filter housing of the zero air system using a Teflon fitting.
F. Ensure the power switch is in the off position, and plug the power cord into the back of this instrument, then into a surge protected outlet.
G. Connect the data logger
    a. Connect one end of a RS-232 null modem cable to the connection on the back of the transfer standard, connecting the other end to the serial comm port 3 on the ESC 8832 data logger.

b. Connect the wiring (see figure 5.4 below for close up on configuration) Control In on the Transfer standard and Relay out on the Data logger

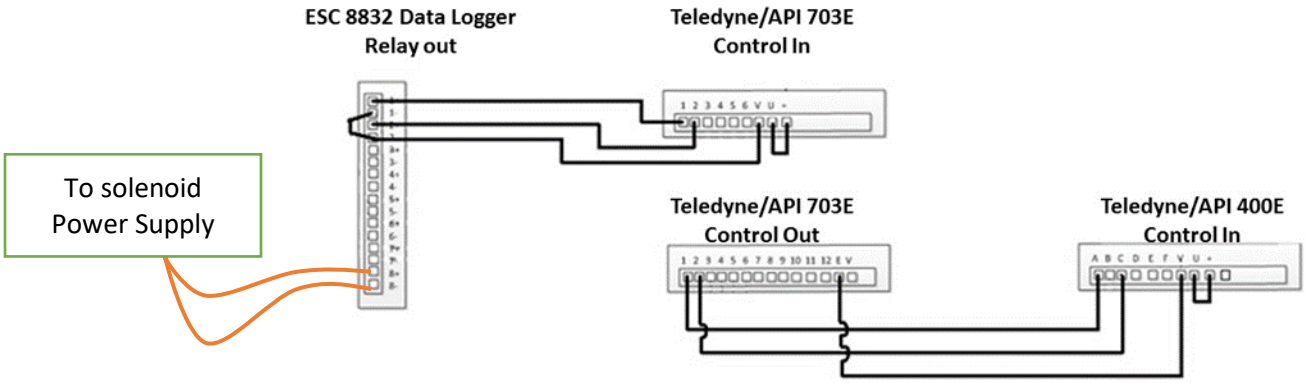


Figure 5-4 Nightly Zero Wiring

H. Power On the instrument.

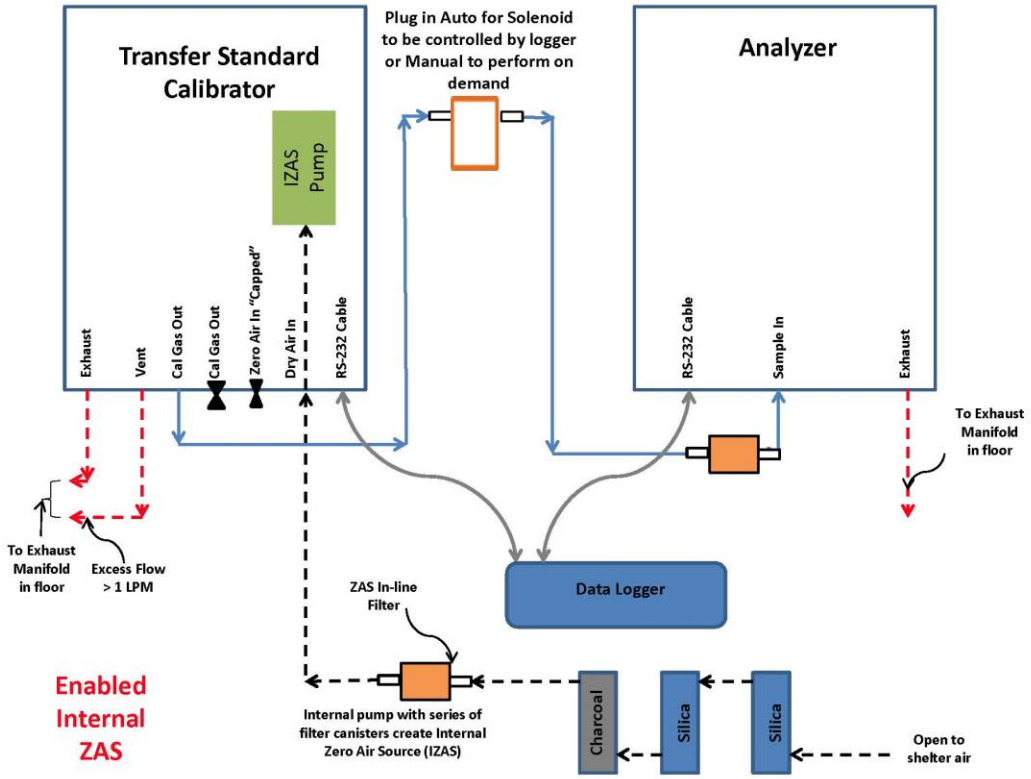


Figure 5-5 Normal Site Set-up

5.4 Leak Check

A leak check shall be performed during the preseason set up before any calibrations or verifications occur. A leak check is also a diagnostic tool for failed checks. The following instructions start after setting the system up as described in 5.2 and 5.3 above.

A. Disconnect the calibration gas line from the “Cal Gas 1” port on the back of the transfer standard.
B. Plug the calibration gas line using a stainless steel or FEP ferrule fitting plug. If no plugs are available, an operator may use their finger to close off the calibration gas line.
C. Plug the Manual Override male connector to the female connector for the solenoid valve to engage to link the sample and calibration gas lines.
D. Turn on the analyzer and wait for the initiation screen to scroll through.
E. On the main screen use the arrows to toggle through the parameters until the “Sample Pressure” is shown. If the sample pressure falls below 10 inHg and remains there the system has no significant leaks.
F. Toggle through the parameters until “Sample Flow” is shown. If the sample flow falls below 10 cc/min and remains there, the system has no signification leaks.
G. Uncap, or release your finger to remove the vacuum. Do not pressurize in this manner for long periods of time.
H. If a significant leak is detected, check all fittings. Tighten or replace if necessary, the inline particulate filter housing should also be tightened and checked carefully. Repeat leak test and investigation of leaks until passing.
I. If no significant leak is found, record the leak check results in the analyzer logbook.
J. Reconnect the Cal Gas line to the Cal Gas 1 Port on the back of the transfer standard.
K. Remove the manual plug from the solenoid power supply and reconnect the automatic plug.

5.5 Line Conditioning

Ozone is a reactive gas that is subject to interferences from a number of other gases and substances. As such it is important to scrub the calibration and sample line of interferences by conditioning the lines. Start with set up as completed through 5.3 and 5.4.

A. On the back of the transfer standard remove the zero-air line from the “dry Air In” port. Cap the port and connect the zero-air line into the “Zero Air In” port.
B. On the Transfer standard disable the internal zero air pump as follows:
    a. Select Setup, then More, then VARS
    b. Enter Password “818”
    c. Use “Next” until “ZA pump ENAB=ON” appears
    d. Select Edit, then “Off” then Enter
    e. Exit to main menu
C. Connect a piece of tubing from an external zero air pump to the bottom of the first silica canister of the zero air system.
D. Turn on the XZAS pump.
E. On the solenoid power supply, unplug the automatic male plug, and plug in the manual plug engaging the calibration gas line connection.
F. Generate 1000ppb ozone concentration on the Transfer standard
    a. Press the “TST” button until “ACT=STANDBY” appears on the display
    b. Press “GEN” button until “GENERATE” is shown on the display.
    c. Press “AUTO” button once.
    d. Toggle the front panel buttons until a concentration of “1000” appears and press “ENTR” button.
G. Allow the concentration to run through the system for 24 hours.
H. After 24 hours, press “STBY” button on the transfer standard to place it in standby mode.
I. Record line conditioning procedure completed in the Analyzer logbook.
J. Proceed to Calibration and Verification Procedures. (Section 6.0)
K. Initial calibration and verification procedures use XZAS, but if not proceeding with additional actions on the same day turn off the XZAS pump. But leave all other plumbing as is for calibrations.

5.6 Data Logger Procedures

The data logger programing is prepared using the Airvision software. Programing is managed and checked by the Air Monitoring Program Manager (AMPM). No changes of programing should occur without explicit knowledge and approval of the AMPM. For programing of automated checks and channel configurations, please see the most recent Airvision training manual as well as the ESC 8832 operating manual.

5.6.1 Log In ESC 8832 – you can use the keypad or attach a keyboard.

A. Press <ESC> key until the login menu screen appears
B. Use the down arrow key to highlight “Login/ Set User Level” and press <Enter>
C. Hold down the white <Ctrl> key and type the password “nerothecat” then press <Enter>.
D. You will be on the home menu screen. The log in does time out after no use and password will need to be re-entered

5.6.2 Procedure for Enabling/ Disabling logger channels.

A. From the home menu screen after logging in use the arrow keys to highlight “Configuration Menu” and press <Enter>
B. Use the arrow keys to scroll down and highlight “Configure Data Channels”. Then press <Enter>
C. Use the arrow keys to scroll and select “Enable/Mark Channel Online” to enable valid logger channels {Or Disable/Mark Channel Off line for disabling logger channels}. Then press <Enter>.
D. Use the arrow keys to highlight the desired channel to Enable or Disable per previous choice and press <Enter>.
E. You will automatically be taken back out to the Channel selection menu where you can choose another channel to mark.
F. After all desired channels are chosen, press <ESC> to back out to the Home Menu.
G. Check status of channels by following the highlight/enter process to select through the following menus “Real-Time Display Menu” -> “Show LARGE TEXT display”. Enabled channels will be in green, disabled in red.

6.0 Calibration and Verification Procedures

Ozone concentrations are not available in gas cylinders due to the reactivity of ozone. Therefore, a certified transfer standard must be maintained to generate ozone for calibrations and verifications. As described in section 4.1 Standards Hierarchy, Air Quality maintains 2 level II standards, the Bench Standard and the Audit Standard. These are certified by USEPA LSASD Region 4. The bench standard is then used to certify the transfer standards used to calibrate the analyzers. The following section describes the various calibration and verification procedures performed. Please make note as there are calibrations of transfer standards to the bench standard as well as calibrations of analyzers to the transfer standards. Much of the terminology is similar so be diligent in following what equipment is being discussed.

Calibration scale versus full scale (or operating range) is an important distinction when discussing calibration of standards. The Teledyne 400 series Analyzers and 703 Calibrators both have a full scale operating range greater than the calibration scale set. The more precise calibration scale tightens the relationship and covers more closely the expected values. The operating range is designated in the instrument manual as well as method designation.

6.1 Qualification, Certification, and Recertification of the Level III Transfer Standards

Qualification, certification and recertification forms described in this section can be found in the excel workbook labelled O3, Bench Forms located on the shared drive. The individual named forms listed in each check below are worksheets within this main workbook.

6.1.1 Qualification

Qualification consists of demonstrating that the transfer standard is sufficiently stable and repeatable to be useful as a site calibrator. First all transfer standards used by Air Quality must meet the requirements established in 40 CFR Part 50 Appendix D for a calibration system which is established prior to purchasing the instrument. Secondly a qualification test is performed on new transfer standards to verify local repeatability for those variables most often encountered in regular performance.

To qualify the equipment the following 5 zero/precision/span comparisons should be completed using the bench standard. See Qualification Instructions and Qualification worksheets found in the O3, Bench Forms excel workbook and Qualification Form found in Appendix A Figure 12.2 of this SOP.

A. The first ZPS shall be performed with the system set up just as in the multipoint bench comparison described in 6.1.2 below using points 400, 70, and 0. Including a 24 hour warm up for instrument. After completion of check, power off instrument, disconnect and cap all lines.
B. Perform a second ZPS after setting up and warming up for less than 24 hours. After completion power down, disconnect and cap lines.
C. Move instrument into a vehicle and drive around the block a few times. This is to ensure the instrument is stable and not prone to leaks. Upon return complete warm up and perform third ZPS. After completion power down, disconnect and cap lines.
D. Repeat Step C for a fourth ZPS comparison. Upon completion do not power down nor disconnect when complete, proceed to E.
E. Perform final ZPS while still warmed up from step D. This ZPS is performed in the reverse order of 0,70,400 rather than descending scale.

In all qualification testing, keep the read times less than 20 minutes apart, closer to 10 minutes when possible, while still achieving the required stability (<1.0ppb) to ensure adequate and accurate response time. To pass the qualification, no test should result in greater than 4 ppb or 4% difference (whichever is greater). If a test results in 3.0ppb/3% different note and discuss with Air Monitoring Program Manager about additional investigation into the variability tested.

6.1.2 Certification

Certification requires the averaging of six comparison between the transfer standard and the Bench Standard. Each comparison covers the range of ozone concentrations in the calibration scale and takes places on separate days. Use form “6 Comparison Evaluation” (Appendix A Figure 12-3) located in the O3,Bench Forms excel workbook to complete the calculations. For each comparison the following equations are used for calculating relationships.

$$m\,(slope) = \frac{\sum xy - \frac{(\sum x)(\sum y)}{n}}{\sum x^2 - \frac{(\sum x)^2}{n}} \quad \ldots\ldots\ldots\

$$\bar{m} = \frac{1}{6}\sum_{i=1}^{6} m_i \quad \text{..... (3)}$$

The average intercept from the six individual intercepts is determined using the equation:

$$\bar{I} = \frac{1}{6}\sum_{i=1}^{6} I_i \quad \text{..... (4)}$$

The relative standard deviation of the six slopes (sm) is determined using the equation:

$$s_m = \frac{100}{\bar{m}}\sqrt{\frac{1}{5}\left[\sum_{i=1}^{6}(m_i)^2 - \frac{1}{6}\left(\sum_{i=1}^{6} m_i\right)^2\right]}\% \quad \text{..... (5)}$$

The standard deviation measures the variation in the data set by determining how far the data values are from the mean, on the average. The value of sm in equation 5 must be ≤ 3.7% to certify. The quantity (sI) for the six intercepts is determined using the equation:

$$s_I = \frac{100}{\bar{m}}\sqrt{\frac{1}{5}\left[\sum_{i=1}^{6}(I_i)^2 - \frac{1}{6}\left(\sum_{i=1}^{6} I_i\right)^2\right]} \quad \text{..... (6)}$$

The value of SI in equation 6 must be ≤ 1.5 to certify.

6.1.3 Recertification

Involves periodic six-point comparisons between the field transfer standard and the Bench standard. Air Quality should recertify each transfer standard to the bench standard at the beginning, middle and end of ozone season. However, Air Quality must recertify at least every 182 days. Recertification uses the same calculations and form as the certification process. As well the acceptance criteria is the same for Sm ≤ 3.7% and Si ≤ 1.5. As Recertification requires one six-point comparison, which is then compared to the 5 most recent on file for statistical analysis. Additional to the Sm and Si acceptance criteria the new slope m, must be within ±0.05 of the previous slope. The form “6 Comparison Evaluation” (Appendix A Figure 12-3) is used to complete this as well.

6.1.4 Performing a Multi-point Comparison between a Transfer Standard and the Bench Standard

Note**:** Unlike site operations, the Bench and Transfer Standard require zero air to be fed from the same source during comparison and calibration procedures. The zero air system for the Bench Standard comparisons consists of (starting at back of the standard) a stainless steel “T” that connects both standards to the system, a Teflon particulate filter, 2 canisters of activated charcoal, 1 canister of purifil, 2 canisters of silica gel and an external pump.

Figure 6.1 below illustrates the set up for the Transfer Standard (Calibrator) to Bench Standard comparison and zero air system set up.

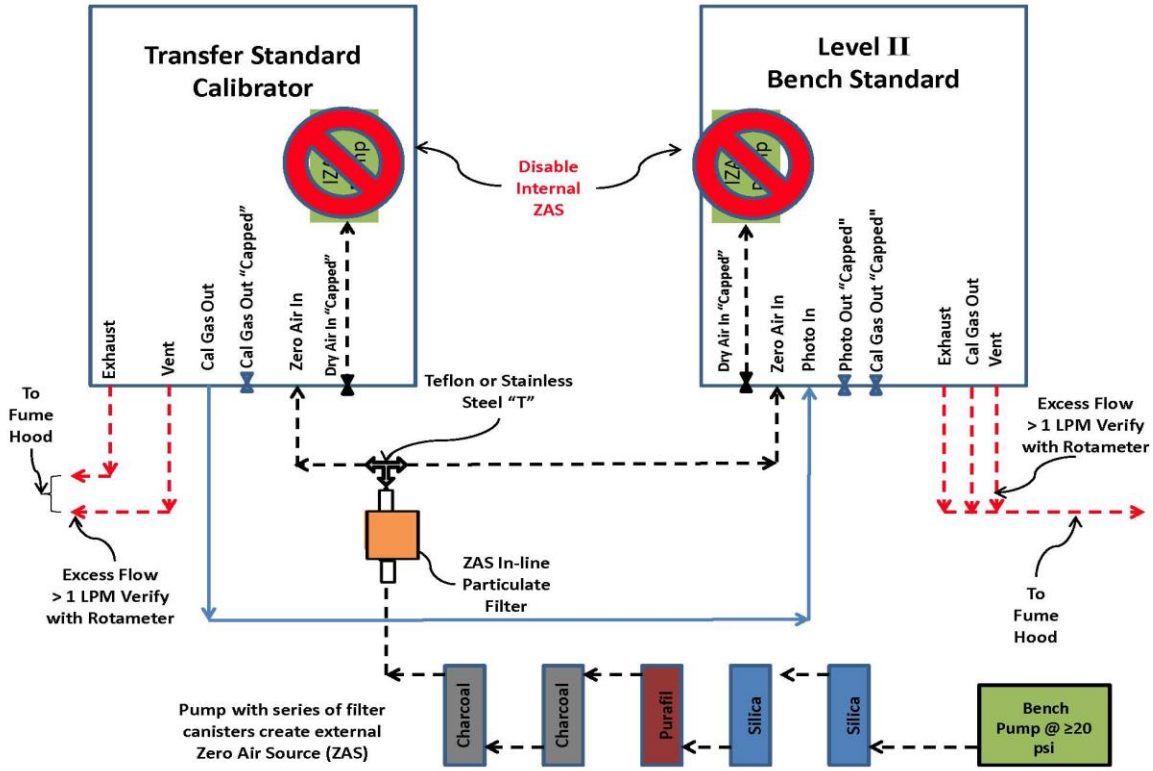


Figure 6-1 Transfer Standard to Bench Comparison Set-up

A. Connect the Bench Standard to the zero air system and the transfer standard calibrator according to Figure 6.1 above.
B. Power on the Bench standard and Calibrator for at least 12 hours for adequate warm up.
C. Power on the zero air system pump at least 10 minutes before generating ozone to allow the pump to warm up. Ensure the pump set to 20-30psi.
D. Confirm that the internal pump is disabled on the Transfer Standard Calibrator as described in 5.5.B of this SOP.
E. On the Transfer Standard Calibrator generate 500ppb ozone for 10 minutes to burn out any contamination in the system
    a. Press <TST> button until “ACT=STANDBY” appears on the display
    b. Press <GEN> button until “GENERATE” is shown on the display.
    c. Press <Auto> button once
    d. Toggle the front panel buttons until a concentration of “500” appears then press <ENTR> button.
F. Set up the Bench Standard to display the photometer readings.
    a. On the home Screen press <SETUP>
    b. Enter Password “818”
    c. Press <O3>
    d. Press<PHOT>
    e. Press <BCAL>
    f. Enter Password “717” press <ENTR>
    g. Press <CAL>
    h. Press <ZERO>
    i. Press <ENTR>
    j. Read ACT=___PPBO3”

G. During the burn out period verify the Bench and Calibrators Reg Pressure is set to near 15 psi and/or the Output Flow is near 5.0 LPM by using the <TST and TST> buttons. If adjustment is required on either, open the front panel and turn the pressure regulator adjustment knob.
H. Attach a rotameter to the vent port on the Transfer Standard Calibrator to verify the excess flow rate is >1.0 LPM. Record on the Form “Bench Printable” located in the O3,Bench Forms Excel workbook. See Appendix A Figure 12-4
I. Verify that the ESC 8832 Data Logger Displays both the Transfer Standard Calibrator and the Bench ozone readings correctly.
    a. The Bench Standard should be connected using an RS-232 cable to the logger Serial Port 2, the Transfer Standard Calibrator should be connected using an RS-232 cable to the logger Serial Port 3.
    b. Log into the ESC 8832 as described in section 5.6.1 of this SOP.
    c. Navigate to the Current Readings Menu, highlight and select “Show Large Text Display.
    d. The “ACT=” value on the Transfer Standard Calibrator should be corresponding to the “O3CAL” readings on the data logger.
    e. The “ACT=” value on the Bench Standard should correspond to the “Level 2” reading on the data logger.
    f. If only one, or neither are displayed, try power cycling the data logger, continuing to trouble shoot if that does not solve the issue, check the RS-232 cables, toggle the DCE/DTE on rear panel of instrument that is not connecting. The DEC/DTE switch relates to the cable used, and how the pins are aligned. This should not need to be changed if the same cables and set up are used each time.
J. Conduct the Comparison of the Transfer Standard Calibrator to the Bench Standard
    a. Press the <GEN> button until “Generate” is shown on the display.
    b. Press the <AUTO> button once.
    c. Toggle the front panel buttons until a concentration of “0000” appears and press the <ENTR> button. Enter the time on the Bench Printable under “Generating Time est”
    d. During the initial zero record the parameters on the Bench Printable Form and verify that all parameters are within acceptable ranges for the comparison.
    e. Record the readings for the initial zero on the Bench Printable for each instrument after the concentration has run through the system for at least 10 minutes and record the “Read est” time to ensure Generating Time vs Read time is at least 10 minutes different.
    f. Repeat steps a-e 6 more times substituting the following concentrations: 400,110,70,50,35 and zero.
    g. On the Transfer Standard Calibrator press the <STBY> button until “STANDBY” appears on the display.
    h. On the Bench Standard press <EXIT> on the Bench.

NOTE: if <Zero> is selected “Are you sure?” will appear on the display. The operator must always select “NO”. Selecting “YES” will adjust the zero point of the Bench Standard and thus losing the Bench Standard certification.

K. Transfer the values gathered for each point from the Bench Printable to the Bench Calculations form (Appendix A 12-5). This will verify that the individual comparison meets basic acceptance criteria. Print, sign and turn in both the Bench Printable and the Bench Calculations form to the QA ozone folder.
L. Use the Bench Comparison Sticky (Appendix B Figure 13-1) to enter the values into the Transfer Standard’s logbook. Logbook entries should contain more information than just the sticky provides, these are used to keep data entry neat.
M. The QA personnel or the operator will then add to previous comparisons on the 6 Comparison Evaluation form to verify the transfer standard is certified. This is printed and signed and placed in the Transfer Standards Comparisons Binder, as well as saved on the shared drive under the subfolder Pollutants->Ozone-> O3 Transfer Comparisons.

6.2 Calibration of the Transfer Standard to the Bench

Calibrations occur when the transfer standard's concentrations drift from the bench standard, after annual maintenance or major repairs. A multi-point bench comparison as described in 6.1.4 in this SOP should be performed prior to calibrating the instrument. This will ensure we have documented the ending state of the previous calibration before any changes are made. The Transfer Standard Calibrator and Bench Standard should be powered on and warmed up for at least one hour prior to performing any adjustments, with 24 hours being ideal.

A. Set up the instrument as described in Section 6.1.4 in this SOP Items A-I. {There should be no change as the instruments should be set up this way for the pre calibration multi-point bench comparison.
B. On the Transfer Standard Calibrator Press <SETUP>, Then <O3> Then <PHOT> then <BCAL>
C. Enter the password 717 and press <ENTR>
D. Press <CAL> then <ZERO> then <ENTR>
E. The Calibrator will begin generating zero allow the zero to run through the system for at least 10 minutes.
F. Record the Parameters of both instruments on the Calibration form located in the O3,Bench Forms excel workbook.
G. After at least 10 minutes, record the pre calibration slope and offset, and the pre calibration values for each instrument on the "Calibration" form (Appendix A Figure 12-6)
H. On the Transfer Standard Calibrator (NOT THE BENCH!!) press the <ZERO> button. "Are you Sure" will come up, select yes to calibrate the instrument zero point.

NOTE: You do not enter an actual value when calibrating zero, if the bench standard is more than ± 2 ppb from zero, investigate back pressure, and the zero air system before calibrating.

I. After calibration (H above) allow the machine to continue to run zero for an additional 10 minutes, repeat if necessary, or if calibrated to the bench record post calibration reading. Do not record post calibration slope and offset as this will change again during span calibration. Press <EXIT> to return to the Calibration menu.
J. Select <SPAN> then <ENTR>
K. Toggle the keys to select 400 as the Target span point. And press <ENTR>
L. Allow the concentration to run for at least 10 minutes then record the pre calibration span values.
M. Press the <SPAN> button, the instrument will ask for the actual concentration, enter the concentration reading on the Bench Standard and press <ENTR>
N. The instrument will ask Are you Sure? Select Yes.
O. Allow the instrument to continue to run the span concentration, as it will self-adjust to meet the target, for at least 10 minutes.
P. Record the post calibration values for the Span Target and the Post Calibration slope and intercept.
Q. Perform a Post Calibration Verification by following the procedures for the Multi-Point Bench Comparison detailed in 6.1.4 of this SOP.

6.3 Calibration of the Site Analyzer to the Transfer Standard

When possible, perform calibrations after the Transfer Standard and Analyzer have been onsite and turned on for a 24-hour warm-up. A minimum 1 hour warm up is required. This procedure uses form "Calibration" located in the O3, Site Forms Excel workbook (Appendix A 12-7). This procedure should never be performed if back pressure is present. Review Section 9.4 of this SOP for determining if a back-pressure compensation is needed. Additionally, if the transfer standard zero does not read within ± 2ppb from zero, troubleshoot leaks, and zero air system prior to calibration.

A. Connect the external zero air system and site instruments according to Figure 6.2 below.
B. Disable the Calibrator's internal zero air pump Confirm that the internal pump is disabled on the Transfer Standard Calibrator as described in 5.5.B of this SOP.
C. Disable the appropriate data logger channels {O3 and O3CAL} according to Section 5.6 of this SOP.
D. Plug in the manual over ride "M" plug for the solenoid valve to activate the calibration gas line.
E. Turn on the external pump for a minimum 10 minute warm up. Adjust, if necessary, the pump pressure to 20-30psi
F. While warming up the pump, generate 500 on the transfer standard to clear lines

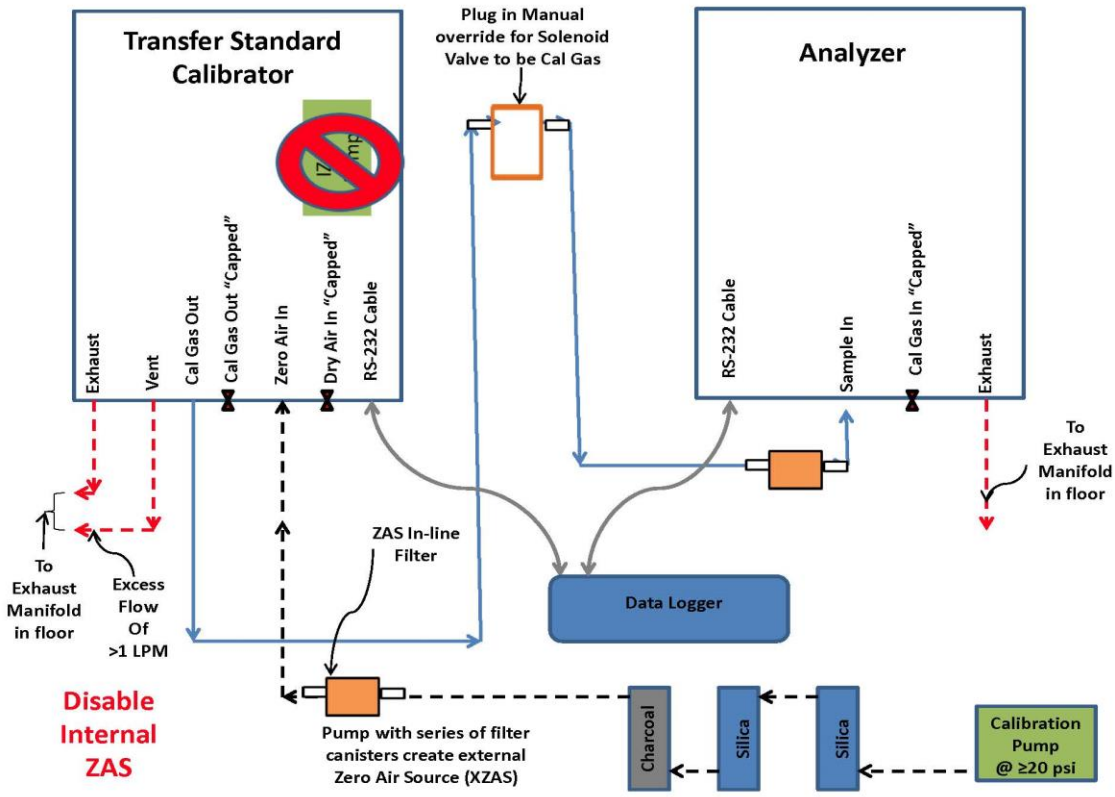


Figure 6-2 Calibration and Verification Set-up

G. Generate zero on the Transfer Standard
   a. On the Transfer Standard, press the <TST> button until "ACT=STANDBY" appears on the display.
   b. Press the <GEN> button, "GENERATE" will appear on the display
   c. Press the <AUTO >button,
   d. Toggle the buttons until a concentration of "0000" appears, then press <ENTR>
   e. Attach a rotameter to the vent port of the Transfer standard and record the vent flow rate on the form.
   f. Using the <TST> buttons, scroll through the parameters on the Transfer Standard, ensuring that the parameters are within the acceptable criteria and record.
   g. Using the <TST> buttons, scroll through the parameters on the Analyzer, ensuring that the parameters are within the acceptable criteria and record.

H. Calibrate Analyzer zero
   a. On the Analyzer press the <CAL> button and allow the zero air to pass through the system for at least 10 minutes.
   b. After the 10 minutes is complete and stability is <1ppb record the "Pre-zero" concentrations for the Calibrator, Analyzer and analyzer's stability on the Calibration Form.
   c. Press the <ZERO> then <ENTR> buttons on the front panel of the Analyzer. This will change the analyzer's equation and adjust the zero of the instrument.
   d. Allow the analyzer to stabilize for 10 minutes at the new zero setting, then record the "post-zero" concentrations for the Transfer Standard and the Analyzer

I. Set the Analyzer's internal span
   a. On the Transfer Standard press the <GEN> button "GENERATE" will be shown on the display.
   b. Press the <AUTO> button
   c. Toggle the buttons until a concentration of "0400" appears and press the <ENTR> button.
   d. Allow the analyzer to sample span gas for at least 10 minutes and reach a stability of <1ppb.

e. Record the “pre-span” concentrations for the Transfer standard, the analyzer and analyzer stability on the Calibration form.
f. On the front of the analyzer press <CONC>
g. Toggle the buttons until the concentration reading from the transfer standard is entered should be within few ppb of what was entered in part c above. Then press <ENTR>
h. Press the <SPAN> button and then <ENTR> to update the analyzer’s equation.
i. Allow the analyzer to stabilize for 10 minutes to the new span setting. Then record the “post-span” concentrations for the Transfer Standard, Analyzer and analyzer’s stability.
j. Press the <EXIT> button on the Analyzer and <STBY> on the Transfer standard to exit the procedure.

J. Complete the Calibration Form (Appendix A 12-7). Print and sign the form, bring in to the office, placing in the QA folder labeled for the site at which the analyzer is located.
K. Record the procedure in the Analyzer’s logbook including the pre and post slope and offsets.
L. Proceed to the calibration verification – skip to 6.4.2 (H) as pump is warm, and lines are clear.

6.4 Calibration Verification of Site Analyzers

6.4.1 Calibration Verification Frequency

Calibration Verification (also known as multi-point verification) procedures are performed after calibrations, but also at other times through the season as a more in-depth quality check than the zero-precision-span. A calibration verification shall be performed in the following situations:

- After a calibration adjustment
- Before the start of ozone season after site installation if no calibration was performed
- Whenever the transfer standard or analyzer is removed or returned to the site (repairs, mid-season and end of season)
- Before and after any repairs to the Transfer Standard or Analyzer.
- Upon failure of a zero-precision-span

6.4.2 Calibration Verification Procedure

The calibration verification involves comparing known concentrations of ozone generated by the transfer standard to the analyzer’s response for 5 upscale concentrations points and a zero. This procedures uses form Calibration Verification and Parameter Check. (Appendix A Figures 12-8 and 12-9)

A. Connect external zero air system to the site monitoring equipment according to figure 6.2 above.
B. Confirm that the internal pump is disabled on the Transfer Standard Calibrator as described in 5.5.B of this SOP.
C. Disable the appropriate data logger channels {O3 and O3CAL} according to Section 5.6 of this SOP.
D. Plug in the manual override “M” plug for the solenoid valve to activate the calibration gas line.
E. Turn on the external pump for a minimum 10 minute warm up.
F. Adjust the pump pressure to 20-30psi and record this value on the Calibration Verification Parameter Check Form (Appendix A Figure 12-9).
G. While warming up the pump, generate 500 on the transfer standard to clear lines. Use this time to also check that the Calibrator’s Reg Pressure is set to near 15 psi and/or the Output Flow is near 5.0 LPM by using the <TST and TST> buttons. Also check that there is no back pressure by reading the photosPres and watching it as the internal solenoid switches that the difference of the two values is not more than 0.1 inch. Additional information on Back Pressure compensation can be found in section 9.4 of this SOP.
H. Attach a rotameter to the vent port on the Transfer Standard Calibrator to verify the excess flow rate is >1.0 LPM. Record on the Calibration Verification Parameter Check form.
I. Set up the AV Trend software to display concentrations at 10 second intervals.
J. Challenge the Analyzer with known ozone concentrations
   a. On the Transfer Standard press <TST> button until “ACT=STANDBY” appears on the display
   b. Press the <GEN> button then <AUTO> button.

c. Toggle the front panel buttons until a concentration of “0400” appears and press the <ENTR> button.
d. After the concentration has run through the system for at least 10 minutes and the Analyzer has stabilized (Indicated by stability ≤ 1ppb) record the Transfer Standard concentration, analyzer concentration, stability and times on the Ozone Calibration Verification Form (Appendix A Figure 12-8). Also record all parameters during the zero point on form Verification Parameter Form
e. Repeat Steps b-d substituting the “0400” for the concentrations of 110,70,50,35 and final 0.
f. After all points are taken press the <STBY> button on the Transfer Standard.

K. After all data has been recorded on the Ozone Calibration Verification and Verification Parameter Form, print and save the spreadsheet according to the records retention guidelines in section 11.0 of this SOP.
L. Use a Verification sticky (Appendix B figure 13-2), or create a small chart to record the times, concentrations of transfer standard and analyzer and stability in the Analyzer logbook.
M. Review the results
a. The verification is successful if the following acceptance criteria are met:
i. The analyzer’s zero concentration is between -3 and +3 ppb.
ii. All analyzer concentrations vary by less than 2% of the best fit line or 1.5ppb whichever is greater from the transfer standard concentrations.
iii. The slope of the linear regression is between 0.9 and 1.1
iv. The intercept of the linear regression is between -5 and +5 ppb.
v. The correlation squared of the linear regression is > 0.95.
b. The Ozone Calibration Verification Form utilizes conditional formatting to check the criteria and should turn any criteria red that does not meet acceptance criteria. However, the Operator shall check the criteria as well.

7.0 Quality Control

Quality control (QC) procedures include automated and manual QC checks at routine intervals. All QC check concentration data must be captured by the data logger and AirVision computer to ensure consistency and accuracy of the check. Air Quality utilizes a few types of QC checks; automated zero span checks, the Precision Point which includes an established QC point and zero, the ZPS which includes the zero, precision, and span points, and final the multi-point verification which includes zero and 5 upscale points. The multi-point verification is identical to the calibration verification, and can be found in section 6.4.2 of this SOP.

In order to collect representative ozone data, it is important to consider the timing for QC checks and maintenance. Depending on the hours of the day, as little as three hours of downtime can render the entire day invalid for establishing a design value. According to EPA guidance the following guideline should be followed whenever possible:

- Automatic nightly zero span checks shall be scheduled from 11:45pm-12:15 am.
- Schedule and complete any maintenance work before 11am EST. This includes the necessary QC checks before and after maintenance.
- Review forecast when scheduling routine procedures that will take longer than 3 hours. Avoid performing on forecast high ozone days, or during peak ozone concentration hours.
- Coordinate with auditors (internal and external) to schedule auditing of instrumentation competition before 11am EST.

7.1 Automated nightly zero / span checks

Automated nightly zero/span checks are initiated by the site data logger. The Transfer Standard Calibrator uses its internal ozone generator and internal zero air system to challenge the analyzer with zero and 400 ppb concentrations. These are delivered through the calibration gas line which is activated by electrical signal to the solenoid valve to switch. The data collected is transmitted to the AirVision computer and should be

checked daily by the Operator. See Figure 5.4 in Section 5.3 Instrument Setup of this SOP for wiring diagram. When setting up the site, upon completion of all of the calibrations and verifications described in section 6.0 of this SOP, ensure that the internal zero air pump is enabled on the transfer standard calibrator in order for the automated checks to work. To enable the pump follow procedures detailed in Section 5.5.B selecting "ON" instead of "Off." Programing or changing the sequence should not be performed by the Operator without the Program Manager's approval. Review the instrument manual, data logger manual, and AirVision manual for guidance on automated sequence programing.

When reviewing the nightly zero / span checks, the Operator shall check for drift. Dirty sample lines or moisture in the lines can cause the 400 value to drift. Back pressure, or poorly maintained zero air system can cause the zero value to drift. Valid nightly zero / span checks must meet the following criteria:

- The sample flow of the analyzer must be maintained at 800 ± 80 cc/min
- Percent difference of analyzer and calibrator values at 400 ≤ ± 7%. Action Point set at ± 5%
- Zero reading ± 3 pbb. Action point at ± 2 ppb.

If the nightly zero/span exceeds any acceptance criteria or reaches the action point, a site visit is required to investigate any problems. Additionally, the Operator shall perform first a Zero / Precision / Span (ZPS) as described in section 7.2 of this SOP and then a line integrity test. The line integrity test consists of disconnecting sample and calibration gas lines, then connecting the cal gas line out from the transfer standard calibrator directly to the sample line in of the analyzer using a short piece of clean tubing and fittings. Then perform a ZPS using the short line. This will determine if the drift is caused by issues in the sampling line or within the instruments.

Percent difference is calculated using the following formula:

$$\frac{Analyzer\ value - Transfer\ Sandard\ value}{Transfer\ Standard\ Value}\ x\ 100\%$$

7.2 Manual Precision and ZPS checks

Precision and Zero / Precision / Span checks are performed manually by the Operator using the transfer standard calibrator with internal zero air. A one-point precision check (70ppb) and a zero / span check are required by the Quality Assurance Project Plan (QAPP) every 14 days. However, checks should occur alternating weekly. For example, the Operator performs a Precision Check (zero and precision point) one week, the following week the Operator performs a ZPS, and continues this pattern throughout ozone season. This provides twice the QC data and reduces the potential data loss from malfunctions.

7.2.1 QC Check Procedure

A. Use the Zero / Precision/ Span form (Appendix A Figure 12-10) and follow the Records Management procedures detailed in Section 10.2.1
B. Disable the O_3 channels on the data logger according to section 5.6.2 of this SOP.
C. Set AV Trend software to display concentrations at 10 second intervals.
D. Plug in the manual over ride "M" plug for the solenoid valve to activate the calibration gas line.
E. Challenge the Analyzer with known ozone concentrations
    a. On the Transfer Standard press <TST> button until "ACT=STANDBY" appears on the display
    b. Press the <GEN> button then <AUTO> button.
    c. Toggle the front panel buttons until a concentration of "0400" {OR 0070 if just performing Precision Point} appears and press the <ENTR> button.
    d. After the concentration has run through the system for at least 10 minutes and the Analyzer has stabilized (Indicated by stability ≤ 1ppb) record the Transfer Standard concentration, analyzer concentration, and stability on the Zero / Precision /Span Form.
F. Repeat step E replacing "0400" for "70" and then again for "0000".
G. While generating zero, conduct and record a parameter check on the Form.

H. Once complete, press the <Standby> button on the transfer standard calibrator.

7.2.2 Evaluating the results and return to ambient monitoring

A. Record the information collected in the Analyzer logbook using a sticky (Appendix B Figure 13-3).
B. Review the results
    a. If the analyzer's zero is between -3ppb and +3 ppb and the percent difference between the transfer standard and the analyzer at 70 ppb and/or 400 is < ± 5%, the precision has passed. Proceed to Step C.
    b. If the precision or span percent difference is >± 5%, but <± 7%, the precision has marginally passed and action is required. Review all parameters, check all connections, perform a leak check and repeat a full ZPS.
    c. If the precision or span point percent difference is >± 7%, the precision check has failed and data may need to be invalidated. Investigate to determine if failed precision can be resolved without performing a calibration adjustment. (as with marginally passed, leak check, repeat)
        i. Discuss failed precision with QA personnel and Program Manager to determine if data invalidation is required.
        ii. Record the date of the last successful QC check.
        iii. Review all parameters, check all connections, perform a leak check, repeating ZPS multiple times as investigated.
        iv. As a last resort perform a calibration adjustment, calibration verification and final ZPS
C. Unplug the manual over ride "M" plug from the solenoid valve, and re-plug in the "A" plug for the automated nightly.
D. Enable the data logger to resume ambient ozone data collection.
E. Bring original signed and dated copy of each form back to the office for QA personnel.
F. Upon returning to the office graph the QC check using AirVision software.

7.3 Site Visit Procedures

An entry must be made in the site logbook for every site visit. Section 7.2.2 of this SOP shall discuss entry protocol.

7.3.1 Station Inspection

Before entering the shelter, the fence and perimeter should be inspected for damage. Weather, vandalism, and vegetative growth may have resulted in required maintenance issues since the operator's last visit. Check that the sample probe is intact and undamaged, as well as clear from debris and insects.

Once inside the shelter the Operator should check for an obvious analyzer malfunctions including but not limited to water in sample lines, low flow, noisy pump, no display. Perform a weekly parameter check using the Ozone Weekly Parameter Check and Maintenance Form (Appendix A Figure 12-11). Record the information in the logbook using the "Weekly sticky" (Appendix B Figure 13-4) and Verify the shelter temperature is between 15°C and 35°C as shown on the front panel of the data logger. If outside the acceptable range, determine if the shelter HVAC system or temperature sensor is malfunctioning. Correct any malfunctions, note in site log book.

7.3.2 Logbooks

The Operator maintains multiple logbooks at each site. Each logbook shall be clearly identified on its cover. All logbook entries shall include the date, time, operator's initials, and details section. The Operator is responsible for detailing activities and observations. These are invaluable for data validation and serve as the official record of events. An operator must never remove pages, use white out, or completely black out an entry. A single line strike through and initials shall be used to signify a

correction. Blank spaces are discouraged, and should be X'ed out to prevent back filling. "Stickies" are printed on clear sticky back paper to provide structure and neatness for regular data entry.

A. Site Logbook – A site logbook must be maintained at each monitoring site and accurately reflect the site operations. The logbook will be identified on the cover and first page with the site name, AQS site number. Additionally, upon installation a start date for the logbook. Site logs shall be scanned at least quarterly. Each site visit must have a site log data entry. Additional information on silica gel maintenance, shelter or site maintenance, nearby construction, sample line cleaning provides a written record of observations of abnormal operations that are invaluable in data validation.
B. Analyzer Logbook – An analyzer logbook must be maintained for each analyzer. The logbook shall be identified with the equipment name and serial number on the cover. Inside entries shall detail maintenance, installation / site location, QA/QC activities.
C. Calibrator Logbook – The calibrator logbook must be maintained for each transfer standard calibrator. The logbook shall be identified with the equipment name and serial number on the cover. Inside entries shall detail maintenance, installation / site location, bench comparisons and calibrations.

7.4 Routine Data Review

The station operator has the primary responsibility for distinguishing valid measurements from invalid measurements caused by malfunction instruments. Each morning the ozone concentration data, shelter temperature data, nightly zero span checks, and flow rate data shall be reviewed using the AirVision computer.

A. Review data for concentrations, temperature and flow rate.
    a. Select the Favorites Tab
    b. Select the My Favorites tab and choose the desired data set
    c. Verify that 1 min average, and the correct site of interest is selected (Daily review choosing both sites at once is helpful for visual comparison)
    d. Select "Retrieve Data" and then "Time Series Graph" from the ribbon
    e. Confirm data is current within two hours. If not current, review AirVision manual for manual polling procedures and communication error help.
    f. Observe any spikes, flat-lines or anomalies. A site visit may be required to determine causes of any abnormalities. Provide explanation in the appropriate logbook for disruptions as applicable.
    g. Click the "Data Back" icon on the ribbon to review the previous day's data.
    h. After weekends or holidays, repeat f and g to review all data gathered.
B. Review the automated nightly zero span check
    a. Select the Favorites Tab
    b. Select the My Favorites tab and choose Calibration Results (Title not indicative – no calibration after check programmed)
    c. Choose the Site of interest
    d. Click Generate Report
    e. Review the results and expected values. Look for drift from 0 and 400.

7.5 Corrective Action Reports

Corrective action reports (CAR) are used to highlight and explain decision making regarding the diagnostic, repair, and data validation. (Appendix A Figure 12-12) A corrective action report can be initiated by an operator, QA personnel or program manager. They are often collaborative with each person providing input and detail for describing an occurrence and the decisions on repair and data quality that follow. The person who initiates the CAR completes and signs the document, but within the document notates the other individuals involved in each step. An example of when to use a CAR would be a failed QC precision point. The CAR would detail the failure, what occurred, the reason, and how data was affected. In this example the

reason for the failure would greatly affect the validation decisions, if the reason was a leak introduced when the operator changed the filter before the process, a review of the nightly would document and a passing ZPS after tightening the filter housing would document that no data would be affected. Conversely if a failing pump were the reason for the failure, data would be invalidated back to the last passing QC check.

The corrective action reports are a useful tool to gather all the information together during or shortly after an incident, rather than trying to piece together multiple logbook entries, notes, emails with instrument service department and strip charts well after the fact.

8.0 Quality Assurance

Quality assurance (QA) procedures are audits performed to identify system deficiencies which would not be detected by quality control procedures. These audits are performed by independent auditors using their own standards and zero air system.

Air Quality performs quarterly internal audits of each ozone monitor. The procedures are documented in the Technical Systems Audit SOP. The operator may be asked to accompany the internal auditor, so that the auditor can view the operator performing routine tasks. The actual concentration auditing by the audit level II standard vs the site analyzer is performed by the internal auditor. The internal auditor will notify the operator and the program manager of any deficiencies or concerns found.

Air Quality participates in the USEPA National Performance Audit Program (NPAP). The operator must meet and provide access to the equipment for NPAP auditors. The operator should disable logger for the duration of the audit, and re-enable upon its completion. As always document in the logbook any actions taken, and provide the logbook for the audit personnel to record into as well. The operator should remain onsite while the NPAP auditors are present to be available if any issues arise. The NPAP auditor frequently provides unofficial results to the operator, so that any results outside of ± 10% can be reviewed systematically.

The Tennessee Department of Environment and Conservation (TDEC) performs audits typically semi-annually. The operator must always accompany TDEC auditors while they perform an audit. TDEC provides a finalized report to the program manager at a later date. The operator should disable logger for the duration of the audit, and re-enable upon its completion. As always document in the logbook any actions taken, and provide the logbook for the audit personnel to record into as well

Operators shall notify the program manager or QA personnel if there are any questions to the procedures performed by any auditors.

9.0 Maintenance and Troubleshooting

9.1 Site maintenance

It is the operator's responsibility to maintain the monitoring station. Routine maintenance includes keeping the interior and exterior of the shelter clean, maintaining the siting criteria for trees and vegetation, and observing potential problems. Potential problems might include, shelter water leak, HVAC problems indicated by shelter temperature, accumulation of dust and debris, vegetative overgrowth, and insect infestation.

9.1.1 Annual Maintenance

Annual maintenance shall be performed before the start of ozone season. Annual maintenance shall be recorded in the site logbook

- Replace and condition sample and calibration gas lines.
- Change charcoal and silica gel in zero air system.
- Change the zero air system particulate filter Teflon 47mm (5UM).

9.1.2 Semi-Annual Maintenance

Semi-annual maintenance shall be performed before the start of ozone season, and midway (June or July through ozone season.

- Change the shelter's HVAC filters
- Change the shelter's HVAC thermostat batteries

9.1.3 Silica Gel Maintenance

Silica gel is utilized in the zero air system detailed in section 5.1.4 of this SOP. The silica gel is color indicating, turning blue to pink when absorbing moisture. The silica gel at each site shall be replaced when > 25% of the silica gel indicates pink in the first canister (the canister closest to the ambient air intake). The silica is then regenerated for reuse.

Silica gel crystals may cause lung irritation. Use of a dust mask and the fume hood while preparing and regenerating silica is required.

A. Collect all the spent silica to be regenerated and spread evenly in an aluminum or metal pan (use fume hood when pouring and spreading silica)
B. Place the prepared pans of silica into the baking oven and securely lock the door.
C. Turn on the oven and set the temperature to 120-200 °C.
D. Bake the silica for 1.5-2 hours.
E. Turn off the oven and allow it to cool – 3+ hours or overnight
F. Scoop regenerated silica into the storage jar.

Do not overheat or overcook the gel. Burnt or overheated gel will appear very dark to black in color. It is better to under-heat and repeat process than damage it.

9.2 Analyzer Maintenance

This SOP is not intended as a replacement for the analyzer's operation manual. Refer to the most recent operation manual for full maintenance and procedure information. All analyzer maintenance must be recorded in the analyzer's logbook.

A. Weekly –parameter checks are performed by the site operator and recorded in the analyzer logbook and on the weekly parameter check form (Appendix A Figure 12-11). The following information is recorded:
    - Analyzer test parameters
    - Minimum and maximum shelter temperatures from back up min/ max thermometer
    - Bi-weekly in-line filter changes
    - Status and maintenance of zero air system silica gel

    Information collected during weekly parameter checks is used to predict and isolate analyzer and site malfunctions. A sticky form has been created to aid in logbook entry (Appendix B Figure 13-4), but if unavailable, handwritten entry of all required information is acceptable.
B. Biweekly – replace the Teflon 47mm (5UM) particulate filter located on the sample line.
C. Semi-Annually – performed before the start of ozone season and midway through the season. Replace the Teflon 47mm (5UM) particulate filter located inside the analyzer. Perform a flow check according to manual section 11.3.5.
D. Annually - Annual maintenance should be performed before the start of ozone season. Refer to sections 11 and 13 of the instrument's operation manual. Clean the UV absorption tube, inspect all pneumatic tubing and fittings, replace the IZS zero air scrubber, perform a vacuum leak check, sample flow check, photometer UV lamp adjustment and rebuild or replace sample pump if needed.

9.3 Transfer Standard Calibrator Maintenance

This SOP is not intended as a replacement for the calibrator's operation manual. Please refer to the most recent operation manual for full maintenance and procedure information. All maintenance should be performed before the start of ozone season and documented in the calibrator's logbook.

A. Clean the absorption tube, verify “photo reference” value is towards the higher end of the acceptable range and adjust if needed.
B. Change dry air system particulate and charcoal filter.
C. Inspect pneumatic tubing and fittings
D. Rebuild or replace internal zero air system pump if needed.
E. Verify / calibrate sample gas and output gas flows.
F. Perform leak check and verify regulator and photometer pressures.

9.4 Back Pressure Compensation

Changes in the pneumatic connections to and from the Teledyne calibrators (700 T & E Series) can cause a difference in the back pressure within the device. As pressures affect the internal calculations, drastic changes can cause an instrument and the instruments calibrated to it to be less accurate. Back pressure issues should be investigated if a calibrator has difficultly with negative 0 values. An operator should perform the following back pressure review when first installing a transfer standard calibrator in the field, as well as when performing bench comparisons, or other instances when the pneumatic connections (length of tubing, different pump, etc) changes.

After the instrument has warmed up and flows are stable, while generating a concentration, review and record in the calibrator logbook the photometer pressure as it switches between the two sampling lines within the instrument. You can hear the click of the switch. If the difference is more than 0.1 in Hg then a back-pressure compensation is needed. Review Teledyne Erratum07428, and instructional video: https://youtu.be/SyB0aKtrvYo

If a compensation is needed, first perform a ZPS as detailed in section 7.2 of this SOP to create an “as-found” starting point.

A. After completion of the ZPS, using Back Pressure Compensation Form (Appendix A Figure 12-13) generate a concentration between 75-100 and allow the instrument time to stabilize, at minimum 5 minutes, but typically 10. Record the Initial parameters and p Δ gain according to the form.
B. While the instrument is generating ozone, navigate the following:
    a. Setup ->More -> DIAG -> Then enter password 929
    b. Click Next until BACKPRESSURE COMPENSATION is shown.
    c. Press ENTR to start the procedure
C. The operation will take a few minutes, once complete, EXIT to the main menu, and record the Final parameters and p Δ gain.

10.0 Data Validation and AQS Entry

Results from quality assurance and quality control events are reviewed to determine if concentration data collected is valid before it is submitted to AQS for use. The operator performs the first round of validation procedures through the daily review of data and the assignment of flags and null codes for routine operations. Further validation and statistical review are performed by QA personnel and detailed in the QAPP and Data Handling SOP. This section details the routine procedures and basic knowledge of other procedures needed by the operator.

10.1 Quality Data

Table 10.1 indicates the quality data goals and corrective action if goals are not met. These goals are in many cases set higher than the validation requirements to ensure corrective action is taken, and problems are resolved before data is deemed invalid.

Data Quality Indicator	Frequency	Objective	Corrective Action
Nightly Zero/Span	Daily	±5% span, ±3 ppb zero	Visit Site to investigate Check warnings Document actions Perform manual calibration verification
Operating Parameters	Weekly	Sample flow 740-860 cc/min, Shelter Temp 15°-35° C	Check for leaks, Investigate, Advise Management Perform Cal Verification
Manual Zero/Precision/Span	Bi-weekly	±5% precision &span, 3ppb zero	Check for leaks, repeat test Check parameters Recalibrate
Quarterly Internal Audit	4 times a season	±7% every point ±3ppb zero	Check for leaks, Investigate, Advise Management Perform Cal Verification
Completeness	Continuous 1-hour averages March – Oct.	80%	Notify Lab Manager if site down for more than 6 hours.
Manual Calibration Verification	Start, mid and End Season	±4% all points. ±3ppb zero, linear regression between 0.9 and 1.1.	Check for leaks, repeat test Check parameters Recalibrate

Table 10-1 DQI Goals

10.2 Validation Requirements

Please refer to the KCDAQM QAPP for additional information on verifying data quality.

A. Verify all equipment certifications / recertification requirements are met.
B. Verify that all QC procedures for nightly zero / span, precision checks, ZPS, multi-point verifications and weekly parameter checks were completed and valid.
C. Review data for flags and null codes assigned in AirVision.
D. If the results from a QA or QC event do not meet the acceptance criteria, concentration data will be invalidated back to the last QA or QC event which does meet the criteria using code AS. See table 10-2 for a list of the most commonly used null data codes.

Code	Meaning	Code	Meaning
AN	Machine Malfunction	BA	Maintenance
AS	Poor QA Results	BC	Calibration Verification
AT	Calibration	BF	ZPS
AV	Power Failure	BL	Audit
AX	Precision Check	BG	Missing O3 Data Not Likely to Exceed Standard
		QV	Multi-point Verification

Table 10-2 Common Null Codes

10.3 Validation Procedures

This procedure is repeated for each ozone site.

A. In the AirVision program select Reports - > Monthly Reports
B. Select the monthly data range which is being validated; 1 hour rolling average, backward. In the parameter selection select the ozone site (East Knox or Springhill), parameter name (03ppm) and click Generate Report
C. The report will have colored squares to indicate null values or flagged data. Print the report.

D. Review each null or flagged hour. Using the EPA's qualifier codes, assign a code, or notate valid data for each null or flagged hour on the printed sheet. Use table 10-2 above for operator assigned null codes. For example, the operator reviews the report, sees a red coded hour and the notation of disabled logger. This corresponds to the QC check they performed of a ZPS which has documentation and logbook entry. The operator shall code this hour as invalid BF.
E. Review the minute data for unknown flagged data, using the average data editor techniques described in Section 7.4 of this SOP by choosing linear data rather than the graph option. This will show the actual values and a column listing the flags applied by the logger.
F. Update the AirVision hourly data with the null code decisions. Click on Data Editors - > Average Data Editor. Again, choose the selected date range, the average interval of 1-hour average of 60 minutes, the site name and parameter. Select Retrieve Data.
G. Locate the hour block to receive a null code. Click on the box under AQS Null code and choose from the drop-down choices. Save Changes
H. Repeat the report generation described in A and B above. This time click "show null codes". Print the report.
I. Sign each printed monthly report and any written documentation of validation decisions and placed in the site and pollutant specific QA folder for review by next level validation.

10.4 Entering Data into AQS

AQS is the data management system maintained by the US EPA. It's assumed in these instructions that the user has a username, required access, and basic training provided by EPA. AQS is accessed through the internet. The website name may change. The EPA also uses the ENSC program to upload data into AQS. This program also requires a user, name password and basic training. The operator does not have the primary responsibility of uploading data into AQS. That function is performed by QA personnel following review of the first level data validation provided in Section 10.3 of this SOP. This section is included to provide a basic overview of the process as a small agency back up duties are shared. Additional information is provided in the draft Data Handling SOP.

A. In AirVision create an AQS file for uploading. Click Reports - > AQS Text Report. This shall be completed at least quarterly upon completion of the validation procedures but can be completed more frequently.
B. Select the date range, the average interval of 001H, and the site. You may hold the control and click multiple sites for one report.
C. Click "Save to File" and place on the shared drive in a file by year, pollutant, Data to AQS. S://Air_Quality_Lab/Air Monitoring XXXX (YEAR)/Pollutants/ Ozone / Data to AQS
D. Log into the ENSC program with username and password.
E. Select Exchange Network Services
F. Select AQS Submit: Send Files to the Air Quality System.
G. Browse to the file created and saved in C above, fill in the user data (email and AQS user id).
H. Then enter
    a. Screening Group: Knox Co, Tn
    b. File type: Flat
    c. Final Processing Step: Post
    d. Stop on Error: No
I. Click send Data. Notify the Program Manager that the data has been exported, initial and date the QA tracking sheet. (Appendix A figure 12-14)

11.0 Records Management

Recordkeeping is a critical part of a quality system. Careful documentation of sampling data will salvage data that may otherwise be lost. This section highlights the specific documentation that must be completed. This shall supplement any documentation elements described in previous sections.

11.1 Documentation

The operator is required to thoroughly document all quality control checks, maintenance, site visits and troubleshooting procedures related to the ozone network. The documentation is used during the data validation process to determine the validity of the ambient ozone concentrations obtained by the analyzer and to determine the appropriate null data code for missing or invalid data.

11.1.1 Logbooks: Site operators must keep logbooks onsite to document all procedures involving the analyzer and the site itself. The logbook shall be bound and page numbered. All logbook entries shall be written in waterproof ink. Corrections to the logbook shall be made with a single line through the entry, the site operator's initial and the date of the correction. Additional information on logbook documentation can be found in the QAPP Section 9 and training How-To Video located on the shared drive.

11.1.2 Excel Spreadsheets: The site operator is required to complete the appropriate excel spreadsheet-based forms during QC checks. All forms are to be completed and printed at the site. These official forms shall be signed and dated promptly. The operator should also save the excel file on the site computer.

11.1.3 AV Trend Software data and Graphs – when a communication problem arises the onsite AVTrend concentration data is used as a backup digital database which can be used to create excel graphs for QA/ QC activities. Excel graphs may be created using 10 second data from the analyzer and the transfer standard calibrator.

11.2 Records Management

Records management is the professional practice of managing the records of an organization throughout their life cycle, from the time they are created to their eventual disposal. This includes identifying, classifying, storing, securing, retrieving, tracking and destroying or permanently preserving records.

11.2.1 Document Integrity

The majority of documents used in the ozone program are formatted through Microsoft Excel. This program performs the calculations and graphs the results. All documents calculations shall be randomly checked annually as part of the records management program for accuracy of the formulas. The master forms are in read-only format to help insure there are no alterations to the approved formulas and format during the year.

All documents produced in the field shall be signed and dated by the operator. An electronic copy shall be saved on the site computer. These along with logbook entries may be used to recreate lost or damaged documents. All documents shall be scanned and saved in the Access database. This is performed and managed by QA personnel and detailed in the Draft Data Handling SOP. The scanned originals serve as an additional electronic record back of in the event of the loss of the physical document.

11.2.2 Records Retention

Upon completion of the ozone season all QC checks, data validation charts and decision documentation are bundled. These bundles are stored in a filing cabinet until the conclusion of the

sampling year. After the year is complete and all required data is uploaded to AQS, these bundles are combined with logbooks and any additional documentation for the year and stored in a filing cabinet. All ozone files are maintained according to the current Records Retention Policy.

The shared drive file server is maintained by the county IT department and backed up nightly. Any problems with lost data should be forwarded to the Computer Help Desk.

12.0 Appendix A – Forms

Knox County Health Department
Every Person. A Healthy Person

Ozone Site Maintenance

Site Name: _____

Site #: _____

PRE-SEASON

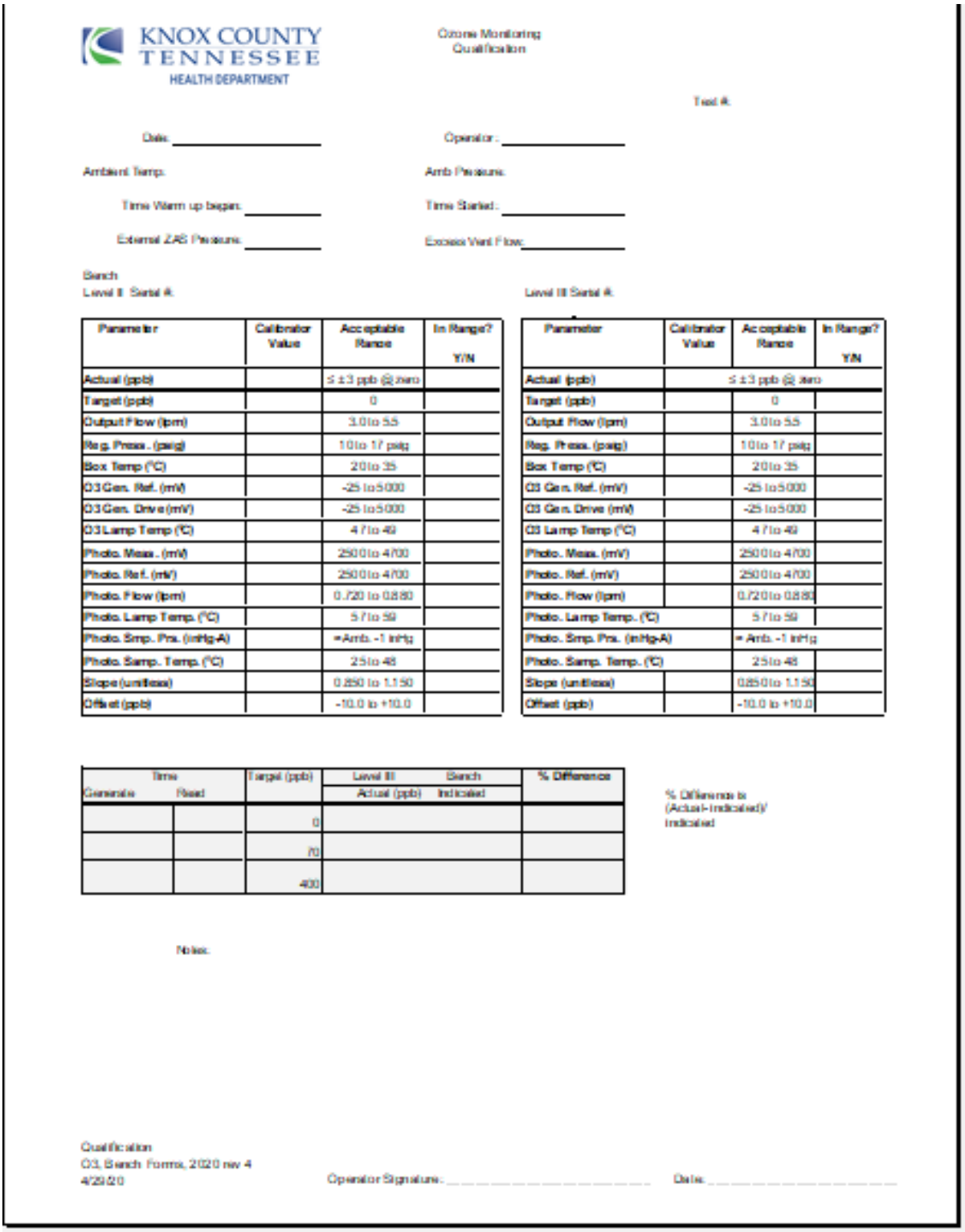
	Date	Operator
Change Sample and Cal Gas Lines		
Change Analyzer's Internal Particualte Filter		
Change/ Check HVAC Filter		
Change Batteries in Thermostat		
Install Shelter Temp. Min. Max. Thermometer		
Change Silica Gel in Both Canisters		
Change ZAS Particulate Filter		

MID-SEASON

	Date	Initials
Change Analyzer's Internal Particualte Filter		
Change/ Check HVAC Filter		
Change Battery in Thermostat		

Site Maintenance
O3, Site Forms 2020 rev 2
9/27/19

Figure 12-1 Site Maintenance Form



KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Ozone Monitoring
Qualification

Test #:

Date: _____ Operator: _____

Ambient Temp: Amb Pressure:

Time Warm up began: _____ Time Started: _____

External ZAS Pressure: _____ Excess Vent Flow: _____

Bench
Level II Serial #: Level III Serial #:

Parameter	Calibrator Value	Acceptable Range	In Range? Y/N
Actual (ppb)		≤ ±3 ppb @ zero	
Target (ppb)		0	
Output Flow (lpm)		3.0 to 5.5	
Reg. Press. (psig)		10 to 17 psig	
Box Temp (°C)		20 to 35	
O3 Gen. Ref. (mV)		-25 to 5000	
O3 Gen. Drive (mV)		-25 to 5000	
O3 Lamp Temp (°C)		47 to 49	
Photo. Meas. (mV)		2500 to 4700	
Photo. Ref. (mV)		2500 to 4700	
Photo. Flow (lpm)		0.720 to 0.880	
Photo. Lamp Temp. (°C)		57 to 59	
Photo. Smp. Pre. (inHg-A)		= Amb. -1 inHg	
Photo. Samp. Temp. (°C)		25 to 48	
Slope (unitless)		0.850 to 1.150	
Offset (ppb)		-10.0 to +10.0	

Parameter	Calibrator Value	Acceptable Range	In Range? Y/N
Actual (ppb)	≤ ±3 ppb @ zero		
Target (ppb)		0	
Output Flow (lpm)		3.0 to 5.5	
Reg. Press. (psig)		10 to 17 psig	
Box Temp (°C)		20 to 35	
O3 Gen. Ref. (mV)		-25 to 5000	
O3 Gen. Drive (mV)		-25 to 5000	
O3 Lamp Temp (°C)		47 to 49	
Photo. Meas. (mV)		2500 to 4700	
Photo. Ref. (mV)		2500 to 4700	
Photo. Flow (lpm)		0.720 to 0.880	
Photo. Lamp Temp. (°C)		57 to 59	
Photo. Smp. Pre. (inHg-A)		= Amb. -1 inHg	
Photo. Samp. Temp. (°C)		25 to 48	
Slope (unitless)		0.850 to 1.150	
Offset (ppb)		-10.0 to +10.0	

Time		Target (ppb)	Level III	Bench	% Difference
Generate	Read		Actual (ppb)	Indicated	
		0			
		70			
		400			

% Difference is (Actual-Indicated)/ Indicated

Notes:

Qualification
O3 Bench Forms, 2020 rev 4
4/29/20

Operator Signature: _____ Date: _____

Figure 12-2 Qualification Form

KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Summary of 6 Ozone Verifications

Date:	Level 3	Level 2	ppb Diff	%Diff	SN:
7/7/2015	-1	0.4	-1.4		
	49	49.8	-0.8	-1.61%	
	90	89.6	0.4	0.45%	
	201	199.8	1.2	0.60%	
	350	348.6	1.4	0.40%	
	450	448.1	1.9	0.42%	

r	slope (m_i)	Intcpt (I_i)	lin reg
1.0000	1.0067	-0.8282	1.004

Date:	Level 3	Level 2	ppb Diff	%Diff	
11/10/2015	0	0.1	-0.1		
	50	50.7	-0.7	-1.38%	Previous Slope difference -0.0042
	90	90.5	-0.5	-0.55%	
	199	199.8	-0.8	-0.40%	
	350	350.1	-0.1	-0.03%	
	450	449	1	0.22%	

r	slope (m_i)	Intcpt (I_i)	lin reg
1.0000	1.0025	-0.6823	1.000

Date:	Level 3	Level 2	ppb Diff	%Diff	
2/23/2016	0	-0.8	0.8		
	450	452.1	-2.1	-0.46%	Previous Slope difference -0.0075
	350	349.8	0.2	0.06%	
	200	199.9	0.1	0.05%	
	90	89.3	0.7	0.78%	
	50	49.6	0.4	0.81%	

r	slope (m_i)	Intcpt (I_i)	lin reg
1.0000	0.9950	0.9593	0.998

Date:	Level 3	Level 2	ppb Diff	%Diff	
2/7/2017	0	-1.1	1.1		
	400	401.8	-1.8	-0.45%	Previous Slope difference -0.0024
	250	251.8	-1.8	-0.71%	
	110	110.2	-0.2	-0.18%	
	70	70	0	0.00%	
	45	44.5	0.5	1.12%	

r	slope (m_i)	Intcpt (I_i)	lin reg
1.0000	0.9926	0.7155	0.995

Date:	Level 3	Level 2	ppb Diff	%Diff	
2/16/2017	0	0	0		
	400	400	0	0.00%	Previous Slope difference 0.0084
	250	250	0	0.00%	
	110	111	-1	-0.90%	
	70	71	-1	-1.41%	
	45	45	0	0.00%	

r	slope (m_i)	Intcpt (I_i)	lin reg
1.0000	1.0010	-0.4752	0.999

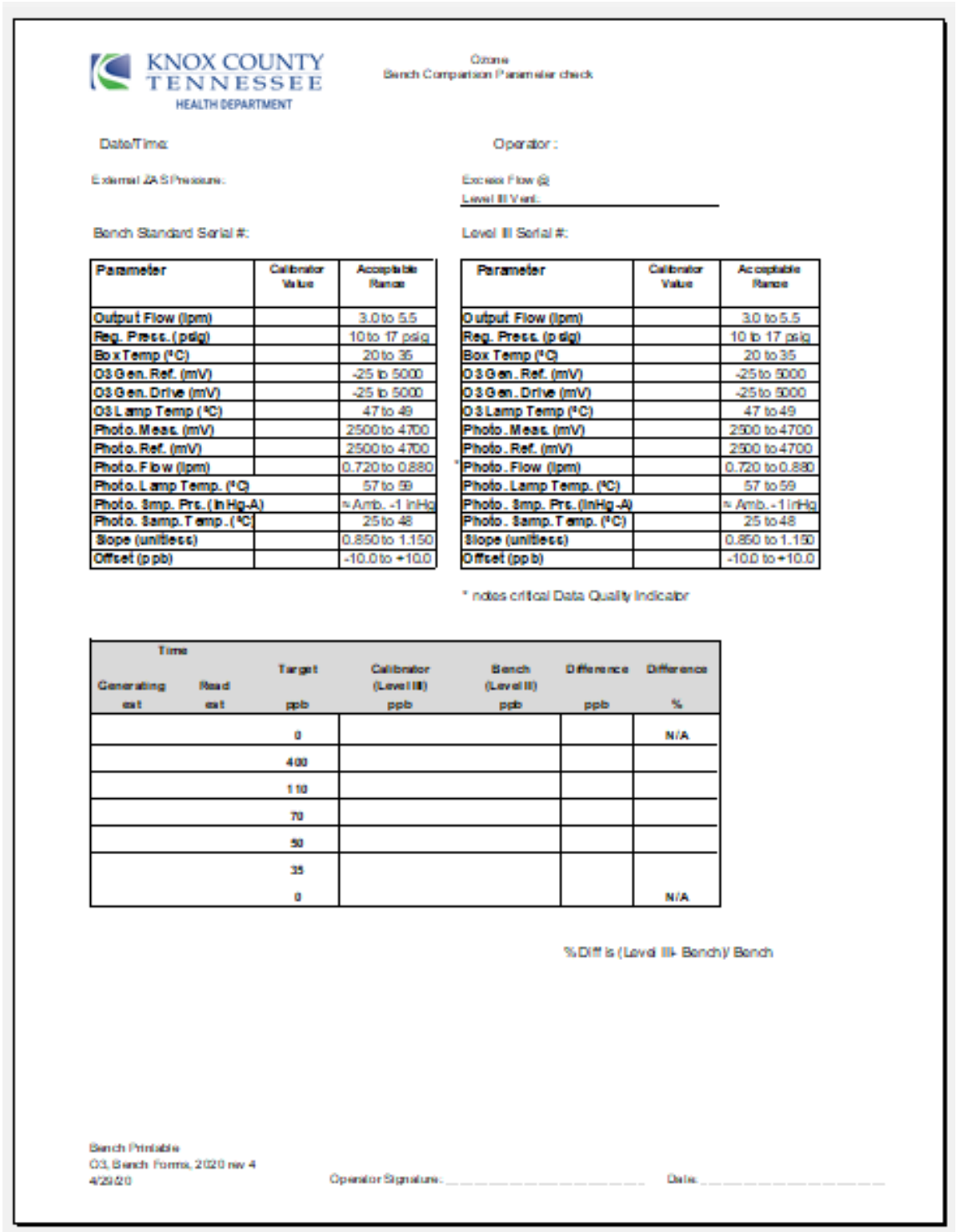
Date:	Level 3	Level 2	ppb Diff	%Diff	
8/7/2017	1	1	0		
	400	399	1	0.25%	Previous Slope difference 0.0010
	249	249	0	0.00%	
	110	109	1	0.92%	
	70	70	0	0.00%	
	45	45	0	0.00%	

r	slope (m_i)	Intcpt (I_i)	lin reg
1.0000	1.0019	0.0539	1.002

Average		1.0000	1.0000	-0.0428	0.9999
RSD	$\$m$	0.52%	$\$I$	0.7484844	

6 Comparison Evaluation rev4 , 4/29/20

Figure 12-3 6 Comparison Evaluation



KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Ozone
Bench Comparison Parameter check

Date/Time:

Operator :

External ZAS Pressure:

Excess Flow @ Level III Vent:

Bench Standard Serial #:

Level III Serial #:

Parameter	Calibrator Value	Acceptable Range
Output Flow (lpm)		3.0 to 5.5
Reg. Press. (psig)		10 to 17 psig
Box Temp (°C)		20 to 35
O3 Gen. Ref. (mV)		-25 to 5000
O3 Gen. Drive (mV)		-25 to 5000
O3 Lamp Temp (°C)		47 to 49
Photo. Meas. (mV)		2500 to 4700
Photo. Ref. (mV)		2500 to 4700
Photo. Flow (lpm)		0.720 to 0.880
Photo. Lamp Temp. (°C)		57 to 59
Photo. Smp. Prs. (InHg-A)		≈ Amb. -1 InHg
Photo. Samp. Temp. (°C)		25 to 48
Slope (unitless)		0.850 to 1.150
Offset (ppb)		-10.0 to +10.0

Parameter	Calibrator Value	Acceptable Range
Output Flow (lpm)		3.0 to 5.5
Reg. Press. (psig)		10 to 17 psig
Box Temp (°C)		20 to 35
O3 Gen. Ref. (mV)		-25 to 5000
O3 Gen. Drive (mV)		-25 to 5000
O3 Lamp Temp (°C)		47 to 49
Photo. Meas. (mV)		2500 to 4700
Photo. Ref. (mV)		2500 to 4700
Photo. Flow (lpm)		0.720 to 0.880
Photo. Lamp Temp. (°C)		57 to 59
Photo. Smp. Prs. (InHg-A)		≈ Amb. -1 InHg
Photo. Samp. Temp. (°C)		25 to 48
Slope (unitless)		0.850 to 1.150
Offset (ppb)		-10.0 to +10.0

* notes critical Data Quality Indicator

Time Generating est	Time Read est	Target ppb	Calibrator (Level III) ppb	Bench (Level II) ppb	Difference ppb	Difference %
		0				N/A
		400				
		110				
		70				
		50				
		35				
		0				N/A

% Diff is (Level III - Bench)/ Bench

Bench Printable
O3, Bench Forms, 2020 rev 4
4/29/20

Operator Signature: _____ Date: _____

Figure 12-4 Bench Printable

KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Ozone Bench Comparison

Level III SN:
Bench (Level II) SN: _____

Percentage Acceptance Value	2%
Calibration Scale (ppb)	400
Point Difference Acceptance Value (ppb)	1.5
Slope Acceptance Criteria	.95 - 1.05

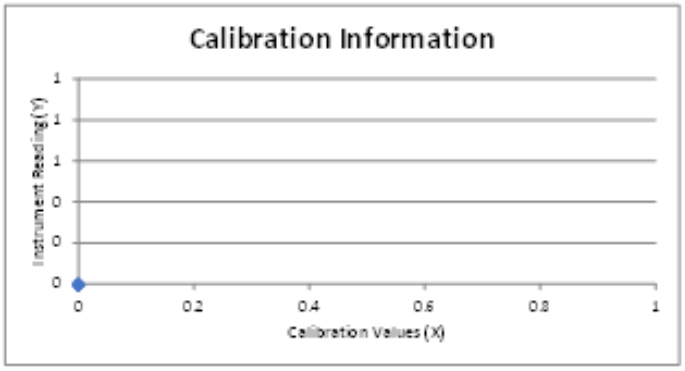
Instructions:
Enter the Level II (bench) actual values in boxes colored
Enter theLevel III actual values in boxes colored
Conditional formating will turn red any failing criteria but entire verification must be complete for formating to be accurate.
First Zero is used for verfying parameters, but not in slope calculations

	Zero Concentration	Concentration 2	Concentration 3	Concentration 4	Concentration 5	Concentration 6	Final Zero
Target Values	0	400	110	70	50	35	0
BenchValue (X) (ppb)							
Level III Values (Y) (ppb)							

Best Fit Concentration	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
Point Difference (Best fit - Average)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
Percent Difference (Best fit Conc vs. Avg Y values)	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!

r	slope (m_0)	intcpt (I_0)	lin reg
#DIV/0!	#DIV/0!	#DIV/0!	#VALUE!

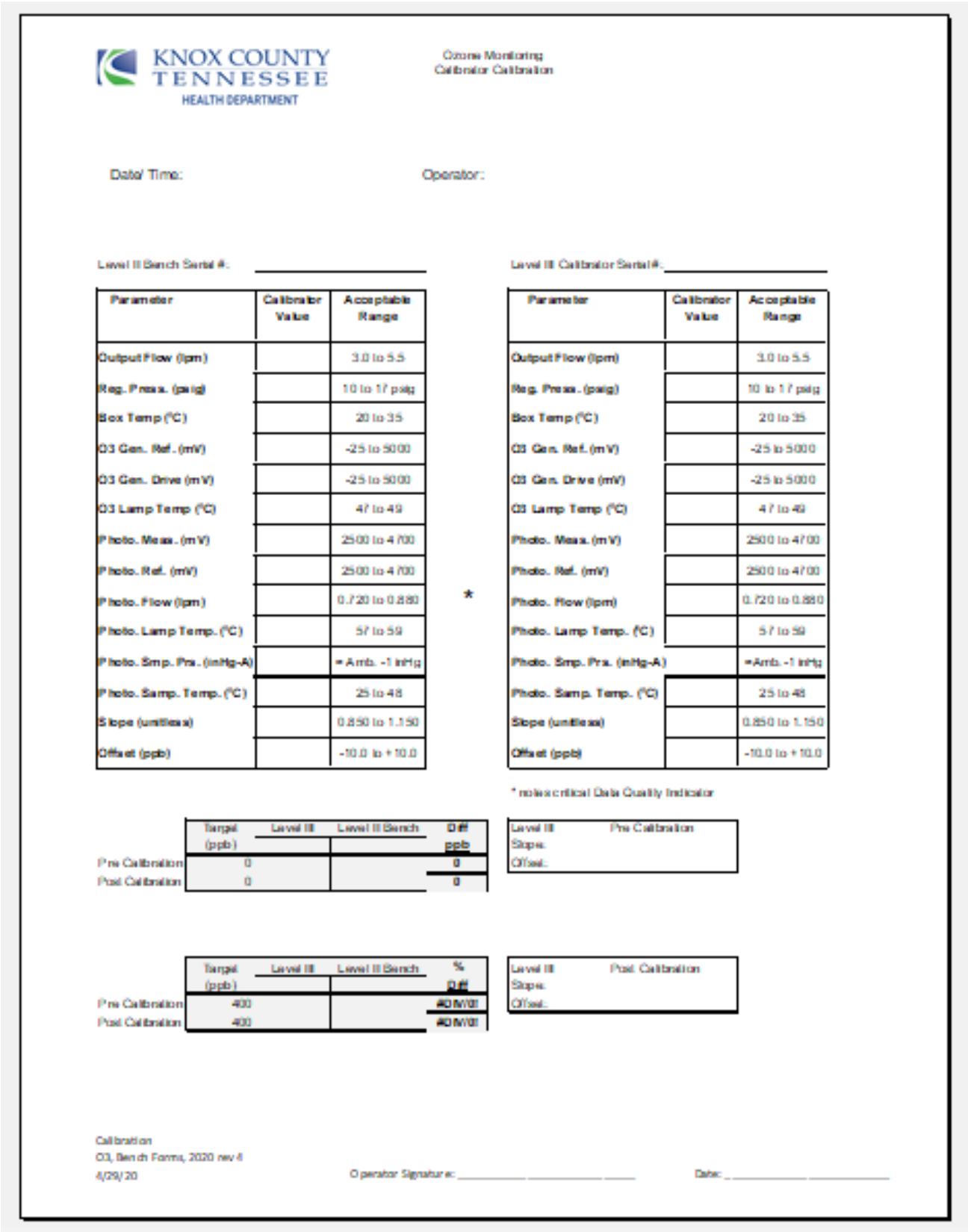
X	Y
0	0.00
0	0.00
0	0.00
0	0.00
0	0.00
0	0.00



Operator Signature and Date

Bench Calculations
O3, Bench Forms, 2020 rev 4
4/29/20

Figure 12-5 Bench Comparison Form



KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Ozone Monitoring
Calibrator Calibration

Date/ Time: Operator:

Level II Bench Serial #: _____

Level II Calibrator Serial #: _____

Parameter	Calibrator Value	Acceptable Range
Output Flow (lpm)		3.0 to 5.5
Reg. Press. (psig)		10 to 17 psig
Box Temp (°C)		20 to 35
O3 Gen. Ref. (mV)		-25 to 5000
O3 Gen. Drive (mV)		-25 to 5000
O3 Lamp Temp (°C)		47 to 49
Photo. Meas. (mV)		2500 to 4700
Photo. Ref. (mV)		2500 to 4700
Photo. Flow (lpm)		0.720 to 0.880
Photo. Lamp Temp. (°C)		57 to 59
Photo. Smp. Pre. (inHg-A)		= Amb. -1 inHg
Photo. Samp. Temp. (°C)		25 to 48
Slope (unitless)		0.850 to 1.150
Offset (ppb)		-10.0 to +10.0

*

Parameter	Calibrator Value	Acceptable Range
Output Flow (lpm)		3.0 to 5.5
Reg. Press. (psig)		10 to 17 psig
Box Temp (°C)		20 to 35
O3 Gen. Ref. (mV)		-25 to 5000
O3 Gen. Drive (mV)		-25 to 5000
O3 Lamp Temp (°C)		47 to 49
Photo. Meas. (mV)		2500 to 4700
Photo. Ref. (mV)		2500 to 4700
Photo. Flow (lpm)		0.720 to 0.880
Photo. Lamp Temp. (°C)		57 to 59
Photo. Smp. Pre. (inHg-A)		= Amb. -1 inHg
Photo. Samp. Temp. (°C)		25 to 48
Slope (unitless)		0.850 to 1.150
Offset (ppb)		-10.0 to +10.0

* notes critical Data Quality Indicator

	Target (ppb)	Level II	Level II Bench	Diff ppb
Pre Calibration	0			0
Post Calibration	0			0

Level II Pre Calibration
Slope:
Offset:

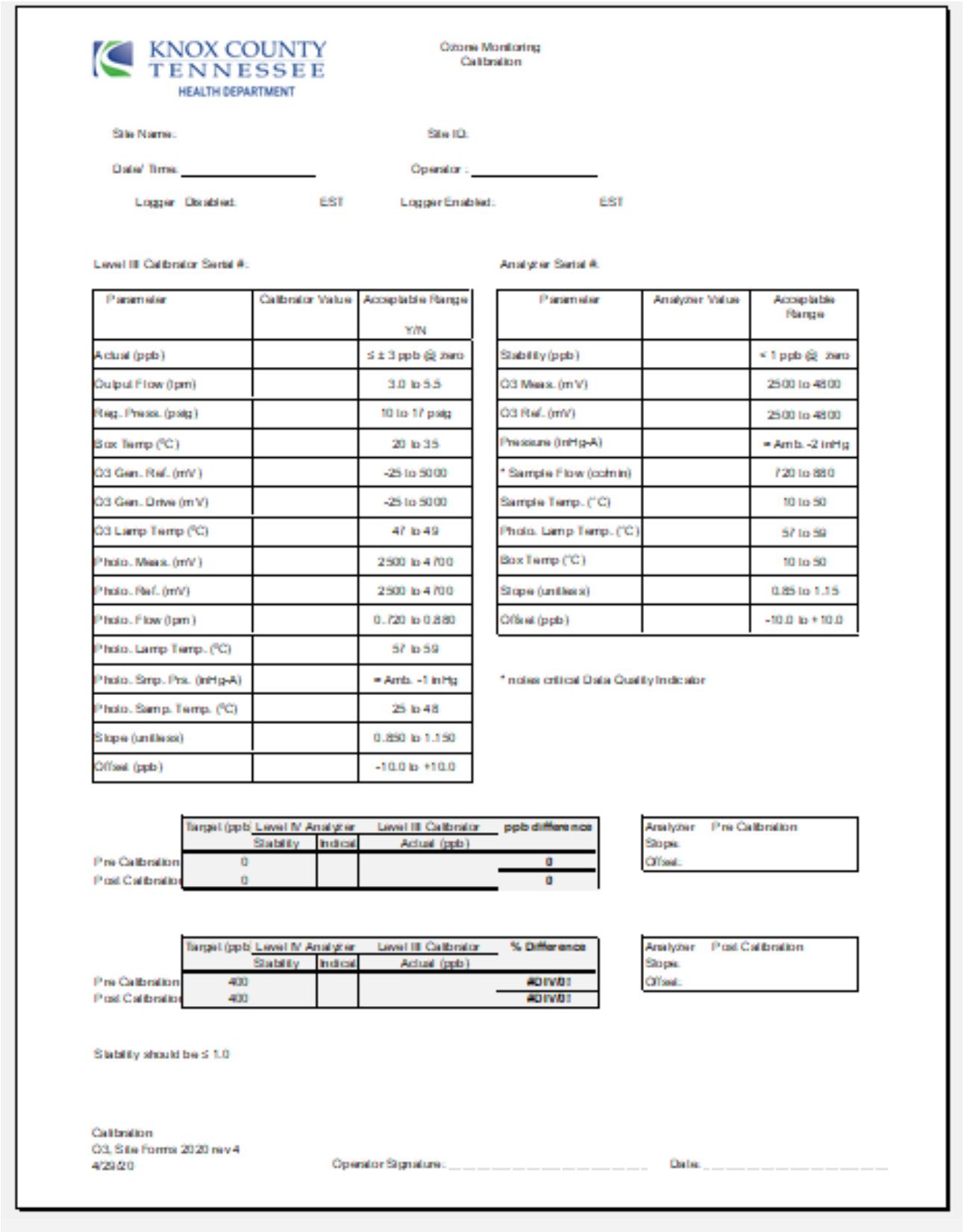
	Target (ppb)	Level II	Level II Bench	% Diff
Pre Calibration	400			#DIV/0!
Post Calibration	400			#DIV/0!

Level II Post Calibration
Slope:
Offset:

Calibration
O3, Bench Forms, 2020 rev 4
4/29/20

Operator Signature: _____ Date: _____

Figure 12-6 Calibration Transfer Standard



KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Ozone Monitoring
Calibration

Site Name: Site ID:

Date/ Time: _____ Operator : _____

Logger Disabled: EST Logger Enabled: EST

Level III Calibrator Serial #:

Parameter	Calibrator Value	Acceptable Range Y/N
Actual (ppb)		≤ ± 3 ppb @ zero
Output Flow (lpm)		3.0 to 5.5
Reg. Press. (psig)		10 to 17 psig
Box Temp (°C)		20 to 35
O3 Gen. Ref. (mV)		-25 to 5000
O3 Gen. Drive (mV)		-25 to 5000
O3 Lamp Temp (°C)		47 to 49
Photo. Meas. (mV)		2500 to 4700
Photo. Ref. (mV)		2500 to 4700
Photo. Flow (lpm)		0..720 to 0.880
Photo. Lamp Temp. (°C)		57 to 59
Photo. Smp. Prs. (inHg-A)		= Amb. -1 inHg
Photo. Samp. Temp. (°C)		25 to 48
Slope (unitless)		0.850 to 1.150
Offset (ppb)		-10.0 to +10.0

Analyzer Serial #:

Parameter	Analyzer Value	Acceptable Range
Stability (ppb)		< 1 ppb @ zero
O3 Meas. (mV)		2500 to 4800
O3 Ref. (mV)		2500 to 4800
Pressure (inHg-A)		= Amb.-2 inHg
* Sample Flow (cc/min)		720 to 880
Sample Temp. (°C)		10 to 50
Photo. Lamp Temp. (°C)		57 to 59
Box Temp (°C)		10 to 50
Slope (unitless)		0.85 to 1.15
Offset (ppb)		-10.0 to +10.0

* noise critical Data Quality Indicator

	Target (ppb) Level IV Analyzer Stability	Indicat	Level III Calibrator Actual (ppb)	ppb difference
Pre Calibration	0			0
Post Calibration	0			0

Analyzer Pre Calibration
Slope:
Offset:

	Target (ppb) Level IV Analyzer Stability	Indicat	Level III Calibrator Actual (ppb)	% Difference
Pre Calibration	400			#DIV/0!
Post Calibration	400			#DIV/0!

Analyzer Post Calibration
Slope:
Offset:

Stability should be ≤ 1.0

Calibration
O3, Site Forms 2020 rev 4
4/29/20

Operator Signature: _____ Date: _____

Figure 12-7 Calibration Analyzer

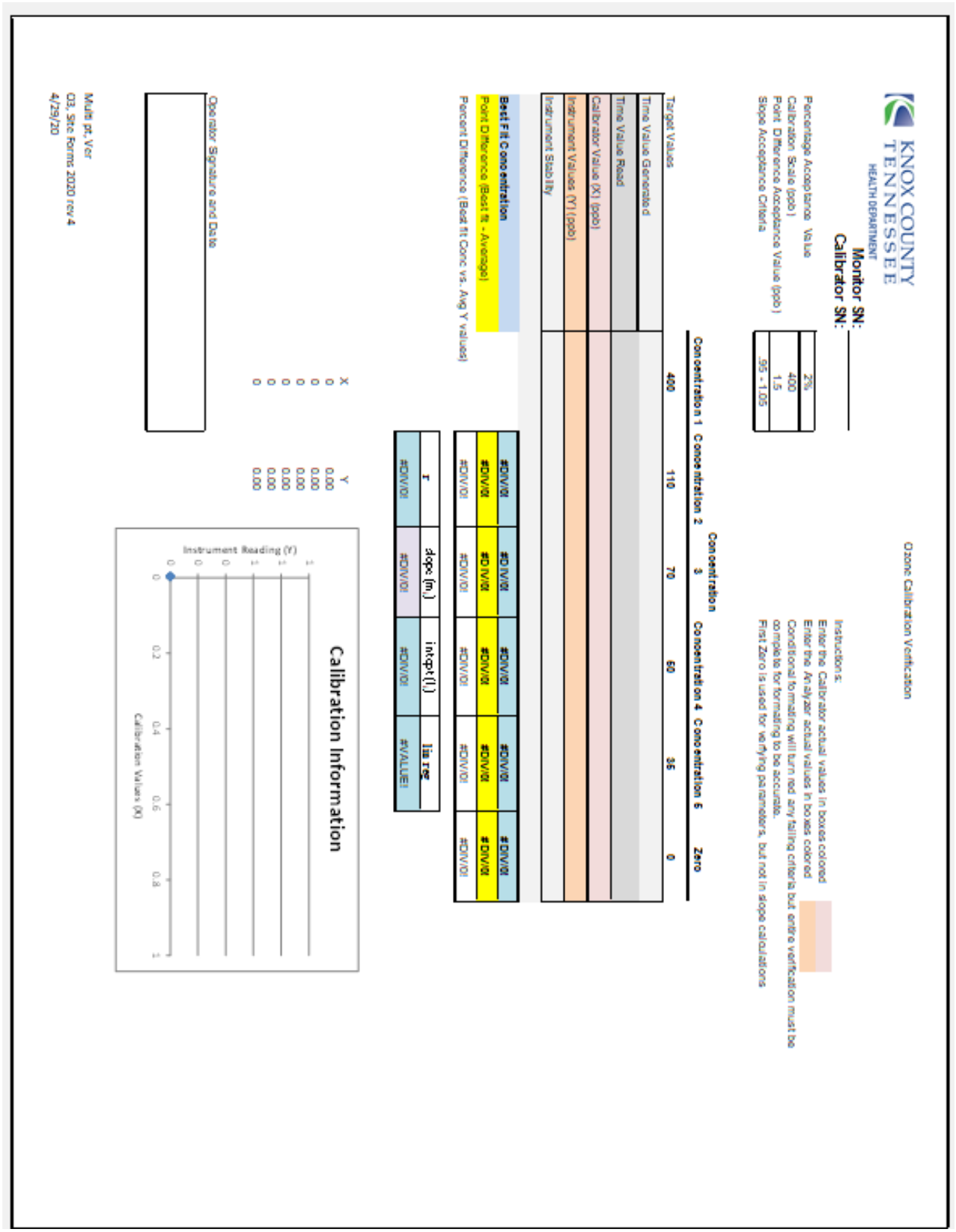
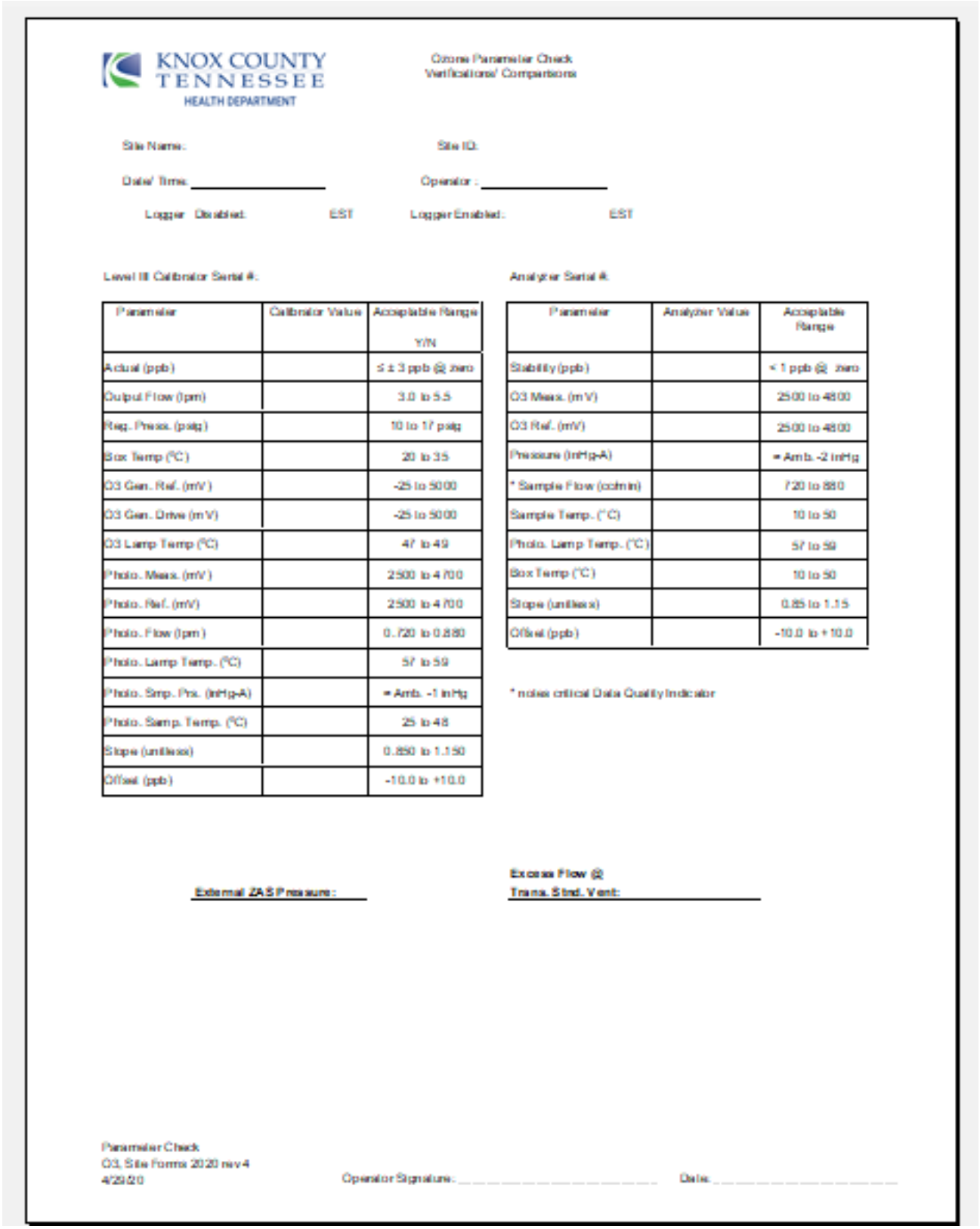


Figure 12-8 Calibration Verification Form



KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Ozone Parameter Check
Verification/ Comparisons

Site Name: Site ID:

Date/ Time: _____ Operator : _____

Logger Disabled: EST Logger Enabled: EST

Level III Calibrator Serial #: Analyzer Serial #:

Parameter	Calibrator Value	Acceptable Range Y/N
Actual (ppb)		≤ ± 3 ppb @ zero
Output Flow (lpm)		3.0 to 5.5
Reg. Press. (psig)		10 to 17 psig
Box Temp (°C)		20 to 35
O3 Gen. Ref. (mV)		-25 to 5000
O3 Gen. Drive (mV)		-25 to 5000
O3 Lamp Temp (°C)		47 to 49
Photo. Meas. (mV)		2500 to 4700
Photo. Ref. (mV)		2500 to 4700
Photo. Flow (lpm)		0.720 to 0.880
Photo. Lamp Temp. (°C)		57 to 59
Photo. Samp. Pre. (inHg-A)		= Amb. -1 inHg
Photo. Samp. Temp. (°C)		25 to 48
Slope (unitless)		0.850 to 1.150
Offset (ppb)		-10.0 to +10.0

Parameter	Analyzer Value	Acceptable Range
Stability (ppb)		< 1 ppb @ zero
O3 Meas. (mV)		2500 to 4800
O3 Ref. (mV)		2500 to 4800
Pressure (inHg-A)		= Amb. -2 inHg
* Sample Flow (cc/min)		720 to 880
Sample Temp. (°C)		10 to 50
Photo. Lamp Temp. (°C)		57 to 59
Box Temp (°C)		10 to 50
Slope (unitless)		0.85 to 1.15
Offset (ppb)		-10.0 to +10.0

* notes critical Data Quality Indicator

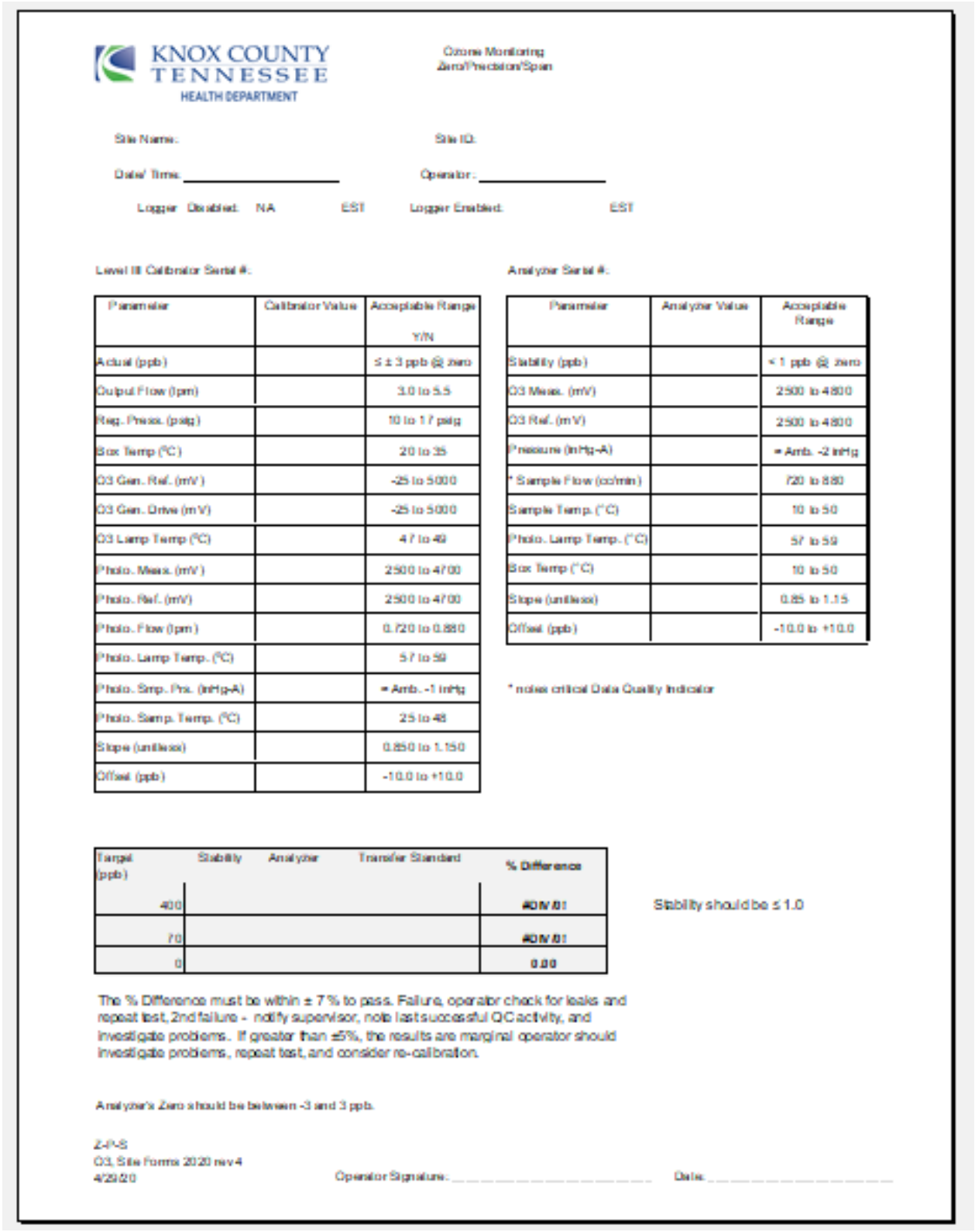
External ZAS Pressure: _____

Excess Flow @ Trans. Std. Vent: _____

Parameter Check
O3 Site Forms 2020 rev 4
4/29/20

Operator Signature: _____ Date: _____

Figure 12-9 Parameter Check



KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Ozone Monitoring
Zero/Precision/Span

Site Name: Site ID:

Date/ Time: _____ Operator: _____

Logger Disabled: NA EST Logger Enabled: EST

Level III Calibrator Serial #: Analyzer Serial #:

Parameter	Calibrator Value	Acceptable Range Y/N
Actual (ppb)		≤ ± 3 ppb @ zero
Output Flow (lpm)		3.0 to 5.5
Reg. Press. (psig)		10 to 17 psig
Box Temp (°C)		20 to 35
O3 Gen. Ref. (mV)		-25 to 5000
O3 Gen. Drive (mV)		-25 to 5000
O3 Lamp Temp (°C)		47 to 49
Photo. Meas. (mV)		2500 to 4700
Photo. Ref. (mV)		2500 to 4700
Photo. Flow (lpm)		0.720 to 0.880
Photo. Lamp Temp. (°C)		57 to 59
Photo. Smp. Prs. (inHg-A)		= Amb. -1 inHg
Photo. Samp. Temp. (°C)		25 to 48
Slope (unitless)		0.850 to 1.150
Offset (ppb)		-10.0 to +10.0

Parameter	Analyzer Value	Acceptable Range
Stability (ppb)		< 1 ppb @ zero
O3 Meas. (mV)		2500 to 4800
O3 Ref. (mV)		2500 to 4800
Pressure (inHg-A)		= Amb. -2 inHg
* Sample Flow (cc/min)		720 to 880
Sample Temp. (°C)		10 to 50
Photo. Lamp Temp. (°C)		57 to 59
Box Temp (°C)		10 to 50
Slope (unitless)		0.85 to 1.15
Offset (ppb)		-10.0 to +10.0

* notes critical Data Quality Indicator

Target (ppb)	Stability	Analyzer	Transfer Standard	% Difference
400				#DIV/0!
70				#DIV/0!
0				0.00

Stability should be ≤ 1.0

The % Difference must be within ± 7 % to pass. Failure, operator check for leaks and repeat test, 2nd failure - notify supervisor, note last successful QC activity, and investigate problems. If greater than ±5%, the results are marginal operator should investigate problems, repeat test, and consider re-calibration.

Analyzer's Zero should be between -3 and 3 ppb.

Z-P-S
O3 Site Forms 2020 rev 4
4/23/20

Operator Signature: _____ Date: _____

Figure 12-10 Zero / Precision / Span Form

KNOX COUNTY
TENNESSEE
HEALTH DEPARTMENT

Ozone Weekly Parameter Check and Maintenance

(Teledyne/API 400 series)

Site: **Month/Year:**

Analyzer Serial #:

	Acceptable Range		
Operator	NA		
Date	NA		
Time (Logger)	± 2 min of NIST		
Time (Analyzer)	± 2 min of NIST		
Time (Calibrator)	± 2 min of NIST		
O3 Meas.	2500 - 4800 mV		
O3 Ref.	2500 - 4800 mV		
Pressure	~ -2 inHg Ambient		
* Sample Flow	720 - 880 cc/min		
Sample Temp.	10 - 50 °C		
Photo. Lamp	57 - 59 °C		
Box Temp.	10 - 50 °C		
Slope	0.85 - 1.15		
Offset	-10.0 - +10.0 ppb		

* Shelter Temp.	15 - 35 °C	Min.		
Min. / Max	(RESET)	Max.		

Changed External Inline Filter			
1st Silica Gel ≥ 25% Blue?			
	Logbook Pg		

Weekly: Record test parameters
Record shelter temperature min. / max. and reset thermometer
Check 1st Silica Gel Canister (≥25% Blue)

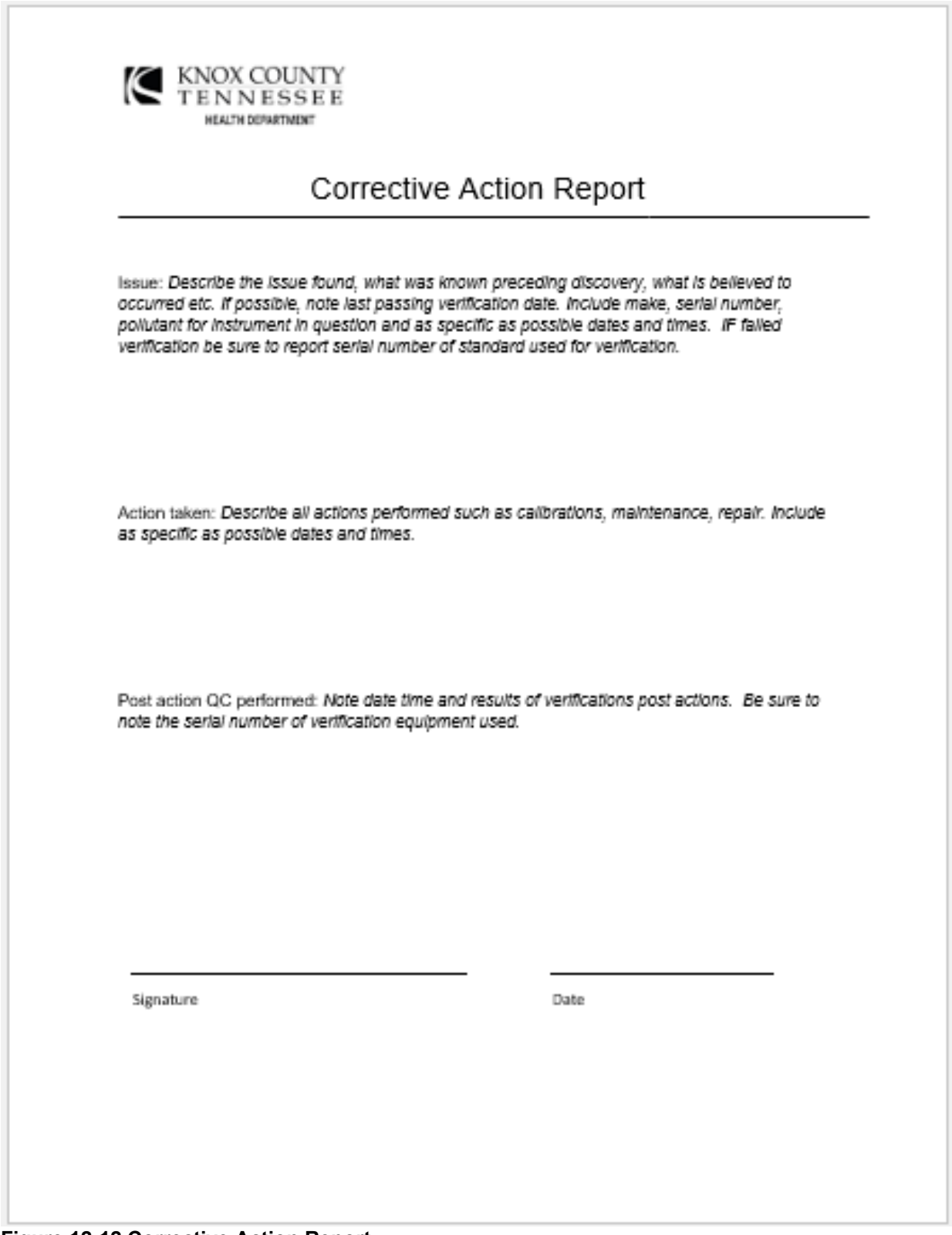
Bi-Weekly: Change external inline particulate filter

* Indicates critical Data Quality Indicator

Date	Comments or Maintenance Performed

Weekly
O3 Site Forms 2020 rev 4
4/29/20

Figure 12-11 Weekly Parameter Check and Maintenance Form



KNOX COUNTY
TENNESSEE
HEALTH DEPARTMENT

Corrective Action Report

Issue: *Describe the issue found, what was known preceding discovery, what is believed to occurred etc. If possible, note last passing verification date. Include make, serial number, pollutant for instrument in question and as specific as possible dates and times. IF failed verification be sure to report serial number of standard used for verification.*

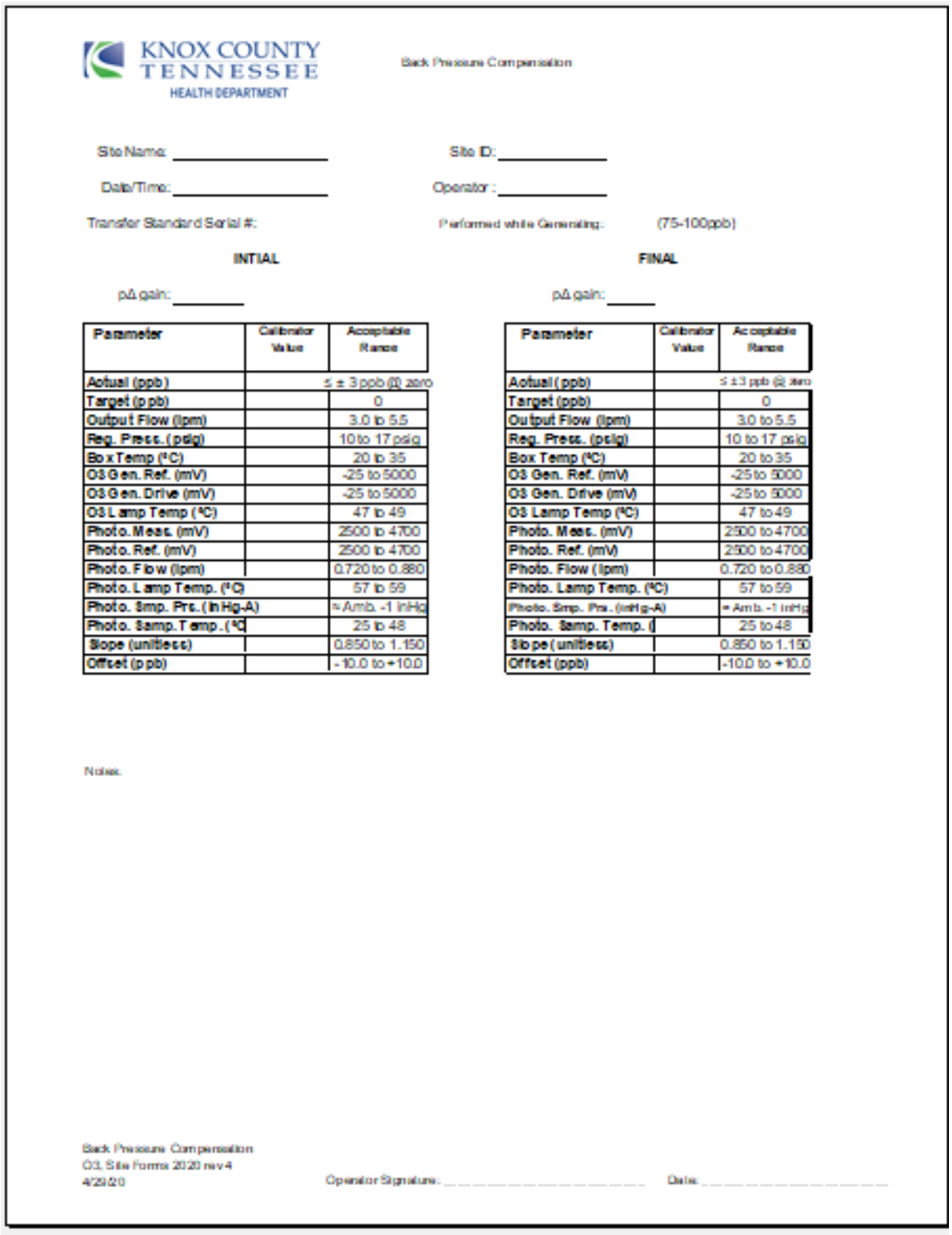
Action taken: *Describe all actions performed such as calibrations, maintenance, repair. Include as specific as possible dates and times.*

Post action QC performed: *Note date time and results of verifications post actions. Be sure to note the serial number of verification equipment used.*

Signature

Date

Figure 12-12 Corrective Action Report



KNOX COUNTY TENNESSEE
HEALTH DEPARTMENT

Back Pressure Compensation

Site Name: _____ Site ID: _____

Date/Time: _____ Operator: _____

Transfer Standard Serial #: Performed while Generating: (75-100ppb)

INTIAL

pΔ gain: _____

Parameter	Calibrator Value	Acceptable Range
Actual (ppb)		≤ ± 3 ppb @ zero
Target (ppb)		0
Output Flow (lpm)		3.0 to 5.5
Reg. Press. (psig)		10 to 17 psig
Box Temp (°C)		20 to 35
O3 Gen. Ref. (mV)		-25 to 5000
O3 Gen. Drive (mV)		-25 to 5000
O3 Lamp Temp (°C)		47 to 49
Photo. Meas. (mV)		2500 to 4700
Photo. Ref. (mV)		2500 to 4700
Photo. Flow (lpm)		0.720 to 0.880
Photo. Lamp Temp. (°C)		57 to 59
Photo. Smp. Prs. (InHg-A)		≈ Amb. -1 InHg
Photo. Samp. Temp. (°C)		25 to 48
Slope (unitless)		0.850 to 1.150
Offset (ppb)		-10.0 to +10.0

FINAL

pΔ gain: _____

Parameter	Calibrator Value	Acceptable Range
Actual (ppb)		≤ ± 3 ppb @ zero
Target (ppb)		0
Output Flow (lpm)		3.0 to 5.5
Reg. Press. (psig)		10 to 17 psig
Box Temp (°C)		20 to 35
O3 Gen. Ref. (mV)		-25 to 5000
O3 Gen. Drive (mV)		-25 to 5000
O3 Lamp Temp (°C)		47 to 49
Photo. Meas. (mV)		2500 to 4700
Photo. Ref. (mV)		2500 to 4700
Photo. Flow (lpm)		0.720 to 0.880
Photo. Lamp Temp. (°C)		57 to 59
Photo. Smp. Prs. (inHg-A)		≈ Amb. -1 InHg
Photo. Samp. Temp. (°		25 to 48
Slope (unitless)		0.850 to 1.150
Offset (ppb)		-10.0 to +10.0

Notes:

Back Pressure Compensation
O3, Site Forms 2020 rev 4
4/29/20

Operator Signature: _____ Date: _____

Figure 12-13 Back Pressure Compensation Form

Month : _____ Site: _____

Monthly Ozone QA Tracking Sheet	Date	Initials
Level 1 – Completed Daily review of minute data in AirVision		
Level 1 – Logbooks scanning for month complete		
Level 1- All QC checks performed, printed with graphs		
Level 1 – Reviewed monthly hourly report, assigned null codes, created AQS file		
Level 1- Corrective Action Report on File {Initial/ date only if applicable for the month)		
Level 2: Review verifications and audits – verified in database and AQS		
Level 2: Reviewed AirVision reports (shelter T and Sample Flow)		
Level 2: Reconciled logbook entries with QC documentation		
Level 2: Reviewed Flags and null codes by operator, updated AQS file		
Level 2: Investigate outliers		
Level 2: Excel form Calc Check **Date on Form Checked :**_____		
Level 3 : Supervisory Review		
Data Uploaded to AQS		

Analyzer value	Calibrator Value	% D on Form	% D Calc

Level 1 items: Completed by the operator
Level 2 items: Completed by QA personnel
Level 3 items: Completed by Program Manager

Figure 12-14 Monthly QA Tracking Form

13.0 Appendix B – Sticky Forms

Time Read	Target	Calibrator (Level III)	Bench (Level II)	Difference (Cal - Bench)	Difference (C - B)/B
	ppb	ppb	ppb	ppb	%
	0				N/A
	400				
	110				
	70				
	50				
	35				
	0				N/A

Figure 13-1 Bench Comparison Sticky

Operator:		Logger Disable:	(Circle one)			
Date:		Logger Enable:	Pass	Fail		
Generate Time	Collection Time	Target	Analyzer Actual	Calibrator Actual	Stability	Difference
EST	EST	ppb	ppb	ppb	ppb	%
		400				
		110				
		70				
		50				
		35				
		0				NA

Figure 13-2 Cal Verification Sticky

Operator:		Logger Disable:		(Circle one	
Date:		Logger Enable:		Pass	Fail
Target (ppb)	Analyzer		Transfer Standard	% Difference	
	Stability				
400					
70					
0					

Figure 13-3 ZPS Sticky

	Acceptable Range		
Operator	NA		
Date	NA		
Time	± 2 min of NIST		
O3 Meas.	2500 - 4800 mV		
O3 Ref.	2500 - 4800 mV		
Pressure	~ -2 inHg Ambient		
Shelter Temp.	15 - 35 ^{0}C	Min.	
Min. / Max	**(RESET)**	Max.	

	Acceptable Range	
Sample Flow	720 - 880 cc/min	
Sample Temp.	10 - 50 ^{0}C	
Photo. Lamp	57 - 59 ^{0}C	
Box Temp.	10 - 50 ^{o}C	
Slope	0.85 - 1.15	
Offset	-10.0 - +10.0 ppb	
Changed External Inline Filter		
1st Silica Gel ≥ 25% Blue?		

Figure 13-4 Weekly Sticky

Appendix F – Thermo 2025 PM2.5 SOP

THERMO Model 2025 Sequential Sampler Standard Operating Procedures

Knox County
Health Department
Every Person, A Healthy Person

Prepared By
Rebecca Larocque

Revision History

Change	Page #	Date of Change	Revision#

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1.0 Introduction

This Standard Operating Procedure (SOP) describes the procedures used to sample particulate matter 2.5 microns or smaller($PM_{2.5}$) using the Partisol ®- Plus Model 2025 Sequential Air Sampler by the Knox County Air Quality Management Monitoring Program. The objective of the SOP is to familiarize the operator with the procedures used in the operation, calibration and maintenance of the sampler and collection, validation and submittal of the air monitoring data. This SOP is to be used as an outline and shall be supplemented with the equipment manufacturer's manuals and procedures. This SOP supports the KCDAQM Quality Assurance Project Plan (QAPP) and is part of the KCDAQM quality system.

1.1 Principles of Operation

The Partisol-Plus 2025 sampler draws ambient air through a PM10 inlet, down tube, and then through the Very Sharp Cut Cyclone (VSCC) particle size separator. Particles in the PM2.5 size range and below are then collected on a 47 mm Teflon filter during a specified 24-hour sampling period. Each sample filter is weighed before and after sampling by a qualified laboratory to determine the net weight (mass) gain of the sample. This is then converted to a mass concentration using the volume of air sampled over the 24-hour period and is reported as micrograms per cubic meter at local conditions. The reference method of PM2.5 sampling is provided in the Code of Federal Regulations 40 CFR 50, Appendix L.

The Partisol-Plus 2025 contains software and hardware that is used to set up and control the sampler during operation, maintain a 16.7 liter per minute (LPM) flow rate and store and display any EPA required run information (i.e. temperature, pressure). For a more detailed explanation of the sampler's theory of operation, please read, "Section 1: Introduction", of the Operating Manual.

1.2 Reference Documents

- *Partisol ® Plus Model 2025 Operating Manual Sequential Air Sampler Revision B,* Thermo Scientific, November 2006
- *40 Code of Federal Regulations Part 50, appendix L, reference Method for Determination of Particulate Matter as PM2.5 in the Atmosphere.*
- *40 Code of Federal Regulations Part 58, appendix A, Quality assurance Requirements for State and Local Air Monitoring Stations (SLAMS)*
- *Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring,* 40 CFR 58 Appendix E
- *Quality Assurance Handbook for Air Pollution Measurements Systems, Volume II, Ambient Air Specific Methods,* U.S. Environmental Protection Agency, Jan 2017
- *Technical Note- Holding Time Requirements for PM2.5 Filter Samples*, EPA OAQPS, October 2015
- *Quality Assurance Guidance Document 2.12*, U.S. Environmental Protection Agency, Jan 2016
- *Quality Assurance Project Plan*, Knox County Air Quality Management Ambient Air Monitoring Program, March 2018

1.3 Sampler Modes of Operation

The Thermo Partisol 2025 sampler has the following modes of operation which will be referenced

throughout this SOP.

Stop Mode – The sampler must be placed into Stop Mode to edit start date, start time, sampler's current time or to enter into the Service Mode. It is also necessary to enter the various calibration screens and conduct calibrations. (This is not to be confused with the flow verifications and leak checks, which are conducted on a routine schedule--the Audit Mode is used for these checks.) It is desirable to not enter the Stop Mode once the sampler is programmed unless it is necessary. Doing so will require the sampler's run date schedule to be reprogrammed.

Audit Mode – This is a Pause Mode used for flow rate verifications, ambient temperature/pressure verifications, filter temperature sensor verification, compartment temperature verification, and external/internal leak checks. It is also used when filter advancement is needed.

Wait Mode (WAIT) – The mode in which the sampler is waiting to operate (start sampling) on the next scheduled date. The sampler should be left in this mode after departure from the site unless it is sampling.

Done Mode (DONE) – In this mode, the filters in the supply magazine have depleted. This will give you an "N" status and blinking indicator light.

Error Mode (ERR) – A significant problem with sampler flow rate or filter exchange.

Sample Mode (SAMP) - In this mode, the monitor will be sampling.

2.0 Installation

This section documents the installation or moving of a sampler. A location change of a sampler either swapping at an existing site location or installing at a new location requires authorization from the Air Monitoring Program Manager and possible updates to the Network Plan.

2.1 List of Tools, Equipment and Materials

- Thermo 2025 Sampler System
- Filter cassette containing laboratory pre-weighed Teflon filters in magazine
- PM10 head and VSCC
- Additional sampler parts and supplies including additional gaskets/seals.
- Miscellaneous hand tools including: hammer, screwdrivers, wrenches, nut drivers, hex key wrenches,
- Power tools, electric drill, power screwdriver, bits
- Extension cord
- Personal protective equipment

2.2 Physical Inspection and Initial Acceptance

Begin a logbook for the sampler. This logbook will follow the sampler throughout its working life in the department. Record the sampler serial number in the logbook.

Upon receipt of all shipped sampling equipment, parts and supplies, inspect equipment and accessories for completeness and/or damage. If a shortage or damage is found, immediately notify the lab manager and/or the equipment vendor to repair or replace damaged equipment or missing supplies. Make note of how the foam packing was installed. If the equipment is found to be damaged repack it in the same way it was delivered.

Follow the procedures in section 2.2 of the manufacturer's operating manual to assemble the sampler. Prior to setting up the sampler in the field, the initial checks in section 6.0 of the manufacturer's manual should be performed at the air monitoring laboratory. Record acceptance testing and initial inspection results in the sampler logbook.

The checks consist of internal and external leak checks, temperature (ambient, filter, compartment) and flow rate checks. The flow rate checks should be conducted at 15.0, 16.7 and 18.3 liters per minute. All checks must be within acceptable limits according to the manufacturer's manual, or corrective action must be taken prior to installation at field site. Document the results in the sampler log book.

2.3 Siting Requirements

All $PM_{2.5}$ samplers must be sited in accordance with US EPA criteria contained in 40 CFR 58, Appendix E. The PM sampler should have unobstructed air flow in all directions. Adequate space should be allowed for installation of a portable FRM performance evaluation (PE) sampler. If samplers are to be collocated, then sufficient area should be provided for this accommodation.

The sampler inlet must be placed at a height of 2 to 15 meters above ground level. If the $PM_{2.5}$ sampler is collocated with another $PM_{2.5}$ sampler, the spacing between must be between 1 to 4 meters. If the $PM_{2.5}$ sampler shares a site with another type of sampler, separation requirements are based on volume of air required by sampler. Samplers drawing 200 liters/min of air or more are considered high-volume samplers and must be at least 2 meters from the Partisol 2025. Samplers drawing less than 200 liters/min are considered low volume and must be at least 1 meter from inlets of other samplers. Follow all requirements found in Table E-4 of 40 CFR Part 58 appendix E (https://www.gpo.gov/fdsys/granule/CFR-2012-title40-vol6/CFR-2012-title40-vol6-part58-appE). See KCDAQM QAPP Section 10.0 for detailed description on KCDAQM's siting and probe placement protocol.

The sampler location should be selected considering ease of access, security for equipment, and space for audit equipment. The sampler location must be situated where the operator can reach it safely regardless of weather conditions. The sampler must be securely anchored to the stand and supporting structure. The site security is to be maintained with a locked fence.

2.4 Sampler Installation and Assembly

The location of the sampler will have already been identified as part of the monitoring site selection. The sampler may be located on a solid, level surface such as a concrete slab or a raised wooden platform/scaffolding. Position the shelter housing to meet the siting requirements as stated above and secure the stand directly to the solid, level surface. Make sure sampler, stand, and the platform are sufficiently secured to withstand wind conditions.

The $PM_{2.5}$ sampler operates at 105 to 125 volts AC and a frequency of 60 hertz. Every effort should be made to provide a stable power source for the monitoring site. The site should be capable to provide sufficient power for the primary sampler, a collocated sampler, and an FRM PE Sampler.

Inspect the electrical cords and connections for signs of wear and have them replaced as needed. Always unplug the power to the sampler prior to removing boards or replacing parts. If monitor is hardwired in, access the breaker box and turn the power off at the breaker (See operating manual.) Be aware of weather patterns; leave the area if storms approach. Sampler is elevated and made of conductive metal.

2.5 Programing the Sampler and Navigating the Screens

The 2025 Sampler is a menu driven instrument with many screens. The system software allows the user to navigate easily through the use of "soft function keys" (<F1> to <F5>) and the <Esc> key. It is relatively easy to switch from one display to another. From the main screen, simply press <F1: Help>, <F2: Stats>, <F3: Filt set>, <F4: Data> or <F5: Setup> to drop down one level in the screen structure. Pressing the <Esc> key causes the current display to be replaced by the next higher screen in the hierarchy. The definition of the "soft function keys" change as different screens appear on the display and different functions are required. See section 4 and 5 in the manufacturer's operating manual for full instructions on each menu item.

Many screens allow the user to change the value of system parameters. The <Edit> key causes the sampler to enter the edit mode. Some displays contain the <F5: More > key indicating that additional options exist. Pressing <F5: More> causes the additional menu options to appear on the bottom line of

the screen. To distinguish between the selections available from the main and second lines of the menu, the function keys in the first line are designated by <F1> to <F5>, and the second line contains the titles <F6> to <F10>. Press <F10> while in the second line to return to the main menu.

The master menu provides another option to gain access the operational screens in the instrument software. It can be accessed by pressing <Menu>. Press <↓> and <↑> to view the "soft function key" options available. Press the desired "soft function key" to make a selection or <Enter> in the absence of any "soft function key" definitions.

Do not select Service Mode unless maintenance or QA operations are being performed. Press <Esc> to return to the Main Screen without making any selection.

2.5.1 Setting the Site Identification

Upon installation at a site the Site identification shall be set in the sampler. This will allow the data to be identifiable by the EPA site identification. Start by pressing <Menu> to enter the Master menu screen. Use the arrow keys to select "System Setup" -> "soft function key" F5 (System) -> "soft function key" F3 (Site ID). Enter the full site identification number in ID1 (this can be found in the front of the logbook). For example, for the Rule site location the full ID would be 47093101701. This represents the State (47), County (093), Site location (1017) and POC (01). Enter the sampler's serial number in Id2. The serial number is printed on the label inside the filter compartment on the left-hand side. For example, 2025B226541005 is listed on the sampler as the serial number, but it is actually the model number, serial number and date of manufacture. It is model 2025B, serial number 22654 and manufactured in 10/05. In this example, only "22654" would be entered in ID2. Entering the full ID and serial number will ensure proper data tracking when downloading and saving the sampler data. Note: YOU MUST PRESS F5 AFTER EACH NUMBER, THE CURSER WILL NOT ADVANCE TO THE NEXT POSITION

2.5.2 Program System Setup Screen

The system setup screen is used to define parameters for operation. See default settings below. The parameters for this screen can be edited only in the Stop Mode. Press <F5: Setup> twice when in the main screen to enter the system setup screen. Once the monitor's initial setup, this information should not need to be changed.

For $Pm_{2.5}$, the monitor must be programmed to sample in local temperature and pressure. These are the default settings for the 2025 sampler. Ensure that each line is as shown in table 2.1 below.

Avg. Temp:99	Standard Temp: 99
Avg. Press: 999	Standard Press: 999
Date Form: yy/mm/dd	Avg. Time: 30
Time Form: Hr/min/sec.	Filter Fan: Auto
Current Time: set to current time	Auto Run : ON
Current Date: set to current date	

Table 2.1 System Setup

To make changes, proceed as follows * Must be in Stop Mode: Press < Edit> on the keyboard panel. Using the arrow keys, scroll to the proper parameter. Enter the correct information using the keypad. Press Enter and confirm the information is correct. Press <ESC> twice to return to the Main Screen.

2.5.3 Program Sample Setup Screen

The Sample definition screen shows the sampling schedule. To modify the sampling schedule from the Main Screen, Press < F5: Setup> to enter the sample definition screen See example in Table 2.2 below.

Parameter	Value
Sample Definition Method	Basic
Default Sample Start Time	00:00
Default Sample Duration	24:00
Default Sample Repeat Time	024:00 (Daily sampling) 072:00 (1 in 3 sampling) 144:00(1 in 6 sampling)
Default Filter Type	T (press “edit”, 20, “enter”)
Sample Flow Rate:	16.7
Flow Error Mode	Err
Separators	No

Table 2.2 Sample Definition Screen Settings

These are the parameters that will be used during routine operation but can easily be modified when in Edit Mode. Use the arrow keys to select the field to be edited, press <Enter> when all fields have the desired data.

3.0 Calibration Procedures

Before starting a calibration, make sure to do a pre-calibration verification if possible as well as a post-calibration verification to ensure the calibration was successful. The pre-calibration verification should be recorded in the sampler log on the monthly maintenance sticky. The post-calibration verification should be recorded on the calibration sheet in the sampler logbook. Note: A multipoint verification is required at least annually. The post-calibration verification section of the calibration sheet is designed for this purpose and should be completed annually. All calibrations must be performed in Service Mode. Reference to the manufacturer's service manual is recommended during the calibration procedures (Section 3). Perform the calibrations in the order presented. There should not be a sampling filter in the sample position during the calibration process. The filter should be advanced during the pre-calibration verification and set aside in a clean magazine or anti-static bag. In the event a pre-calibration was not possible follow the procedures in 4.2.C to advance the filter.

3.1 Calibration Frequencies

To ensure accurate measurement of $PM_{2.5}$ concentrations, calibrate the Partisol-Plus 2025 upon installation, annually, after major repairs, and upon failure of monthly verification or audit.

3.2 Calibration Equipment

- Streamline Pro (SLP) transfer standard or flow audit transfer standard.
- Streamline Pro or aneroid barometer (e.g., a climber's or engineer's altimeter), capable of accurately measuring ambient barometric pressure within ± 1 mm Hg resolution and referenced within ±5 mmHg to a barometer referenced to a NIST standard.
- Streamline Pro or thermometer capable of accurately measuring temperature over the range of - 30°C to +50°C (243 K to 323 K) to the nearest ± 0.1°C and referenced to an NIST thermometer within ± 0.5°C at least annually.
- "Calibration Sheet" (Appendix A.1) and the station logbook.
- An empty filter cassette (empty ring)

Transfer standards or certified reference device such as the Streamline Pro, thermometers, barometers, or other flow meter must have a recorded certification date valid within one year. The make, serial number, and certification date shall be recorded in the sampler logbook with the calibration or verification for which the standard was used.

3.3 Analog I/O Calibrations

Analog I/O calibrations are performed automatically when the sampler enters the Stop Operating Mode upon power-up. At an interval of every 30 minutes, the Partisol-Plus Sampler conducts an automatic calibration of its analog input channels. The user can also manually instruct the unit to calibrate the analog input and output channels if necessary. Unless notified, a manual analog I/O calibration will not be necessary.

3.4 Ambient Air Temperature Probe Calibration

The manufacturer has certified that each Sensor is linear, and therefore requires only a one-point calibration.

A. Press <Menu> and enter the Service Mode. With the cursor pointing to Calibration/Audit, press <F3: Sens Cal> to enter the Sensor Calibration Screen
B. Place the reference temperature probe as close to the sampler probe as possible without touching (within 0.5 inches) or within the radiant heat shield. Allow temperatures to equilibrate.
C. Press <Edit> then enter the measured reference temperature in the Actual column of the Ambient Temp row of the Sensor Calibration screen.
D. Record the displayed "Current" and "Actual" temperature in the sampler logbook using the "Calibration Sheet" (See Appendix A.1)
E. Press <Enter>.The sampler automatically adjusts the corresponding offset based upon the input. The two temperatures should now be in agreement.
F. Record the new offset on the "Calibration Sheet"

3.5 Filter Temperature Probe Calibration

A. Place an empty cassette ring in the sampling position. Open the sampler top and remove the very sharp cut cyclone (VSCC)
B. Insert the reference temperature probe down through the empty filter cassette ring to the exposed filter temperature probe.
C. From the Service Menu, enter the Calibration/Audit Screen. Press <F4: Filt Cal> to enter the Filter Temp Calibration Screen.
D. Allow the temperatures to equilibrate for at least 5 minutes.
E. Press <Edit> and enter the measured reference temperature in °C in the Actual column on the filter row of the Filter Temperature Calibration Screen.
F. Record the displayed "Current" and "Actual" temperature in the sampler logbook using the "Calibration Sheet" (See Appendix A.1)
G. Press <Enter>. The sampler automatically adjusts the offset based on input. The temperatures should now agree. Record the new offset in the sampler logbook using the "Calibration Sheet" sticky form (See Appendix A.1)
H. Place the VSCC back onto the unit.

3.6 Ambient Pressure Calibration

A. From the service Menu, go to the Calibration/Audit Selection and press<Enter>. Press <F3: Sens Cal>.
B. Using the transfer standard determine the current ambient station pressure in mm Hg (not corrected to sea level).
C. Press <Edit>, and enter the measured ambient pressure in the Actual column of the Ambient Pres row of the Sensor Calibration screen.
D. Record the displayed "Current" and "Actual" pressure in the sampler logbook using the "Calibration Sheet" (See Appendix A.1)
E. Press <Enter>. The sampler adjusts the offset. The two (2) pressure measurements should agree. Record the new offset in the sampler logbook using the "Calibration Sheet" form (See Appendix A.1)

3.7 External Leak Check

A. Advance the audit/verification filter cassette (fig 4.1) into the sampling position by pressing <F4> "FiltAdv".

B. Remove the 1st stage inlet by grasping near rain collection jar and pulling straight up with a twisting motion.

C. Install the flow audit adapter on the sample down tube and close the valve on the flow audit adapter.

D. Press <F5: Leak Chk>, <F2: Start>, and follow the instructions displayed on the screen. A pass or fail message will be displayed.

E. If a leak check is failed, check the flow adapter valve, seating of the external sample tube, and the VSCC to ensure that it is assembled snuggly. (Also check the integrity of the cassette as they tend to crack, scratch and not snap together well.)

F. Repeat the leak check. After a successful external leak check, slowly open the valve on the flow audit adapter. If the leak check failed, refer to the manufacturer's service manual or check with technical support for guidance. *Trouble shooting tip: The V-Seals are a common cause of leaks.

G. Record the results on the "Calibration Sheet" in the logbook (Appendix A.1).

3.8 Flow Calibration

A. Insure that the calibration/verification filter cassette (see figure 4.1) is in the sampling position.

B. From the Service Menu, go to the Calibration/Audit Selection and press <Enter>. Then press<F5: Flow Cal> to enter the Flow Calibration Screen. To set flow rates to EPA Calibration requirements (15.0, 16.7, and 18.3), press <More> and then press <EPA Cal.>. The required range will be automatically set.

C. Attach the reference device to the down tube after removing the stage-1 inlet from the sampler.

D. Press <F4: Start>. Note: During the calibration procedure, the sampler begins with a 16.7 flow rate.

E. Wait for the flow to stabilize. Enter the flow from the reference device in the Actual Flow Field (measured flow).

F. Record "Current" and "Actual" flows on the "Calibration Sheet"

G. Press enter and record Offset and Span on "Calibration Sheet"

H. Repeat steps D through G for the remaining (2) flow rates.

I. The Sampler automatically adjusts the offset and span values in the Flow Calibration Screen once it performs measurements at all flow rate plateaus. Record the displayed information in the sampler logbook using the "Calibration Sheet" (See Appendix A.1)

J. Upon completion of the Calibration perform a Multipoint Flow Verification according to section 4.4. After a Calibration, all flow should be within 2%. Record results and investigate if values

exceed acceptance criteria.

3.9 Filter Compartment Temperature Calibration

Make sure the unadjusted span and offset prior to calibration is recorded on the previous "Calibration Sheet" if not; record the value on the current "Calibration Sheet" under comments.

A. Press <Menu> and enter the Service Mode. With the cursor pointing to Calibration/Audit, press <F4: Filter Cal> to enter the Sensor Calibration Screen
B. Determine the current temperature (°C) at the location of the filter compartment temperature probe using a calibrated reference thermometer.
C. Press <Edit> then enter the measured filter compartment temperature in the Actual column of the Ambient Temp row of the Sensor Calibration screen.
D. Record the displayed "Current" and "Actual" temperature in the sampler logbook using the "Calibration Sheet" (See Appendix A.1)
E. Press <Enter>.The sampler automatically adjusts the corresponding offset based upon the input. The two temperatures should now be in agreement.
F. Record the new offset on the "Calibration Sheet"
G. Return to the Main Screen and perform necessary post calibration verifications.

* Note with all calibrations, that if a problem occurs during the calibration causing radical change and errant results, it may be useful to review the previous calibration for the span and offset. Enter the previous span and offset and begin again.

4.0 Verification Procedures

4.1 Verification Frequencies

To ensure accurate measurement of $PM_{2.5}$ concentrations, calibrate the Partisol-Plus following guidelines in section 3.0; then perform verifications according to the frequencies in table 4.1.

Procedure	Frequency	Acceptable Limits
Flow Rate	Monthly	± 4% actual/ ±5% design
Filter Temperature	Monthly	± 2.0°C
Ambient Temperature	Monthly	± 2.0°C
Ambient Pressure	Monthly	± 10 mmHg
Clock	Monthly	± 2 minutes
Leak Check internal	If External failed	140 mmHg (Pass)
Leak check External	Monthly	25 mmHg (Pass)
Multipoint verification	After calibration	± 2% each point

Table 4.1 Verification Frequencies

4.2 Preparing for Monthly Verification

A. Placing the monitor in Audit Mode: if the monitor is in “Samp” or “Wait” mode: Press <Run/Stop>, select “audit” from the “soft function keys”. If the monitor is in “Stop” mode press <Menu> scroll down to “service mode” and press <Enter>, then <Esc> to main menu. Press <Menu> and select “audit” from the “soft function keys”.

B. Install a supply magazine with an empty cassette (no screen or Teflon filter), filter cassette (with a screen and Teflon filter), and an internal leak check cassette (with a solid disk) in a magazine on the left side (the supply side) of the filter exchange mechanism**.** See figure 4.2 for illustration.

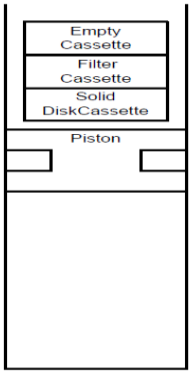


Figure 4. 2 Verification Magazine Set up

C. Install a clean, empty storage magazine on the right side of the filter exchange mechanism. Press <F4: Filter Advance > to advance the empty cassette into the sampling position and

capture the sample filter in the supply magazine. Cap it and set this magazine aside to be used later.

4.3 Performing the Monthly Verification

Verifications shall be completed in the following order External Leak Check, Ambient Pressure, Ambient Temperature, Flow, and Filter Temperature. A multipoint flow check should be substituted for the one-point Flow verification in the sequence when required.

4.3.1 External Leak Check

A. Advance the external leak check/ filter cassette into the sampling position by pressing <F4> "FiltAdv".
B. Remove the 1st stage inlet by grasping near rain collection jar and pulling straight up with a twisting motion.
C. Install the flow audit adapter on the sample down tube and close the valve on the flow audit adapter.
D. Press <F5: Leak Chk>, <F2: Start>, and follow the instructions displayed on the screen. A pass or fail message will be displayed.
E. If a leak check is failed, check the flow adapter valve, seating of the external sample tube, V-seals and the VSCC to ensure that it is assembled snuggly. (Also check the integrity of the cassette as they tend to crack, scratch and not snap together well.)
F. Repeat the leak check. After a successful external leak check, slowly open the valve on the flow audit adapter. If the leak check failed, conduct an internal leak check according to section 4.3.6 and refer to the manufacturer's service manual or check with technical support for guidance.
G. Record the results on the "Monthly Maintenance Sticky" in the logbook (appendix A.2).

4.3.2 Ambient Pressure

A. Compare the sampler's ambient pressure to the current ambient station pressure (in mmHg) measured by a calibrated reference device.
B. Verify the ambient pressure displayed in the Audit screen is within $\pm$10 mmHg of the measured barometric pressure.
C. Record the results on the "Monthly Maintenance Sticky" in the logbook (appendix A.2). If the readings are not within $\pm$10 mmHg, the sampler requires re-calibration. Refer to section 3.6 of this document, the manufacturer's service manual or check with technical support for guidance.

4.3.3 Ambient Temperature

A. Place the reference thermometer's temperature probe in the solar shield with the ambient temperature sensor of the 2025 unit.
B. Allow time for equilibration. Compare the measured temperature with the ambient temperature displayed on the Audit Screen.
C. Record the results on the "Monthly Maintenance Sticky" in the logbook (appendix A.2). If

the measured temperature and ambient temperature readings are not within $\pm$ 2° C of each other, an ambient temperature calibration is necessary. Refer to section 3.4 of this document, the manufacturer's service manual or check with technical support for guidance.

* Trouble shooting tip: Before calibration, look for insect infestations on temperature probe and corrosion on the pins of the connector.

4.3.4 Flow Rate

A. Ensure the filter used in the external leak check is still in the sample position.
B. Install the transfer standard reference unit on the sample tube.
C. Confirm that 16.7 l/min. is the set flow in the Set Flow field. Press <F1: Pump> and then <F2: Valve>.
D. Wait for the flow rate displayed in the "Cur Flow" column to stabilize.
E. Record the current value from sampler and the actual value from the reference device on the "Monthly Maintenance Sticky" in the sampler logbook (Appendix A.2). If the percent difference between the current indicated flow (sampler) and measured flow (reference device) are within $\pm$4%, the results are acceptable.
F. If these results exceed $\pm$4%, sampler requires re-calibration. Refer to section 3.8 of this document, the manufacturer's service manual or check with technical support for guidance.
G. Press <F2 Valve> then <F1: Pump> after completion of the flow rate check.
H. Replace the 1st stage inlet on the sample tube by pressing down until it hits a stop.
I. Remove the audit magazine from the supply position.
J. Replace the filter magazine containing the filter removed from sampling position at the start of the verification in the supply side (left side) of monitor.
K. Connect the air hose to the supply magazine. Press <F4: Filt Adv> to move the cassette into the sampling position.

4.3.5 Filter Temperature

A. Unlatch and open the sampler's top cover. Pull straight up to remove the VSCC.
B. Insert a calibrated reference thermometer into the sampling chamber. Allow time for equilibration.
C. Compare the measured temperature with the filter temperature displayed on the audit screen. Record the results on the monthly maintenance sticky in the logbook (appendix A.2). If the measured temperature and filter temperature are not within $\pm$ 2° C of each other, a calibration is necessary. Refer to section 3.5 of this document or the manufacturer's service manual for guidance. * Trouble shooting tip: Before calibration, look for insect infestations near cooling fans and corrosion on the pins of the connector.
D. Reinstall the VSCC. Lower the top cover and re-latch it.

4.3.6 Internal Leak Check

Performed if an external leak check failed.

A. Press <F4: Filt Adv> to advance the cassette with the solid disk into the sampling position.
B. Press<F2: Start> and follow the instruction on the screen.

C. A pass or fail message will appear on the screen. If the check is failed, refer to manufacturer's service manual or check with technical support for guidance.
D. Record the results on the monthly maintenance sticky in the logbook (appendix A.2).
E. Remove the audit magazine from the supply position.
F. Replace the filter magazine containing the filter removed from sampling position at the start of the verification in the supply side (left side) of monitor.
G. Connect the air hose to the supply magazine. Press <F4: Filt Adv> to move the cassette into the sampling position.
H. The internal leak check cassette will advance to the storage magazine. Reorganize audit cassettes in audit magazine back to their original position.
I. Press <Esc> twice to return to the Main Screen. Press <Run/Stop> to return to "Wait" or "Samp" Mode. Press <Menu>, then scroll down to "exit service menu" to reenter "Stop" mode.

4.4 Multipoint Verification of Flow Rate

Multipoint Verification of a sampler's flow rate measurement system is performed annually or when single flow rate verification indicates a variation of more than ± 4%. The multipoint verification must be conducted while in the Audit Mode. The flow rates are checked at three points: 15.0, 16.7 and 18.3 liters per minute. By conducting the verification of the flow rate in the Audit Mode, no adjustments of the sampler's flow rate span or offset are made. All indicated flow rates must be within ±2% of the measured flow rate using a certified reference device. The multipoint verification should typically follow the annual calibration. The form for the multipoint verification is located on the calibration sheet. Note: If only a multi-point verification is being performed, the calibration sheet should still be used, filling in only the appropriate boxes.

A. Ensure the filter used in the external leak check is still in the sample position.
B. Install the transfer standard reference unit on the sample tube.
C. Confirm that 16.7 l/min. is the set flow in the Set Flow field. Press <F1: Pump> and then <F2: Valve>.
D. Wait for the flow rate displayed in the Cur Flow column to stabilize.
E. Record the current value from sampler and the actual value from reference device in the sampler logbook. If the percent difference between the current indicated flow (sampler) and measured flow (reference device) are within ±2%, the results are acceptable.
F. If these results exceed ±2%, sampler requires re-calibration. Refer to section 3.8 of this document, the manufacturer's service manual or check with technical support for guidance.
G. Return to the Menu to change the set flow field to 15.0. Press <F1: Pump> and then <F2: Valve>.
H. Wait for the flow rate displayed in the Cur Flow column to stabilize.
I. Record the current value from sampler and the actual value from reference device in the sampler logbook. If the percent difference between the current indicated flow (sampler) and measured flow (reference device) are within ±2%, the results are acceptable.
J. Return to the Menu to change the set flow field to 18.3. Press <F1: Pump> and then <F2: Valve>.
K. Wait for the flow rate displayed in the Cur Flow column to stabilize.
L. Record the current value from sampler and the actual value from reference device in the sampler logbook. If the percent difference between the current indicated flow (sampler) and measured flow (reference device) are within ±2%, the results are acceptable
M. Press <F2 Valve> then <F1: Pump> after completion of the flow rate check.

N. Return to the Menu to change the set flow field back to 16.7 l/min. Press Enter.
O. Replace the 1st stage inlet on the sample tube by pressing down until it hits a stop.
P. <ESC> to main menu

4.5 Quality Control and Corrective Actions

Minor problems that affect a few filters at a site, shall be notated on the "PM2.5 Filter Cassette Transport Sheet" (Appendix A.4). When failed audits, verifications, multiple down days for service, or other significant event that affects multiple filters occurs a "Corrective Action Report" (Appendix A.7) should be completed by the Operator, Quality Assurance Officer, or Air Monitoring Program Manager. This report shall detail the issue, the actions taken, and quality control procedures performed. Additionally, this report should include the last passing QC check.

Table 4.2 below details action points, more stringent than acceptance criteria of Table 4.1 and corrective action to be followed.

Data Quality Indicator	Frequency	Objective	Corrective Action
Flow verification	**Monthly**	**±3.5% of transfer standard ±4.0% of design value**	**Leak Check Document actions Perform multi point Calibration/ Verification**
Temperature Verification	**Monthly**	**±2°C**	**Investigate Check damage/ blockage of fan. Document actions Recalibrate**
Pressure verification	**Monthly**	**±10 mmHg**	**Investigate Document actions Recalibrate**
Quarterly Internal Audit	**4 times a year**	**±4% of transfer standard ±5 % of design value**	**Leak Check Document actions Perform multi point Calibration/ Verification**
Completeness	**Scheduled Sampling Days**	**80% each quarter**	**Schedule make up run if missed Notify Lab Manager if site down for more than 1 run day**
Laboratory verification	**At least Quarterly**	**Lab QC checks and filter handling verified**	**Contact Lab**
Internal Leak check	**Monthly**	**Pass**	**Investigate, Check cassette ring, seals, O-rings. Replace or lubricate as necessary. Repeat test.**
Multipoint Verification	**Annually**	**Same as monthly Flow/ Temperature/ Pressure verifications**	**Re-calibrate**
Collocated Samples	**Every 6 days, aggregated quarterly, annually**	**CV ± 10% for samples > 3.0 µg/m3**	**Notify Lab Manager, review flow rates of collocated monitors**
Calibration	**At installation, post-major repair, after failed verification,)**	**Operation Manual**	**Contact Equipment Manufacture**

Table 4.2 Quality Control and Corrective Actions

5.0 Sample Procedures

5.1 List of Tools, Equipment and Materials

- EPA Ambient Particulate Monitoring Sample-Day Schedule
- KCDAQM's sample schedule
- Clean, unexposed, EPA approved Teflon filters in cassettes
- Disposable gloves for handling the filters (powder free)
- Logbook and ink pens
- Filter magazine
- Refrigerator

5.2 Preparing Filters

The laboratory will supply KCDAQM with pre-weighed Teflon filters installed in filter cassettes. Filter cassettes should be stored in a secure environment to maintain chain of custody. Although there are no control limits on humidity or temperature for the storage environment of unexposed filters, an area with a reasonable degree of temperature control shall be designated. The PM2.5 desk and adjacent shelving in the clean room is designated for PM2.5 filter handling. This area must be kept clean of dust and debris.

The laboratory will provide the: filter number, cassette number, and expiration date. (The expiration date is the use by date but is labeled by the supplying lab as the expiration date). This information will be listed on the "Packing List", (Appendix A.3) which also serves as a chain of custody. The information on the "Packing List", will be transferred to the "PM2.5 filter Cassette Transport Sheet" (Appendix A.4) for each site.

Operator will create a filter exchange schedule which will indicate sampling and pick-up dates. All samples should be picked up on the assigned schedule within 96 hours. Refer to the KCDAQM QAPP Table 7.2 for sample recovery requirements. The schedule is entered on a calendar and kept in the PM2.5 gear bag. The sites are represented by site name abbreviations followed by the number of filters in the magazine; for example, A-3 is Air lab site with 3 filters in the magazine). A line under the number represents a field blank is in the (A-3). A highlighted slash through the number indicates the presence of an empty ring in position #1(A-3).

NOTE: The filters are never to be separated from their cassette. Staff and previous documents may inter-change the terminology and use filter, filter cassette, cassette interchangeably at times.

5.3 Loading Filters

Filters are loaded into magazines prior to the filter exchange. Operators shall use the schedule calendar to determine how many filters in each magazine are necessary for the next filter exchange.

A. Prepare for Filter loading by cleaning the desk and surrounding area in the clean room by wiping surfaces with an alcohol wipe.
B. Put on static free gloves for handling filters.

C. Count out the number of filters needed for a sampling site according to the sampling calendar. Remove appropriate number of anti-static bags containing the filter cassettes from the larger zip-loc bag they were shipped in. Make note if empty ring, or field blank is needed.
D. Record the filter numbers and cassette numbers on the "$PM_{2.5}$ filter Cassette Transport Sheet" (See Appendix A.4). Get a clean magazine and record the magazine number on the form as well.
E. Remove the filter cassettes from their anti-static bags and place them in the magazine. Remember to place them in reverse order so that the filter scheduled to run on the most recent day will be on top, and the last filter scheduled to run will be on the bottom. The filter is upside down if the silver screen support if visible. Cap the filter magazine and place in the metal transport case.
F. Make sure each storage bag is sealed. Mark each storage bag with initial of the site filter will be sampled at, then paperclip anti-static bags together by sampling site and set aside for return shipping.

5.4 Magazine Installation and Retrieval

Each magazine will be transported in a metal transport case designed for this purpose. Upon arrival at the monitoring site, unlock the security fencing and inspect the equipment. Note any items needing maintenance or any irregularities that may affect data quality in the comment section on the log sheets.

A. **Upon Arrival** Check Status light on top of unit.
    a. If **on** or **flashing** press <Stats> then <St Code> from the "soft function keys" across the bottom of the screen. A code will appear in the upper left-hand corner. Refer to sampler's manual section 7.1 for help. If the problem is minor (**Not "N" stat)** according to the manual (example: power failure) press <Reset> and <Yes> then <ESC> to main menu. Record all status codes on the "$PM_{2.5}$ filter Cassette Transport Sheet" and sampler logbook
    b. **If** the stat is "N" and the monitor **is not** scheduled to be running on the day of exchange, DO NOT CLEAR STATS. This will be addressed later in this section under "Exchanging the filters for a monitor that is not running upon arrival". If the monitor is scheduled to be running and an "N" status is displayed, the monitor has encountered a filter exchange error. See operator's manual for assistance.

B. **Inputting a filter list:**
    a. Input the Filter List by selecting <FiltSet> from the "soft function keys" on the bottom of the screen. Select <FiltLst>, Press <Edit>.
    b. Using the arrow keys scroll over past the "T" to the filter numbers list. Input filter number, scroll over using the arrow key to cassette number and enter the cassette number. Note: The letter "T" can be changed if need be. Each letter is represented by a number via its place in the alphabet. Place the curser over the letter, press <Edit>, type "20" then <Enter>. The letter will appear as "T" as "T" is the 20th letter of the alphabet.
    c. If the filter is a blank scroll over again using the arrow key and press the <+list> to change the "NO" to a "YES" (do not press <Enter> until you have completed updating all the information for the filter list. If you accidentally do press <Enter>, simply press <Edit> again to continue entering information.)
    d. Press <Enter> when done and <ESC> to main menu.

C. **Exchanging the filters for a monitor that is running upon arrival**
   a. "Samp" will be shown in the upper right-hand corner of the screen. Remove empty supply magazine from left side (magazine hooked to air hose).
   b. Place new supply magazine on the left side and reconnect the air hose.
   c. Take care to shelter the filters after orange cap is removed from magazine and quickly place it in the monitor and reconnect the air hose.
   d. Remove storage magazine from right side and place the orange cap on it. Again, take care to shelter the filters from debris and place orange cap **immediately** on the magazine.
   e. Place the empty storage magazine on the right side. Make sure the piston is in the **top** most position to prevent filter exchange errors or contamination.
   f. Place the magazine with the removed exposed filters in the metal storage container and place the container in the cooler for transport.

D. **Exchanging the filters for a monitor that is not running upon arrival**
   a. If the monitor is not running, "Done" or "Wait" should be displayed in the upper right-hand corner.
   b. Remove air supply hose from supply magazine on the left, then remove empty magazine. Get new supply magazine out and ready for installation.
   c. A field blank or an empty cassette ring will be in the top position of the new supply magazine.
   d. Place the supply magazine with new filters in the left side of monitor, and then reconnect the air hose. Make sure to consult the scheduling calendar if unsure about when to install an empty ring or a field blank.

      i. <u>If field blank **is not** being installed</u> (an empty ring will be in the top position): Press <Stats> then <St Code>.
         1. Select <Filtadv> or <F5>. This will take you to the filter exchange screen. Press <Start> and the empty ring should advance into sampling position.
         2. Remove the storage magazine on the right side of cabinet and replace orange cap.
         3. Place the empty magazine on the right side as a storage magazine; make sure the piston is in the top position. <ESC> to the main menu and press <Run/Stop> to place monitor in Wait Mode. it is imperative the monitor is in Wait Mode. Place removed magazine with exposed filters into metal storage container and place in the cooler for transport.

      ii. <u>If a field blank **is** to be installed:</u> Press <Run/Stop> until sampler is in "Wait" mode.
         1. The field blank will advance into sample position. The 1st exchange will move the field blank into sample position, pushing the last filter that ran into the storage magazine.
         2. Quickly remove the storage magazine on the right side of cabinet and replace orange cap.
         3. Place the empty magazine on the right side as a storage magazine. Make sure the piston is in the top position.
         4. A 2nd exchange will occur to push the field blank through into the

storage magazine. The field blank will stay in the monitor until the next scheduled pickup.

5. Place the magazine with the removed filters in a metal storage container and place the container in the cooler for transport.

5.5 Post Sampling Filter Handling

Post sampling filter handling is critical to collecting valid data. The transport and storage temperatures affect the time period allowed for weighing of samples.

5.5.1 Preparing / Transporting

A. Store ice packs in a cooler and put the cooler in a freezer capable of freezing the ice packs solid overnight.
B. To prepare for the trip to the sampler: Place metal transport cases into chilled cooler.
C. Take two frozen-solid ice packs and pack them around the metal case(s) so they fill in as much void space as possible. This is to maintain a minimum internal temperature.

The cooler does not need to be taken up on elevated monitoring platforms and may be kept in the passenger compartment of the operator's vehicle. After completion of all required tasks involving the sampler, place the magazine containing exposed filters in chilled metal storage container and then place the metal storage container in the cooler.

Note: Care must be taken to maintain the cooler in an upright position when opening the lid or removing and replacing the chilled metal transport case(s).

5.5.2 Post Sampling Handling in AQM Lab

The $PM_{2.5}$ filter storage refrigerator is located in the designated clean room. The temperature of the refrigerator must be maintained between 0°C - 4° C.

A. Record the operator initials, date, Action (Filters in/ Filters out) current T, Max T, Min T and time in the Filter storage refrigerator log book 2.5 Storage Temperature Record.
B. Carefully remove all metal transport case(s) from cooler and place them on the counter beside the refrigerator.
C. Remove magazines from transport cases
D. Place the magazines in the refrigerator designated for $PM_{2.5}$ samples. Carefully close the door. These steps minimize the loss of cold air and the introduction of warm air, thus helping to prevent an increase in the temperature of the refrigerator's interior.
E. Reset the Max/Min thermometer. Note: Any exceedances of the holding temperature minimum (0 ° C) or maximum (4° C) shall be recorded in the Storage Temperature Record Logbook, as well as transferred to the note section of the "$PM_{2.5}$ Filter Cassette Transport Sheet" for all filters affected by the temperature excursion.
F. Also, record the start time (if known), end time and cause of the temperature increase from events such as general power failures, defrosting, accidental unplugging, etc.

5.6 Shipping Procedures

Filters are shipped overnight, usually on a Thursday, every two weeks, or more frequently to Inter-Mountain Labs in Wyoming. Personnel should review the sampling dates, holidays, and an estimated receiving date to ensure that the samples have time to be received, processed, conditioned and weighed within the 30-day window. Additional review will be necessary during data validation if storage and shipping temperatures exceed 4 ° C.

A. Retrieve filter storage bags and remove magazines from refrigerator. In an effort to decrease time outside of refrigeration consider removing all the magazines for one site, then moving on to the next.
B. Put on static free/lint free gloves.
C. Remove the magazines orange cap.
D. Connect the air pump to the fitting at the base of the magazine. Pump the bulb of the air pump, to move filters up to the top. Once the top most filter is exposed, stop pumping.
E. Gingerly remove exposed filter and place it back in its designated storage bag.
F. **Ensure the bag is sealed!** Repeat for each remaining filter.
G. Rubber band together the filter bags in groups (approx. 10 at a time) so they don't jostle unnecessarily during shipping.
H. Place banded groups of filters into larger (gallon sized) zip lock bag and seal.
I. Return filters in bags to the sampler refrigerator. Fill out the "Chain of Custody" (Appendix A.5). List the Date sampled, filter number under sample identification, filter's void status and field blanks under comments. This should be typed and saved on L: Drive.
J. Print COC and verify number of samples sent is correct. Make a copy of the COC and give to Data Validator.
K. Pack shipping cooler with blue ice freezer packs, filter samples in bags and min/max thermometer. Note: Be sure to reset min/max upon placement in cooler.
L. Place original COC in Ziploc bag and place in cooler.
M. Thoroughly seal the shipping cooler with packing tape. Place shipping label on and take for shipping. Note: Shipping currently occurs via UPS.

5.7 Downloading Sampler Data

Data can be downloaded in several different ways. The preferred way is by remotely accessing the instrument via modem. Data can also be downloaded to a laptop using RPCOMM or HyperTerminal software and a RS232 cable. As a last resort filter data can be written down manually. Data should be downloaded or recorded at least every two weeks.

Data must be saved to a flash drive installed on the MTL computer (F: drive) and also to the shared network drive (L: Drive). On F: Drive the data is saved into "PM25 Data YEAR", then sorted into folder, by quarter, month and site name. Name the file with the following format: two-digit year, two-digit month, two-digit date, and site name abbreviation. Make sure the file is saved as a text file. For example; data downloaded on April 1, 2016 at Spring Hill would be 160401sh.txt and would be saved under file path F: PM25 Data 2016/ 2nd Quarter/ April/ Springhill. When saving on L: Drive (the network drive) use the same format as above and follow this path to get to the folder PM25 Data YEAR: L:\Air Lab Data\Ambient Air Monitoring\Pollutants\PM 25 Monitoring\PM 2.5 Datawands.

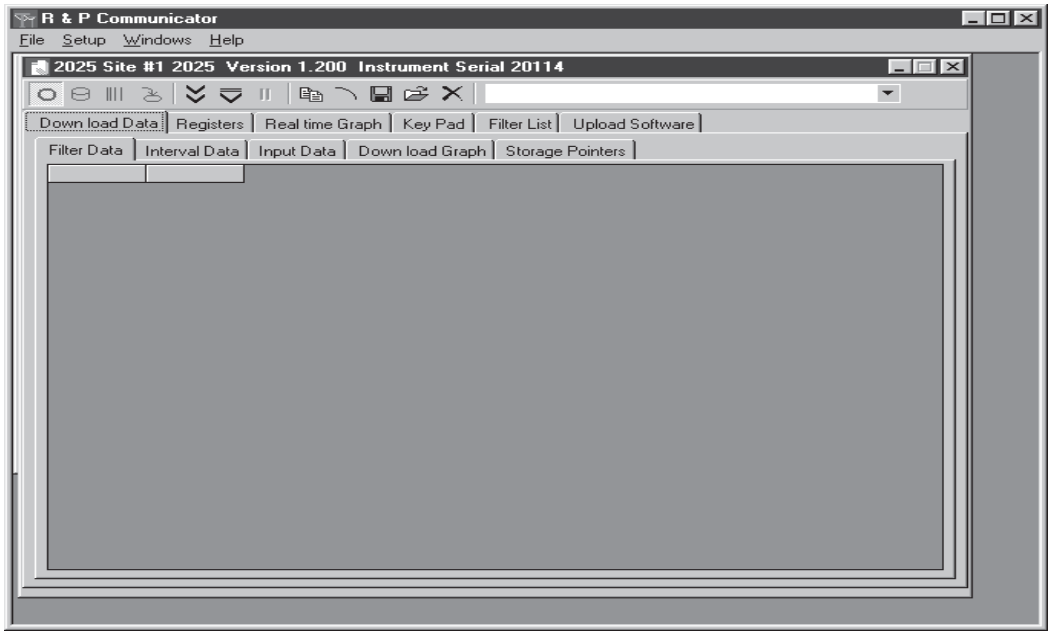
If interval data is needed, it is labeled in the same format as filter data with an "i" in front of it (i070401sh.txt). Interval data is labeled with an "ip" in front of (ip070401sh.txt)]. Once filter data has been saved, open the file to make sure the data downloaded properly. If not, try once more. If still unsuccessful, try downloading data via laptop computer.

Operator should check monitors status by remotely accessing the unit. Most sites are equipped with a phone line and a modem. See Appendix J in "Partisol® -Plus Model 2025 Sequential Air Sampler Operating Manual" for detailed instructions on setting up communication via modem.

5.7.1 Using Modem to Check Monitor Status

A. From the MTL compute at KCDAQM's Air Lab, initiate RPCOMM. Click "File", "Open Group", "2025 network" Select monitor from list.

This screen will appear



B. Click to connect to monitor. Clicking on the "Key Pad" tab will show the main screen of the selected 2.5 monitor. Confirm the monitor is operating correctly. The user may also navigate through the menus and make adjustments as needed.
C. To check the previous run dates select <Data> from the "soft function key"s across the bottom of the monitors main screen. Dates are listed from present to past. The date is in the upper right-hand corner and the elapsed time in the middle at the bottom. Press <-Rec> or <+Rec> to scroll through the dates. Make sure all filters ran for **at least** 23:00 hours and **no more** than 25:00 (unless the filter was a blank). Note any inconsistencies on the "$PM_{2.5}$ Filter Cassette Transport Sheet" and take action to make up incomplete or missing data.

5.7.2 Downloading Data Remotely

A. Open RPComm on MTL computer, click <File>, “Open Group”, “2025 network” and Select monitor from the list.
B. Click to connect to monitor
C. Choose (Download Filter Data) from tabs
D. Click (Download all stored data)
E. Once data has finished downloading, click (Save). Data must be saved with the date downloaded and site initials in the name. Additionally, be sure to save as .txt file.

5.7.3 Downloading Data via Laptop

A. Connect RS232 cable to the port on the sampler and the “RS232 to USB adaptor”.
B. Connect “RS232 to USB adaptor” to the laptop computer.
C. Initiate RP COMM Click <File>, “Open Group”, “2025 Air Monitoring Network”
D. Click to connect to monitor
E. Choose (Download Filter Data) from tabs
F. Click (Download all stored data)
G. Once data has finished downloading, click (Save). Data must be saved with the date downloaded and site initials in the name. Additionally, be sure to save as .txt file.
H. If the data did not download properly use HyperTerminal (which is located under the start menu → all programs →accessories → communications) to download the data. Call technical support at 1-866-282-0430 to help resolve any problem. They can also give pointers on using HyperTerminal if unfamiliar.

6.0 Maintenance

All maintenance should be performed while the unit is in Wait, Audit, or Stop Modes. The "audit filter" should be used during maintenance to limit the chance of damage or contamination to the sample filter. All maintenance activities should be recorded on the monthly maintenance sticky in the sampler logbook including a brief description of the activity and the date of the activity.

6.1 Routine Maintenance

The monitoring station shall be kept clean. The operator shall inspect the station for any potential problems each time he/she visits the site. Examples of potential problems include:

- Storm or weather-related damage including accumulation of dirt and debris
- Insect or rodent infestations
- Vegetation overgrowth, weeds impeding on gates or trees that may impact siting criteria

6.2 Monthly Maintenance

A. The operator should complete a leak check and flow verification prior to performing maintenance but must complete at least a leak check. See Section 4.3 for instructions on performing these checks.
B. Clean the first stage inlet figure 6.1: The inlet is removed by gently lifting the complete inlet upward from the sample tube. Grasp near the rain collection jar and turn and lift upward. Disassemble, clean with alcohol wipe, check O-rings, and reassemble. Refer to Appendix G of the Manufacturer's Operating Manual for detailed directions on cleaning the inlet.

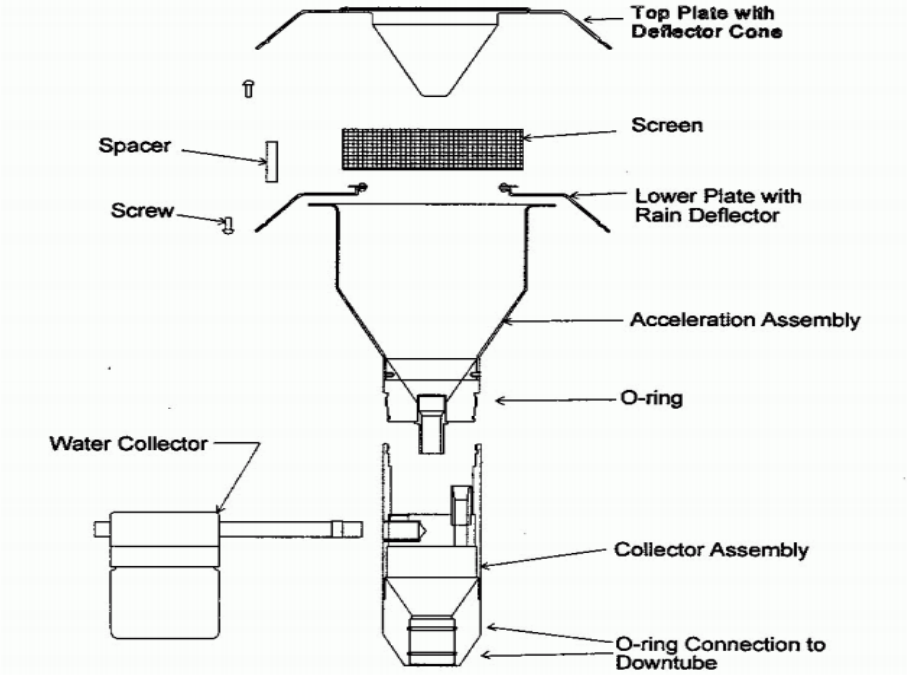


Figure 6.1 First Stage Inlet Diagram (PM_{10} head)

C. Clean the very sharp cut cyclone (VSCC) figure 6.2

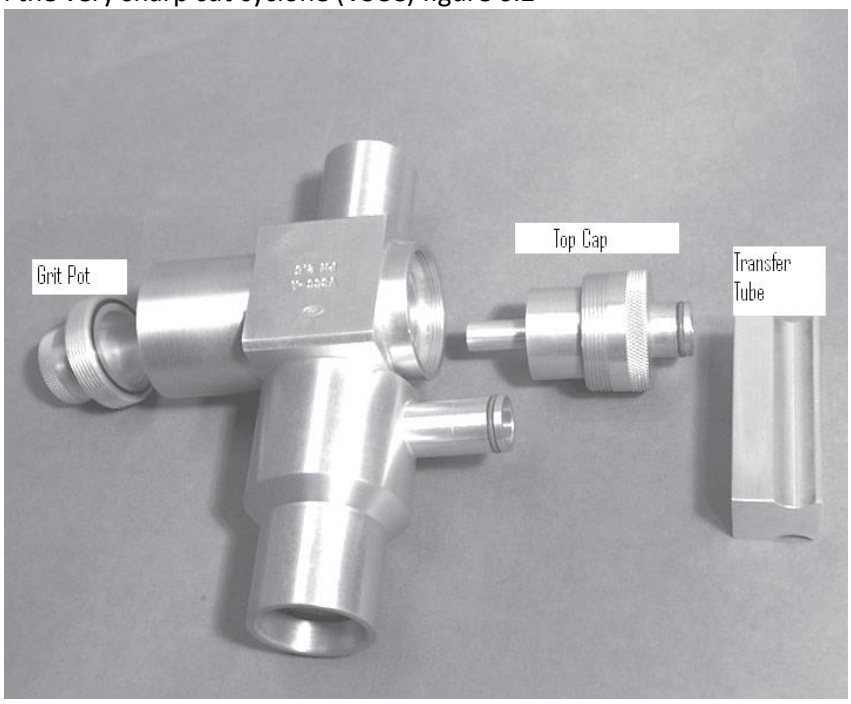


Figure 6.2 Very Sharp Cut Cyclone Diagram

a. Remove the VSCC and transfer tube.
b. Unscrew the top cap and grit pot from the VSCC. Use a moist, lent free cloth and remove all visible deposits from the inside of the VSCC.
c. Inspect all O-rings for shape and integrity. Replace if necessary.
d. Lubricate the O-rings with a light silicone grease.
e. Install the grit pot and top cap into the VSCC. Install the transfer tube in the VSCC and reinsert the VSCC onto the sampler.
f. Perform an additional external leak check after maintenance is complete.
g. Replace the PM_{10} inlet.

6.3 Quarterly Maintenance

A. The operator should complete a flow verification and leak check prior to performing maintenance but must complete at least a leak check. See Section 4.3 for instructions on performing these checks.
B. Clean sample down tube: Make sure the audit filter is in sample position and that the leak check adaptor is placed over the inlet of the VSCC before removing down tube. Some water usually collects at base of tube and will run out when it is removed, so take care to cover the sample filter.
    a. Remove the stage-1 inlet and loosen the large plastic nut at base of sample down tube. In an upward twisting motion, pull upward firmly on the down tube until it is free from the sampler.
    b. Swab inside of tube with a clean, lent-free cloth. Clean the area where the down tube is

seated in the hatch and check O-rings.
c. Lubricate O-rings if necessary and reinstall down tube and making sure it is seated completely.
d. Hand tighten the plastic nut at base of down tube.

C. Check/Clean V-Seals: Ensure the instrument is in Service Mode.

a. On the Service Menu press <F4:Filtchg> to go to the Filter Exchange Screen.
b. Turn on the pump, turn on the pressure vent valve, and turn on the lift/push valve.
c. Unlatch and open the top cover of the sampler. Lift the down tube out of the upper part of the sampler enclosure and locate the down tube mount on the top cover.
d. While supporting the ring on the underside of the top cover, remove the four screws that secure the down tube mount to the top cover.
e. Remove the down tube mount and ring. Locate the down tube “V” seal.
f. Examine the seal for drying and cracking. Replace if necessary or clean undamaged seals with alcohol wipe.
g. Replace the down tube mount and ring onto the top cover and ensure the side of the o ring with the chamber is facing down.
h. Remove the 4 screws on the top head mounting plate of the filter exchange assembly.
i. Lift the plate and remove from the enclosure. Example the 2 “V” seals exposed. Replace if necessary or clean undamaged seals with alcohol wipe.
j. Remove the three screws that secure the exit cylinder base to the filter exchange assembly. Remove the assembly from the enclosure.
k. Inspect the exit cylinder “V” Seal. Replace if necessary or clean undamaged seals with alcohol wipe.
l. Return the exit cylinder bases on the exchange assemble and secure with the screws.
m. Refer to section 12 in the operations manual for detailed directions on checking and cleaning the V-seals.

D. Perform an External Leak Check upon completion of Quarterly maintenance. See Section 4.3.1.

6.4 Semi-Annual Maintenance

A. The operator should complete a flow verification and leak check prior to performing maintenance but must complete at least a leak check. See Section 4.3 for instructions on performing these checks.
B. Clean/replace the fan filters: There are two fan filters. One in the filter compartment and one in the pump compartment.
    a. Snap off the covers enclosing the air intake filters. Take out the filters and clean them by lightly brushing or shaking the dust off.
    b. Filters can also be replaced, and dirty filters returned to the office for cleaning in mild soap and drying. Reinstall the filters in their holders and remount the cover.
    c. Refer to section 3 of the Service Manual for detailed directions on cleaning/replacing the fan filters.
C. Exchanging particle trap filter:
    a. Turn off the sampler.
    b. Locate the bowl-style filter behind the filter exchange assembly.
    c. Remove the filter bowl from the filter manifold.

d. Carefully remove the filter stand by unscrewing it from the filter.
e. Remove the gasket and top disk from the filter stand.
f. Remove the filter element from the filter stand and install a new element.
g. Install the top disk and gasket into the filter stand.
h. Install the filter stand into the filter manifold and O-ring.
i. Install the filter bowl into the filter manifold.
j. Do not over tighten the filter manifold.
k. Refer to section 12 in the operations manual for detailed directions on changing the particle trap filter.

D. Replace Batteries: Three alkaline AA batteries provide backup power for internal data storage and the clock/calendar.
   a. Remove the three screws securing the pump compartment cover, slide the cover up and remove.
   b. Open the electronics compartment and locate the batteries. Remove the clip that holds the batteries in their mounting.
   c. Pull the old batteries out and replace with 3 new batteries.
   d. Complete this process within 5 minutes to avoid the loss of data stored in the RAM.
   e. Reinstall the clips, Close the compartment.
   f. Refer to section 3 of the Service Manual for detailed directions on testing the batteries if there are suspected problems with the battery backup.
E. Perform an External Leak Check upon completion of Semi-Annual Maintenance. See Section 4.3.1

6.5 18-month Maintenance

The pump in the Partisol-Plus Sampler has a lifetime of approximately 12-18 months. If the pump's performance deteriorates pumps may be rebuilt but should be replaced after several rebuilds. Wipe down the interior of the sampler's case to remove any bugs or dirt. See Thermo's replacement parts sheet or call customer service for part number.

7.0 Audit Procedures

Please also consult the most recent Knox County Air Quality Management Air Monitoring Division Audit SOP. The most recently updated SOP should be used for auditing procedures and forms. Use form field sheet PM2.5 (Appendix A.6). Audit equipment to be used and acceptability outlined in Table 7.1 below.

Equipment	Recertification	Acceptable Range
Flow rate Transfer Std	1/yr	±2% of NIST Traceable Std
Field Thermometer	1/yr	±0.1°C resolution, ±0.5°C Accuracy
Field Barometer	1/yr	±1mmHg resolution, ±5mmHg accuracy
Audit Magazine	N/A	With Audit filter
New Storage Magazine	N/A	
Static free Gloves	N/A	
Clock	N/A	Cell phone updated to towers acceptable

Table 7.1 Audit Equipment

7.1 Filter Handling

The Auditor shall observe or verify the filter handling procedures for both clean and exposed filter. The clean filters shall be stored in anti-static bags and handled with anti-static gloves. The work area shall be a clean surface for loading the magazines. The operator shall enter the filter and cartridge number on the field sheets and load the magazines with an organized method to insure the filters are aligned to run on the correct dates.

The Auditor shall inspect the Air Sample Storage Log Book. Each entry should be complete and it shall be up to date. The Auditor shall verify that the maximum temperature recorded from the Max/Min Thermometer in the log book has not exceeded 4°C. Exceedances shall be recorded in the log book and notated on field sheets.

7.2 Field Check

The Auditor shall inspect the very sharp cut cyclone (VSCC) and PM 10 head to ensure they are clean and properly maintained. The Auditor shall perform or observe the operator complete the following quality control measure and insure the results are within the corresponding limits according to table 7.2. Section 12: Routine Maintenance and Verification Procedures" in the Partisol-Plus Model 2025 Non-continuous Air Sampler, Operating Manual is a good reference for the procedures.

Measure	Acceptance Criteria
Time	± 1 minute
Filter Temperature	±2°C
Ambient Temperature	±2°C
Ambient Pressure	± 10mmHg
Flow Rate	± 4% of transfer standard

Table 7.2 Audit Acceptance Criteria

A. Time should be compared to audit clock and recorded. If outside limits notate and continue with audit. Operator should update clock after audit is completed.
B. Place monitor in Audit Mode. From the wait mode, press <Run/Stop> once to enter the Audit Mode. The unit will display the Audit Confirmation Screen. Press <F1:Audit> and then press <Menu> key twice and select <audit>, then press <enter>.
C. Remove storage magazine, cap and set aside.
D. Remove supply magazine and place on storage side.
E. Install audit magazine on supply side (Audit magazine consists of a filter cassette with filter not used for sampling and an empty ring on top).
F. Advance filter –Press <F4: Filter Advance> to advance the empty ring cassette into the sampling position allowing the sampling filter to be in the top most position of the Supply magazine. Remove and Re- cap Supply magazine. Filters used for flow rate audits shall not be used for further sampling.
G. Remove the PM10 Head by pulling straight up on the sample tube. Unlatch and open the sampler's top cover. Pull straight up to remove the VSCC.
H. Verify the sampler's ambient temperature by measuring the current temperature at the ambient temperature sensor using a calibrated reference thermometer. Compare the temperature from the audit standard to the average ambient temperature displayed on the Audit Screen. If the two readings are not within ± 2°C of each other the ambient temperature audit has failed. Record on Field Sheet PM 2.5. Complete the audit and notify Operator and Lab Manager of deficiency for further investigation.
I. Verify the sampler's filter temperature by inserting the calibrated reference thermometer into the sampling chamber. Compare the measured temperature (°C) with the filter temperature displayed in the Audit screen. If the measured and filter temperature readings are not within ± 2°C of each other the filter temperature audit has failed. Record on Field Sheet PM 2.5. Complete the audit and notify the Operator and Lab Manager of deficiency for further investigation.
J. Verify the sampler's ambient pressure by measuring the current ambient pressure in mm Hg with a calibrated reference barometer. The average value for ambient pressure displayed on the Audit screen should be within ±10mmHg of the average audit standard's barometric pressure reading. If out of range, the ambient pressure audit has failed. Record on Field Sheet PM 2.5. Complete the audit and notify the Operator and Lab Manager of deficiency for further investigation.
K. Reinstall the VSCC & Lower the top cover and re-latch it.
L. Advance Filter –Press <F4: Filter Advance> to advance the audit filter cassette into the sampling position. Catch the empty ring as it advances to storage side and set aside. *If the monitor was not running at the start of audit, the pump must warm up for 10-15 minutes before completing the flow rate audit.
M. Perform an external leak check.
    a. Install the flow audit adapter on the sample down tube and close the valve on the flow audit adapter.
    b. Press <F5:Leak CHK>,<F2:Start>, and follow the instructions displayed on the screen.
    c. A pass or fail message will be displayed
    d. If a leak check failed, check the flow adapter valve, seating of the external sample tube and the VSCC to ensure it has been reassembled snuggly. Also check the audit filter cassette to ensure cassette isn't damaged. Repeat test.
    e. After a second failure the flow audit fails and operator shall perform internal leak check

and investigate problems further

f. If leak check passes on the first or second time, record on field sheet and proceed to Flow rate audit.

N. Place the flow sensor on the top of the monitor's inlet. Confirm that 16.7 L/min is the set flow in the Set Flow field. Press <F1:Pump> and then <F2: Valve>/ wait for the flow rate displayed in the Cur Flow column to stabilize. *If monitor was not running at the start of audit, the pump must warm up for 10-15 minutes. Record the displayed value and the audit standard value. If the percent difference between the sampler flow and the audit flow are within ± 4% the results are acceptable. If not, the flow audit has failed. Record results on Field Sheet PM 2.5. Complete the audit and notify the Operator and Lab Manager of deficiency for further investigation.

O. Remove audit magazine from the supply position and place it on the storage side. Re-install the supply magazine, verifying visually that the previously removed sampling filter cassette is in the top most position. Connect the air hose. Press <F4: Filt Adv> to move the cassette into sampling position. The audit filter will advance to the audit magazine. Remove the audit magazine, place the empty ring back on top and replace it with the original storage magazine from 4.1 E. Cap audit magazine for use at next location.

P. Replace the PM10 head on the sample tube by pressing down until it hits a stop. Press <Esc> twice to return to the Main Screen. Press <Run/Stop> to return to the Wait Mode or place the machine back in sample mode if interrupted.

7.3 Evaluation of Results

If a parameter value is outside the acceptance criteria, the Auditor shall make note of the last successful verification from the instrument log book. The Operator shall investigate the cause of the deficiency and follow current SOP procedures for proper corrective action. All information will be forwarded to the QA officer and Lab Manager for evaluation

The QA Officer shall review field sheet PM SP for completeness for each sampler. The data shall be entered into the Quarterly Audit Workbook. The Auditor prepares a summary of the audit including any suggested corrective action. The summary should include any borderline results which required operator intervention to correct onsite but must include any results outside the acceptance criteria. See Section 4.5 for quality control and corrective actions.

8.0 Data Validation and AQS Entry

Data must be validated before submitted to AQS. The QA personnel shall perform validation procedures at three points. First, upon shipment of filters to the Inter-Mountain Laboratories (IML) which includes sampler data to be included in final report from IML. Second, by reviewing calibration/ verification data monthly as completed and finally upon receipt of weight data and AQS file format from IML. Data validation is the confirmation, through objective evidence, that the requirements are met for the intended use. We validate with the intended end use of NAQQS comparison as the standard with the weight of evidence approach. Section 8.1 represents the established criteria and validation action for specific typical events.

8.1 Validation Criteria

The following chart indicates the first round of; validation criteria, void codes typically used, whether this will affect a single sample or batch, and whether evaluated with shipment, verification or post weigh data return. QA personnel shall use this as a guide, as well as follow Table 7.2 $PM_{2.5}$ Measurement Quality Objectives found in the KCDAQM QAPP.

Process	Acceptance Criteria	Void Code/ Flag	Single or Batch (S/B)	Evaluation Point
Sampling				
	Sample Period 1380-1500 minutes	AG	S	Shipment
	Average Flow rate 15.84-17.5 L/min	AH	S	Shipment
	Flow CV ≤2%	AH	S	Shipment
	No Flow Rate Excursions >±5% for >5min	Flag W	S	Shipment
	No Filter Temp excursions >5°C for > 30 min	Flag X	S	Shipment
Filter Handling				
	Sampled before Expiration date	TS	S	Shipment
	Samples recovered ≤7 days 9 hours	Flag 2, HT	B	Shipment
	Transport Temperature ≤ 4° C * Review Average T by filter,		B	Post Weigh
	Final weighing ≤30 days from sample at 4° C or < Avg sample T	TS	B	Post Weigh
Conditioning				
	Field Blank ± 30 µg	FB	B	Post Weigh
	Lab Blank ± 15µg	LB	B	Post Weigh
	Temperature 24hr mean 20°-23°, ±2°C st.d	Flag 1, TR	B	Post Weigh
	Humidity 24hr mean 30-40%RH, ±5% St.d	Flag 1, HR	B	Post Weigh

Table 8.1 Validation Criteria

Process	Acceptance Criteria	Void Code/ Flag	Single or Batch (S/B)	Evaluation Point
Verifications				
	Monthly separated by 14 days	Flag 6,	B	verification
	External Leak check <25 mmHg	Flag 2	B	verification
	Flow rate ±4%of Transfer standard	AH	B	verification
	Temperature ±4° of standard	Flag 2, TP	B	verification
	Pressure ± 10mmHg of standard	Flag 2, BP	B	verification
	Flow rate ± 5% of design flow 16.67	AH	B	verification

Table 8.1 Validation Criteria cont.

The batch sample voids/flags for Filter Handling and Conditioning are applied to all affected filters. The verification batch analysis the data validator shall invalidate/flag all data collected back until a documented passing/acceptable verification occurred, and forward until an acceptable verification occurred.

Each validation decision must be documented on the Filter Cassette Transport Sheets". Any additional documentation shall be attached to the affected "Filter Cassette Transport Sheet." The initial data review and validation is performed by an environmental specialist/ Quality Assurance Officer. The data will then receive a second-level review and validation by the Air Monitoring Manager.

8.2 Validation Procedures

KCDAQM employees the use of a software program called MTL. There is a demo CD for use if unfamiliar with the software. The MTL software is installed on the MTL computer in the computer room located at KCDAQM's Air Lab.

8.2.1 Raw Data

The PM2.5 Operator according to Section 5.6 of this SOP shall download the sampler data and save it on the L: Drive (shared drive). Once the PM2.5 Operator ships filters, they shall give the QA personnel the Chain of Custody and the associated Filter Cassette Transport Sheets. The QA personnel shall review the data according to the follow steps.

A. Prepare sampler data for emailing to IML. Review the Chain of Custody for each sampling site. Copy and Paste the sampler data from the downloaded files and save a new file that just contains the data from the current shipment. A file is created for each sampler.

B. Review the Filter Cassette Transport Sheets for notes from the operator which may include a notation of skipped filter, or machine malfunction which would invalidate some data, and/ or cause sampler to associate the incorrect data with the filter. The most frequent error found is failure to enter the Filter and Cassette numbers. This is corrected in the newly

saved file. Enter the Filter numbers and cassettes as indicated on the Filter Cassette Transport Sheets for the run days. The sampler data would just list "000000000" if this were the case. The saved files should be placed in a folder named for the date sent. This is a sample location, which path would be updated each year. L:\Air Lab Data\Ambient Air Monitoring\Pollutants\PM 25 Monitoring\PM 2.5 Datawands\PM25 Data 20xx\To IML

C. After insuring the prepared sampler data file is complete. Upload the file into the MTL program.
   a. Open the program on the desktop
   b. Click Sampler Data then File.
   c. Choose the sampler file you completed in Section B. and click save.
   d. After the file has uploaded, the filter list will be displayed on the screen. Verify that the filters uploaded represent all of the filters shipped for the sampler. Remember this is done PER SAMPLER and repeated for each.
   e. After the files for each sampler have been loaded and verified. Click Return and the top left, and then Reports.
   f. Under the Reports tab, search for the filters to validate using the site and the sampling dates.
   g. Review each filter on the screen for elapsed time, sample flow rate, and filter Temperature. A Red X should be indicated in the Sampling Conditions section to indicate a value out of range.
   h. Follow the guidelines from Table 8.1 and note any flags or voided filters on the Filter Cassette Transport Sheet.
   i. Repeat for each sampler/site location.

D. Upon completion of the preliminary validation email the sampler files created in Section B. Prepare and email to the staff at IML & copy lab manager noting all flags and void codes with corresponding filter number. Attach the sampler files to the email.

8.2.2 Weigh Data

IML will routinely email a weigh report to KCDAQM. This report contains all the weighing related data for filters analyzed. These reports are saved on to L:\Air Lab Data\Ambient Air Monitoring\Pollutants\PM 25 Monitoring\20xx IML Data\Reports. As these reports are received, they should be saved to the above location but following the correct year for the final folder. Our QA personnel shall review each report for prominent issues; such as transport temperatures, lab conditioning, or holding time issues. However, the full weigh data QA review is completed with the IML Quarterly Report.

IML also produced an AQS file which should contain the weigh data, and all void and flag codes applied at the first level of review. The report includes conditioning environment, flags and codes from laboratory issues, and confirms weigh dates from sample dates. QA personnel must review the reports for the additional QA criteria in Table 8.1. Then they shall note on the Filter Cassette Transport Sheets any QA decisions made.

The IML quarterly report includes the control charts for replicates and lab blank analyses for ease of evaluating the laboratory's quality system.

8.2.3 Final Validation

After uploading the AQS file provided by IML the QA personnel must complete a final review of data. A "PM2.5 Data Validation Check List" Figure 8.2 is used to ensure that each review step is completed.

PM 2.5 Data Validation Check List	Date	Initials
Review Lab data and flags		
Reviewed tracking sheets		
Reviewed log books		
Reviewed verifications and audits		
Investigate outliers		

Figure 8.2 Data Validation Check List

A. Review the QA activities performed per sampler such as monthly verifications and audits to ensure all of the verifications were within acceptable range.

B. Review any "Corrective Action Reports" (Appendix A.7)

C. Assign any necessary post weigh voids/flags. Document on "Filter Cassette Transport Sheets"

D. Review outliers: Create line, or bar graph for all samples organized by site. An outlier may be abnormally high or low in comparison to the rest of the network. If identified, further review of the data, and discussion with Air Monitoring Program Manager to evaluate the weight of evidence to determine if sample is valid. See Figure 8.3 below for sample graph. Graph is printed.

E. Enter into AQS any of the post weigh Void codes and flags added.

F. Print the AQS report AMP350 Raw Data report. Check that each sample date has an associated value or void code. Check that the void codes, flags, and sample values match the IML report and the Filter Cassette Transport Sheets. Enter all void filters into the QA Database Void Filter table, note

G. Bundle the PM2.5 Data Validation Check List, AMP350, Outlier graph, Printed Weigh Report, and Filter Cassette Transport sheets, by sampler and quarter to be reviewed by the Lab Manager during final data certification.

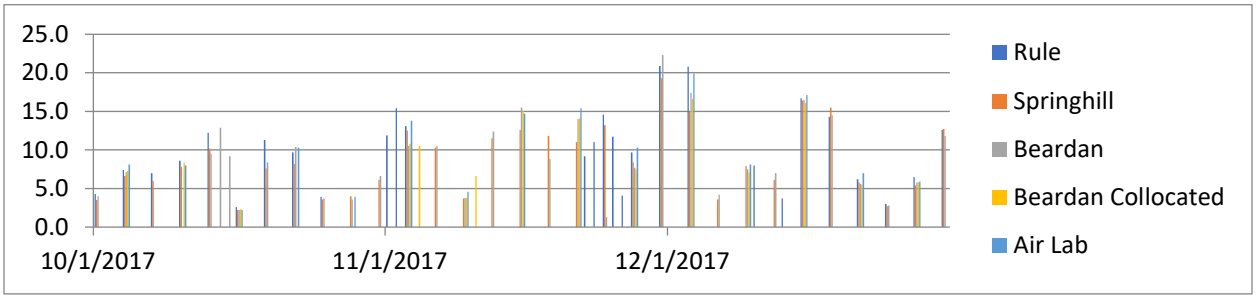


Figure 8.2 Outlier Graph

8.3 Entering Data into AQS

AQS is the data management system maintained by the US Environmental Protection Agency (EPA). It is assumed in these instructions that the user has a user name, required access, and basic training provided by DEP. AQS is accessed through the internet. The website name may change. ENSC and AQS are currently accessed through https://www.epa.gov/aqs

8.3.1 Uploading using ENSC

ENSC is an exchange server that uploads bulk data in to AQS. The ENSC exchange server requires a username and password for access and additional requires AQS user name and credentials for uploading data.

A. Select Exchange Network Services and choose Send Information

B. Select AQS submit

C. Complete the Express Request form for AQS submit as follows:

   a. Select the File to upload
   b. Enter email address
   c. Enter you AQS user ID
   d. Select Knox, TN as the Screening Group
   e. File type: Flat (typical with IML but verify)
   f. Final processing step: Post
   g. Stop on Error: No

D. Complete the next page as needed for additional email address to notify (Lab Manager) and notes on what data in included in the submittal.

Click Send Data

8.3.2 Entering QA into AQS

The QA for PM2.5 is recorded in the sampler logbooks. The Operator shall bring in the logbooks upon completion of a QA assessment for the QA personnel to review and enter in to AQS.

A. Enter the QA results into AQS under

a. Select
b. Maintain
c. QA Assessments
d. Flow Rate Verifications or Semi-Annual Flow Rate Audits

B. Enter Data as follows for Flow Rate Verifications

- State Code: 47
- County Code: 093
- Site ID: 1017, 0028, 1020, 1013
- Parameter Code: 88101
- POC: 1 for official monitors, 2 for collocated monitors
- Performing Agency: 0581 for Knox County Air Quality Management, 1025 for TDEC
- Assess. Date: Date in format YYYYMMDD
- Assess. Number: 1 unless assessment was repeated, then 2, 3 etc.
- Method Code: 145
- Unit: 118 for Liters/min. May change depending on device used
- Assessment Flow Rate: Enter Flow rate provided by Streamline Pro or Transfer Standard
- Monitor Flow Rate: Enter value reported by the monitor.

C. It is recommended that you save after each line entered. AQS can have connectivity issues and frequent saves reduces the data lost.

D. After completing AQS entry, the log books shall be returned to the operator for placement back in the field.

The QA personnel shall make every effort to complete this with in one day of logbooks presented. If delay necessary, return the logbooks to the field and retrieve them when data entry is possible.

9.0 Documentation and Records Management

Recordkeeping is a critical part of the QA program. Careful documentation of sampling data will salvage samples that may otherwise be lost. The sheer repetition of recording data may result in errors. This section is to highlight specific documentation that must be completed. This shall supplement any documentation elements described in previous sections.

9.1 Documentation

There are many levels of documentation in PM2.5 sampling. Additions to this list may occur. It is easiest said to document everything.

9.1.1 Sample Setup

The operator shall completely fill out and maintain the Filter Cassette Transport Sheets for each sampler organized by month. The operator shall provide the Filter Cassette Transport Sheets to the QA personnel at the close of each quarter.

9.1.2 Sample Retrieval

A. Onsite: The operator shall note in the logbook and on the Filter Cassette transport sheets time and date of sample retrieval. They shall also note any stack codes or warnings provided by the sampler.

B. In Lab: The operator shall record the refrigerator temperature in the logbook and note on the Filter Cassette Transport Sheets for filters in the fridge if there was an excursion over 4°C.

9.1.3 Chain of Custody

The operator shall fill out the chain of custody record provided by IML (appendix A.5) and include in the remarks if Field Blank, or any additional notes. Additionally, the operator shall save a copy of the chain of custody L:\Air Lab Data\Ambient Air Monitoring\Pollutants\PM 25 Monitoring\PM 25 filters, Chain of Custody

9.1.4 QA Documentation

All QA decisions shall be thoroughly documented on the “Filter Cassette Transport Sheet” (Appendix A.4) including any calculations, comparisons, or special circumstances (staple additional documentation if necessary to the “Filter Cassette Transport Sheet”). Each decision should be obvious to external personnel reviewing the decision. Additionally, the QC checks (verifications), audits and void data are all entered into the Access file- AQM QA database by the Quality Assurance Officer. The void data log shall also include comments if void requires further explanation.

9.2 Records Management

Records Management is the professional practice of managing the records of an organization throughout their life cycle, from the time they are created to their eventual disposal. This includes identifying, classifying, storing, securing, retrieving, tracking and destroying or permanently preserving records.

9.2.1 Document Integrity

The majority of the documents, used in the PM2.5 program, are hand entered logs. Each operator shall be responsible for obtaining and maintaining the appropriate field and lab logbooks. These logbooks will be bound, uniquely numbered and associated with a specific monitor or operation. Document integrity of handwritten logbooks is maintained by the following procedures.

- Error in recording data: one-line strike thru on incorrect data, initial and date change. Fill in correct data.
- Skipped lines, or pages: X'ed out to ensure backfilling does not occur.

The laboratory provides reports in excel format. A few documents are created in MS Excel. This program performs the calculations and graphs the results. All MS Excel reports shall be checked annually, by the QA Officer or Air Monitoring Manager, as part of the records management program for accuracy of formulas and applicability to procedures. The master forms are in read-only format to help insure there are no alterations to the approved formulas and format during the year.

9.2.2 Records Retention

IML maintains the filters in cool storage for one year. They then ship the filters back to KCDAQM and we store for an additional 4 years. Filters are maintained for a total of 5 years. Upon completion of the data validation from section 8.2.3 the QA documents and Filter Cassette Transport sheets are bundled by quarter. These bundles are stored in a filing cabinet until the conclusion of the sampling year. After the year is complete, and all sampling and QA/QC data are entered and validated in AQS, the quarterly bundles are combined with any other logbooks and documentation for the year and placed in a permafile box marked with the year and pollutant. The box is stored in the data room. All PM2.5 files are maintained at least 5 years. Refer to the KCDAQM Records Retention Policy Appendix B of the KCDAQM QAPP for full records schedule.

9.2.3 Digital Records

KCDAQM continues to increase their ability to digitally store and track data. The sampler data is stored on the shared drive, and on a flash drive. The data stored on the shared drive location L:\Air Lab Data\Ambient Air Monitoring\Pollutants\PM 25 Monitoring are the true documents. The additional saved sampler data on the flash drive, serves as a secondary firewall to loss of data of the main system. All computing systems that store PM2.5 data are on a regular back up schedule. Additionally, the sampler data is stored thru email servers when transmitted to the laboratory pre-weighing. Digital copies of logbooks, certification documentation, and other documents serve as additional back up but shall not replace the physical retention of files.

10.0 Data Completeness

KCDAQM is required to have at least 75% data completeness annually. In the event KCDAQM, may not meet this requirement the Tennessee Division of Air Pollution Control and U.S. EPA- Region 4 APTMD will be contacted as soon as possible to assist KCDAQM.

Appendix A Forms

A.1 Calibration Form / Sticky

Technician	Date / Time	Site Name / Site ID / Sampler ID
		Rule
		47-093-1017-01
		2025B26451005

Ambient Temperature (°C)		
Current	Actual	Offset

Compartment Temperature (°C)		
Current	Actual	Offset

Filter Temperature (°C)		
Current	Actual	Offset

Barometric Pressure		
Current	Actual	Offset

External Leak Check Passed?	
Yes	No

Post Calibration Verification				
	System	Refrence	Diff.	Criteria
Temp. Amb.				±2°C
Temp. Filt.				±2°C
Bar. Press.				±10mmHg
Flow: 16.67				±2%
Flow: 15.0				±2%
Flow: 18.4				±2%

16.67 L/min	Current	Actual	Offset	Span

15.0 L/min	Current	Actual	Offset	Span

18.4 L/min	Current	Actual	Offset	Span

Std. ID:	Std. Type:	Cert. Date:

Rev. 1 PM2.5 2025 Calibration sheet sticker 4/4/2014

A.2 Monthly Maintenance Sticky

Site Name	Air Lab
Site ID	47-093-1013-02
Operator	

Month	
Year	2016
Sampler ID	2025B225760909

Operator Instructions: Unless otherwise noted, times are EST.

At Sample Unload: Record and review sample run data; inspect/drain inlet water jar and clean interior of sampler.

Every 10 Runs: Install Field Blank.

Monthly: Clean PM-10 inlet, first stage inlet and VSCC assembly.
Perform single point Temp., Pressure, and Flow QC check plus an internal and external leak check
Check clock time (<+/-1min).
Date / / Actual : : Unit : : Δ : :

Quarterly: Inspect "V" Seals and O-Rings, and clean down t Due Date:_____

Semi-annually: Exchange partical trap filter, test/replace batteries and clean/replace air intake filters. Due Date:_____

Annually: Perform Temp., Pressure and Flow calibration. Due Date:_____
Certified QC reference standards Temp. Std. / / (cert. date) Pressure Std. / / (cert. date) Flow Std. / / (cert. date)

Other: Rebuild piston pumps every 18 months Due Date:_____

Maintenance

Maintenance	Operator														
	Date														
	Field Blank Installed (x)														
	Monthly Maintenance (x)														
	Quart. Maintenacne (x)														
	Semi-Annual Maint. (x)														
	Annual Maintenacne (x)														

Sampler Temperature, Pressure and Flow Rate Check

Date performed:	Temperature		Pressure	Flow	Leak Check	
/ /	Ambient	Filter	Pressure Standard	Flow Rate Standard	Internal Leakcheck	External Leakcheck
System:						
Reference:					mmHg	mmHg
Difference:					Pass	Pass
Standard Name/Type:						
Standard ID:					Fail	Fail
Acceptance Criteria:	<+/- 2 ºC		≤+/-10 mmHg	≤+/- 4%	Pass/Fail	Pass/Fail

A.3 Packing List

KCAQM
9.8.16
2:00 est
BJW

PACKING LIST

Inter-Mountain Laboratories, Inc.
Air Science
555 Absaraka
Sheridan, WY 82801
(307) 674-7506

Ship To:
Amber Talgo
Knox County
140 Dameron Ave.
Knoxville, TN 37917

UPS 2nd Day
RS Standard Overnight
16 lbs
15 X 11 X 13
6 Ice Packs

Send Empty rings

Ship Date: 2-Sep-16

Quantity	Description
54	$PM_{2.5}$ Teflon Membrane Filters in cassettes and Anti-Static Bags ID# T6641264,T6641265,T6641266,T6641267,T6641268,T6641269,T6641270,T6641271,T6641272,T6641273,T6641274,T6641275,T6641277,T6641278,T6641279,T6641281,T6641282,T6641283,T6641285,T6641286,T6641287,T6641288,T6641289,T6641290,T6641291,T6641293,T6641294,T6641295,T6641296,T6641297,T6641298,T6641299,T6641300,T6641301,T6641303,T6641305,T6641306,T6641307,T6641308,T6641309,T6641310,T6641311,T6641312,T6641313,T6641314,T6641315,T6641316,T6641317,T6641318,T6641319,T6641320,T6641321,T6641322,T6641323

A.4 PM2.5 Filter Cassette Transport Sheet

$PM_{2.5}$ Filter Cassette Transport Sheet

Filter ID	Cass. ID	Expiration Date	T B	F B	S	Unit ID	Mag. In	Set-up Date	Tech	Run Date	Removed Date	Tech	Mag. Out	Shipping Date	Tech	Data Sent	Comments
				1		22654				9/15/16							
					2					9/15/16							
					3					9/16/16							
					4					9/17/16							
					5					9/18/16							
					6					9/19/16							
					7					9/20/16							
					8					9/21/16							
				1		22654				9/22/16							
					2					9/22/16							
					3					9/23/16							
					4					9/24/16							
					5					9/25/16							
					6					9/26/16							
					7					9/27/16							
					8					9/28/16							
				1		22654				9/29/16							
					2					9/29/16							
					3					9/30/16							
					4					10/1/16							
					5					10/2/16							
					6					10/3/16							
					7					10/4/16							
					8					10/5/16							

September-16

Reviewed by: / /

Site: R-95

A.5 IML Chain of Custody

Inter-Mountain Labs
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.

Page of
#WEB

Client Name	Project Identification	Sampler (Signature/Attestation of Authenticity)	Telephone #
Knox County Air Quality Management			865-215-5943 /865-215-5942

Report Address	Contact Name
aetalgo@aqm.co.knox.tn.us bjwhite@aqm.co.knox.tn.us	Barron White Email bjwhite@aqm.co.knox.tn.us Phone
Invoice Address	**Purchase Order #** / **Quote #**
140 Dameron Ave. Knoxville, TN 37917	

ITEM	LAB ID (Lab Use Only)	DATE SAMPLED	TIME	SAMPLE IDENTIFICATION	Matrix	# of Containers	ANALYSES / PARAMETERS: PM 2.5 Gravimetric	REMARKS
1					FT	1	X	
2					FT	1	X	
3					FT	1	X	
4					FT	1	X	
5					FT	1	X	
6					FT	1	X	
7					FT	1	X	
8					FT	1	X	
9					FT	1	X	
10					FT	1	X	
11					FT	1	X	
12					FT	1	X	
13					FT	1	X	
14					FT	1	X	

LAB COMMENTS	Relinquished By (Signature/Printed)	DATE	TIME	Received By (Signature/Printed)	DATE	TIME

SHIPPING INFO	MATRIX CODES		TURN AROUND TIMES	COMPLIANCE INFORMATION	ADDITIONAL REMARKS
☒ UPS	Water	WT	Check desired service	Compliance Monitoring ? N	
☐ FedEx	Soil	SL	☒ Standard turnaround	Program (SDWA, NPDES,…)	
☐ USPS	Solid	SD	☐ RUSH - 5 Working Days	PWSID / Permit #	
☐ Hand Carried	Filter	FT	☐ URGENT - < 2 Working Days	Chlorinated? Y / N	
☐ Other	Other	OT	*Rush & Urgent Surcharges will be applied*	Sample Disposal: Lab Client X	

Inter-Mountain Labs, Inc. www.intermountainlabs.com Rev 4.6 Web

A.6 Field Sheet PM2.5 (Audits)

Knox County Health Department
Every Person, A Healthy Person

KNOX COUNTY AIR QUALITY MANAGMENT
QUARTERLY AUDIT FIELD SHEET

PM2.5	**Please circle Reference device used for Audit**		
DeltaCal	Tri Cal	Tetra Cal	Streamline

Serial Number: _____ Calibration Date: _____

Site Name: Date:

Sampler ID:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 1 Min.
Filter Temperature				+/- 2° C
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow (16.67 l/min) [(actual-indicated) / actual] * 100				+/- 4%

Site Name: Date:

Sampler ID:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 1 Min.
Filter Temperature				+/- 2° C
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow (16.67 l/min) [(actual-indicated) / actual] * 100				+/- 4%

Site Name: Date:

Sampler ID:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 1 Min.
Filter Temperature				+/- 2° C
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow (16.67 l/min) [(actual-indicated) / actual] * 100				+/- 4%

Site Name: Date:

Sampler ID:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 1 Min.
Filter Temperature				+/- 2° C
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow (16.67 l/min) [(actual-indicated) / actual] * 100				+/- 4%

Site Name: Date:

Sampler ID:	System	Reference	Difference	Acceptance Criteria
Time (in EST)				+/- 1 Min.
Filter Temperature				+/- 2° C
Ambient Temperature				+/- 2° C
Barometric Pressure				+/- 10 mmHg
Sample Flow (16.67 l/min) [(actual-indicated) / actual] * 100				+/- 4%

A.7 Corrective Action Report

Knox County Health Department
Every Person, A Healthy Person

Serial Number: _____

Date: _____

Page: _____ of _____

Corrective Action Report

Issue: *Describe the issue found, what was known preceding discovery, what is believed to have occurred etc. If possible, note last passing verification date. Include make, serial number, pollutant for instrument in question and as specific as possible dates and times. If failed verification be sure to report serial number of standard used for verification.*

Action taken: *Describe all actions performed such as calibrations, maintenance, repair. Include as specific as possible dates and times.*

Post action QC performed: *Note date time and results of verifications post actions. Be sure to note the serial number of verification equipment used.*

Signature

Appendix G – IML QAPP

IML QAPP link : or - https://knoxcountymail.sharepoint.com/:b:/s/AirQualityLab2/EVKZC6TdiXlLkU4YnK-wAvAB6dQEO_Jo_rnt2xoB3xOvtA?e=QEi4Z5

Appendix H – Support for the EPA National Contract for Lead Analysis

SUPPORT FOR THE EPA NATIONAL CONTRACT FOR LEAD ANALYSIS

Contract No. EP-BPA-15-D-0004

2015

Quality Assurance Project Plan
Category 1

Eastern Research Group, Inc.
601 Keystone Park Drive, Suite 700
Morrisville, NC 27560

2015 Quality Assurance Project Plan, Category 1
National Contract for Lead Analysis (Contract No. EP-BPA-15-D-0004)

Approved by:

U.S. EPA Project Officer: _____ Date: 12/10/15

U.S. EPA QA Manager: _____ Date: 12/10/15

U.S. EPA Delivery Order Manager: _____ Date: 12/10/15

ERG Program Manager: _____ Date: 12/21/15

ERG Program QA Officer: _____ Date: 12/21/15

DRI Project Manager: _____ Date: 12/15/15

DRI Project QA Officer: _____ Date: 12/15/2015

DISCLAIMER

This Category 1 Quality Assurance Project Plan has been prepared specifically to address the operation and management of the U.S. EPA National Contract for Lead Analysis for the National Ambient Air Quality Standards (NAAQS). The contents have been prepared in accordance with Level 1 Specifications of the EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5.

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A ERG Standard Operating Procedures

ERG-MOR-017	Standard Operating Procedure for Developing, Documenting, and Evaluating the Accuracy of Spreadsheet Data
ERG-MOR-022	Standard Operating Procedure for Preparation of Standards in the ERG Laboratory
ERG-MOR-033	Standard Operating Procedure for Hazardous Waste
ERG-MOR-045	Standard Operating Procedure for Sample Receipt at ERG Chemistry Laboratory
ERG-MOR-057	Standard Operating Procedures for Project Peer Review
ERG-MOR-079	Standard Operating Procedure for Sample Login to the Laboratory Information Management System

ERG-MOR-084	Standard Operating Procedure for the Preparation and Analysis of High Volume Quartz Filters for Metals by ICP-MS using Method IO 3.5 and FEM Method EQL-0512-201
ERG-MOR-085	Standard Operating Procedure for the Preparation and Analysis of 47mm Filters for Metals by ICP-MS using Method IO 3.5 and FEM Method EQL-0512-202
ERG-MOR-098	Standard Operating Procedure for the Preparation of Monitoring Data for AQS Upload
ERG-MOR-099	Standard Operating Procedures for the Laboratory Information Management System

B DRI Standard Operating Procedures

DRI SOP #2-209.8	DRI Standard Operating Procedure for X-Ray Fluorescence (XRF) Analysis of Aerosol Filter Samples (PANalytical Epsilon 5)
DRI SOP #4-1117r1	DRI Standard Operating Procedure for General EAF Internal Audit Procedures
DRI SOP #6-0015r1	DRI Standard Operating Procedure for Demonstration of Capability
DRI SOP #6-017r0	DRI Standard Operating Procedure for Corrective Action

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SYMBOLS AND ABBREVIATIONS

AMTIC	Ambient Air Monitoring Technical Information Center
AQS	Air Quality Subsystem
BPA	Blank Purchase Agreement
BS/BSD	Blank Spike/Bland Spike Duplicate
CAR	Corrective Action Report
CCB	Continuing calibration blank
CCV	Continuing calibration verification
COC	Chain of Custody
CSN	Chemical Speciation Network
CV	Coefficient of Variation, precision
DOC	Demonstration of Capability
DPR	Daily Performance Report
DQOs	Data Quality Objective(s)
DRI	Desert Research Institute
DUP	Duplicate (used for Replicate Analysis)
EAF	Environmental Analysis Facility
EPA	U.S. Environmental Protection Agency
ERG	Eastern Research Group, Inc.
EDXRF	Energy Dispersive X-ray Fluorescence
FACA	Federal Advisory Committee Act
FEM	Federal Equivalency Method
FRM	Federal Reference Method
HSV	High standard verification
IC	Initial Calibration Standards (ICP-MS)
ICB	Initial Calibration Blank
ICP-MS	Inductively Coupled Plasma/Mass Spectrometer
ICSA	Interference Check Standard A
ICSAB	Interference Check Standard B
ICV	Initial calibration verification
ISTD	Internal Standard
LCV	Low Calibration Verification
LIMS	Laboratory Information Management System
LOQ	Limit of Quantitation
LQL	Lower Quantifiable Limit

LRB	Laboratory Reagent Blank
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MB	Method Blank
MDLs	Method Detection Limit(s)
mm	Millimeter
MQOs	Measurement Quality Objective
µg/L	Micrograms per liter
$\mu g/m^3$	Microgram per cubic meter
NAAQS	National Ambient Air Quality Standard
NAREL	National Air and Radiation Environmental Laboratory
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
ng/L	Nanogram per liter
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
OSHA	Occupational Safety and Health Administration
Pb-TSP	Lead for Total Suspended Particles
Pb-PM_{10}	Lead for PM_{10}
PDS	Post digestion spike
PE	Performance Evaluation
POC	Parameter Occurrence Code
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RB	Raw Blank
RD	Raw Data
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SOPs	Standard Operating Procedure(s)
SRD	Serial dilution
SRM	Standard Reference Material
TCEQ	Texas Commission on Environmental Quality
TSAs	Technical System Audits
TSP	Total Suspended Particles
XRF	X-ray Fluorescence

DISTRIBUTION LIST

Copies of this plan and all revisions will be provided to:

- Jeff Yane, Work Assignment Manager, U.S. EPA, C404-02, RTP, NC
- Laurie Trinca, Project Manager, U.S. EPA, C304-06, RTP, NC
- Greg Noah, AT QA Coordinator, U.S. EPA, C304-06, RTP, NC

U.S. EPA Regional contacts may obtain a copy of the QAPP by contacting the ERG Program Manager. It is the responsibility of each Regional contact to make copies of the plan for appropriate State personnel or to refer them to ERG Program Manager.

PROJECT MANAGEMENT
SECTION 1
PROJECT/TASK ORGANIZATION

1.1 Assignment of Program Personnel

Table 1-1 presents the program organization for each aspect of the Environmental Protection Agency (EPA) Analysis for Lead in TSP and PM_{10} Filters. The program organizational chart is presented in Figure 1-1. All Eastern Research Group (ERG) and Desert Research Institute (DRI) staff working on this contract are provided access to a current electronic copy of this signed, EPA approved Quality Assurance Project Plan (QAPP).

ERG's primary support on this contract includes analysis for lead for Total Suspended Particles (Pb-TSP) or PM_{10} (Pb-PM_{10}) by Inductively Coupled Plasma/Mass Spectrometer (ICP-MS). DRI's primary support includes analysis for Pb-PM_{10} filters by X-ray Fluorescence (XRF) analysis.

ERG is responsible to the client for the work of the subcontractor, DRI. The subcontractor will meet the Data Quality Objectives (DQOs) requirements for the appropriate method. ERG shall maintain a record of subcontractor compliance, including documentation of subcontractor's Method Detection Limits (MDLs).

1.1.1 Program Manager

Ms. Julie Swift, an ERG Vice President, is the Program Manager for this contract and will serve as the primary contact. In this role, she has the primary responsibility for understanding EPA's and their clients' (i.e., State, local, and tribal agencies) needs at the program level. Ms. Swift coordinates with the ERG Quality Assurance (QA) Officer and metal's Task Lead to provide EPA client perspective and communicate technical issues and needs. As the Program Manager, Ms. Julie Swift is responsible for the technical operation and the quality

of the program on a day-to-day basis. She leads the analytical tasks and provides technical direction and support. She assists in the resolution of technical issues and serves as a resource for the Task Lead regarding any project issues. Ms. Swift also performs an overall review of the data that is reported.

1.1.2 Program QA Coordinator

Ms. Donna Tedder, the Program and Laboratory QA Coordinator, is responsible for ensuring the overall integrity and quality of project results. Ms. Tedder, or her designee, will do a 10 percent QA review for all sample analyses delivered for reporting to the Program Manager. In the case of subcontracted work, 20 percent of data from subcontractor will be reviewed. The lines of communication between management, the Program QA Coordinator, and the technical staff are formally established and allow for discussion of real and potential problems, preventive actions, and corrective procedures. On major quality issues, Ms. Tedder reports independently to Ms. Mary Willett, ERG's corporate QA Officer.

1.1.3 Task Leaders

Ms. Jennifer Nash, ERG's Metals Task Lead, is responsible for meeting the project objectives, meeting report schedules, and directing the technical staff in execution of the technical effort for their respective task(s). She will review 100 percent of all sample analyses and will deliver 10 percent of sample analyses that the QA Coordinator requests for review prior to data reporting by the Program Manager. Ms. Nash will assess and report on the project's progress and results (e.g., recordkeeping, data validation procedures, sample turnaround time) and ensure timely, high-quality services that meet the requirements in this QAPP.

ERG will subcontract the analysis for the XRF analysis of PM_{10} filters to DRI. Dr. Richard Tropp will serve as DRI's Principal Investigator and Project Manager while Steve Kohl, from DRI, will be the DRI Task Leader responsible for leading the XRF analysis of PM_{10} filters. Dr. Tropp will serve as primary contact at DRI, with Mr. Kohl as secondary. Dr. Tropp

will also be responsible for overseeing the project and Quality Assurance/Quality Control (QA/QC) of the Energy Dispersive X-ray Fluorescence (EDXRF) data and its reporting to ERG. Mr. Kohl will be responsible for overseeing the receipt, EDXRF analysis, data processing, data validation and reporting for the Teflon PM_{10} samples.

Table 1-1
Program Organization

Program Assignment	Program Personnel Assigned	Phone Number	Email Address
ERG Program Manager	Julie Swift	(919) 468-7924	julie.swift@erg.com
Task Lead – ERG ICP-MS Analysis	Jennifer Nash	(919) 468-7881	jennifer.nash@erg.com
DRI Project Manager	Richard Tropp	(775) 674-7094	Richard.Tropp@dri.edu
Task Lead – DRI XRF Analysis	Steve Kohl	(775) 674-7056	Steve.Kohl@dri.edu
Program QA Coordinator	Donna Tedder	(919) 468-7921	donna.tedder@erg.com
Project Administrator	Kerry Fountain	(919) 468-7962	kerry.fountain@erg.com

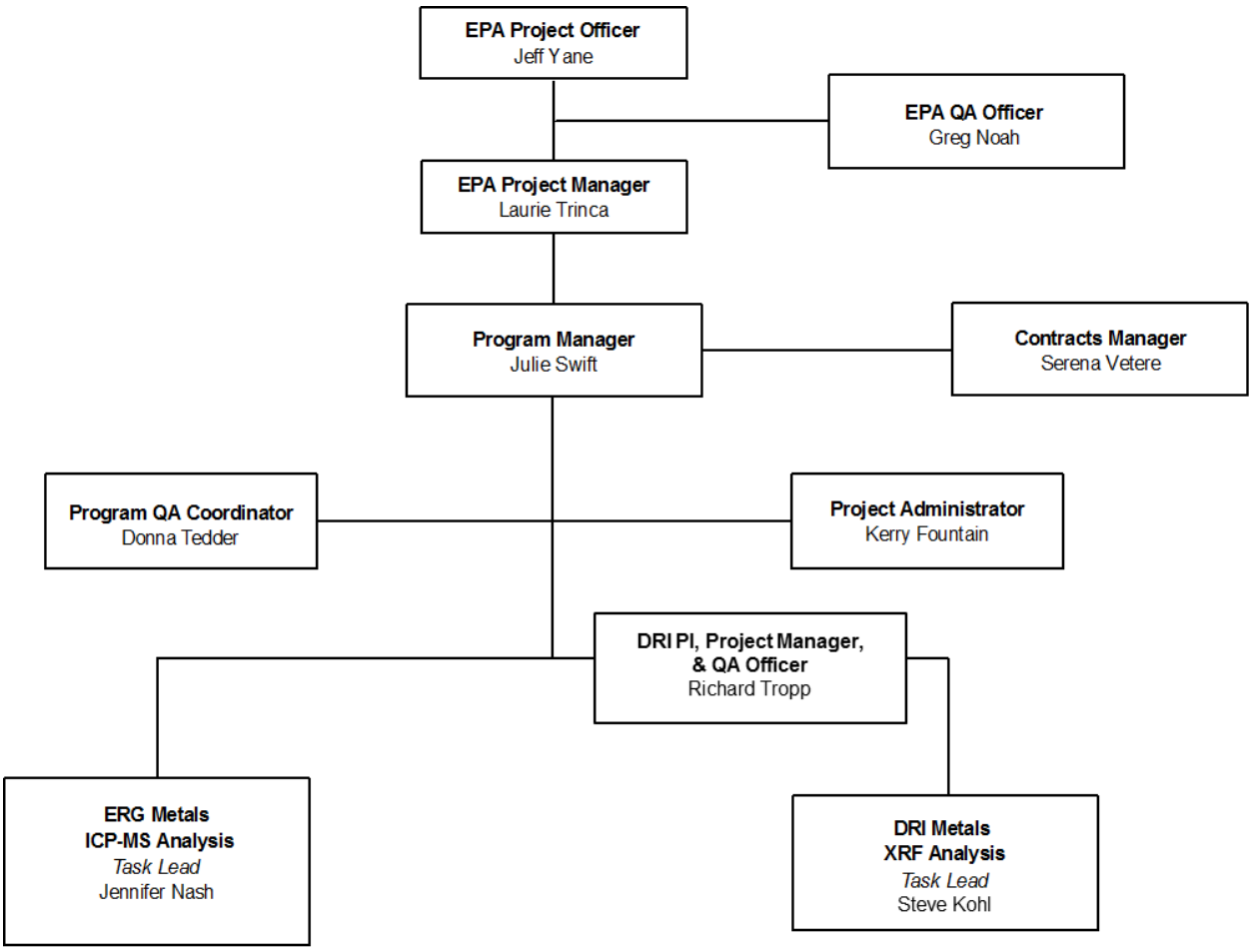

Figure 1-1. Lead Analysis Organizational Chart

SECTION 2
PROBLEM DEFINITION/BACKGROUND

On November 12, 2008, the EPA issued a rule strengthening the primary and secondary National Ambient Air Quality Standards (NAAQS) for lead and associated monitoring requirements (Federal Register Volume 73, Number 219, (73 FR 66964) [(1)] to provide necessary protection for public health and welfare. The EPA revised various elements of the primary standard to provide increased protection for children and at-risk populations against adverse health effects, most notably including neurological effects in children. EPA revised the primary (health-based) standard from 1.5 micrograms per cubic meter ($\mu g/m^3$) to 0.15 $\mu g/m^3$, as total suspended particles (TSP). EPA is revising the secondary (welfare-based) standard to be identical in all respects to the revised primary standard.

The EPA also promulgated a new Federal Reference Method (FRM) for the sampling and analysis of Pb-PM_{10} as Code of Federal Regulations Title 40 Part 50 (40 CFR Part 50), Appendix Q[(2)]. The averaging time was revised to a rolling 3-month period with a maximum (not-to-be-exceeded), evaluated over a 3-year period.

EPA revised the data handling procedures, including allowance for the use of Pb-PM_{10} data in certain circumstances, and the treatment of exceptional events, and ambient air monitoring and reporting requirements for Pb, including those related to sampling and analysis methods, network design, sampling schedule, and data reporting. Depending on specific circumstances outlined in the rule, States have the option of monitoring for Pb-TSP or Pb-PM_{10} following approved FRMs or Federal Equivalent Methods (FEMs) to meet the monitoring requirements.

SECTION 3
PROJECT/TASK DESCRIPTION

This section describes the activities performed under the national contract for the analysis of Pb-TSP and Pb-PM_{10} for state, local, and tribal monitoring agencies. Sampling and analysis schedules are prepared when a Blank Purchase Agreement (BPA) is provided by EPA.

3.1 NAAQS for Lead Analysis

The NAAQS national contract for the analysis of Pb-TSP and Pb-PM_{10} was created so that the State, Local and Tribal monitoring agencies could access a laboratory to provide their analysis following EPA approved FRM/FEM analysis specifications. The filters are supplied by the state/local agencies for this program. The EPA provides the agencies, through their Regional Coordinators, access to this contract to provide the analysis. A list of the analyses, EPA FRM/FEM, and laboratory Standard Operating Procedures (SOP) are listed in Table 3-1. ERG can prepare the data in the Air Quality Subsystem (AQS) database format for quarterly upload if requested.

Table 3-1
List of Analytical Services

Analysis	Method	SOP
ICP-MS • Pb-TSP • Pb-PM_{10}	 EQL-0512-201[3] EQL-0512-202[4]	 ERG-MOR-084 ERG-MOR-085
XRF • Pb-PM_{10}	40 CFR Part 50, Appendix Q[2]	DRI SOP #2-209

SECTION 4
DATA QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

Because ERG performs analysis services only, DQOs for defining a network program are not identified in this QAPP. This section will discuss the Measurements Quality Objectives (MQOs) of ERG and DRI laboratory analyses, emphasizing the levels of uncertainty the decision maker is willing to allow/accept from the analytical results.

Once a sampling DQO is established, the quality of the data must be evaluated and controlled to ensure that data quality is maintained within the established acceptance criteria. MQOs are designed to evaluate and control various phases (sampling, preparation, analysis) of the measurement process to ensure that the total measurement uncertainty is within the range prescribed by the DQOs. MQOs can be defined in terms of the following data quality indicators:

Precision - a measure of mutual agreement among individual measurements of the same property usually under prescribed similar conditions. This is the random component of error.

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

Detectability - the determination of the low range critical value of a characteristic that a method-specific procedure can reliably discern.

Comparability - a measure of the level of confidence with which one data set can be compared to another.

Bias has been the term frequently used to represent closeness to “truth” and includes a combination of precision and bias error components. The MQOs listed will attempt to separate measurement uncertainties into precision and bias components. Bias will be assessed in the quarterly EPA audits which will be reported into AQS.

Analytical Precision is calculated by comparing the differences between replicate analyses (two analyses of the same sample) from the arithmetic mean of the two results as shown below. Replicate analyses with low variability have a lower Relative Percent Difference (RPD) (better precision), whereas high variability samples have a higher RPD (poorer precision).

$$RPD = \frac{|X_1 - X_2|}{\bar{X}} \, x \, 100$$

Where:

X_1 = Ambient air concentration of a given compound measured in one sample;

X_2 = Concentration of the same compound measured during replicate analysis;

$\overline{X}$ = Arithmetic mean of X_1 and X_2.

Table 4-1 lists the MQOs for Pb-TSP and Pb-PM_{10} using ICP-MS and XRF.

Table 4-1

Measurement Quality Objectives for the National Lead Analysis Program

Analyte/ Instrument	Reporting Units	Precision from analysis of Replicate Samples (RPD)*	Precision from collection of Collocate Samples*	Comparability/ Based on Method	Minimum Detection Limit
Pb-TSP ICP-MS	µg/filter	± 10%	± 20%	EQL-0512-201	See Table 11-4
Pb-PM10 ICP-MS	µg/filter	± 10%	± 20%	EQL-0512-202	See Table 11-4
Pb-PM10 XRF	µg/filter	± 10%	± 20%	40 CFR Part 50, Appendix Q	See Table 11-4

* Sample value is ≥ 10 times the MDL

SECTION 5
SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

The activities of EPA's National Contract for Lead Analysis is performed using accepted EPA, National Institute for Occupational Safety and Health (NIOSH), and Occupational Safety and Health Administration (OSHA) analytical protocols for the analytical laboratory staff.

5.1 ERG Analytical Laboratory Personnel

ERG analytical laboratory personnel involved in this project have been trained in their tasks and have up to 28 years of experience in the duties they will be performing in the analytical laboratory. Training of ERG laboratory personnel is recorded in ERG Training Records in an Excel® database and filed as a hardcopy. Technical training includes general techniques and specific training based on the appropriate SOP, method, and program QAPP. After training, an initial demonstration of each personnel's ability to perform the analytical task involves repeated measurements of a standard. These records are kept by the ERG QA staff. Currently, no special certifications are needed for the analysis of the ambient samples received for this programs. Health and Safety training is performed annually. The laboratory personnel will adhere to the ERG Corporate Health and Safety manual.

ERG maintains appropriate SOPs for each analytical method. These SOPs are presented in Appendix A. All SOPs document equipment and/or procedures required to perform each specific laboratory activity. Laboratory staff will be subject to on-site surveillance by the Quality Assurance staff and quarterly audit samples.

5.2 DRI Analytical Laboratory Personnel

DRI Environmental Analysis Facility (EAF) personnel involved in this project have an education background and up to 35 years of experience in the duties they will be performing at DRI EAF. The DRI Project Manager, Dr. Tropp, has been overseeing sampling and analysis

projects for over 35 years, including the QA/QC of XRF analysis results. DRI's EAF Analysis Task Leader, Mr. Kohl, has been performing EDXRF analysis of PM samples for more than 20 years. All laboratory personnel performing tasks according to EAF SOPs have passed a demonstration of capability (DOC) initially then annually according to *DRI SOP for Demonstration of Capability*, DRI SOP #6-0015r1 in Appendix B. These records are kept by the EAF QA Manager. DRI EAF personnel are subject to periodic internal QA audits by the EAF QA Manager. In addition, DRI EAF personnel will perform periodic PE sample analyses as required. All DRI EAF personnel are required to undergo DRI laboratory safety training annually and adhere to the Institute's health and safety plans.

SECTION 6
DOCUMENTATION AND RECORDS

The EPA National Contract for Lead analysis generates a number of documents and records that need to be retained/archived. The PM will provide all staff working on this contract access to a current electronic copy of this signed, EPA approved QAPP. In this QAPP, ERG's reporting package (defined as the information required to support the data reported) includes all data required to be collected as well as support data deemed important by ERG and/or DRI.

6.1 Data Management

ERG has a structured records management retrieval system that allows for the efficient archive and retrieval of records. The analytical information that is collected by the laboratory will be managed in this system. The laboratory paper copies of all analyses are stored on site in a secured temperature-controlled laboratory area for up to five years after the close of the contract. Data obtained from DRI will be imported into the LIMS data server so that the same reports will be generated for all samples submitted for analysis. ERG also archives the data in the Laboratory Information Management System (LIMS) data server which is backed up weekly, monthly, and biannually. The backed-up data is stored at an off-site ERG facility. Data Storage and Retrieval is discussed in more detail in Section 15.6. The Program Manager has final authority for the storage, access to, and final disposal of all records kept for this program.

6.2 Data Reports

Data reports, sent in Adobe and Excel formats to EPA and appropriate state/local/tribal agencies, will include the sample name, filter ID number, collection date, received date, analysis date, analytical method, analytical data reported in µg/filter, MDL, and qualifiers. This report will be submitted within 30 days from the receipt of the samples. The data reports will include analytical results for individual samples as well as associated QC samples, associated MDLs, and qualifiers.

6.3 Records and Supporting Data

Data used for the measurement of lead and the associated QA/QC data are collected electronically or on data forms. Table 6-1 presents the location of the data records for laboratory operations stored at the ERG and DRI laboratories.

Table 6-1
Data Documentation and Records

Item	Record	Short Term Location Storage	Long Term Location Storage
Field Operations			
Chain of Custody (COC)	ERG COCs	In Laboratory with Samples	Copy scanned and stored on ERG LIMS
Laboratory Records - ERG			
Sample Prep Data	Bench sheets	Hardcopy filed, LIMS	Hardcopy archived, LIMS
Sample Management Records (sample receipt, handling, storage, etc.)	COCs	LIMS, with bench sheets	LIMS, with bench sheets
Test Methods	SOPs	Hardcopy filed, shared network drive	Shared network drive
QA/QC Reports (General QC records, MDL information, calibration, etc.)	Individual records for each analysis	Hardcopy filed, shared network drive	Hardcopy archived, shared network drive
Corrective Action Reports	Individual records for each analysis	Original copy filed, copy in data package	All copies archived
Laboratory Records - DRI			
Sample Management Records (sample receipt, handling, storage, etc.)	COCs	Shipping & Receiving	Hard Copy Archiver
Test Methods	SOPs	Analytical; Lab	LAN & QA Files
QA/QC Reports (General QC records, MDL information, calibration, etc.)	Individual records for each analysis	Analytical Lab	LAN
Corrective Action Reports (CAR)	Individual records for each analysis	Analytical Lab	LAN and EAF QA Manager *
Data Reduction, Verification, and Validation			
Electronic Data (used for AQS data entry if requested)	Excel® and Access®	Shared network drive	Shared network drive

* Archive for formal CARs

6.3.1 Notebooks

ERG issues laboratory notebooks to each laboratory division upon request. This notebook is uniquely numbered and associated with the laboratory personnel. Although LIMS data entry forms are associated with all routine environmental data operations, the notebooks can be used to record additional information about these operations. All notebook entries are filled out in indelible ink. Corrections are made by inserting one line through the incorrect entry, initialing the correction (ERG and DRI maintain a signature log), and placing the correct entry alongside the incorrect entry, if this can be accomplished legibly, or by providing the information on a new line.

Field Notebooks - Field notebooks are the responsibility of the EPA, States, local or tribal agencies as ERG is not responsible for the collection of samples.

Laboratory Notebooks - Notebooks are associated with general procedures such as temperature records for the refrigerators, calibration of analytical balances, sample preparation logs, calibration of analytical instruments, preventive maintenance and repairs, receipt of standards and other supplies, etc., used in this program.

Sample Shipping and Receipt - ERG's LIMS system is used to record samples received. Hard copies of COC records are also stored for one year; however, electronic copies are scanned and stored in LIMS and on a shared network drive.

6.3.2 Electronic Data Collection

In order to reduce the potential for data entry errors, automated systems are utilized (where appropriate) and record the same information that is found on data entry forms. Whenever possible, DRI utilizes barcode-based hardcopy forms, data processing routines to process data from sampling and analytical instruments directly, and standardized data processing routines. Information available from multiple sources (e.g., hardcopy forms, sampler files, and

analytical instruments) are cross-checked for consistency and revised or flagged when questions arise. Instrument and laboratory environmental data are stored on instruments and computers in the laboratories and transferred to a database on the EAF LAN.

6.4 Data Reporting Package Archiving and Retrieval

In general, all the information listed above will be retained for at least 5 years from the date of the end of the closed contract with EPA. If any litigation, claim, negotiation, audit or other action involving the records has been started before the expiration of the 5-year period, however, the records will be retained until completion of the action and resolution of all issues which arise from it. The long-term storage at ERG is located in the laboratory in a locked climate-controlled file room with limited access. The project secretary keeps a record of documents entering and leaving long-term storage. Access to the facility storage area is limited to authorized personnel only.

DRI EAF uses a phased approach for long-term storage of hard-copy records. Recently archived records are located in a hardcopy storage area at DRI with limited access and checkout logs. Older records, generally more than five years old after project completion, may be moved to a climate-controlled offsite storage facility. It also has limited access and checkout logs. For electronic records, DRI EAF laboratory computers house raw data stored on a RAID 1 (Mirror) system. Raw data is automatically backed up to a virtual file and database server, which is run on a physical clustered RAID 1 (Mirror) server, once a day. Once data is on the server it is stored in an instantly accessible, un-modifiable directory for 35 days and an instantly accessible, modifiable directory for 10 days. All data in these locations begin as exact copies of data that was on each individual laboratory computer. After data is safely in those locations, the raw data is extracted from the files and imported to the database server for possible modification. After data has been on the server for 35 days, it is automatically written to tape and stored indefinitely. Daily e-mails are automatically generated to confirm backups and notify computer personnel of data processing and data management issues. All hard drives and tape, once filled, are stored in a special media storage room. The room is secure, accessible only by assigned personnel, with

entry through a security system. The room has no windows, no drop ceilings, and is buried in the side of a hill in the lower section of the DRI building. It also contains UV filters on the lights to prevent damage to media. In addition, there are separate keyed lockers for each DRI laboratory. As part of an Institute-wide disaster recovery plan, data stored on networks at DRI's Reno campus are backed up to DRI's Las Vegas campus and vice versa.

6.5 Quality System Document Control

To ensure the use of the most current version of quality system documents, all quality documents (QAPP, SOPs, etc.) generated at the ERG Laboratory must be uniquely identified. Original documents shall include the date of issue, revision number, page number, total number of pages, and appropriate signatures. Copies of quality documents shall be controlled and include the date of issue, revision number, page number, total number of pages, and copy control number. When an original quality document is updated, the QA Coordinator or designee will ensure that the copy documents are also updated and old versions are disposed. During the course of the project, revised QAPPs will be circulated to the EPA and to ERG's laboratory staff. For copies of documents out of the laboratory's control, a stamp or watermark stating "Uncontrolled" or "Draft", if applicable, will be applied. Each approved QAPP will be posted on EPA's Ambient Air Monitoring Technical Information Centers (AMTIC) Website.

MEASUREMENT DATA ACQUISITION
SECTION 7
SAMPLING PROCESS DESIGN

ERG is not responsible for the collection of samples nor the design of the samplers used in the NAAQS program. Pb-TSP sampling that meets the requirements of Appendix B to Part 50, Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High-Volume Method)[5] is acceptable as a FRM sampler. Low-volume PM_{10} samplers that meet the requirements (as described in Appendix O of Part 50[6]) can be used for Pb-PM_{10} monitoring intended to meet NAAQS comparison objectives. ERG is responsible only for the analysis of the samples it receives from the sites.

SECTION 8
SAMPLING METHOD REQUIREMENTS

Because ERG is not responsible for actual execution of the field sampling in this program, the method support for the site's samplers are not discussed in this QAPP.

SECTION 9
SAMPLE HANDLING AND CUSTODY REQUIREMENTS

ERG's Shipping and Receiving Task Leader will ensure that sample media that is received in the laboratory follow all of the procedures listed in this QAPP and the individual SOPs. The Task Leader will also advise the Project Manager of any issues or obstacles regarding sample receipt, login and storage. The sample custodian working under the Shipping and Receiving Task Leader will receive custody of samples, complete COC receipt information, document sample receipt, and enter COC information into LIMS to create a work order. Samples for XRF analysis will be received and logged in at ERG's laboratory before they are sent to DRI for analysis.

9.1 Analysis Chain of Custody Forms

Field testing personnel will record data on the COC forms (Figure 9-1). The COC form documents time, date, location, and other field parameters. Because the sites supply the filters used for metal analysis, COC forms are generated by the State, local or tribal agency for these samples. If needed, however, the COC forms provided on the AMTIC website at: http://www3.epa.gov/ttn/amtic/pb-monitoring.html. Samples are received at ERG's laboratory as presented in the *SOP for Sample Receipt at ERG Chemistry Laboratory*, ERG-MOR-045, in Appendix A.

The sample specific information from the COC is then entered into the LIMS (example login page is shown in Figure 9-2). The sample is logged into the LIMS database following the *SOP for Sample Login to the Laboratory Information Management System*, ERG-MOR-079 found in Appendix A. The sample is given a unique LIMS identification number.

The LIMS ID number is recorded on all ERG copies of the COC. The COC is scanned (the PDF is stored in the LIMS system) and is kept with the samples until analysis is complete.

Chain of Custody TSP/PM_{10} Lead Analysis (EP-BPA-15-D-0004)

601 Keystone Park Drive, Suite 700, Morrisville, NC 27560

Page of

SITE							Relinquished by:		
AQS Code							Date/Time:		
Submitter:			ANALYSES				Received by:		
							Date/Time:		
#	Filter ID	Date	TSP (ICP	PM10 (ICP	PM10 (XR	Sample Volume (m^3)	Comments	ERG LIMS ID (Lab use only)
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								

Figure 9-1. Metals COC

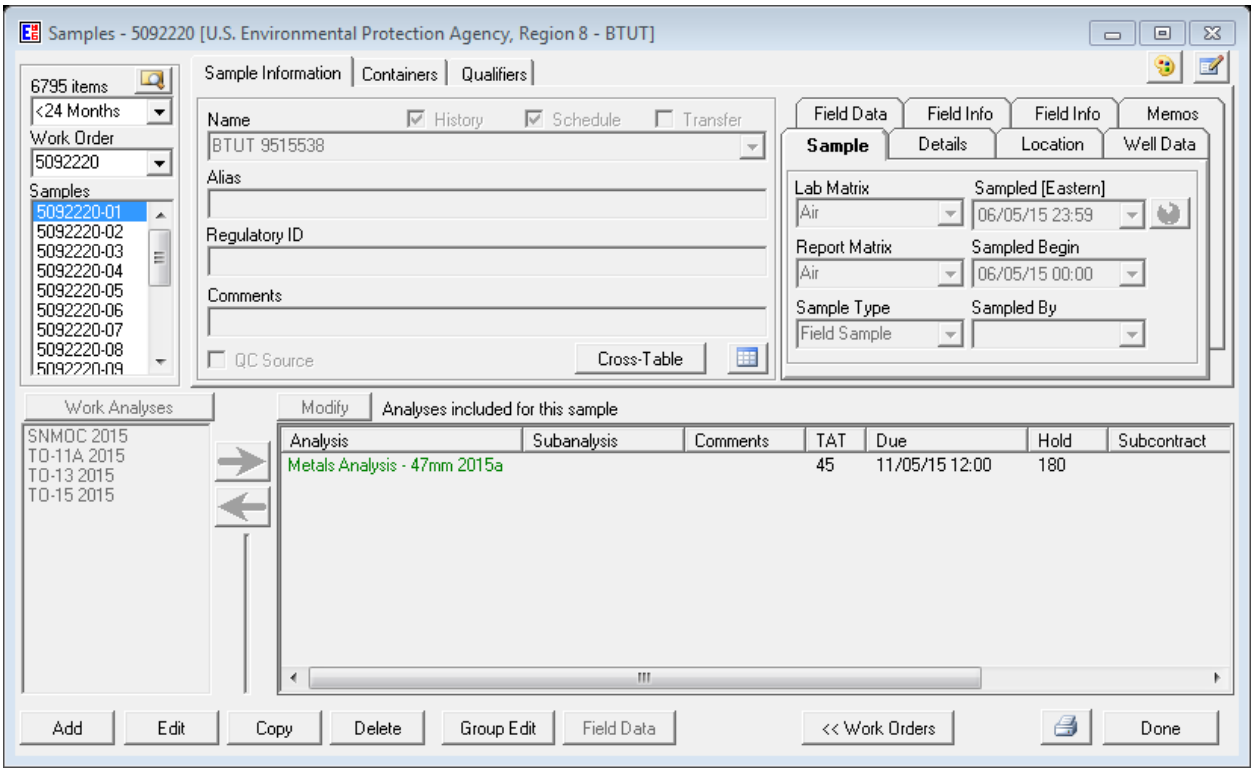

Figure 9-2. Example ERG LIMS Login Page

9.2 Invalid Samples

The sample can be determined invalid at the site or in the laboratory. SOP ERG-MOR-045 describes the sample receiving procedure and sample acceptance. Individual sites will be contacted if there are any questions about the samples upon receipt. When a sample is designated as invalid, the assigned LIMS ID number is notated as a void and the sample is invalidated on the individual respective COC form. The sites will also be notified of any invalid samples in the analytical data reports.

9.3 Analytical Data

All laboratory electronic records will be stored for archive on shared network drive which is backed-up daily, weekly, and monthly. The back-ups are stored off-site for added safety. Raw data will be included in the project archive files stored in an ERG long-term storage location.

DRI EAF laboratory computers house raw data stored on a RAID 1 (Mirror) system. Raw data is automatically backed up to a virtual file and database server, which is run on a physical clustered RAID 1 (Mirror) server once a day. Once data is on the server it is stored in an instantly accessible, un-modifiable directory for 35 days and an instantly accessible, modifiable directory for 10 days. All data in these locations begin as exact copies of data that was on each individual laboratory computer. After data is safely stored in those locations, the raw data is extracted from the files and imported to the database server for possible modification. After data has been on the server for 35 days, it is automatically written to tape and stored indefinitely. Daily e-mails are automatically generated to confirm backups and notify computer personnel of data processing and data management issues. All hard drives and tape, once filled, are stored in a special media storage room.

All records generated are signed or initialed by the person performing the work and reviewed by an appropriate Task Leader. Measurement results become part of a project report, of which 10 percent is chosen and reviewed by the QA Coordinator or a reviewer designated by the QA Coordinator.

9.4 Sampling Monitoring Data

All COC forms from the monitoring sites will be stored with the extraction bench sheet. The COC forms will be reviewed by the Analysts, Task Leaders and Program Manager. The original field data for all the samples, TSP by ICP-MS, PM_{10} by ICP-MS and PM_{10} by XRF will remain in ERG custody and eventually will be stored on file until 5 years after the end of the contract. DRI will receive a copy of the COC forms from the sites with the ERG LIMS ID number added. ERG will contact the individual site if necessary information is not completed on the COC forms.

SECTION 10
ANALYTICAL METHODS REQUIREMENTS

Analytical procedures are laboratory-specific because of the different methods being used at ERG and DRI. The ERG analytical methods for of Pb-TSP and Pb-PM_{10} uses ICP-MS. The analytical method used at DRI utilizes the XRF for the analysis of Pb-PM_{10}. All analytical method SOPs are provided in Appendix A for ERG and Appendix B for DRI. Corrective action for analytical system failures realized at time of analysis is initiated by the Analyst, supported by the Task Leader for that method.

The SOPs for these analytical methods are reviewed annually and updated as necessary. The QA Coordinator, Program Manager and Writer/Editor will review, sign and date it before distributing to the particular laboratories satellite file areas. The previous copies will be replaced with the revised edition. The original, and all previously revised edits, are stored in a historical file maintained by ERG's Document Administrator.

10.1 Lead Analysis Using an ICP-MS Analytical System

Sample preparation and analysis procedures are based on NAAQS FEM for the analysis of Lead (EQL-0512-201[3] for TSP and EQL-0512-202[4] for PM_{10}) using ICP-MS analysis techniques. Upon receipt from the field, the samples are checked against the COC forms and then logged into the LIMS system. Each sample component is examined to determine if damage occurred during travel. Color, appearance, and other particulars of the samples are noted. A complete description of the preparation and analytical procedures for glass fiber (8x10") filters (ERG-MOR-084) and for Teflon® 47mm filters (ERG-MOR-085) are presented in Appendix A. Analysis hold time for metals filters is 180 days.

The ICP-MS consists of an inductively coupled plasma source, ion optics, a quadrupole mass spectrometer, a recirculator and an autosampler. The mass spectrometer will be mass calibrated and resolution checked. Resolution at low mass is indicated by magnesium isotopes

24, 25, and 26. Resolution at high mass is indicated by lead isotopes 206, 207, and 208. Instrument stability must be demonstrated by running a tuning (daily performance check) solution (1 nanograms per liter (ng/L) of barium, bismuth, cerium, cobalt, indium, lead, lithium, and uranium; and 10 ng/L of magnesium) five times with the resulting Relative Standard Deviation (RSD) of absolute signals for all analytes of less than 3 percent. Sample and waste disposal procedures are outlined in ERG-MOR- 033, the *SOP for Hazardous Waste*.

10.2 Lead Analysis Using an X-Ray Fluorescence Analytical System

DRI will analyze the PM_{10} filters for Pb by XRF. Upon receipt from ERG, the samples are checked against the COC forms. EDXRF analysis will be performed on Teflon-membrane filters for Pb PM_{10}, although the system may be used for up to 51 elements including the 33 elements currently reported to AQS for EPA's Chemical Speciation Network (CSN). XRF analyses are performed on a PANalytical Epsilon 5 EDXRF analyzer. Seven XRF conditions are normally used by the PANalytical instrument on each sample to optimize the detection limits for the specified elements. However, since Pb has no significant interferences from other elements that are accounted for in the deconvolution software for the condition used, if EPA and ERG agree, DRI will modify its normal multi-element procedure to use only the one condition for Pb and increase the condition's analysis time by a factor of four to improve the Pb detection limit by a factor of two.

Two types of EDXRF standards are used for calibration, performance testing, and auditing: (1) vacuum-deposited thin-film elements and compounds from Micromatter Co. (Vancouver, BC), and (2) polymer films. The vacuum deposit standards cover most elements and are used as calibration standards. The polymer film and NIST standards are used as QC standards. During EDXRF analysis, filters are removed from their Petri slides and loaded into holders for entry into the x-ray analysis chamber. The vacuum in the x-ray chamber and the heat induced by the absorption of x-rays may evaporate some materials, such as ammonium nitrate. A QC standard and a replicate from a previous analysis will be analyzed with each set of 10 filters. When a QC value differs from specifications by ± 10% or more, or when a replicate

value differs from the original value (where values exceed 10 times the detection limits) by ±10% or more, the problem is identified and filters may be reanalyzed. If further tests of standards show that the system calibration has changed by more than ±5%, the instrument is recalibrated. In addition, DRI will maintain a set of laboratory blanks that will be analyzed periodically (~1 blank for every 20 filters analyzed) to test for baseline shifts in blank values.

After EDXRF analysis, the Teflon-membrane filters are returned to their Petri slides and stored until the XRF data validation is completed and indicates that the runs are acceptable.

Detailed information on the EDXRF analysis of Teflon-membrane filters is given in DRI *SOP for for X-Ray Fluorescence (XRF) Analysis of Aerosol Filter Samples (PANalytical Epsilon 5)*, DRI SOP #2-209.8, in Appendix B.

SECTION 11
QUALITY CONTROL REQUIREMENTS

This section describes the quality control requirements for each of the analytical methods. The MDLs presented in this section were performed in 2015.

11.1 Standard Traceability

The standards used for all analytes are vendor-supplied National Institute of Standards and Technology (NIST) standards or vendor-supplied referenced to a NIST standard. All analytical methods are also certified by comparison to a second source NIST-traceable standard. The ERG-MOR-022, *SOP for the Preparation of Standards in the ERG Laboratory*, provides direction for preparing standards (Appendix A).

11.2 Accuracy and Acceptance

Because ambient air measurements encompass a range of compounds and elements whose individual concentrations are unknown, defining absolute accuracy is not possible. Instead, accuracy is determined by comparing the analysis of standards of known concentration. The criteria for the analysis of collocate samples and their replicate analyses are found in Section 4. Each instrument calibration is discussed by method in Section 13 of this QAPP. Accuracy of analysis is based on the accuracy of the calibration, including the accuracy of the calibration standards. Accuracy is monitored throughout the program using QC and quarterly audit (or proficiency) samples. Routine analysis of proficiency lead test strips/filters for TSP by ICP-MS, PM_{10} by ICP-MS and PM_{10} by XRF is discussed in Section 16.1.3. Required QC samples and their criteria and corrective actions are discussed by the methods listed below.

11.2.1 Lead Analysis by ICP-MS

Daily, the mass spectrometer used for metals analysis must have an acceptable daily performance check using the tuning solution. Performance specifications are presented in Table 11-1. Analysis of lead will be performed by ICP-MS. Bismuth and Holmium are the internal standard used for the analysis of lead. Internal standard responses must be evaluated for stability. Daily calibration, using a calibration blank and a minimum of 4 non-zero standards prepared from NIST-traceable stock solutions, is performed to ensure that the analytical procedures are in control. To be considered acceptable, the calibration curve must have a correlation coefficient ≥ 0.998. After calibration, an Initial calibration Verification (ICV), Initial Calibration Blank (ICB), Low Standard Verification (LCV), High Standard Verification (HSV) to ensure quality by verifying the initial calibration before the analysis of the samples and throughout the day. Interference Check Standard A (ICSA), and Interference Check Standard B (ICSAB) are also analyzed daily to ensure the accurate measurement of lead.

Table 11-1.

ICP-MS Instrument Performance Specifications

Mass Number	Sensitivity/Ratio	RSD
KED Mode		
24Mg 25Mg 26Mg	> 3,000 cps > 500 cps > 600 cps	< 5% RSD
59Co	> 30,000 cps	< 2% RSD
115In	> 30,000 cps	< 2% RSD
206Pb 207Pb 208Pb	> 60,000 cps > 50,000 cps > 80,000 cps	< 2% RSD
238U	> 80,000 cps	< 2% RSD
140CeO/140Ce	< 0.01	N/A
59Co/35Cl.16O	< 18.0	N/A
Background	< 0.5 cps @ Mass 4.5 < 2.0 cps @ Mass 220.7	N/A

*cps – Counts per second

If the initial calibration checks do not meet criteria, a second calibration check analysis is performed. If the second set does not pass, or if one or more of the daily QC checks do not meet criteria, a new calibration curve is prepared and analyzed. All samples analyzed with the unacceptable QC check will be reanalyzed or flagged appropriately when necessary. During the analysis of the samples, the Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) are analyzed immediately before the analysis of samples, every ten samples, and at the end of every analysis batch. The ICSA and ICSAB are analyzed every eight hours and at the end of every analysis. The LCV is analyzed at the end of every analysis. Quality procedures for metals analysis by ICP-MS are shown in Table 11-2.

Table 11-2
Summary of Quality Control Procedures for ICP-MS Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Daily Performance Check (DPR)	Daily, prior to samples	See Table 11-1 for acceptance criteria.	1)Repeat analysis of DPR 2)Re-optimize instrument tuning parameters 3) Reprepare DPR standard 4) Perform instrument maintenance
Initial Calibration Standards (IC)	Daily, at least 5 calibration points (blank included as one calibration point)	Correlation coefficient ≥ 0.998 & %RSD ≤ 10. RSDs > 10 are acceptable for the CAL2 standard.	1) Repeat analysis of calibration standards 2) Reprepare calibration standards and reanalyze
ICV	Immediately after calibration	Recovery 90-110%	1) Repeat analysis of ICV 2) Recalibrate ICV standard 3) Recalibrate and reanalyze
ICB	Immediately after ICV	Absolute value must be < MDL	1) Locate and resolve contamination problems before continuing 2) Reanalyze or recalibrate or flag failing elements for the entire analysis when appropriate
HSV	After ICB and before ICS	Recovery from 95-105%	1) Repeat analysis of HSV 2) Reprepare HSV
ICSA/IFA	Following the HSV, every 8 hours and at the end of each run	Within ±3 times Limit of Quantitation (LOQ) from zero or from the standard background contamination when present	1) Repeat analysis of ICSA 2) Reprepare ICSA and analyze 3) Adjust correction equation(s) and reprocess entire analysis

Table 11-2, Continued
Summary of Quality Control Procedures for ICP-MS Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
ICSAB/IFB	Following each ICSA	Recovery 80-120% of true value plus standard background contamination when present	1) Repeat analysis of ICSAB 2) Reprepare ICSAB and analyze 3) Adjust correction equation(s) and reprocess entire analysis
CCV	Analyze before samples, after every 10 samples, and at the end of each run	Recovery 90-110%	1) Reanalyze CCV 2) Reprepare CCV 3) Recalibrate and reanalyze samples since last acceptable CCV
LCV	At the beginning and end of each analysis, between the CCV and the CCB	Recovery 70-130%	1) Reanalyze LCV 2) Reprepare LCV 3) Recalibrate and reanalyze samples since last acceptable LCV
CCB	Analyzed after each CCV	Absolute value must be < MDL	1) Reanalyze CCB 2) Reanalyze samples since last acceptable CCB
LRB/BLK1	1 per 20 samples, a minimum of 1 per batch	Absolute value must be < MDL	1) Reanalyze 2) If > MDL, but < 5x MDL, sample results for that element must be flagged for the entire analysis 3) If > 5x the MDL then sample results for that element must be blank subtracted
MB/BLK2	1 per 20 samples, a minimum of 1 per batch	Absolute value must be < MDL. Note: The MB is used only for the purpose of MDL generation	This standard is not required by the method and there is no corrective action
Standard Reference Material (SRM)	1 per 20 samples, a minimum of 1 per batch	Recovery 80-120%	1) Reanalyze 2) Flag sample data 3) Re-extract batch
LCS/BS (and BSD for 47mm Teflon® filters only)	1 per 20 samples, a minimum of 1 per batch	Recovery 80-120%	1) Reanalyze 2) Flag data if recovery for only one or two elements fail criteria 3) Reprepare sample batch if recovery for most elements fail criteria, if possible

Table 11-2, Continued
Summary of Quality Control Procedures for ICP-MS Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Replicates (DUP) (Replicate Analysis)	1 per 20 samples, a minimum of 1 per batch	≤ ±10% RPD for 47 mm Teflon filters and 20% RPD for 8x10" Quartz filters values ≥ 10 times the MDL	1) Check for matrix interference in the case of DUP1 2) Repeat replicate analysis 3) Flag data
Collocated Samples (C1/C2)	10% of samples annually, if applicable	≤ ±20% RPD for sample and collocate values ≥ 10 times the MDL	1) Flag C2 data if associated replicate analysis are within criteria 2) Repeat analysis if replicate analysis fail
Matrix Spike (MS) and Matrix Spike Duplicate (MSD) for 8x10" Quart filters only	1 per 20 samples per sample batch	Recovery 75-125%	1) Flag data if recovery for only one or two elements fail criteria, or when a matrix interference is confirmed by SRD and/or PDS results 2) Reanalyze 3) Reprepare sample batch if recovery for most elements fail criteria or contamination is evident
Post Digestion Spike (PDS)	1 per 20 samples, minimum of 1 per batch	Recovery 75%-125%	1) Flag failed elements for parent sample and PDS 2) Reprepare PDS if preparation issue is suspected reason for failure
Serial Dilution (SRD)	1 per batch	Recovery 90-110% of undiluted sample if the element concentration is minimally a factor of 50 above the MDL in the original sample	1) Reprepare dilution if preparation issue is suspected reason for failure 2) Flag failed analytes
Internal Standards (ISTD)	Every Calibration, QC and Field Sample	Recovery 60-125% of the measured intensity of the calibration blank	1) If drift suspected, stop analysis and determine cause, recalibrate if necessary 2) Reprepare sample 3) If recovery > 125% due to inherent ISTD, dilute sample and reanalyze

11.2.2 Lead Analysis by XRF

Table 14-1 summarizes the QC measures for elemental analysis by EDXRF.

Two types of EDXRF standards are used for calibration, performance testing, and auditing: (1) vacuum-deposited thin-film elements and compounds from Micromatter Co. (Deer Harbor, WA), and (2) polymer films. The vacuum deposit standards cover all elements except for Ir, Ta, Zr, and Hf (which may be determined by interpolation) and are used as calibration standards. The polymer film and NIST standards are used as QC standards. During EDXRF analysis, filters are removed from their Petri slides and loaded into the carousel for entry into the x-ray analysis chamber.

The vacuum in the x-ray chamber and the heat induced by the absorption of x-rays may evaporate some materials, such as ammonium nitrate. Two QC standards are run once per day. Ten percent replicates are also analyzed. When a QC value differs from specifications by ± 10% or more, or when a replicate value differs from the original value (where values exceed 10 times the detection limits) by ±10% or more, the previous filters are reanalyzed. If further tests of standards show that the system calibration has changed by more than ±5%, the instrument is recalibrated. In addition, DRI will maintain a set of laboratory blanks that will be analyzed periodically (~1 blank for every 20 filters analyzed) to test for baseline shifts in blank values.

More detailed information on the QC measures for elemental analysis by EDXRF may be found in DRI SOP #2-209.8, in Appendix B.

Table 11-3
Summary of Quality Control Procedures for XRF Analysis

Requirement	Frequency	Calibration Standard	Performed By	Acceptance Criteria	Corrective Action
Multipoint Calibrations	Annually	QC standards	XRF lab supervisor	± 5%	Recalibrate
Minimum Detection Limit (MDL)	Initially, then quarterly or after major instrument change	Lab blanks	XRF lab supervisor, Project Manager	Within ± 10% of previous limits	Troubleshoot instrument and check filter lots
Lower Quantifiable Limit (LQL)	Quarterly	Field blanks	XRF lab supervisor, Project Manager	Within ± 10% of previous limits	Troubleshoot instrument and check filters
QC Samples					
Lab blanks	1/20 samples	N/A	Analyst	Within 3 σ of MDLs	Check instrument and filter lots
QC standards	Daily	Micromatter thin films	Analyst	± 10%	Samples before QC standard and previous standards reanalyzed
NIST-traceable standards	Annually	Micromatter thin films	Analyst	± 10%	Samples before QC standard and previous standards reanalyzed
Replicates	10% of samples	N/A	Analyst	± 10% when value >10*MDL	Reanalysis of previous samples
Level 1 Review	Every sample	N/A	XRF lab supervisor	Per SOP	Reanalysis of problem samples or flagging per SOP

11.3 Precision

Analytical precision is estimated by repeated analysis of approximately 10 percent of the samples. The second analysis is performed in the same analytical batch as the first analysis. Precision estimates are calculated in terms of absolute percent difference. Because the true concentration of the ambient air sample is unknown, these calculations are relative to the average sample concentration. The precision criteria for all parameters were listed previously in Table 4-1.

Precision is determined as the RPD using the following calculation:

$$RPD = \frac{|X_1 - X_2|}{\overline{X}} \times 100$$

Where:

X_1 is the ambient air concentration of a given compound measured in one sample;

X_2 is the concentration of the same compound measured during duplicate/collocate/replicate analysis; and

$\overline{X}$ is the arithmetic mean of X_1 and X_2.

11.4 Sensitivity (Method Detection Limits)

MDLs are determined different ways for each analytical system. For the ICP-MS, one MDL is determined for glass fiber filters, and another for Teflon® filters. The detection limits for metals by ICP-MS is determined by the FACA[7] method using compiled method blank data. The XRF MDLs are determined by DRI as three times the standard deviation of laboratory blanks. They are updated periodically, usually quarterly. The MDLs are shown in Table 11-3 and are based on an average sampling volume of 2000 m^3 for the glass fiber filters and 24.04 m^3 for the Teflon® filters.

Table 11-4. 2015 Method Detection Limit

Element	47 mm Teflon MDL (ng/m^3)	8x10" Glass Fiber Filter MDL (ng/m^3)
Lead – ICP-MS	0.029	0.089
Lead – XRF	2.0*	

* If only the one condition for Pb is used and the analysis time in increased by a factor of four, the MDL could become 1.0 ng/m^3.

NOTE: For total metals: Assumes total volume of 24.04 m^3 for Teflon® filters and 2000 m^3 for Glass Fiber filters.

SECTION 12
INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

To ensure the quality of the analytical equipment, ERG and DRI conduct performance checks for all equipment used. The laboratories monitor the performance of the analytical instrumentation to ensure proper operation while maintaining a spare parts inventory to shorten equipment downtime. Table 12-1 details the maintenance items, how frequently they will be performed, and who is responsible for performing the maintenance. All checks, testing, inspections, and maintenance done on each instrument are recorded in the appropriate Maintenance Logbook or LIMS Instrument Maintenance Logs for each instrument. Following instrument maintenance, a calibration must be passed to ensure the instrument is performing properly prior to analyzing samples. Because ERG's instrument is under full service contracts, service is rendered swiftly and instrument down time is kept to a minimum. DRI does not have a full service contract for its XRF instrument but keeps a wide variety of spare parts on hand for most common maintenance issues and make our own repairs. Historical maintenance on worst case instrument failure (multiple components), it has taken less than 2 weeks to get parts and repair and recalibrate the instrument. There is also a mutual aid agreement with UC Davis for analyzing XRF samples in case of XRF instrument failure.

12.1 ICP-MS

For ICP-MS analysis, preventive maintenance is performed by competent technical service representatives as needed. ERG personnel perform minor maintenance, such as detector maintenance, on an as-needed basis. Spare pump tubing, autosampler probes, nebulizers, spray chambers, torches, and cones should all be kept in reserve in the lab for the ICP-MS. More procedures, checks, and scheduled maintenance checks are provided in ERG's SOP ERG-MOR-084 and -085 for metals analysis by ICP-MS, in Appendix A.

Table 12-1
Preventive Maintenance in ERG & DRI Laboratories

Item	Maintenance Frequency	Responsible Party
ICP-MS Instrument		
Computer Backup	Daily	Manufacturer Service Contractor/Analyst
Change Roughing Pump Oil	Annually	Manufacturer Service Contractor/Analyst
Chiller Fluid Top-off and Cleaning	As Needed	Manufacturer Service Contractor/Analyst
Replace Peripump Tubing	Daily	Analyst
Clean/Replace Sample Introduction Components	As Needed	Analyst
Replace Autosampler Housing & Instrument Air Filters	Annually	Manufacturer Service Contractor/Analyst
XRF Instrument		
Run QC standards	Daily	Analyst
Fill LN2 container	Weekly	Analyst
Perform detector energy calibration	Weekly after LN_2 fill	Analyst
Check/fill x-ray tube cooling water vessel	As needed	XRF Lab Supervisor
Check/change pump oil	As needed	XRF Lab Supervisor
Multipoint calibrations	Annually or after certain equipment replacement or repair	XRF Lab Supervisor

12.2 XRF

For XRF analysis, most preventive maintenance is performed by competent analysts while the multipoint calibration is performed by the XRF Laboratory Supervisor. The instrument tends to run with minimal maintenance and major components tend to operate for years before needing replacement. Additional procedures and checks are described in DRI's SOP #2-209 for lead analysis by XRF in Appendix B.

SECTION 13
INSTRUMENT CALIBRATION AND FREQUENCY

Because the requirements for analytical system calibrations differ, the laboratories are discussed separately in this section. Analytical instruments and equipment are calibrated when the analysis is set up, when the laboratory takes corrective action, following major instrument maintenance, or if the continuing calibration acceptance criteria have not been met. Appropriate standards are prepared by serial dilutions of pure substances, accurately prepared concentrated solutions. In preparing stock solutions of calibration standards, great care is exercised in measuring weights and volumes, since analyses following the calibration are based on the accuracy of the calibration.

DRI's three types of standards used with the DRI XRF are: (1) elemental thin film standards from μMatter; (2) multiple element thin film standards from μMatter; and (3) NIST certified standards. None of these standards require preparation; they are used as received from the supplier. The μMatter standards are stored in PetriSlides and kept in a cool dark cabinet when not in use to retard oxidation and loss of volatile elements. NIST standards are stored in the XRF room in the standards cabinet at ambient conditions. Certificates of elemental concentrations are provided by the manufacturer and are filed in the XRF lab. The DRI XRF system is recalibrated approximately every 12 months using the μMatter thin film standards. Recalibration is also performed whenever the QA standard indicate a drift of >±5% in calibration. Standards including elements from Na to U are analyzed in standard 47 mm filter holders under the filter analysis application (Filterxxxyy, where yyy = month and xx = year). Calibration factors in μg/cm2 per counts per second per milliamp are calculated for each element using linear regression analysis by the Epsilon 5 software package. The μMatter standards are accurate to ±5% relative, as stated by the manufacturer. The NIST standards are used to verify the multipoint calibration that is performed annually or after major component repairs or replacement.

Each calibration analysis is stored, electronically and hardcopy, with the samples analyzed using that calibration. All programs store the calibration information separately with all pertinent information (raw data, control charts, and/or any summary statistics) together with the analyzed samples.

13.1 ICP-MS Calibration

Calibration requirements for ICP-MS is shown in Table 13-1.

13.2 XRF Calibration

Calibration requirements for the XRF are also shown in Table 13-1.

Table 13-1
ICP-MS and XRF Analytical Calibration Requirements

Analytical Parameter	Quality Parameter	Method of Determination	Frequency	Acceptance Criteria
ICP-MS	Calibration – Quantitative	Initial analysis of a calibration blank plus 4 levels of standards	Prior to sample analysis	Correlation coefficient ≥ 0.998 & RSD ≤ 10. RSDs > 10 are acceptable for the CAL2 standard.
	ICV	Analysis of a second source standard	Immediately following calibration	Recovery 90-110%
	ICB	Analysis of an ASTM Type I water acidified with the same acid matrix as is present in the calibration standards.	Immediately following the ICV	Absolute value must be ≤ MDL
	HSV	Analysis of high concentration standard	Immediately following the ICB	Recovery 95-105%

Table 13-1, Continued
ICP-MS and XRF Analytical Calibration Requirements

Analytical Parameter	Quality Parameter	Method of Determination	Frequency	Acceptance Criteria
	ICSA	Analysis of blank solution containing interfering elements	Following the HSV, every 8 hours, and at the end of each run	Analyte must be ±3x the QL from zero or from the standard background contamination when present.
	ICSAB	Analysis of standards containing interfering elements	Immediately following each ICSA	Recovery 80-120% of true value plus standard background contamination when present
	CCV	Analysis of mid-range calibration standard to verify initial calibration	Analyze before the 1st sample, after every 10 samples and at the end of the run	Recovery 90-110%
	LCV	Analysis of low-range calibration standard	Analyze at the beginning and end of each analysis	Recovery 70-130%
	CCB	Analysis of an ASTM Type I water acidified with the same acid matrix as is present in the calibration standards.	Analyze after each CCV	Absolute value must be ≤ MDL
XRF Analysis	Accuracy	QC µMatter standards	Daily	± 5% of standard
	Detector Signal	Internal tungsten beam	Weekly	Internal automated adjustment

SECTION 14
INSPECTION/ACCEPTANCE FOR SUPPLIES AND CONSUMABLES

14.1 Purpose

The purpose of this element is to establish and document a system for inspecting and accepting all supplies and consumables that may directly or indirectly affect the quality of the data. By having documented inspection and acceptance criteria, consistency of the supplies can be assured. This section details the supplies/consumables, their acceptance criteria, and the required documentation for tracing this process.

14.2 Critical Supplies and Consumables

Table 14-1 details the various components for the laboratory operations.

14.3 Acceptance Criteria

Acceptance criteria must be consistent with overall project technical and quality criteria. It is the laboratory analyst's responsibility to update the criteria for acceptance of consumables. As requirements change, so do the acceptance criteria. Observation of damage due to shipping can only be performed once the equipment has arrived on site.

All supplies and consumables are inspected and either accepted or rejected upon receipt in the laboratory. The employees who ordered the supply are responsible for verifying that the order is acceptably delivered, stored and dispersed upon receipt in the laboratory. Some supplies or consumables listed in Table 14-1 have to be deemed acceptable, through testing or blanking, such as with the filters used for blank analysis. Any changes in standards and sample media must meet the acceptance criteria outlined in Section 11 for that particular method. Such testing and blanking data is kept with the sample data. Staff should not use supplies or consumables of different model numbers or from different vendors without first discussing it with the Program Manager and testing the supply or consumable.

Table 14-1
Critical Field and Laboratory Supplies and Consumables

Area	Item	Description	Vendor	Model Number
Laboratory Supplies and Consumables (Laboratories listed below)				
ICP-MS	Argon	Coolant Gas	Air Gas	LARS-65
ICP-MS	Helium	Collision Gas	Air Gas	HE R80A
ICP-MS	Acid	High Purity HNO_3 High Purity HCl High Purity HF	Fisher	A497-2 A466-500 A463-250
ICP-MS	Inorganic standards	Individual Metals Standards	High Purity Spex Inorganic Ventures	Various
ICP-MS	Nalgene 60oz. bottles	Sample containers	Fisher	16058-043
ICP-MS	Extraction consumables	Sample Vials FilterMate Filters Reflux Caps Autosampler Vials	SCP Science Environmental Express CPI	010-500-264 SC0408 SC506 P/N 4092-316
ICP-MS	Glass Fiber/ Teflon Filters	Filters	Whatman MTL	1822-866 PT47AN
XRF	Liquid Nitrogen	Detector coolant	Air Gas	NA
XRF	Mylar sheets	2”x 2” 3.6 µm thick	Somar Spectrofilm	3615-33
XRF	Tweezers	For handling filters	Millipore	62-000067
XRF	Kimwipes	Large & small	VWR	34255 & 34155
XRF	Methanol (in squeeze bottle)	Cleaning solvent for sample holders & filter loading area	Fisher Scientific (VWR)	A454-4 (16649-945)
XRF	Epsilon 5 XRF Analysis Logsheet	Analysis Logsheet	DRI	NA

Consumables and supplies with special handling and storage needs must be handled and stored as suggested by the manufacturer. Consumables with expiration dates, such as standards, must be labeled with a receipt date, date opened, and the initials of the person that opened the consumable and standard expiration dates must be entered into the standards section of LIMS. To decrease waste, the oldest supplies or consumables should be used first.

14.4 Tracking and Quality Verification of Supplies and Consumables

Tracking and quality verification of supplies and consumables have two main components. The first is the need of the end user of the supply or consumable to have an item of the required quality. The second need is for the purchasing department to accurately track goods received so that payment or credit of invoices can be approved. In order to address these two issues, at ERG the following procedures outline the proper tracking and documentation procedures to follow:

- Receiving personnel will perform a rudimentary inspection of the packages as they are received from the courier or shipping company. Note any obvious problems with a receiving shipment such as crushed box or wet cardboard.
- The package will be opened, inspected, and contents compared against the packing slip.
- If there is a problem with the equipment/supply, note it on the packing list and notify the Purchasing Agent who will immediately call the vendor.
- If the equipment/supplies appear to be complete and in good condition, sign and date the packing list and give it to the Purchasing Agent so that payment can be made in a timely manner.
- Notify appropriate personnel that equipment/supplies are available. For items such as filters, it is critical to notify the laboratory manager of the weight room so sufficient time for processing of the filters can be allowed.
- Stock equipment/supplies in appropriate pre-determined area.

Standards and reference materials are purchased from vendors who certify the purity and traceability of these standards. Certificates of analysis and/or purity are maintained in project files or in the LIMS standards log.

SECTION 15
DATA MANAGEMENT

15.1 Data Recording

Data management for sample data is presented in Figure 15-1. The sample data path is shown from sample origination to data reporting and storage. The LIMS allows the laboratory to manage and track samples, instrument workflow, and reporting. The LIMS stores the raw instrument data and performs the conversion calculations to put the data into final reporting units. These calculations are reviewed and documented annually by ERG's QA coordinator and kept in the QA files in Room 102. The main procedures are described in the *SOP for the Laboratory Information Management System* (ERG-MOR-099). The main functions of the LIMS system include, but are not limited to:

- Sample login;
- Sample scheduling, and tracking;
- Sample processing and quality control; and
- Sample reporting and data storage.

All LIMS users must be authorized by the LIMS Administrator and permitted specified privileges. The following privilege levels are defined:

- Data Entry Privilege – The individual may see and modify only data within the LIMS that he or she has personally entered.
- Reporting Privilege – Without additional privileges.
- Data Administration Privilege – Data Administrators for the database are allowed to change data as a result of QA screening and related reasons. The Data Administrator is responsible for performing the following tasks on a regular basis:
  - Merging/correcting the duplicate data entry files;
  - Running verification/validation routines, correcting data as necessary; and
  - Generating summary data reports for management.

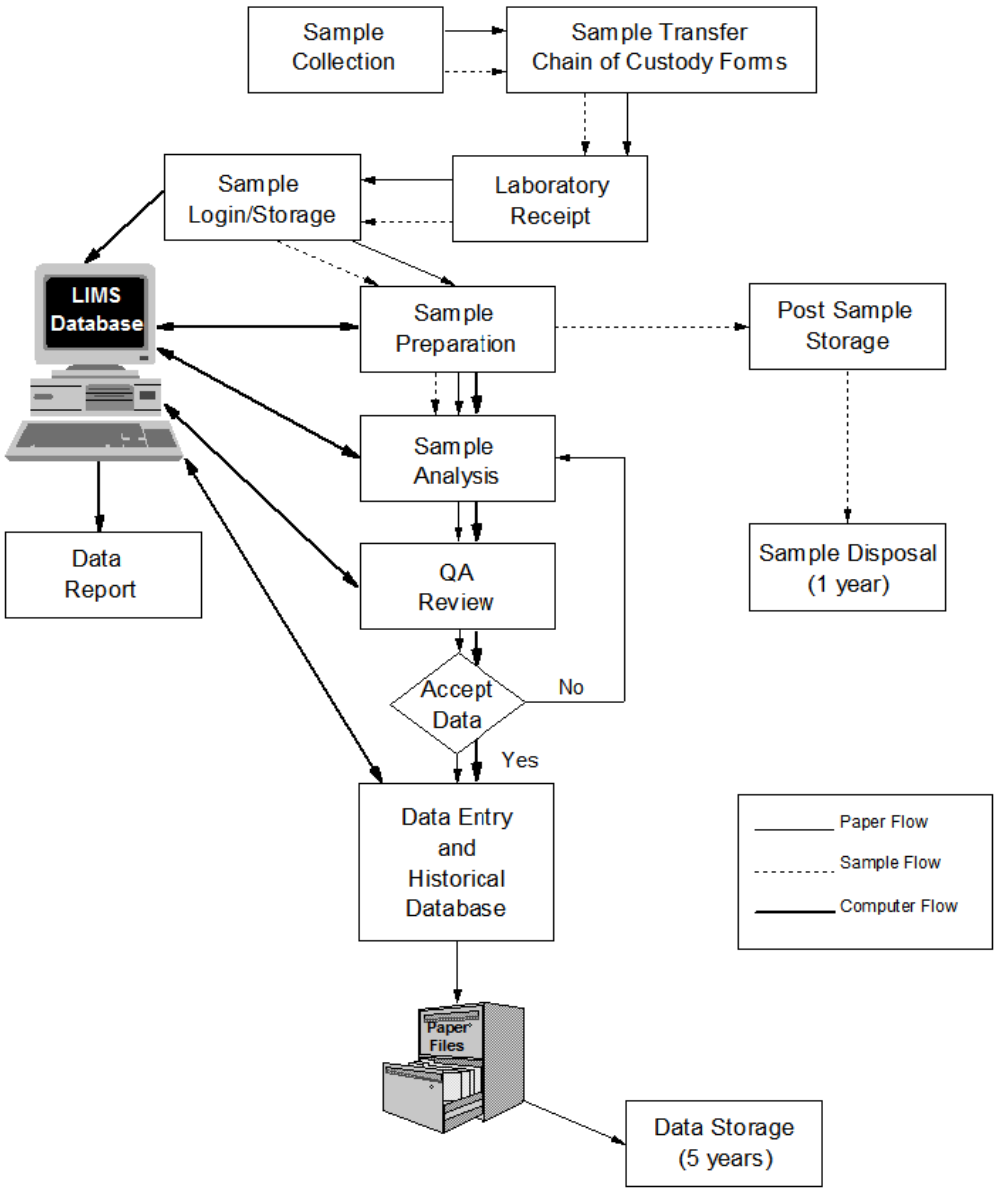

Figure 15-1. Data Management and Sample Flow Diagram

15.2 Data Validation

Data validation is a combination of checking that data processing operations have been carried out correctly and of monitoring the quality of the field operations. Operations checked include collection information on COCs, sample receipt entry into LIMS, sample volume entry

into bench sheets, and upload of data into LIMS. Data validation can identify problems in these areas. Once problems are identified, the data can be corrected or invalidated, and corrective actions can be taken for laboratory operations.

15.3 Data Reduction and Transformation

Calculations for transforming raw data from measured units to final concentrations use standardized procedures listed in the individual ERG or DRI SOPs. The equations for transforming raw data are set up to automatically calculate to final concentrations in the LIMS system. The initial units for ICP-MS Pb results are ng/L with final reporting units of µg/filter. The initial units for XRF Pb results are µg/cm^{2}with the final results in µg/m^3. For XRF Pb, the associated MDLS and uncertainties are reported along with the final concentrations. For ICP-MS, the associated MDLs are reported along with the final concentrations, and the MDLs are adjusted for dilution (for ICP-MS) and actual prep volumes before reporting.

The electronic data file is uploaded onto a network server (which is backed-up daily) and into the LIMS. DRI data files are imported onto the network server. Once the data is in LIMS, the ERG Task Leader reviews it following the checklists presented in Section 16 using instrument software and method specific controls set up in LIMS. Ten percent of the total data is reviewed by the QA Coordinator or designee following the checklist and method specific acceptance criteria in the summary quality control procedure tables outlined in Section 11. After the data has successfully completed both reviews and the checklists have been signed, it is available for reporting by the Program Manager.

The *SOP for Project Peer Review* uses manual calculations and visual verification to review all data reported to EPA and State/ local/tribal agencies following guidelines outlined in SOP ERG-MOR-057 (see Appendix A). *SOP for Developing, Documenting, and Evaluating the Accuracy of Spreadsheet Data*, presented in SOP ERG-MOR-017 (see Appendix A), is consulted in special cases where the calculations are performed via spreadsheets instead of the LIMS system.

Reporting formats are designed to fulfill the program requirements and to provide comprehensive, conventional tables of data. The LIMS data reporting format includes any required data qualifiers, footnotes, detection limits for each analyte, and appropriate units for all measurements. The LIMS can produce Adobe and Excel data reports, which is standard for this program. The report is reviewed by the Program Manager or designee before it is sent to the client. As mentioned in Section 15.5, a comprehensive annual data report is prepared including data statistics and characterization.

15.4 Data Transmittal

Data transmittal occurs when data are transferred from one person or location to another or when data are copied from one form to another. Some examples of data transmittal are copying raw data from a notebook onto a data entry form for keying into a computer file and electronic transfer of data over a computer network. Each individual SOP listed in Appendix A & B discusses the procedures for determining the calculations of concentrations as well as data entry.

If requested, ERG will report all ambient air quality data and information specified by the AQS User's Guide and other documents located at the website http://www.epa.gov/ttn/airs/airsaqs/manuals/ coded in the AQS format. Such air quality data and information will be fully screened and validated and will be submitted directly to the AQS via electronic transmission, in the format of the AQS, and in accordance with the annual schedule. The *SOP for the Preparation of Monitoring Data for AQS Upload* is presented in Appendix A (SOP ERG-MOR-098).

15.5 Data Tracking

The ERG LIMS database contains input functions and reports necessary to track and account for the status of specific samples and their data during processing operations. The following input locations are used to track sample and sample data status:

- Sample Control
  - Sample collection information (by Work Order);
  - Sample receipt/custody information;
  - Unique sample number (LIMS ID);
  - Storage location;
  - Required analyses;
  - Project due dates/hold times.

- Laboratory
  - Batch/bench assignment;
  - Sequence assignment;
  - Data entry/review;
  - Query/update analysis status;
  - Standards/calibration information.

15.6 Data Storage and Retrieval

Data archival policies are shown in Table 15-1.

All data are stored on the ERG LIMS server. This system has the following specifications:

- Operating System: Windows 2008 Server
- Memory: 6G RAM
- Hard Drives: Three drives of 450G each configured as RAID 5;
- Network card: Gigabit card (10/100/1000)
- Tape Drives for Backup: Two tape drives are daisy chained (Compaq SDLT 600 & HP SureStore DLT 818).
- Security: Network login password protection on all workstations; Additional password protection applied by application software.

Security of the data in the database is ensured by the following controls:

- Password protection on the data base that defines three levels of access to the data;
- Regular password changes (quarterly);
- Logging of all incoming communication sessions, including the originating telephone number, the user's ID, and connect times; and
- Storage of media, including backup tapes, in an alternate location that is at a locked, restricted access area.

Table 15-1. Data Archive Policies

Data Type	Medium	Location	Retention Time	Final Disposition
Laboratory notebooks	Hardcopy	Laboratory	5 years after close of contract	N/A
LIMS Database	Electronic (on-line)	Laboratory	Backup media after 5 years	Backup tapes retained indefinitely

ASSESSMENT/OVERSIGHT
SECTION 16
ASSESSMENTS AND RESPONSE ACTIONS

An assessment is defined as an evaluation process used to measure the performance or effectiveness of the quality system or the establishment of the monitoring network and sites and various measurement phases of the data operation.

The results of QA assessments indicate whether the control efforts are adequate or need to be improved. Documentation of all QA and QC efforts implemented during the data collection, analysis, and reporting phases are important to data users, who can then consider the impact of these control efforts on the data quality. Both qualitative and quantitative assessments of the effectiveness of these control efforts will identify those areas most likely to impact the data quality. ERG will perform the following assessments in order to ensure the adequate performance of the quality system.

The Response/CAR will be filed whenever a problem is found such as an operational problem, or a failure to comply with procedures that affects the quality of the data. A CAR is an important ongoing report to management because it documents primary QA activities and provides valuable records of QA activities. A CAR can be originated by anyone on the project, but must be sent to the Program QA Coordinator and Program Manager. Any problem that affects the quality of the overall program will be discussed with the EPA.

On the numbered CAR, the description of the problem, the cause of the problem, the corrective action, and the follow-up are documented. The follow-up assists the QA coordinator in determining if the corrective action was successful and if it was handled in a timely manner. ERG's CAR is recorded on a three-part form, the white copy goes into the project file, the yellow copy goes into the QA file (Room 102), and the pink copy goes to the facilitator. A copy of the ERG CAR Form is shown in Figure 16-1.

Corrective Action Report – 2015-01

Originator:	**Date:**	
Project Number:	**Corrective Action Number:**	
Is Immediate Stop of Work required?	Yes	No
Description of the Problem: (Provide date and time identified)		
State cause of Problem: (An investigation can reveal the cause, may fill after investigation, provide dates and time frame and if multiple approaches have been used to identify the cause)		
State Corrective Action Planned: (Include persons involved in action and date action is to be completed, include all approaches and dates)		
Close Out Details: (Fill when the corrective action successfully provides an effective long-term solution, include all approaches attempted)		

Signature and Date	Comments
QA Officer:	
Project Manager:	
Originator:	

Figure 16-1. ERG Corrective Action Report Form

DRI's process for dealing with corrective actions is governed by DRI *SOP for Corrective actions,* DRI SOP #6-017, and its CAR is shown in Figure 16-2.

EAF CORRECTIVE ACTION REQUEST

CAR No. _____ **Date** _____

To **From**	
How Identified	
Reported (Nonconformance) Conditions **Answer Due Date**	**Signature**
Root Cause	
Corrective Action (Give Steps & Expected Completion Dates)	
Preventive Action	
Completed by	**Date**
Accepted by	**Date**
Follow-up Results **Performed by**	**Date**
Closed Out (Y/N) _____	If not closed out on follow-up, issue a new CAR
New CAR No.	**Date**

Figure 16-2. DRI Corrective Action Report Form

Each recommendation addresses a specific problem or deficiency and requires a written response from the responsible party. Each also requires the Program QA Coordinator to verify that the corrective action has been implemented. A summary of the past years' CARs are discussed during the annual QA Management Systems Review.

The following actions are taken by the laboratory QA Coordinator and Program Manager when any aspect of the testing work, or the results of this work, does not conform to the requirements of the quality system or testing methods:

- Identify nonconforming work and take actions such as the halting of work, the withholding of test reports;
- Evaluate of the impact of nonconforming work on quality and operations;
- Take remedial action and make decision about the acceptability of the nonconforming work (resample, use as is with qualification, unable to use);
- Notify the client, and if necessary, recall the work; and authorize the resumption of work.

ERG and DRI are responsible for implementing the analytical phase of this program and are not responsible for the overall DQOs. Therefore, this QAPP tries to ensure that analytical results are of adequate quality to ensure the achievement of the various program DQOs.

16.1 Assessment Activities and Project Planning

16.1.1 External Technical Systems and Data Quality Audits

A Technical Systems Audit (TSA) is a thorough and systematic on-site qualitative audit, where facilities, equipment, personnel, training, procedures, subcontractor systems, and record keeping are examined for conformance to the QAPP. The TSAs will be performed by EPA or its designee at the ERG Laboratory. The TSAs of the contract are conducted approximately every five years. The EPA QA Office will implement the TSA either as a team or as an individual

auditor. ERG will participate in any data quality audits by EPA or designee at the discretion of the EPA QA Coordinator.

The EPA audit team will prepare a brief written summary of findings for the Program Manager and Program QA Coordinator. Problems with specific areas will be discussed and an attempt made to rank them in order of their potential impact on data quality. ERG will work with EPA to solve required corrective actions. As part of corrective action and follow-up, an audit finding response letter will be generated by the Program Manager and Program QA Coordinator. The audit finding response letter will address what actions are being implemented to correct the finding(s) of the TSA. This summary from EPA and the following response from ERG are filed in the QA/QC file in Room 102. The findings and the follow-up corrective actions are discussed in the annual QA Management Systems Review.

As part of ongoing National Environmental Laboratory Accreditation Conference (NELAC) certification, TSAs are performed at ERG by Florida Department of Health or designee every two years. A summary of findings is sent to ERG, specifically the QA Coordinator. The QA Coordinator sends its response of corrective actions which is either accepted or denied by Florida Department of Health. This documentation is stored in the QA/QC file in Room 102. The findings and the follow-up corrective actions are discussed in the annual QA Management Systems Review.

As a contractor laboratory performing analyses for EPA's $PM_{2.5}$ CSN, DRI has external TSAs performed roughly every three years by an audit team from EPA's National Air and Radiation Environmental Laboratory (NAREL). In addition, as part of its NELAP accreditation program through the Texas Commission on Environmental Quality (TCEQ), the EAF is audited roughly every three years by a TCEQ contractor.

16.1.2 Internal Technical Systems Audits

At ERG, an internal TSA is performed examining facilities, equipment, personnel, training, procedures, and record keeping for conformance to the individual SOPs and this QAPP. The TSAs will be performed by the Program QA Coordinator and will be conducted at least once per year. The checklists for the internal TSAs are based on the NATTS TSA or National Environmental Laboratory Accreditation Program (NELAP) checklists with additional areas addressing the individual SOPs and this QAPP. The content of the checklists vary episode to episode to ensure comprehensive in-depth coverage of procedures over time. Such elements will be included in the checklists:

- Criteria listed in Section 11 of this QAPP
- SOP specifications
- Method specifications
- Supporting equipment specifications
- Other laboratory wide QA systems in place (ex. Satellite SOP notebooks)

The Program QA Coordinator will report internal audit findings to the Program Manager within 30 days of completion of the internal audit in the form of a report. The EPA Delivery Order Manager will be informed if issues from the internal audit impact the quality of this program. The report is filed in the QA/QC file in Room 102. All corrective actions are addressed and implemented as soon as they are determined. The findings and the follow-up corrective actions are discussed in the annual QA Management Systems Review to assess effectiveness of the corrective actions.

At DRI, internal audits are conducted by the EAF QA Manager using the procedures contained in DRI *SOP for General EAF Internal Audit Procedures*, DRI SOP #4-117.1. The checklists for the internal TSAs are based on the EPA CSN TSA and NELAP checklists with additional areas addressing the individual SOPs, this QAPP, and the QAPPs for other projects.

16.1.3 Proficiency Testing

The PT is an assessment tool for the laboratory operations. Routine 'blind' samples (Pb Test strips/filters) are sent to the laboratory, where they follow the normal handling routines that any other sample follows. The results are sent to the Program Manager and Program QA Coordinator for final review and reporting to the auditing agency. The auditing agency prepares a PT report and sends a copy of the results to the Program Manager, Program QA Coordinator, and the EPA QA Office(s). Any results outside the acceptance criteria are noted in the PT report. Repeated analyte failures are investigated to determine the root cause and documented on a CAR. The PT reports are filed in the QA/QC file in Room 102. The performance on these audits is discussed in the annual QA Management Systems Review.

As a CSN contractor laboratory, DRI receives annual XRF PE samples from NAREL. However, these are ambient PM2.5 samples (of different sampling durations) and the XRF analysis is not exclusively for Pb. Pb one of several elements typically used for the annual inter-comparison. The results of these intercomparisons may be found at: http://www3.epa.gov/ttn/amtic/pmspec.html.

16.2 Documentation of Assessments

16.2.1 TSA, Data Quality Audit, and PT Documentation

All reports from EPA or designated contractors regarding ERG's performance on TSAs, Data Quality Audits, and PTs are filed in the QA/QC file in Room 102. PT reports are dispersed and discussed with contributing staff.

Reports from internal TSAs are prepared and discussed with the Program Manager, and filed in the QA/QC file in Room 102. A similar procedure is used by DRI for internal TSAs, external TSAs, and PE results and intercomparisons.

16.2.2 Internal Data Review Documentation at ERG

Internal data review is performed on 100 percent of the data by the Task Leader and 10 percent of the data by the Program QA Coordinator or designee against the criteria in the individual SOPs and this QAPP prior to being reported each month. The assessment is documented on the data review checklist, which is returned to the Task Leader for minor correction action and inclusion in the data package. The checklists used for analyses are shown in their respective SOPs (Appendix A) as follows:

- **Lead by ICP-MS** – ERG-MOR-084, *SOP for the Preparation and Analysis of High Volume Quartz Filters for Metals by ICP-MS using Method IO 3.5 and FEM Method EQL-0512-201* and ERG-MOR-085, *SOP for the Preparation and Analysis of 47mm Filters for Metals by ICP-MS using Method IO 3.5 and FEM Method EQL-0512-202.*

During the internal data review, major QC problems identified are brought to the attention of the Program Manager and are documented on a CAR. The final project report also addresses QA considerations for the whole project.

16.2.3 Internal Data Review Documentation at DRI

The procedures used at DRI are similar to those used by ERG. The SOP used for these analysis are presented in Appendix B.

- **Lead by XRF** – DRI *SOP #2-209.8, X-Ray Fluoresence (XRF) Analysis of Aerosol Filter Samples (PANalytical Epsilon 5), IO 3.3, and 40 CFR Part 50, Appendix Q.*

SECTION 17
REPORTS TO MANAGEMENT

This section describes the quality-related reports and communications to management necessary to support monitoring network operations and the associated data acquisition, validation, assessment, and reporting. Important benefits of regular monthly reports to EPA provide the opportunity to alert EPA to data quality problems, to propose viable solutions to problems, and to procure necessary additional resources.

Effective communication among all personnel is an integral part of a quality system. Regular, planned quality reporting provides a means for tracking the following:

- Adherence to scheduled delivery of data and reports;
- Documentation of deviations from approved QA and test plans, and the impact of these deviations on data quality; and
- Analysis of the potential uncertainties in decisions based on the data.

Frequency, content and distribution of reports for monitoring are shown below.

17.1 Data Reports

Analytical data reports prepared by the Program Manager are sent to EPA, State, Local and Tribal agencies within 30 days after the samples are received at ERG's laboratory. These reports include the monthly analytical data for each sample collected includes sample results, sample information (sample information, sample volume, etc.) and a QA data.

17.2 Internal Technical System Audit Reports

The Program QA Coordinator or designee performs an internal technical system audit at least once a year. The findings are listed in reports which are presented to the Program Manager

and filed in the QA/QC storage file cabinet located in Room 102. These reports are available to EPA personnel during their TSA. More detail on internal TSAs is provided in Section 16.

DATA VALIDATION AND USABILITY
SECTION 18
DATA REVIEW AND VERIFICATION

Data verification is a two-stage process to determine if the sampling and analytical data collection process is complete, consistent with the DQOs discussed in this QAPP and associated SOPs, and meets the program requirements. First the data is reviewed for completeness, accuracy, and acceptability. Then the data is verified to meet the quality requirements of the program.

18.1 Data Review Design

Information used to verify air toxics data, includes:

- Sample COCs, holding times, preservation methods.
- Multi-point calibrations – the multipoint calibrations are used to establish proper initial calibration and can be used to show changes in instrument response.
- Standards – certifications, identification, expiration dates.
- Instrument logs – all activities and samples analyzed are entered into the LIMS logs (batches, sequences, etc.) to track the samples throughout the measurements procedures.
- Supporting equipment – identification, certifications, calibration, if needed.
- Blank, CCVs, replicate and spike results – these QC indicators can be used to ascertain whether sample handling or analysis is causing bias in the data set.
- Review Checklists – these record monthly data quality review performed on all data by Task Leader and on 10 percent of the data by the QA Coordinator or designee. Data is reviewed using the checklists derived from the SOPs and this QAPP.
- Summary Reports – data reports present the data to the EPA and respective State/local/tribal representatives including data qualifiers.

The reliability and acceptability of environmental analytical information depends on the rigorous completion of all the requirements outlined in the QA/QC protocol. During data analysis and validation, data are filtered and accepted or rejected based on the set of QC criteria listed in the individual SOPs included in Appendix A (ERG) and B (DRI).

The data are critically reviewed to locate and isolate spurious values. A spurious value, when located, is not immediately rejected. All questionable data, whether rejected or not, are maintained along with rejection criteria and any possible explanation. Such a detailed approach can be time-consuming but can also be helpful in identifying sources of error and, in the long run, save time by reducing the number of outliers.

18.2 Data Verification

Data verification confirms by examination that specified requirements have been fulfilled. The specific requirements are QC checks, acceptable data entry limits, etc. as presented in Section 4 and Section 11. The analytical procedures performed during the monitoring program will be checked against those described in the QAPP and the SOPs included in Appendix A (ERG) and B (DRI). Deviations from the QAPP will be classified as acceptable or unacceptable, and critical or noncritical. During review and assessment, qualifiers will be applied to the data as needed; data found to have critical flaws (such as low spike or surrogate recoveries, contaminated blanks, etc.) will be invalidated and a CAR filled out and implemented, if needed. All of the data management guidelines followed for this contract are presented in Section 15.

18.3 Data Review

The chain of custody forms are checked to ensure accurate transcription. The results are double-checked by the Task Lead and a 10% check is performed by the QA Coordinator. The collected data are reviewed by the Analyst and the Task Leader. The data are scrutinized daily to eliminate the collection of invalid data. Invalid samples are discussed in more detail in Section 9.2. The analyst records any unusual circumstances during analysis (e.g., power loss or

fluctuations, temporary leaks or adjustments, operator error) on the LIMS bench sheet and notifies the analytical Task Leader.

QC samples and procedures performed during the monitoring program will be checked against those described in Section 4 of the QAPP. If QC is found unacceptable by these criteria, corrective actions described in the same section are implemented. Prior to reporting, 100 percent of the data is reviewed by the Task Leader. To verify accuracy, at least 10 percent of the database is checked by the QA Coordinator or designated reviewer. Items checked include original data sheets, checks of all calculations (from calibration to sample analysis), and data transfers. As the data are checked, corrections are made to the database as errors or omissions are encountered. If major errors are found, all of the data is checked to verify data quality. The Program Manager reviews all of the data before it is reported to EPA or the State/local/tribal agencies.

18.4 Data Reduction, Validation, and Reporting

Data validation is confirmed by examination of objective evidence that the particular requirements for a specific intended use are fulfilled. Intended use deals with data of acceptable quality to permit making decisions at the correct level of confidence. This section outlines data validation and usability requirements.

All samples received are given a LIMS ID number that corresponds to that sample's information. An analysis logbook is maintained to detail pertinent sample information at the time of analysis. Entries include site code, sample date, analysis date, LIMS ID, and electronic file names. Electronic copies of the data are stored on the ERG LIMS server.

Data summaries are distributed to the participating EPA technical staff, administrators, and to the administrators of the State/local/tribal agencies involved in the study. Each report is prepared within 30 days from receipt of the samples at ERG's laboratory. Cumulative listings are periodically generated upon request. Any changes made in the data as a result of subsequent

data validation processes performed by EPA and/or State/local/tribal agencies are noted in the cumulative project data summaries for each specific sampling site. The data summaries include:

- Site code;
- Sample identifications;
- Sample dates;
- Target compound list;
- Final concentrations; and
- Method detection limits.

Data summaries are emailed to the program participants. The Program Manager reviews all data before they are reported to EPA and/or the State/local/tribal agencies.

18.5 Air Quality System

ERG can submit the data collected for the Lead Analysis to the AQS database if requested by the sites. This data can be either submitted by ERG or by the State, Local or Tribal Agency.

Prior to ERG's submittal of data to AQS, the State/local/tribal agency would have to submit, at a minimum, Basic Site Information transactions (Type AA) for each sampling site, and Site Street Information (Type AB) and Site Open Path Information (Type AC), if necessary. ERG then submits monitor transactions to prepare the AQS database for data upload. Data that are uploaded into AQS include Raw Data (RD) transactions, QA transactions and Raw Blank (RB) transactions.

The submittal process involves the following steps:

- The raw data are formatted into pipe-delimited (|) coding that is accepted by the AQS. Raw data, data generated by single sample episodes, by the primary sample

(D1) of a duplicate episode, or by collocates (C1 and C2) are submitted using RD transactions. Precision data, data generated by duplicate and replicate samples (R1, D2, and/or R2) are submitted using QA transactions, specifically duplicate and replicate transactions. Accuracy data, generated for lead-FEM audit results, are submitted using Pb Analysis Audit transactions.

- The RD, QA (specifically duplicate, replicate and Pb Analysis Audit), and RB coding is generated and reviewed following guidelines listed in the *SOP for the Preparation of Monitoring Data for AQS Upload (ERG-MOR-098)* to ensure that the proper monitor ID (including state, county, site, parameter, and Parameter Occurrence Code [POC] codes), sampling interval, units, method, sample date, start hour, and sample values are correct. The transactions are stored as text files for upload into the AQS database.

- The transaction files are loaded under the appropriate screening group.

- The transactions are edited to remove any errors found by AQS and then are resubmitted. This step is repeated until the transactions are free of errors.

- AQS performs a statistical check on the data submitted to validate the data and determine if there are any outliers based on past data.

- The data transactions are then posted into the AQS database.

18.5.1 AQS Flagging and Reporting

Air toxics data submittals may be submitted with flags to indicate additional information related to the sample. There are two qualifier flag types that may be applied: Null codes and Qualifier codes.

- **Null Code —** assigned when a scheduled sample is not usable (e.g., damaged filter, improper sampling time, etc.).

- **Qualifier Code —** used to note a procedural or quality assurance issue that could possibly affect the uncertainty or concentration of the value.

Qualifier Codes can be used in combination, with up to 10 possible codes applied. If a Null code is used, no other flag should be used since no results are reported. Table 18-1 presents the Qualifier codes and Table 18-2 presents the Null codes available to AQS users. These flags

are applicable to the various steps of sample collection and analysis such as field operations, chain of custody, and laboratory operations.

Blank issue flags are qualifier flags used if reported blank values are above the limits set by the method SOPs or QAPP. If high blank values are associated with samples, the sample values are reported but appropriately flagged. Samples will not be invalidated due to high blank values. Blank issue flags are included in Table 18-1.

Table 18-1
Qualifier Codes

Qualifier Code	Qualifier Description
1	Deviation from a CFR/Critical Criteria Requirement
2	Operational Deviation
3	Field Issue
4	Lab Issue
5	Outlier
6	QAPP Issue
7	Below Lowest Calibration Level
9	Negative value detected - zero reported
CB	Values have been Blank Corrected
CC	Clean Canister Residue
CL	Surrogate Recoveries Outside Control Limits
DI	Sample was diluted for analysis
EH	Estimated; Exceeds Upper Range
FB	Field Blank Value Above Acceptable Limit
HT	Sample pick-up hold time exceeded; data questionable
IA	African Dust
IB	Asian Dust
IC	Chemical Spills & Industrial Accidents
ID	Cleanup After a Major Disaster
IE	Demolition
IF	Fire - Canadian
IG	Fire - Mexico/Central America
IH	Fireworks
II	High Pollen Count
IJ	High Winds
IK	Infrequent Large Gatherings
IL	Other
IM	Prescribed Fire
IN	Seismic Activity
IO	Stratospheric Ozone Intrusion
IP	Structural Fire

Table 18-1, Continued
Qualifier Codes

Qualifier Code	Qualifier Description
IQ	Terrorist Act
IR	Unique Traffic Disruption
IS	Volcanic Eruptions
IT	Wildfire-U. S.
J	Construction
LB	Lab blank value above acceptable limit
LJ	Identification Of Analyte Is Acceptable; Reported Value Is An Estimate
LK	Analyte Identified; Reported Value May Be Biased High
LL	Analyte Identified; Reported Value May Be Biased Low
MD	Value less than MDL
MS	Value reported is ½ MDL substituted
MX	Matrix Effect
ND	No Value Detected
NS	Influenced by nearby source
QX	Analyte does not meet QC criteria
SQ	Values Between SQL and MDL
SS	Value substituted from secondary monitor
SX	Does Not Meet Siting Criteria
TB	Trip Blank Value Above Acceptable Limit
TT	Transport Temperature is Out of Specs
V	Validated Value
VB	Value below normal; no reason to invalidate
W	Flow Rate Average out of Spec.
X	Filter Temperature Difference out of Spec.
Y	Elapsed Sample Time out of Spec.

Table 18-2
Null Codes

Null Code	Qualifier Description
AA	Sample Pressure out of Limits
AB	Technician Unavailable
AC	Construction/Repairs in Area
AD	Shelter Storm Damage
AE	Shelter Temperature Outside Limits
AF	Scheduled but not Collected
AG	Sample Time out of Limits
AH	Sample Flow Rate out of Limits
AI	Insufficient Data (cannot calculate)
AJ	Filter Damage
AK	Filter Leak
AL	Voided by Operator
AM	Miscellaneous Void

Table 18-2, Continued
Null Codes

Null Code	Qualifier Description
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	Q C Control Points (zero/span)
AZ	Q C Audit
BA	Maintenance/Routine Repairs
BB	Unable to Reach Site
BC	Multi-point Calibration
BD	Auto Calibration
BE	Building/Site Repair
BF	Precision/Zero/Span
BG	Missing ozone data not likely to exceed level of standard
BH	Interference/co-elution/misidentification
BI	Lost or damaged in transit
BJ	Operator Error
BK	Site computer/data logger down
BL	QA Audit
BM	Accuracy check
BN	Sample Value Exceeds Media Limit
BR	Sample Value Below Acceptable Range
CS	Laboratory Calibration Standard
DA	Aberrant Data (Corrupt Files, Aberrant Chromatography, Spikes, Shifts)
DL	Detection Limit Analyses
FI	Filter Inspection Flag
MB	Method Blank (Analytical)
MC	Module End Cap Missing
SA	Storm Approaching
SC	Sampler Contamination
ST	Calibration Verification Standard
TC	Component Check & Retention Time Standard
TS	Holding Time Or Transport Temperature Is Out Of Specs.
XX	Experimental Data

ERG submits data to AQS using qualifier flags to show where the data are with respect to the detection level. A variety of terms and acronyms are used for defining the lowest level that can be detected for each analytical method. These terms and applications are presented below:

- **Quantitation Limits (QL) —** the lowest level at which the entire analytical system must provide a recognizable signal and acceptable calibration point for the analyte.

- **Detection Limits (DL) —** the minimum concentration of an analyte that can be measured above instrument background.

- **MDL —** the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (Part 136, App. B).

- **SQL —** the lowest concentration of an analyte reliably measured within specified limits of precision and accuracy during routine laboratory operating conditions. Normally, the SQL is determined as a multiplier of the method detection limit (e.g., 3.18 times) and is considered the lowest concentration that can be accurately measured, as opposed to just detected.

The qualifier flags associated with quantitation and detection limits are also included in Table 18-1, while Table 18-3 summarizes how they are applied to the data. ERG and DRI MDLs will be uploaded into AQS to provide an accurate representation of the data.

Table 18-3
Summary of Quantitation and Detection Limit Flags and Applications

If Concentration is:	Value to Report	Flag Applied
> SQL	Value	None
> MDL ≤ SQL	Value	SQ
≤ MDL	Value	MD
Not Detected	0	ND

SECTION 19
DATA VALIDATION, VERIFICATION AND ANALYSIS

Many of the processes for verifying and validating the measurement phases of the data collection operation have previously been discussed in Section 18. If these processes as written in the QAPP are followed, and the sites are representative of the boundary conditions for which they were selected, one would expect to achieve the DQOs. However, exceptional field events may occur, and field and laboratory activities may negatively affect the integrity of samples. In addition, it is expected that some of the QC checks will fail to meet the acceptance criteria. This section will outline how ERG will take the data to a higher level of quality analysis by performing software tests, plotting, and other methods of analysis.

19.1 Process for Validating and Verifying Data

19.1.1 Verification of Data

For the analytical data, the entries are reviewed to reduce the possibility of entry and transcription errors. Once the data are transferred to the ERG LIMS database, the data will be reviewed for routine data outliers and data outside acceptance criteria. These data will be flagged appropriately. All flagged data will be "re-verified" to ensure that the values are entered correctly.

19.1.2 Validation

Records of all samples will be retained on file for 5 years, valid or invalid. Information will include a brief summary of why the sample was invalidated along with the associated flags. This record will be available on a network server. When the laboratory analyst reviews the COC forms, he/she will look for possible problems. Filters that have flags related to obvious contamination, filter damage, or field accidents will be examined immediately. Upon concurrence of the associated laboratory analyst and the Program Manager, these samples will be invalidated.

19.2 Data Analysis

Data analysis refers to the process of interpreting the data that are collected. This section will describe how the laboratories will begin to analyze the data to ascertain what the data illustrate and how the data should be applied. The analyst should note any data that needs flags (dilutions, collocate/replicate precision agreement). This is verified by the Task Leader, QA Coordinator, and the Program Manager. The Program Manager has the ultimate authority to invalidate any sample, given acceptable reasons.

19.2.1 Analytical Tests

ERG will employ software programs, described below, to help analyze the data.

Spreadsheet – Select ERG employees perform analysis on the data sets using Excel® spreadsheets (analysts, Task Leaders, and QA reviewers) and Access® databases (AQS data entry). Spreadsheets and databases allow the user to input data and statistically analyze, graph linear data. This type of analysis will allow the user to see if there are any variations in the data sets. In addition, various statistical tests such as tests for linearity, slope, intercept or correlation coefficient can be generated between two strings of data. Time series plots and control charts can help identify the following trends:

- Large jumps or dips in concentrations;
- Periodicity of peaks within a month or quarter; and
- Expected or unexpected relationships among species.

SECTION 20
RECONCILIATION WITH DATA QUALITY OBJECTIVES

The project management team, QA Coordinator, and sampling and analytical team members are responsible for ensuring that all measurement procedures are followed as specified and that measurements data meet the prescribed acceptance criteria. Prompt action is taken to correct any problem that may arise.

20.1 Conduct Preliminary Data Review

A preliminary data review will be performed as discussed in Section 16 to uncover potential limitations to using the data, to reveal outliers, and generally to explore the basic structure of the data. The first step is to review the quality assurance reports. The second step is to calculate basic summary statistics, generate graphical presentations of the data, and review these summary statistics and graphs.

ERG will review all relevant quality assurance reports, internal and external, that describe the data collection and reporting process. Particular attention will be directed to looking for anomalies in recorded data, missing values, and any deviations from standard operating procedures in a qualitative review.

20.2 Draw Conclusions from the Data

If the sampling design and statistical tests conducted during the final reporting process show results that meet acceptance criteria, it can be assumed that the network design and the uncertainty of the data are acceptable.

SECTION 21

REFERENCES

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4. U.S. Environmental Protection Agency. Standard Operating Procedure for the Determination of Lead in PM10 by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Hot Block Dilute Acid and Hydrogen Peroxide Filter Extraction, Method EQL-0512-202.
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7. U.S. Environmental Protection Agency. Federal Advisory Committee Act (FACA).
Can be found at: *http://www.epa.gov/waterscience/methods/det/*

Appendix A

ERG Standard Operating Procedures

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ERG
EASTERN RESEARCH GROUP, INC.

CONFIDENTIAL
Standard Operating Procedure
Procedure Number: ERG-MOR-017
Revision Number: 2
Revision Date: May 12, 2015
Page: 1 of 9

ENGINEERING AND SCIENCE DIVISION

TITLE: Standard Operating Procedure Developing, Documenting, and Evaluating the Accuracy of Spreadsheet Data	EFFECTIVE DATE: MAY 20 2015
REFERENCES: ERG-MOR-009	
SATELLITE FILES: N/A	
REVISIONS: Removed outdated statements, Added document control of spreadsheets	
WRITER/EDITOR: NAME/DATE Donna Tedder 5/20/15	PROJECT MANAGER: NAME/DATE Quia L. Swift 5/20/15
PROJECT QUALITY ASSURANCE MANAGER: NAME/DATE Donna Tedder 5/20/15	NEXT SCHEDULED REVIEW: 1/31/17

1.0 IDENTIFICATION AND PURPOSE

This standard operating procedure (SOP) describes the procedures to follow when developing spreadsheets that are intended to be a project deliverable, not spreadsheets used as a general check. It prescribes the minimum standards to be maintained to help ensure data quality and reproducibility. With the use of the laboratory information management system (LIMS), project deliverable spreadsheet use is somewhat limited.

2.0 MATRIX OR MATRICES

NA

3.0 METHOD DETECTION LIMIT

NA

4.0 SCOPE AND APPLICATION

This SOP applies to all spreadsheets developed during project work at the laboratory located in Morrisville, NC.

5.0 METHOD SUMMARY

To ensure the quality of data delivered to client in the form of a spreadsheet, the developer must consider multiple components of the spreadsheet, including the data, calculations, and presentation of the spreadsheet. The data, subsequent calculations and assumptions must be reviewed by another project team member before the spreadsheet can be finalized and sent to the client.

6.0 DEFINITIONS

Spreadsheet – An electronic table that is used to process or present data. A spreadsheet can be used to store and manipulate data, as well as present data in report-quality, tabular format.

Spreadsheet Developer (Developer) – The person responsible for the overall accuracy and quality of a spreadsheet. The Developer ensures that data are entered correctly and that mathematical functions are accurately executed.

Reviewer – The person not associated with the development of the spreadsheet that verifies the accuracy, completeness, and reasonableness of the data in the spreadsheet.

7.0 INTERFERENCES

NA

8.0 SAFETY

NA

9.0 EQUIPMENT AND SUPPLIES

NA

10.0 MATERIALS

NA

11.0 CHEMICALS, REAGENTS, AND STANDARDS

NA

12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

NA

13.0 CALIBRATION AND STANDARDIZATION

NA

14.0 PROCEDURE

14.1 Summary of Responsibilities

14.1.1 The Spreadsheet Developer:

- Ensures that all original data are transcribed (entered) to the spreadsheet correctly.
- Ensures that all equations used to generate results are entered correctly; ensures that all equations are used appropriately.
- Ensures that all conversion factors and constants used in equations are described.
- Ensures that all sources of original data are referenced in the spreadsheet.
- Ensures that all variables within equations are defined.
- Ensures that all spreadsheet tables are developed (content and format) according to the directions provided by the Project Manager or Task Leader, QAPP or SOP.
- If possible, keeps a log of spreadsheet revisions. If different versions of a spreadsheet are created, the Developer should maintain a log that describes the changes made to the different versions. With two or more people editing one spreadsheet, control can be hard to maintain. One approach is to allot a tab in the spreadsheet where it can be logged who edits the spreadsheet and what was done (short description), including the date of the edits.

14.1.2 The Project Manager or Task Leader:

- Determines when the use of spreadsheets (rather than database technology) are appropriate.
- Determines if a specific format must be used and specifies what information should be included in each spreadsheet.
- Ensures that methods and technical approaches used to produce a desired result are technically sound.
- Ensures that spreadsheet documentation is included in the project file or within the spreadsheet.
- Provides guidance on how to present data in the spreadsheet (based on client's needs, project goals and objectives and QAPP QC acceptance criteria if applicable).

14.1.3 The Reviewer:

- Verifies that the Developer's technical approach is reasonable and logical.
- Verifies that calculations and results are accurate.
- Verifies that documentation is complete and clear.
- Ensures that assumptions and procedures used are reasonable.
- Provides timely, constructive, and direct comments to the Developer.
- Assigns a document version control number (SOP ERG-MOR-009).

14.2 To maintain acceptable data quality, it is important to practice adequate QC measures during the development and review of spreadsheets. The information presented in a spreadsheet should be evaluated to determine if input data are transcribed correctly, calculated results are technically sound, and the final results are reported in a manner that will allow the data to be evaluated.

14.2.1 Spreadsheet Identification:

- Save the spreadsheet using a descriptive label and include the Work Assignment number or unique project number, if applicable, for example, “WA 1-09 Method Detection Limits”.

- Include a title in the spreadsheet, at the beginning. Make the title descriptive enough to clearly identify the data presented and the project.

- All spreadsheets should contain a tab to log the person(s) making edits to the spreadsheet and a short description of the edits, including date of edits, labeled Version Control, or similar.

- Identify the reviewer and the date (month/day/year) the spreadsheet was reviewed in the log tab.

- As needed, include headers or footers that identify the name of the electronic spreadsheet file, the page number, and total number of pages (e.g., Page 1 of 2), and the date the spreadsheet was last revised. The name of the disk or drive on which the file is stored may also be included with the file name.

- Assign a unique name and number to the revised version of the spreadsheet.

- Include a footer or header in the spreadsheet (or a note at the top or bottom of the spreadsheet) that indicates the date the spreadsheet was generated and the revision number, not the date that printing occurred.

14.2.2 Spreadsheet Development:

- Describe all equations, using footnotes or a comments field, where appropriate. (e.g., if gram/kilogram are being converted to pound/ton, the equation performing the calculation should be explained as: “Convert g/kg to lb/ton: 1 g/kg x 1 lb/453.59 g x 1 kg/1,000 g x 453.59g/lb x 2,000 lb/ton, which is equivalent to multiplying by 2”). If detailed descriptions exist in project notebooks, then a reference to that notebook (e.g., notebook and page number) should be made in the comments field.

- Identify any constants or conversion factors used.

- Avoid using specific values in equations, except for easily recognizable conversion factors or constants. Enter values within a cell. Equations that use the value should reference the cell.

- Hand (manually) verify equation cells.

- Protect verified equation cell regions of spreadsheet to avoid accidentally over writing.

14.2.3 Supporting Data Requirements

The original raw data used in the spreadsheet should be retained in the project file and in the project archive.

14.2.4 Project Data File Requirements

Maintain an electronic backup copy at an identified location on the network and in hard copy in the project file.

15.0 CALCULATIONS

NA

16.0 QUALITY CONTROL

Spreadsheet Quality Control Responsibilities

16.1 The Spreadsheet Developer:

- Checks the accuracy of data transcriptions.

- If the spreadsheet is being given to someone who will make revisions or enter data, data cells that should not be changed should be locked. Locking data cells in this manner will help prevent inadvertent changes to the spreadsheet.

16.2 The Project Manager or Task Leader:

- Determines the level of QC necessary. For example, the Project Manager or Task Leader must decide if all data points and all calculations should be checked, or if only a percentage should be checked. It may be appropriate to initially check a percentage and, based on the number of discrepancies identified, decide if additional QC is required.

16.3 The Reviewer:

- Verifies at least one calculation for each equation or combination of equations used.
- Verifies the accuracy of total values, means, and statistical evaluations of the data.
- With the Project Manager or Task Leader, determines the amount of data to check; the number of errors found will dictate the amount of data evaluated for accuracy. The higher the error rate, the more data points to be checked. If numerous errors are found, the spreadsheet should be returned to the data generator with a note that includes a description of the review procedure and percentage of errors found. The error rate is a good indicator of the accuracy of all of the information in the spreadsheet. If needed, the site QA Coordinator should be consulted for guidance in determining the most effective way to determine which and how many values to recalculate.
- Verifies that original data were input correctly.
- Evaluates the technical soundness of methods and approaches used.
- Ensures that equations in the spreadsheet produce the correct result and that equations were entered into the spreadsheet accurately.
- Ensures that adequate documentation is included in the spreadsheet and that the documentation supports the data in the spreadsheet.
- Discusses all discrepancies with the Developer and Project Manager or Task Leader, as appropriate. Actual spreadsheet errors identified by the Reviewer should be corrected by the Developer.
- Summarizes the review, inputs their findings in the Version Control tab and comments on how to make changes to calculations in the Description tab indicating the errors or problems found, and the recommendations for revisions. The summary should also include the reviewer's name, data of QC review (month/day/year), name of file, type of data reviewed, and the percentage of each type reviewed in the Version Control tab.
- Assigns a document version control number (SOP ERG-MOR-009).

17.0 PREVENTION

NA

18.0 CORRECTIVE ACTION

NA

19.0 WASTE MANAGEMENT

NA

20.0 MAINTENANCE

NA

21.0 SHORTHAND PROCEDURE

1. Label a tab as Version Control and enter name, date and description of what was done
2. Label a tab as Description and include information about the project, the source of the raw data and what calculation are to be included in the spreadsheet. Define constants and equations here
3. In the working tab add a title on top of the page that includes a description of the table content
4. Ensure all raw data was entered correctly
5. Hand calculate some data points to review the spreadsheet results
6. Pass the spreadsheet to Technical Reviewer
7. The Technical reviewer will enter any comments or changes to the Control Version tab and provide a revised version to the Developer and Manager

22.0 DOCUMENTATION AND DOCUMENT CONTROL

All relevant information concerning Version Control, Description of calculations, should be included in separate tabs within the spreadsheet. However, accompanying documentation can be used such as hand calculations, more in detailed project descriptions and raw data collection comments. A separate tab within the spreadsheet will contain information about the edits to the spreadsheet contents, indicating editor's name, date, short description of edits including reviews. Once reviewed the spreadsheet is assigned a document version control number (SOP ERG-MOR-009).

23.0 REFERENCES

Food and Drug Administration (FDA) Good Laboratory Practice (GLP) standards, 40 CFR Part 58.81(a), Subpart E – Testing Facility Operation, Standard Operating Procedures

Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) GLP standards, 40 CFT Part 160.81(a), Subpart E – Testing Facility Operation, Standard Operating Procedures

Toxic Substance Control Act (SCA) GLP standards, 40 CFR Part 792.81(a), Subpart E – Testing Facility Operation, Standard Operating Procedures

24.0 TABLES, DIAGRAMS, FLOWCHARTS, VALIDATION DATA

NA

ERG
EASTERN RESEARCH GROUP, INC.

CONFIDENTIAL
Standard Operating Procedure
Procedure Number: ERG-MOR-022
Revision Number: 1
Revision Date: May 8, 2012

ENGINEERING AND SCIENCE DIVISION

TITLE: Standard Operating Procedure for the Preparation of Standards in the ERG Laboratory	EFFECTIVE DATE: May 21, 2012
REFERENCES: ERG-MOR-031, ERG-MOR-033, ERG Health and Safety Manual	
SATELLITE FILES: All	
REASON FOR REVISION: Update to increase applicability	
DATE OF LAST REVIEW:	NEXT SCHEDULED REVIEW:
WRITER: NAME/DATE Donna Tedder 5/16/12	TECHNICAL DIRECTOR: NAME/DATE Julie L. Swift 5/27/12
QUALITY ASSURANCE COORDINATOR: NAME/DATE Donna Tedder 5/16/12	MANAGER: NAME/DATE Julie L. Swift 5/27/12

1.0 IDENTIFICATION AND PURPOSE

This standard operating procedure (SOP) presents the procedure to be followed when preparing standards for use in the Sample Preparatory Laboratories. This procedure should be followed to ensure that only high-quality data are generated in the laboratory.

2.0 MATRIX OR MATRICES

This SOP addresses procedures used to prepare liquid standards/stocks from solid or liquid chemicals.

3.0 METHOD DETECTION LIMIT

NA

4.0 SCOPE AND APPLICATION

This SOP is a general guidance to standard preparation. Some procedures may not be applicable to all analyses preformed at this location. The SOP was originally intended for use in the Organic Sample Preparation Laboratory however, the general procedures outlined in this SOP can be applied to preparing standards for any method.

The calibration standards are used to establish or verify the instrument calibration which is used to calculate the concentrations of the analytes.

The internal standard solution is used to measure the relative responses of analytes (and surrogates). When used, internal standards are added to all samples, standards, and quality control (QC) samples at the same concentration.

The matrix spiking solution is added to a matrix prior to processing and is used to assess the efficiency of the preparative technique and analysis methodology.

The surrogate spiking solution containing one or more compounds, different from the method analytes but similar in physical and chemical behavior, that can be used to measure extraction and analysis efficiency without interfering with the analysis.

5.0 METHOD SUMMARY

NA

6.0 DEFINITIONS

COA	certificate of analysis
g	gram
ID	identification
LIMS	laboratory information management system
µg	microgram
µL	microliter
mg	milligram
mL	milliliter
ng	nanogram
QA	quality assurance
QC	quality control

7.0 INTERFERENCES

To ensure the standards are free from interfering contaminants, all glassware should be cleaned according to the glassware cleaning SOP (ERG-MOR-031). Syringes should be

cleaned by rinsing the plunger and the cylinder at least 10 times with the solvent to be used.

8.0 SAFETY

Follow normal laboratory safety procedures as outlined in the ERG Health and Safety Manual. The appropriate personal protection should be used during the preparation of standards/stocks.

9.0 EQUIPMENT

NA

10.0 MATERIALS

The following equipment should be obtained: volumetric glassware, including volumetric flasks and pipettes, and syringes in the appropriate size(s). All volumetric glassware should be class A. A balance should be used that is appropriate to the degree of precision desired. In most cases, either a four- or a five-place balance should be used. Document the calibration check in the balance notebook prior to using the balance.

11.0 CHEMICALS, REAGENTS, AND STANDARDS

All reagents used to prepare standards should be reagent grade or better and be identified by their manufacturer and lot number. Solvents should be "high purity" or "pesticide residue grade." If chemicals used in standards are less than 96% pure, then the percentage of purity must be factored into the calculations. All chemicals should be logged into the laboratory following the chemical inventory SOP (ERG-MOR-037). As chemicals are depleted, the chemical inventory should be updated so that chemicals can be replaced as needed. If a chemical has an expiration date, it should not be used past that date.

12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

Store the standard/stocks as recommended by the vendor, or by the method.

13.0 CALIBRATION AND STANDARDIZATION

NA

14.0 PROCEDURE

14.1 Standards Preparation - Stock Solutions

This section gives a general explanation of how to prepare standards.

14.1.1 For small amounts of liquids or solids, the chemicals should be measured by weight. Tare a small vial. Add the desired amount of chemical to the vial. Record the final weight of the vial with the chemical.

14.1.1.1 For solids, the desired chemical can be added with a small spatula. Calculate the amount needed to obtain the desired concentration using the following equation:

Final Concentration * Total Volume = Weight Added

Example: A final concentration of 1000 µg/mL is required for a standard. 100 mL of the solution is to be made up.

1000 µg/mL * 100 mL = 100,000 µg (100mg)

14.1.1.2 For liquids, the desired chemical can be added with a syringe, a pipette, or can be weighed. If the liquid is to be added with a syringe or pipette, calculate the volume needed with the following equation:

Density (µg/mL) * X mL Neat/Final Volume (mL) = µg/mL

Example: A final concentration of 1000 ng/µL is required for a standard. 100 mL of the solution is to be made up. The density of the liquid is 0.500 g/mL.

The approximate amount of liquid required is shown below.

500,000 µg/mL * X mL Neat/1 mL = 1000 µg/mL

X = 0.002 mL (2 µL)

14.1.2 Dissolve each of the weighed chemicals in the appropriate solvent. Some chemicals may not dissolve readily and may require sonication to promote dissolution.

14.1.3 Fill a clean, solvent-rinsed volumetric flask approximately ½ to ⅔ full of the solvent to be used for the standard. Add the dissolved chemicals from Step 14.1.2 to the volumetric flask. Rinse each vial three times with the solvent to make sure that all of the dissolved chemical is transferred to the volumetric flask.

14.1.4 Carefully fill the volumetric flask up to the final desired volume with the solvent. The bottom of the meniscus should be at the volume line on the volumetric flask. Stopper the volumetric flask with a ground glass stopper and invert the flask 10 to 15 times to make sure that the solution is well mixed.

14.1.5 Transfer the stock standard to a vial with a Teflon®-lined screw cap lid for storage in the standards refrigerator.

14.2 Working Standards Preparation - Dilutions of Stock Solutions

This section gives a general explanation of how to prepare dilutions of standard solutions.

14.2.1 When the concentration of a chemical is lower than can be obtained using the methods described above, dilutions of the stock solutions can be made. Initially, a calculation must be done to determine what dilution is needed using the equation below:

14.2.2 Fill a solvent-rinsed volumetric flask approximately ½ to ⅔ full with the solvent to be used for the standard. Measure the amount of the stock solution needed with a volumetric pipette or with a syringe. Add the stock solution to the solvent in the volumetric flask, and dilute to the volume needed with the solvent required.

14.2.3 Transfer the working standard to a vial with a Teflon®-lined screw cap lid for storage in the standards refrigerator.

15.0 CALCULATIONS

Many of the necessary calculations are presented in Section 14.0.

Final Concentration = Weight Added/Total Volume

Final Concentration = (Volume Added * Concentration)/Total Volume

In all calculations, check the units carefully to make sure that the final concentration is given in the proper units.

Note: If a chemical used was less than 96% pure, the final concentration should be multiplied by the purity to obtain the actual concentration. For example, if a chemical used in a stock solution is 95% pure, the final calculated concentration of the stock solution should be multiplied by 0.95.

To calculate the molarity of a standard solution:

Molarity = Number of Moles/Total Volume (liters)

Note: If solute is a percentage solution, the weight percentage must be taken in account.

16.0 QUALITY CONTROL

16.1 The preparation of all standards and stocks must be documented including the identification (ID) number, type, concentration, amount of solute, total volume, preparer, preparation dates, expiration dates, stock or chemical lot numbers, and solvent lot numbers.

16.2 When preparing a standard with calculations out of the normal for that analysis, the calculations must be validated by Task Lead or quality assurance (QA) staff before proceeding.

16.3 The following expiration dates should be used for prepared standards/stocks if one is not indicated in the associated method:

- Stock solutions: 1 year from date of preparation;
- Standard solutions: 6 months from date of preparation;
- Standard solutions expiration dates cannot extend past the expiration date of the stock solution from which they were prepared.

17.0 PREVENTION

When possible, minimize the amount of chemicals used in the preparation of the standards/stocks. Prepare the smallest volume of standard/stock that can be used before the expiration date.

18.0 CORRECTIVE ACTION

NA

19.0 WASTE MANAGEMENT

Hazardous waste disposal is discussed in SOP ERG-MOR-033.

20.0 MAINTENANCE

NA

21.0 SHORTHAND PROCEDURE

NA

22.0 DOCUMENTATION AND DOCUMENT CONTROL

On each standard vial, a label must be affixed with the standard name (e.g., base, neutral, acid, or BNA surrogate), identification (ID) number, the standard expiration date, and the initials of the person who made the standard.

The preferred method of standards documentation is the laboratory information management system (LIMS). The LIMS system has a Standards section to document ID number, type, concentration, amount of solute, total volume, preparer, preparation dates, expiration dates, lot numbers of chemical and solvent, and link the information to the appropriate QC samples. For commercial standards or stocks, the certificate of analysis (COA) can be scanned, imported, and linked with the appropriate standard/s. A sample Standards page from the LIMS system is presented in Figure 1.

A notebook can be used for standards and stocks. Write the standard number and description so they are clearly visible, preferably at the top of a page in the notebook. Follow this with a short description of how the standard was made, including the amounts added, the glassware used, and the solvent(s) used. If a solid compound was used, note how it was dissolved. The brand and lot numbers of all chemicals, including the solvents, must be recorded as well as the equations used to calculate the final concentrations. If a stock solution was used, also record the concentration. Lastly, record the final concentrations of the chemicals in the standard solution.

23.0 REFERENCES

NA

24.0 TABLES, DIAGRAMS, FLOWCHARTS, VALIDATION DATA

Figure 1. Sample LIMS Standards Page

Analytical Standard Record

Eastern Research Group

2D27011

Description:	IO3.5 Teflon Calibration Standard 1	Expires:	08/03/12
Standard Type:	Calibration Star	Prepared:	04/27/12
Solvent:	0.3% HCl, 1.11% HNO3, 0.1% HF	Prepared By:	Randy Mercurio
Final Volume (mls):	500	Department:	Inorganics
Vials:	1	Last Edit:	04/27/12 11:27 by RJM

Analyte	CAS Number	Concentration	Units
Yttrium	NA	0.07	ug/mL
Scandium		0.11	ug/mL
Indium		0.07	ug/mL
Gold	NA	5	ug/mL
Gallium	na	0.2	ug/mL
Bismuth	NA	0.07	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
1A25002	Fisher Optima Hydrofluoric Acid;	01/25/11	** Vendor **	03/31/13	02/21/11 12:52 by RM	0.5
1H22003	10,000 mg/L Gold Stock; Expires	08/22/11	** Vendor **	02/18/13	08/22/11 13:48 by RM	0.25
1I19003	J.T. Baker Ultrex II Nitric Acid;	E09/19/11	** Vendor **	09/17/12	09/19/11 14:58 by RM	5.55
1I29009	Aristar Ultra Hydrochloric Acid;	E09/29/11	** Vendor **	05/21/13	09/29/11 14:47 by RM	1.5
2A04003	ICP-MS IO3.5 Internal Standard;	I01/04/12	Randy Mercurio	08/03/12	01/04/12 11:17 by RM	1

Reviewed By Date

Page 1 of 1

ERG
EASTERN RESEARCH GROUP, INC.

CONFIDENTIAL
Standard Operating Procedure
Procedure Number: ERG-MOR-033
Revision Number: 4
Revision Date: December 10, 2012
Page: 1 of 16

ENGINEERING AND SCIENCE DIVISION

TITLE: Standard Operating Procedure for Hazardous Waste	EFFECTIVE DATE: 2/4/13
REFERENCES: 40 CFR Parts 260 through 299; ERG-MOR-037, ERG-MOR-038, Hazardous Waste Management Plan	
SATELLITE FILES: GC, GC/MS, ICP-MS, Prep	
REASON FOR REVISION: Updated to agree with Hazardous Waste Management Plan updates	
WRITER: NAME/DATE Tracey Foote 2/4/13	PROJECT MANAGER: NAME/DATE Julie L. Swift 2/5/13
QUALITY ASSURANCE COORDINATOR: NAME/DATE Donna Tedder 2/4/13	NEXT SCHEDULED REVIEW: 1/31/14

1.0 IDENTIFICATION AND PURPOSE

The purpose of this SOP is to provide guidelines for the correct handling and disposing of hazardous waste at the ERG analytical laboratory located at 601 Keystone Park Drive, Suite 700, Morrisville, NC. ERG's analytical laboratory is licensed as a small-quantity hazardous waste generator (SQG). ERG will maintain the SQG status as specified by our Environmental Management System (EMS). These guidelines have also been developed to ensure compliance with federal and North Carolina hazardous waste regulations. A description of the overall program is contained in the Hazardous Waste Management Plan. Also included in the Hazardous Waste Management Plan are the emergency procedures listed in the Emergency Preparedness and Response Plan section (Appendix C).

2.0 MATRIX OR MATRICES

N/A

3.0 METHOD DETECTION LIMIT

N/A

4.0 SCOPE AND APPLICATION

This document presents procedures for personnel who generate, handle, or are involved in the disposal of waste.

5.0 METHOD SUMMARY

N/A

6.0 DEFINITIONS

A hazardous waste handler is an individual who regularly transfers hazardous waste from the satellite accumulation points (SAPs) to the 180-day storage room.

A hazardous waste generator is any laboratory personnel who generates hazardous waste.

The health and safety coordinator (HSC) is responsible for managing health and safety programs at the ERG laboratory.

A satellite accumulation point (SAP) is a designated location near the point of generation where waste is temporarily stored.

A small-quantity generator (SQG) is a facility that generates less than 1,000 kg (2,200 lbs) of hazardous waste and less than 1 kg (2.2 lbs) of acutely hazardous waste in a calendar month.

7.0 INTERFERENCES

N/A

8.0 SAFETY

Proper personal protective equipment (PPE), as outlined in the Hazardous Waste Management Program, the SOP for PPE (ERG-MOR-038), and/or Section 14.2.3 of this document, is required to be worn when handling hazardous waste.

9.0 EQUIPMENT

N/A

10.0 MATERIALS

N/A

11.0 CHEMICALS, REAGENTS, AND STANDARDS

N/A

12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

Because waste disposal contractors are not currently permitted to accept dioxin-containing wastes, there are no disposal options for the dioxin-containing chemicals that ERG has archived. The following criteria define dioxin waste. The dioxin waste categories described below are subject to the RCRA regulations contained in 40 CFR Parts 261, 264, 265, 270, and 271:

- Waste containing tetra- and pentachloro-dibenzo-*p*-dioxins and -dibenzofurans;
- Process wastes from the manufacturing use of tetra-, penta-, or hexachlorobenzenes under alkaline conditions;
- Wastes from the production and manufacturing of tri-, tetra-, and pentachlorophenols and their chlorophenoxy derivatives;
- Discarded, unused formulations containing tri-, tetra-, and pentachlorophenols or formulations containing compounds derived from these chlorophenols; and
- Soil contaminated with any of the wastes listed above.

The dioxin-containing analytical standards that ERG has on-site are to remain segregated and stored in the refrigerator in the 180-day storage room, which requires a key for entry.

13.0 CALIBRATION AND STANDARDIZATION

N/A

14.0 PROCEDURE

Any chemical or item contaminated with a chemical, including samples, should be initially considered hazardous waste until the Project Manager and/or HSC determines otherwise.

14.1 Determination, Classification, and Packaging of Hazardous Waste

14.1.1 Determination

Not all waste is hazardous by EPA's definition. Project Managers, with the help of the HSC or designee, must determine whether generated waste is hazardous. Section 24.1 has a summary of questions to help determine the applicability of the regulations from Title 40 CFR 262.11.

14.1.2 Classification

The Morrisville facility typically generates the following classifications of hazardous wastes:

- F = from non-specific sources;
- U = hazardous compounds from discarded commercial products;
- D = characteristic (toxic, corrosive, ignitable, or reactive) wastes; and
- P = acutely hazardous from discarded commercial products.

The waste disposal contractor uses these classification codes to correctly group and dispose of the waste. The HSC or designee, while reviewing the hazardous waste manifests, ensures that the correct classification codes are used for waste generated at ERG laboratory.

14.1.3 Packaging

Hazardous waste generated by ERG is disposed of in two major packaging categories:

- Bulked wastes are produced routinely in relatively large volumes. This waste group includes spent solvents, aqueous acids, discarded environmental samples (large volumes), and contaminated sample vials. Approved hazardous waste handlers bulk these wastes every 4-6 weeks. Note: although these are considered bulked wastes for our purposes, for DOT purposes, these are still non-bulk containers (49 CFR 171.8)

- Lab pack wastes are chemical reagents contained in jars, vials, jugs, bottles, and cans. This waste is best disposed of in its primary container. Lab packs are 20- or 30-gal fiber drums that hold individual containers of chemically compatible waste. Individual containers are packed in a fiber drum surrounded by layers of

absorbent vermiculite material, which prevents shifting or breakage of small containers during transport.

Hazardous Waste Handlers should use the following list to determine which types of wastes can be bulked or lab-packed. The waste types with an asterisk (*) are typical wastes generated by the ERG facility.

- Bulked Waste
  - Halogenated flammable solvents*
  - Nonhalogenated flammable solvents*
  - Inorganic acids*
  - Organic acids
  - Aqueous waste contaminated with hazardous waste

- Lab-Packed Waste
  - Glass and metal contaminated with hazardous waste*
  - Paper and plastic contaminated with hazardous waste*
  - Poison A (poisonous gases)
  - Flammable gases
  - Non-flammable gases
  - Flammable liquids (i.e., those that cannot be bulked with solvents)
  - Oxidizers
  - Flammable solids
  - Corrosive liquids
  - Corrosive solids
  - Irritants
  - Combustible liquids

When determining whether to group the waste as either bulked or lab-packed waste, Hazardous Waste Handlers should consider the following factors:

- Bulk whenever possible (it is less expensive.). Use the same drum of an appropriate size for compatible wastes until it is full (it is less expensive.).

- For lab-packed waste, set the waste aside, generally in the chemical storage (Room 139) or a designated location in the labs. The waste disposal contractor packages these wastes at the time of pick-up.

- Do not mix wastes that can be bulked with lab-packed wastes. If the two are mixed, all the waste will need to be lab-packed.

- Never mix non-hazardous waste with hazardous waste. The result is a larger quantity of hazardous waste.

14.2 Hazardous Waste Collection and Storage Procedures

ERG is allowed to store hazardous waste for up to 180 days, provided safe storage practices, as defined by the state and federal governments, are followed. The dedicated storage area is Room 139, the 180-day storage area. The 180-day counting begins when the first drop of waste is transferred to the 180-day storage area.

14.2.1 Collection and storage of in-laboratory waste

Hazardous Waste Generators must follow these steps to store waste properly in the SAPs:

- Label all containers with Hazardous Waste labels, including the constituents or at least the process generating the wastes (i.e., TO-13 waste) on the bottles;

- All containers of hazardous waste must be sealed except when pouring;

- A maximum of 55 gal of hazardous waste and 1 kg acutely toxic waste is allowed at any one SAP; and

- If waste storage presents a clutter problem in the laboratory hoods, contact a Hazardous Waste Handler to transfer the waste to the 180-day storage area.

The following are additional requirements for properly storing specific types of wastes in the SAP and 180-day storage area:

- Glass and Metal Waste

  - All contaminated glass and metal waste will be collected in closed containers labeled Waste Glass and Metal. These containers must be stored in the SAP. A Hazardous Waste

Handler will periodically dispose of these in fiber drums located in the 180-day storage area.

- Place uncontaminated broken pipettes, test tubes, and other items of broken glass in the designated Broken Glass bins or containers, not in the regular trash cans. This practice will prevent the janitorial staff from cutting themselves on broken laboratory glassware.

- Solvent Waste

  - All organic solvent wastes are collected in 4 liter amber glass bottles in designated SAP areas. These are often transferred from the hood to a flammables or vented cabinet in the same SAP area when full before being bulked in the 180-day storage area.

- Contaminated Paper and Plastic Storage

  - Paper and plastic waste material that is contaminated with chemicals (hazardous or nonhazardous) will be placed inside thick plastic bags and stored inside the fume hood until the bag is transported to the 180-day storage area by a Hazardous Waste Handler.

- Aqueous Waste and Environmental Samples

  - If possible, environmental samples and other aqueous wastes should be stored in the original glass sample containers. NOTE: No Aqueous/Acid waste will be put in the solvent waste drum.

  - When storing liquid waste, an adequate supply of spill materials (vermiculite and/or absorbent padding) must be on hand to control and clean up potential spills. Refer to the Hazardous Waste Management Program for proper spill procedures.

- Acids and Corrosives

  - All acids and other corrosives must be stored in a separate area with adequate secondary containment to ensure that if

the chemical spills or leaks, it will not come into contact with containers of organic chemicals.

- When not in use or considered waste the acids or corrosives are stored in segregated and labeled storage areas under the laboratory hood in the storage cabinets or in vented flammables cabinets.

14.2.2 Collection of Field-Generated Hazardous Waste

Project Managers are responsible for planning the collection, storage, and transportation of hazardous waste generated in the field. Typical field wastes will include:

- Discarded PPE; and
- Chemicals used on site (e.g., glassware rinsate).

Prior to the start of the job, Project Managers shall arrange for the client to accept the waste or contract with a waste disposal firm to pick up hazardous waste from the site.

NOTE: Because ERG does not have the necessary DOT license, employees are not allowed to transport hazardous wastes.

14.2.3 Bulking waste in the 180-day storage area

- General Requirements

  General Requirements for the Hazardous Waste Handlers to transfer the waste to 180-day storage area are:

  - All containers must be approved hazardous waste containers.

  - Use a dolly or cart to transport accumulated waste from the SAPs to the 180-day storage area. If one is not available, carry all liquid waste inside secondary containers. Paint cans, buckets, and plastic tote bottles are acceptable secondary containers.

- Prior to pouring waste materials, all flammable waste drums must be grounded with a clip wire to a grounding rod located in the area.

- For all hazardous waste delivered to the 180-day storage area, the following information must be recorded in the disposal logbook (Refer to Section 24.2 for a sample page):

  - Date;
  - Quantity of waste;
  - Waste description;
  - Date disposed;
  - Initials of personnel disposing of waste.

- Proper Packaging and Labeling

All wastes placed in the same container must be compatible with each other and the material with which the drum is constructed. The container must be labeled with a yellow hazardous waste label and the information on the label completed to include:

- Description of waste;
- Generator's name and address;
- EPA identification number (found in 40 CFR); and
- Accumulation start date.

All containers and boxes of chemical waste to be disposed of in a lab pack must be marked with the following information:

- Name of the generator (ERG employee responsible for waste);
- Project or process description (i.e., TO-13 waste); and
- Date that the waste was placed into the 180-day storage area.

- Proper Handling Procedures

  The Hazardous Waste Handlers disposing of hazardous waste must use proper personal protection. PPE required for pouring liquid wastes are defined below:

- Pouring acids: Face shield (or full-face respirator), gloves, lab coat, long pants, and closed-toe shoes must be worn at all times.

- Pouring solvents: Respiratory protection, eye protection (unless full-face respirator is worn), lab coat, long pants, and protective gloves must be worn at all times.

Hazardous Waste Handlers should never dispose of waste alone; always dispose of the waste with two people ("buddy system") in case of an emergency. An alarm system is installed and tested annually to notify other laboratory personnel if a problem occurs.

14.3 Disposal of Non-Hazardous Waste

Only limited types of waste may be poured down the sanitary sewer or placed in municipal waste dumpsters. If a laboratory employee has any doubt on proper disposal of any type of waste, the employee should check with the one of the Project Managers.

The Morrisville laboratory routinely disposes and recycles glass containers. The containers are first allowed to dry under a laboratory hood to evaporate remaining chemical residues, and then are triple rinsed with water. The glass bottles are then either recycled (if they are reagent bottles) or disposed of in the dumpster (if the bottles previously contained solvent waste or acids).

15.0 CALCULATIONS

N/A

16.0 QUALITY CONTROL

16.1 Performance

Hazardous waste regulations require that the dedicated storage room (the 180-day storage room) is inspected on a weekly basis by the HSC or designee. This inspection has historically been performed every Tuesday. In addition, the HSC or designee also inspects the SAPs on a weekly basis to verify safe waste storage practices and track waste accumulation levels. Periodic inspections performed by Lab Managers are held to ensure the procedures are followed. Periodic training is held to ensure personnel remain familiar with the procedures.

16.2 Data Assessment

All waste storage procedures in the ERG laboratory will be reviewed by the ERG Hazardous Waste Specialist and periodically subject to inspection by an inspector from the North Carolina Division of Solid Waste Management.

17.0 PREVENTION

N/A

18.0 CORRECTIVE ACTION

N/A

19.0 WASTE MANAGEMENT

19.1 Waste Minimization

ERG will maintain the SQG status as required by our EMS. To minimize waste, ERG maintains a chemical inventory database which must be consulted prior to the purchase of any chemical. The database was created to minimize waste and to track materials that are in stock to prevent duplicate orders, and thus avoid unnecessary chemical storage and waste generation. See ERG-MOR-037 for more information on the chemical inventory procedures.

19.2 Waste Management Organization

The waste management program involves the following personnel, each with a set of specific responsibilities:

19.2.1 Project Manager Responsibilities

- Adequately plan and budget for hazardous waste management; and
- Ensure the laboratory is in compliance with all applicable regulations.
- Project Manager is responsible for ensuring that project and lab personnel:
  - Correctly label hazardous waste;

- Segregate, collect, and store waste properly in the laboratory and in the field;
- Have access to and wear appropriate PPE; and
- Are ready to respond to emergencies.

- Give accurate records to project secretary that document worker training in hazardous waste handling procedures; and

- Ensure that adequate supplies are available and procedures are in place for prompt emergency response.

19.2.2 Health and Safety Coordinator (or designee) Responsibilities

- Conduct weekly inspections of both the SAPs and the 180-day storage area to ensure that employees are disposing of hazardous waste in a timely manner to prevent excess and unsafe accumulation; complete and maintain inspection logs.

- Compile an inventory list of wastes to be picked up and coordinate with the hazardous waste disposal contractor;

- Review and sign manifests prepared by the hazardous waste disposal contractor prior to off-site shipment of hazardous waste and ensure the signed manifests are returned within 60 days after the disposal of the wastes;

- Conduct periodic hazardous waste training for all personnel working with hazardous waste;

- Complete and file all reports with the State of North Carolina, any disposal States as required, and EPA; and

- Retain all records.

19.2.3 Waste Generator Responsibilities

Waste generators are responsible for:

- Disposing of waste in a timely fashion, labeling each type of waste as it is generated;

- Segregating waste, placing in appropriate containers, and storing in designated storage locations (SAPs);

- Being familiar with emergency response procedures, including containment and clean-up of hazardous waste spills and the location of emergency flushing facilities; and

- Notifying the hazardous waste handler when waste needs to be moved to the 180-day storage area.

19.2.4 Hazardous Waste Handler Responsibilities

In addition to having all the Hazardous Waste Generator responsibilities (Section 19.2.3), Hazardous Waste Handlers are responsible for:

- Safely transferring hazardous waste from the SAPs to the 180-day storage area (see Section 14.2.3), including using the "buddy system" and wearing appropriate PPE during waste bulking/transfer operations;

- Labeling each container in the 180-day storage area;

- Entering all wastes transferred to the 180-day storage area into the Waste Disposal Log;

- Notifying the HSC when one month remains before disposal is required of the waste in the 180-day storage area;

- Notifying the disposal contractor to order additional disposal containers and spill cleanup supplies for the 180-day storage area as needed; and

- Cleaning up any spills in the 180-day storage area.

20.0 MAINTENANCE

N/A

21.0 SHORTHAND PROCEDURE

N/A

22.0 DOCUMENTATION AND DOCUMENT CONTROL

The Waste Disposal Log is located in the 180-day storage room (Room 139). All 180-day and SAP inspection logbooks currently in-use are stored in the Supply Room (Room 102). Archives of these logbooks are kept indefinitely in the Data Archives Room (Room 137). Signed hazardous waste manifests are stored in the Supply Room (Room 102) in a white binder labeled "ERG's Hazardous Waste Records," as instructed in SOP 089, "SOP for Hazardous Waste Inspections and Recordkeeping".

23.0 REFERENCES

N/A

24.0 TABLES, DIAGRAMS, FLOWCHARTS, VALIDATION DATA

24.1 Determination of Hazardous Waste

24.2 Waste Disposal Logbook

SECTION 24.1
Determination of Hazardous Waste

In Title 40 CFR 262.11, the regulations provide the following list of questions for proper identification of waste:

- Is the waste excluded from regulation under Title 40 CFR 261.4?
- Is the waste listed in Subpart D (40 CFR 261.30 through 261.35)?
- If the waste is not listed, does it exhibit any of the characteristics listed in Subpart C, Characteristics of Hazardous Wastes (40 CFR 261.20 through 261.24)?

See the referenced regulations for more hazardous waste determination details.

SECTION 24.2
Waste Disposal Logbook

Eastern Research Group, 601 Keystone Park Drive
Waste Disposal Logbook

Date	Operator Intitals	Waste Type	Volume Added (gal)	Cumulative Volume (gal)	Waste Type	Volume Added (gal)	Cumulative Volume (gal)	Waste Type	Volume Added (gal)	Cumulative Volume (gal)	Waste Type	Volume Added (gal)	Cumulative Volume (gal)
Drum #													

Date Started:_____ Date Waste Picked up:_____ Initials:_____

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CONFIDENTIAL
Standard Operating Procedure
Procedure Number: ERG-MOR-045
Revision Number: 8
Revision Date: Feb. 16, 2015
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ENGINEERING AND SCIENCE DIVISION

TITLE: Standard Operating Procedure for Sample Receipt at the ERG Chemistry Laboratory	EFFECTIVE DATE: APR 28 2015
REFERENCES: ERG-MOR-079	
SATELLITE FILES: Shipping/Receiving	
REASON FOR REVISION: Minor Updates	
WRITER/EDITOR: NAME/DATE Donna Tedder 4/27/15	PROJECT MANAGER: NAME/DATE Gabriel Swift 4/28/15
QUALITY ASSURANCE MANAGER: NAME/DATE Donna Tedder 4/27/15	NEXT SCHEDULED REVIEW: 1/31/2016

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to formalize the laboratory procedures for sample receiving and sample log-in. This procedure also documents the sample acceptance policy.

2.0 MATRIX OR MATRICES

This SOP applies to samples of any matrices received at the ERG Research Triangle Park Laboratory. Sample acceptance policies are included for the common sample matrices.

3.0 METHOD DETECTION LIMIT

Not applicable.

4.0 SCOPE AND APPLICABILITY

All samples received in the shipping/receiving area of the ERG Research Triangle Park Laboratory must be handled and distributed following these procedures. In most cases, the samples are collected on/in media that was prepared, labeled and shipped to the site

by the ERG Research Triangle Park Laboratory. If samples are received that do not meet these requirements appropriate corrective action must be taken.

5.0 METHOD SUMMARY

When samples arrive, they are unpacked, inspected, accepted or voided and logged-in into the Laboratory Information Management System (LIMS). The information from the chain-of-custody (COC) forms for each batch of samples received at ERG is entered into the LIMS according to its respective project number (ERG-MOR-079). Information relative to the samples (number, type, collocated or duplicate sample number, analysis to be performed) should be provided prior to arrival by the Task Leader (TL) or Project Manager (PM).

6.0 DEFINITIONS

COC	Chain-of-Custody
C	Collocated
D	Duplicate
FB	Field Blank
Hg	Mercury
IR	Infrared
LIMS	Laboratory Information Management System
NIST	National Institute of Standards and Technology
P	Primary
PM	Project Manager
PUF	Polyurethane Foam
SOP	Standard Operating Procedure
SVOC	Semivolatile Organic Compounds
TL	Task Leader

7.0 INTERFERENCES

Samples should be handled in a way to avoid contamination of samples.

8.0 SAFETY

8.1 The TL or PM should provide information on samples prior to their arrival so that the proper safety precautions can be taken.

8.2 Liquid samples should be handled with gloves; if a sample container is broken, it should be transferred immediately to a fume hood for proper handling and disposal.

9.0 EQUIPMENT

9.1 Heiss Gauge – capable of being zeroed and reading the vacuum or pressure of canister samples; or Weiss Gauge – capable of reading the vacuum or pressure of canister samples and calibrated annually by manufacturer.

9.2 IR Thermal sensor gun – calibrated annually by manufacturer and capable of measuring temperatures from 4 to -25 °C.

10.0 MATERIALS

Not applicable.

11.0 CHEMICALS, REAGENTS, AND STANDARDS

Not applicable.

12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

12.1 Canister samples are stored at room temperature in the air toxics laboratory.

12.2 Carbonyl cartridge samples are stored in refrigerator R-11.

12.3 PUF/XAD cartridge samples are stored in refrigerator R-7.

12.4 Hexavalent chromium filter samples are stored in freezer F-11.

12.5 Metals filter samples are stored at room temperature in the inorganic laboratory.

12.6 Other media should be stored according to the procedures described in the appropriate method or analysis SOP.

12.7 The cold packs must be stored in the freezer in the shipping and receiving area in time to refreeze prior to the next media shipment.

13.0 CALIBRATION AND STANDARDIZATION

13.1 The Weiss vacuum/pressure gauge is sent to the manufacturer annually for National Institute of Standards and Technology (NIST) traceable calibration.

13.2 The Omegascope® thermometers are sent to the manufacturer annually for calibration. The calibration and subsequent testing of the devices are performed using instrumentation and standards that are traceable to NIST.

14.0 RECEIVING and LOG-IN PROCEDURE

14.1 Visually inspect each sample and each sample COC upon receipt. COC must include full, proper, and complete documentation (including but not limited to sample identification, collection site, time and date of sample collection, collector's initials, and any comments regarding the sample). The sample acceptance criteria are listed in Section 16.0. Pay particular attention to the contents of coolers. Remove all items from coolers (including icepacks) to assure that no sample is left in the cooler.

14.1.1 Verify that the site code, collection date, any sample designation (Primary (P), Duplicate (D), Collocated (C) 1 or 2, Field Blank (FB)), and container number (container number or canister number, if it is a canister, hexavalent chromium, or PUF/XAD sample) on COC are same as sample/sample container. If one of these parameters does not match, notify the PM.

14.1.2 If the sample falls outside of acceptable criteria stated in the program instructions, or in Section 16.0 of this SOP (e.g., sample not at the proper temperature, sample container is broken, etc.), document on COC and immediately notify the PM so that a decision can be made whether to invalidate the sample.

14.1.3 The receiver shall initial the COC and record the date of receipt. The condition of the sample upon receipt (such as temperature, canister pressure, color) should also be recorded on the COC.

- In the case of a carbonyl cartridge, hexavalent chromium filter, or PUF/XAD cartridge sample, the receiver checks and records the sample temperature and the thermometer ID upon receipt on COC.

- In the case of a canister sample, the receiver records the canister pressure upon receipt.

14.2 Invalidated samples are listed as VOID on the COC and in the LIMS. The reason for invalidation is recorded in the LIMS and on the COC if not already there.

14.3 The LIMS log-in person assigns the next available unique number in the ERG LIMS. Samples are logged-in as they are listed on the COC forms.

14.4 Following LIMS log-in, each sample COC is labeled with its unique LIMS ID number. Copies of each COC are to be separated (or, if not multiple copies, copied). Original COCs go with sample, copies of each COC are scanned and either given to PM or put into a centralized location by the person logging in the samples.

14.5 Also following LIMS log-in, the original COC is paired with each sample. Labels with a unique LIMS ID and sample date are printed for each carbonyl, metals, and canister sample. Once paired with their original COCs, the samples are placed in the appropriate storage area as listed in Section 12.

14.6 After the log-in of samples is complete, work orders are printed so the PM or their designee must verify that the samples were logged-in correctly according to the copies of each COC.

14.7 Sample hold times vary by method; refer to appropriate analysis SOPs for required hold times. Those listed is this SOP refer to extraction hold times from the day of collection or analysis hold time when there is no associated extraction for that method. Adherence to specified hold times is documented by the LIMS and conveyed to the TL via LIMS Hold Time Alerts. Turnaround times are conveyed via program instructions and/or scheduled project meetings.

14.8 Any special procedures which apply to a set of samples should be made known to the analysts by the TL or PM.

15.0 CALCULATIONS

Not applicable.

16.0 QUALITY CONTROL

The PM or their designee verifies that the sample information has been logged into the LIMS correctly. The COC must be filled out in ink and include full, proper, and complete documentation (including but not limited to, time and date of sample collection, collector's initials, and any comments regarding the sample). Listed below is a summary of the **sample acceptance criteria**, specific to common sample media.

16.1 Special Projects Sample Acceptance Criteria

16.1.1 When samples are received for a new or special project, notify the PM and the recipient immediately. Follow the PM's direction for sample acceptance if method is unknown.

16.2 Canister Sample Acceptance Criteria

16.2.1 If the canister valve is open upon arrival, the sample is invalid.

16.2.2 The site, collection date, and canister ID number (written in indelible ink) must match the accompanying chain of custody.

16.2.3 The canister pressure upon receipt:

- If the gauge needle doesn't move (stays on zero), the sample may be void. If the canister vacuum is zero, there is a possibility that the canister leaked during transport. Notify the PM so that a decision can be made whether to invalidate the sample;

- If the canister vacuum is greater than 15" Hg, notify the PM so that a decision can be made whether to invalidate the sample. Depending on the collection time (3 hour vs. 24 hour), the sample may be valid;

- If the canister vacuum is more than 3 inches Hg different from the final field vacuum, notify the PM so that a decision can be made whether to invalidate the sample. This does not apply to positive pressure readings, in psig. Note: canisters from sites at higher elevations may have larger pressure differences, check with PM;

- There is no maximum positive pressure, psig, for a valid canister sample.

16.2.4 The sample is invalid if the sampling duration is less than 22 or greater than 26 for a 24 hour sample. Add a comment about the sample in LIMS if it has a sampling duration between 22-23 hours or between 25-26 hours, so that the PM can make the decision whether to invalidate the sample.

16.2.5 The sample is flagged in LIMS if the sample is received out of hold time of 30 days or the duration between cleaning and sampling is greater than 30 days.

16.3 Carbonyl Cartridge Sample Acceptance Criteria

16.3.1 The cartridge envelope must be labeled in indelible ink with sample identification that includes the collection site ID and time/date of sample collection. This information is checked to make sure it matches the accompanying COC.

16.3.2 If the sample cartridge and envelope is visibly damaged, the sample is invalid. Sample cartridges are not removed from the envelope for inspection unless the envelope is damaged.

16.3.3 If the sample is received above room temperature the sample should be flagged in LIMS. Note: As stated in the Note in Section 10.12 of Method TO-11A, if samples are to be shipped to a central laboratory for analysis, the duration of the non-refrigerated period should be kept to a minimum, preferably less than two days.

16.3.4 The sample is invalid if the sampling duration is less than 22 or greater than 26 for a 24 hour sample.Add a comment about the sample in LIMS if it has a sampling duration between 22-23 hours or between 25-26 hours, so that the PM can make the decision whether to invalidate the sample.

16.3.5 The sample is flagged in LIMS if the sample is received out of hold time of 14 days.

16.4 PUF/XAD Cartridge Sample Acceptance Criteria

16.4.1 If the site operator has noted the glass thimble is broken on the COC, the sample is invalid. Glass thimbles are not inspected in the shipping/receiving area unless there is visible damage to the plastic canister.

16.4.2 Likewise, if XAD has escaped from the cartridge, the sample is invalid.

16.4.3 The collection site, time/date of sample collection, , and container number must be written on the plastic canister label in indelible ink. This information is checked to ensure it matches the accompanying COC.

16.4.4 If the cartridge is received at a temperature > 21 °C the sample is flagged in LIMS.

16.4.5 The sample is invalid if the sampling duration is less than 22 or greater than 26 for a 24 hour sample. Add a comment about the sample in LIMS

if it has a sampling duration between 22-23 hours or between 25-26 hours, so that the PM can make the decision whether to invalidate the sample.

16.4.6 The sample is flagged in LIMS if the sample is received out of hold time of 14 days.

16.5 Hexavalent Chromium Filter Sample Acceptance Criteria

16.5.1 If the site operator has noted a tear, hole or water damage to the filter on the COC, the sample is invalid. Hexavalent chromium filters are not inspected in the shipping/receiving area unless there is visible damage to the plastic canister.

16.5.2 The collection site, time/date of sample collection, and container number must be written on the plastic canister label in indelible ink. This information is checked to ensure it matches the accompanying COC.

16.5.3 If the cartridge is received at a temperature > 15 °C the sample must be flagged in LIMS.

16.5.4 The sample is invalid if the sampling duration is less than 22 or greater than 26 for a 24 hour sample. Add a comment about the sample in LIMS if it has a sampling duration between 22-23 hours or between 25-26 hours, so that the PM can make the decision whether to invalidate the sample.

16.5.5 The sample is flagged in LIMS if the sample is received out of hold time of 21 days.

16.6 Metals Filter Sample Acceptance Criteria

16.6.1 If the site operator has noted water damage or a tear/hole to the filter that occurred prior to or during sampling, the sample is invalid. Metals filters are not inspected in the shipping/receiving area unless there is visible damage to the sample container.

16.6.2 The filter container or envelope must be labeled with a collection site and, time/date of sample collection written in indelible ink. This information must be checked against the accompanying COC to be sure it is correct and that all samples were received. Flow rate information must be included on either the sample COC or the sample container.

16.6.3 If no COC is present (e.g. Oklahoma metals filters), create a COC listing each sample received.

16.6.4 The sample is invalid if the sampling duration is less than 22 or greater than 26 for a 24 hour sample. Add a comment about the sample in LIMS if it has a sampling duration between 22-23 hours or between 25-26 hours, so that the PM can make the decision whether to invalidate the sample.

16.6.5 The sample is flagged in LIMS if the sample is received out of hold time of 180 days.

16.7 Other Media, Aqueous Sample Acceptance Criteria

16.7.1 If the sample container is broken or leaking, the sample is invalid.

16.7.2 If the sample is received at a temperature higher than the method requirements or out of method specified hold time, notify the PM. The sample may be invalid.

16.7.3 The sample identification, collection site, and time/date of sample collection and container number must match the accompanying COC.

16.8 Other Media, Solid Sample Acceptance Criteria

16.8.1 If the container is broken or the sample has spilled from an open or broken container, the sample is invalid.

16.8.2 If the sample is received at a temperature higher than the method requirements or out of method specified hold time, notify the PM. The sample may be invalid.

16.8.3 The sample identification, collection site, and time/date of sample collection and container number must match the accompanying COC.

17.0 PREVENTION

Not applicable.

18.0 CORRECTION ACTION

If samples are received that are not valid or acceptable based on this SOP, the PM for the project is informed of the samples involved and the issues. The PM contacts the client

and/or resolves the issue and all data related to the samples are flagged with the appropriate caution regarding use and applicability.

19.0 WASTE MANAGEMENT

Not applicable.

20.0 MAINTENANCE

Not applicable.

21.0 SHORTHAND PROCEDURE

Read the entire SOP before following the shorthand procedure, as quality control is not thoroughly covered in this section.

21.1 Special Projects

21.1.1 Notify the PM and the recipient immediately upon receiving samples for a new or special project.

21.1.2 Follow the directions of the PM for proper receiving of the samples.

21.2 Canister Samples

21.2.1 Verify the site code, collection date, any sample designation (P, D, C 1 or 2, FB), and canister number;

21.2.2 Initial and date COC;

21.2.3 Check and document pressure on COC;

21.2.4 Notify the PM if at a vacuum > 15 inches Hg;

21.2.5 Notify the PM if at a vacuum of zero inches Hg;

21.2.6 Invalidate if canister valve is open or partially open;

21.2.7 Invalidate and notify PM if sampled less than 22 or greater than 26;

21.2.8 Flag in LIMS if past hold time or time between canister cleaning and sampling is greater than 30 days;

21.2.9 Store in Air Tox Lab.

21.3 Carbonyl Cartridges

21.3.1 Verify the site code, collection date, any sample designation (P, D, C 1 or 2, FB);

21.3.2 Initial and date COC;

21.3.3 Check and document receipt temperature and thermometer ID on COC;

21.3.4 Invalidate if cartridge is damaged;

21.3.5 Invalidate and notify PM if sampled less than 22 or greater than 26;

21.3.6 Flag in LIMs if past hold time;

21.3.7 Store in refrigerator R-11 with the accompanying COCs.

21.4 PUF/XAD Cartridges

21.4.1 Verify the site code, collection date, any sample designation (P, D, C 1 or 2, FB), and container number;

21.4.2 Initial and date COC;

21.4.3 Check and document receipt temperature and thermometer ID on COC;

21.4.4 Flag in LIMS if cartridge is > 21°C;

21.4.5 Invalidate if glass thimble is broken;

21.4.6 Invalidate and notify PM if sampled less than 22 or greater than 26;

21.4.7 Flag in LIMs if past hold time;

21.4.8 Store in refrigerator R-7; COCs are clipped to the refrigerator door.

21.5 Hexavalent Chromium Filters

21.5.1 Verify the site code, collection date, any sample designation (P, D, C 1 or 2, FB), and container number;

21.5.2 Initial and date COC;

21.5.3 Check and document receipt temperature and thermometer ID on COC;

21.5.4 Flag in LIMS if cartridge is > 15°C;

21.5.5 Invalidate if filter is torn;

21.5.6 Invalidate and notify PM if sampled less than 22 or greater than 26;

21.5.7 Label the accompanying COC with the LIMS ID and attach COC to the container with the sample inside.

21.5.8 Flag in LIMs if past hold time;

21.5.9 Store in freezer F-11.

21.6 Metals Filters

21.6.1 Verify the site code, collection date, any sample designation (P, D, C 1 or 2, FB);

21.6.2 The receiver initials and dates the COC, if present (if no COC present, create a COC listing each sample received). COC should also be labeled with the LIMS IDs;

21.6.3 Invalidate if filter is torn;

21.6.4 Invalidate and notify PM if sampled less than 22 or greater than 26;

21.6.5 Flag in LIMs if past hold time;

21.6.6 Store in Inorganic Lab.

21.7 Other Media

21.7.1 Verify the site code, collection date, any sample designation (P, D, C 1 or 2, FB), and container number (as needed);

21.7.2 Initial and date COC;

21.7.3 Check and document receipt temperature and thermometer ID on COC;

21.7.4 Invalidate and notify PM if temperature exceeds method requirements; if sampled less than specified collection time; if container is broken or leaking;

21.7.5 Flag in LIMs if past hold time;

21.7.6 The LIMS ID is recorded on COC and a label with the LIMS ID is affixed to the sample container.

21.7.7 Store according to PM instructions.

22.0 DOCUMENTATION AND DOCUMENT CONTROL

The need for proper and complete documentation is essential. The documentation is the most reliable resource the laboratory has for knowing how to deal with the multitude of samples that come into a laboratory. If a sample is received with incomplete documentation, every effort should be made to complete the information necessary for the analysis of a sample.

23.0 REFERENCES

Datasystem User's Manual, Promium, LLC.

24.0 TABLES, DIAGRAMS FLOWCHARTS, AND VALIDATION DATA

Not applicable.

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CONFIDENTIAL
Standard Operating Procedure
Procedure Number: ERG-MOR-057
Revision Number: 1
Revision Date: March 3, 2011
Page: 1 of 7

ENGINEERING AND SCIENCE DIVISION

TITLE: Standard Operating Procedure for Project Peer Review	EFFECTIVE DATE: APR 25 ~~2010~~ 2011 KY
REFERENCES: ERG-MOR-017	
SATELLITE FILES: All Labs	
REASON FOR REVISION: Update procedures and update to NELAC format	
DATE OF LAST REVIEW:	NEXT SCHEDULED REVIEW:
WRITER: NAME/DATE Donna Tedder 4/18/11	TECHNICAL DIRECTOR: NAME/DATE Quita C. Swift 4/19/11
QUALITY ASSURANCE COORDINATOR: NAME/DATE Donna Tedder 4/25/11	MANAGER: NAME/DATE Donna Tedder 4/25/11

1.0 IDENTIFICATION AND PURPOSE

The purpose of this standard operating procedure (SOP) is to present the guidelines for selecting a project peer reviewer and conducting the peer review function as it relates to projects conducted at the Morrisville office. This document is not intended to supersede any corporate-level peer review SOP; rather, it is intended to supplement other such documents.

2.0 MATRIX OR MATRICES

NA

3.0 METHOD DETECTION LIMIT

NA

4.0 SCOPE AND APPLICATION

This SOP covers general peer review requirements and does not cover project-specific requirements. Project-specific peer review requirements will be incorporated in project instructions or work plans.

This SOP describes how to select a peer reviewer and the required involvement of the peer reviewer throughout the course of a specific project.

5.0 METHOD SUMMARY

NA

6.0 DEFINITIONS

DQO	data quality objective
PE	professional engineer
PM	project manager
QAPP	quality assurance project plan
QC	quality control
SOP	standard operating procedure
TL	task leader

7.0 INTERFERENCES

NA

8.0 SAFETY

NA

9.0 EQUIPMENT

NA

10.0 MATERIALS

NA

11.0 CHEMICALS, REAGENTS, AND STANDARDS

NA

12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

NA

13.0 CALIBRATION AND STANDARDIZATION

NA

14.0 PROCEDURE

14.1 Selection of a Peer Reviewer

The selection of the peer reviewer is critical to maintaining proper project quality control (QC) of technical work. At project inception, the Project Manager (PM) and the Task Leader (TL) must evaluate the requirements of the specific project to assess the level of experience needed for assignment of a peer reviewer. The peer reviewer must be qualified to understand the technical aspects of the project and to comprehend the client's expectations of the project. Other requirements include an understanding of the priorities, needs, and constraints to complete the project. It is also necessary for the peer reviewer to be aware of ERG's contractual commitments and potential areas of liability and/or exposure.

Normally, the choice of peer reviewer will be governed by the type of work conducted. Experience in a subject area is desired most often, but is not always a necessity.

For those engineering projects requiring final approval by a registered professional engineer (PE), the Coordinating Professional Engineer for the office will guide the selection of the appropriate peer reviewer. For such projects, peer reviewer selection will be based upon knowledge of the subject area, level of experience, and availability throughout the duration of the project.

14.2 Responsibilities

This section describes the various responsibilities of the peer reviewer throughout the duration of a project. The peer reviewer should be involved in project planning, kick-off, routine progress meetings, results, and reports (preliminary, draft, and final).

14.2.1 Project Initiation

At project initiation, the peer reviewer selected must be qualified to understand the technical aspects of the project and the technical content

and quality needs and expectations from the client's perspective. The peer reviewer must understand the timing and scope of his or her duties during the course of the project. Further, the peer reviewer must make the commitment of time and resources to perform proper peer review.

14.2.2 Project Instructions

Project instructions should be peer reviewed carefully before they are distributed to the project team to ensure the following:

- Project objectives are clearly stated, are appropriate, and are realistic.

- All information is clearly communicated. The project instructions should clearly communicate expectations of all project team members with regard to budget, schedules, individual responsibilities, and milestones.

14.2.3 Review of Technical Work Products

All technical work products should be subjected to peer review. These documents include, but are not limited to:

- Technical notes;
- Test plans;
- Quality Assurance Project Plans (QAPPs);
- Preliminary reports;
- Draft reports;
- Final Reports;
- Calculations;
- Field data; and
- Laboratory data.

The areas to be covered in reviewing the technical work products include:

- The project objectives should be clearly stated, as in the QAPP data quality objectives (DQOs). Of equal importance in the peer review is whether the project met its stated objectives. This should be documented on a data review checklist if pertinent to the project.

- Where applicable, all national or local codes, standards, methods, SOPs, and/or contractual constraints should be employed and cited.

As above, the report must be technically defensible in the light of all relevant regulatory or codified requirements.

- The work products must be reviewed to ensure that proper documentation (appendices containing information generated during the course of the project) has been included and that assumptions and supplemental data sources are properly referenced.

- The peer reviewer must consider carefully the conclusions and recommendations made in work products. The conclusions and recommendations must address the client's needs, must be complete, must be supported by the data and work, and should be implementable.

Adequate time should be budgeted for each peer reviewer to ensure that proper review is conducted and that peer review is not compromised as a result of schedule constraints.

All peer review comments should be retained as a part of the project files. Peer review comments must be addressed by the PM or project team. It is recommended that the PM review how the peer review comments were addressed with the peer reviewer(s) prior to completing the subsequent draft of the work product.

14.2.4 Routine Project Data Review

At a minimum, 10% of laboratory data must be reviewed. However, if a systematic or large number of errors are found in the 10% review, more than 10% of the technical work products should be reviewed. As an option, an additional 1% of the technical work products can be reviewed by a second peer reviewer designated by quality staff or PM. The review should be documented on a data review checklist, located in the SOP if pertinent to the project. All peer review comments should be retained as a part of the project files. Peer review comments must be addressed by the PM or project team.

Most projects involve calculations of some form; the selected peer reviewer will ensure that these calculations are adequately reviewed for mathematical accuracy. The selected peer reviewer will check a random number of these calculations personally to ensure that the proper review is being conducted. See SOP ERG-MOR-017 for evaluating the accuracy of spreadsheet data.

14.3 Problem Resolution

The peer reviewer is charged with providing comments and insights into the project throughout the course of the project. Any feedback on project progress or staff performance should be directed to the PM/TL, as appropriate. Any changes made or actions taken to address the concerns should be communicated to the peer reviewer, as well as reviewer concerns that will not be addressed and the reason the comments or concerns will not be addressed.

15.0 CALCULATIONS

NA

16.0 QUALITY CONTROL

As noted in Section 14.1, peer review is critical to proper QC of technical work. The responsibilities of peer reviewers throughout the duration of a project are detailed in Sections 14.2.1 through 14.2.4. In addition, Section 14.2.4 refers to SOP ERG-MOR-017 for evaluating the accuracy of spreadsheet data.

17.0 PREVENTION

NA

18.0 CORRECTIVE ACTION

NA

19.0 WASTE MANAGEMENT

NA

20.0 MAINTENANCE

NA

21.0 SHORTHAND PROCEDURE

NA

22.0 DOCUMENTATION AND DOCUMENT CONTROL

As noted in Section 14.2.3, all peer review comments should be documented on appropriate data review checklists and retained as part of project files.

23.0 REFERENCES

NA

24.0 TABLES, DIAGRAMS, FLOWCHARTS, VALIDATION DATA

NA

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ERG
EASTERN RESEARCH GROUP, INC.

CONFIDENTIAL
Standard Operating Procedure
Procedure Number: ERG-MOR-079
Revision Number: 4
Revision Date: April 27, 2015

ENGINEERING AND SCIENCE DIVISION

TITLE: Standard Operating Procedure for Sample Login to the Laboratory Information Management System	EFFECTIVE DATE: MAY - 6 2015
REFERENCES: ERG-MOR-045, Element Datasystem User's Manual, Promium LLC., Promium website, www.promium.com	
SATELLITE FILES: N/A	
REASON FOR REVISION: Updated procedure	
WRITER/EDITOR: NAME/DATE Jennifer Nash 5/4/15	PROJECT MANAGER: NAME/DATE Gabriel Swift 5/6/15
PROJECT QUALITY ASSURANCE MANAGER: NAME/DATE Donna Tedder 4/29/15	NEXT SCHEDULED REVIEW: 11/31/16

1.0 IDENTIFICATION AND PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to introduce the reader to procedures for entering sample information into the Laboratory Information Management System (LIMS) after receiving samples at the laboratory.

2.0 MATRIX OR MATRICES

N/A

3.0 METHOD DETECTION LIMIT

N/A

4.0 SCOPE AND APPLICATION

The instructions provided in the SOP are applicable to the login of all samples received from clients at ERG's laboratory facilities. This SOP assumes that a user has some familiarity with the Element Datasystem LIMS software.

5.0 METHOD SUMMARY

All samples received at the ERG Laboratory are logged into the LIMS database. The LIMS generates a unique identification (ID) number for each sample, and maintains the pertinent collection information and data for each sample.

6.0 DEFINITIONS

COC - Chain of Custody
Element - The name of the LIMS software used by ERG and sold by Promium, Inc.
ID - Identification
LIMS - Laboratory Information Management System
QC - Quality Control
SOP - Standard Operating Procedure
TAT - Turnaround Time

7.0 INTERFERENCES

N/A

8.0 SAFETY

N/A

9.0 EQUIPMENT

9.1 Desktop computer connected to ERG's Laboratory Intranet server BART (Y://).

9.2 Promium Element Software.

10.0 MATERIALS

N/A

11.0 CHEMICALS, REAGENTS, AND STANDARDS

N/A

12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

N/A

13.0 CALIBRATION AND STANDARDIZATION

N/A

14.0 PROCEDURE

14.1 Run the Element software and log on to the LIMS by entering your username and password.

14.2 Under the **Sample Control** option of the main menu, select the choice **Work Order**. A window will appear on the screen with information on the Work Orders already in the LIMS.

14.3 Click the **Add** button to create a new Work Order with a number generated by the LIMS. The scheme for the Work Order number is: YMMDDXX. Work Orders are generated on a project-by-project basis, i.e. only one project may be entered for every Work Order.

14.4 The following fields on the Project and Receipt tabs must be completed before the LIMS will allow the Work Order to be saved. The Project and Receipt tabs of the Work Order window are presented in Table 24.1 and 24.2. Fields that must be manually updated are denoted with a solid bullet, while fields that auto-complete are denoted with an open bullet. It should be noted that it is sometimes necessary to change fields that are automatically populated (e.g. "Received by Person"), so care must be taken to make sure this information is correct.

Project Tab:

- Client
- Project
    - Client Project Manager
    - Lab Project Manager
    - Project Number
    - Date Received

Receipt Tab:

    - Date Logged In
    - Date Due
    - Received By Person
    - Logged In By Person
    - Turnaround Time (TAT)
- Shipper Container Temperature (°C)
- Shipped By

Once the Work Order has been completed with the correct information, click the green **Save** button to save the information.

14.5 To modify information in a Work Order already created, click the **Edit** button, make the necessary corrections, and then click the green **Save** button to save the changes.

Note: Only the last Work Order created can be deleted by clicking the **Delete** button in the Work Order window. All others may only be modified.

14.6 The Work Order window may be closed by clicking the **Done** button. If individual sample information for a Work Order is going to be created or modified, the **Samples>>** button can be clicked to access this information.

14.7 Input individual sample information by selecting the **Samples** option in the **Sample Control** menu or by clicking the **Samples>>** button in the Work Order window.

14.8 Select the appropriate Work Order number to the left of the window if the correct number does not appear.

14.9 Enter the sample information from the COC form into the fields on the Sample Information and Containers tabs listed below:

Sample Information Tab

- Sample Name
- Lab Matrix
  - Report Matrix (Automatically populates based on Lab Matrix)
- Sample Type
- Analysis
- Sample Begin Date/Time/Zone
- Sample End Date/Time/Zone
- Sample Comments
- Applicable User Fields for canister number and pressure

Containers Tab

- Sample Container
- Sample Location

The Sample Information and Container tabs of the Samples window are presented in Table 24.3 and 24.4. Keep in mind that available analyses will not appear in the window until a Matrix is chosen. Also, information entered into certain fields

such as Sample Name and Comments may be specific to a particular client and/or project.

Note: Recurrent sampling events can be logged in using archived sample information by selecting the pull down arrow from the "Name" field and selecting the appropriate sample name. LIMS will import the information from the previous sample logged in under the same name. Edit the imported sample information appropriately and select "Save."

14.10 Each time a sample is added, Element generates a LIMS ID number with the code YMMDDXX-YY. Record this number in the upper right corner on the respective COC form.

Note: For multiple samples collected at the same site on the same day, new samples can be added to the work order by selecting "Copy" at the bottom of the screen and editing the appropriate sample information.

14.11 Once the Work Orders and Sample Information have been entered for the day, print the Work Orders using the **Work Order** option under the **Print** menu (or by clicking the print icon from the Sample window). Multiple Work Orders may be printed simultaneously by clicking the **Multi** button and selecting multiple Work Order numbers to print. The print template most used is the wko_withstorage template, but other templates may also be used. The Print Work Order window is presented in Table 24.5.

14.12 Print sample labels using the **Sample Labels** option under the **Print** menu (or, if using the print icon from the Sample window, the Sample Labels Print window will automatically come up upon closing the Work Order Print window). Multiple sample labels may be printed simultaneously using the **Multi** button and choosing samples from the present day or other time periods. Canister sample labels use the lsx_m@5163ERG.rpt template and all others (carbonyl and metals samples) use the lsx_m@5160.rpt template. Labels are printed using the Avery 5163 and 5160 varieties, respectively. The Print Sample Labels window is presented in Table 24.6.

14.13 After the Work Orders and Sample Labels have been printed, separate the white and yellow copies of the COC forms and photocopy any white COC forms missing the yellow copy. Give the sample labels and white COC forms to the sample receipt person so they can be placed with the samples. Scan the yellow CoC forms and pass them along to the appropriate project manager with the printed work orders so that sample login can be reviewed.

14.14 Save the scanned copies of the CoCs to a designated location and insert into their respective work order in LIMS. This is achieved by returning to the **Work Order**

option and selecting the **PDF icon** next to "Chain Of Custody". A new window will open, select **Add** and choose the appropriate location and COC scan, then select **Done**. The COC PDF window is presented in Table 24.7.

14.15 The Project Manager or person designated by the Project Manager will compare the Work Orders against the COC forms and indicate the errors to be corrected by the Sample Login person.

14.16 The Project Manager will update the status of the newly logged samples in the Update Status window of the Laboratory menu. Samples may be queried in a variety of ways and displayed by Analysis or by Work Order. The samples or Work Orders to be updated are selected on the screen and updated to a status of Available, Invalid, or Cancelled. Invalid samples should have a comment in the Samples window including the reason for invalidation.

14.17 After the status updates and the necessary corrections have been made, the yellow COC forms and Work Orders can be filed in the appropriate places.

15.0 CALCULATIONS

N/A

16.0 QUALITY CONTROL

16.1 After completing the login of samples for the day, the Work Orders generated should be checked against the COC forms to find any discrepancies. Errors are to be corrected in a timely fashion.

16.2 Work Orders are checked by the respective Project Manager or designee to provide another level of QC.

17.0 PREVENTION

N/A

18.0 CORRECTIVE ACTION

LIMS entries are checked by the Project Manager or designee. Corrections to entries are indicated on the Work Order printouts, and the sheets are returned to the sample logger so that the logger can make the necessary corrections.

19.0 WASTE MANAGEMENT

N/A

20.0 MAINTENANCE

Should any issues with the LIMS software arise, contact the ERG LIMS administrator. An annual service agreement is maintained for the Promium Element.

21.0 SHORT-HAND PROCEDURE

21.1 Create a Work Order for each project/site for which samples are received. Add individual samples to each Work Order using information from the COC forms.

21.2 Record LIMS ID numbers on the COC forms.

21.3 When all Work Orders and samples have been added to the LIMS, print the Work Orders and sample labels.

21.4 Separate the white and yellow copies of the COC forms. The yellow copies are scanned and given to the Project Manager responsible for the samples along with the Work Orders.The white copies are returned to samples.

21.5 PDF scans of the COC forms are inserted into the respective LIMS work order.

21.6 The Project Manager checks the Work Orders against the COC forms.

21.7 The yellow CoC copies are then filed in a designated storage area.

22.0 DOCUMENTATION AND DOCUMENT CONTROL

22.1 White copies of COC forms are filed with the sample data packages and stored according to each project's requirements. Yellow copies of COC forms are filed according to LIMS ID number in the laboratory's central COC file cabinet. The yellow copies are transferred to long-term storage periodically.

22.2 Work Order printouts are mainly used to check the accuracy of data entry into the LIMS. Work Order printouts are stored and/or disposed of according to the applicable Project Manager's instructions.

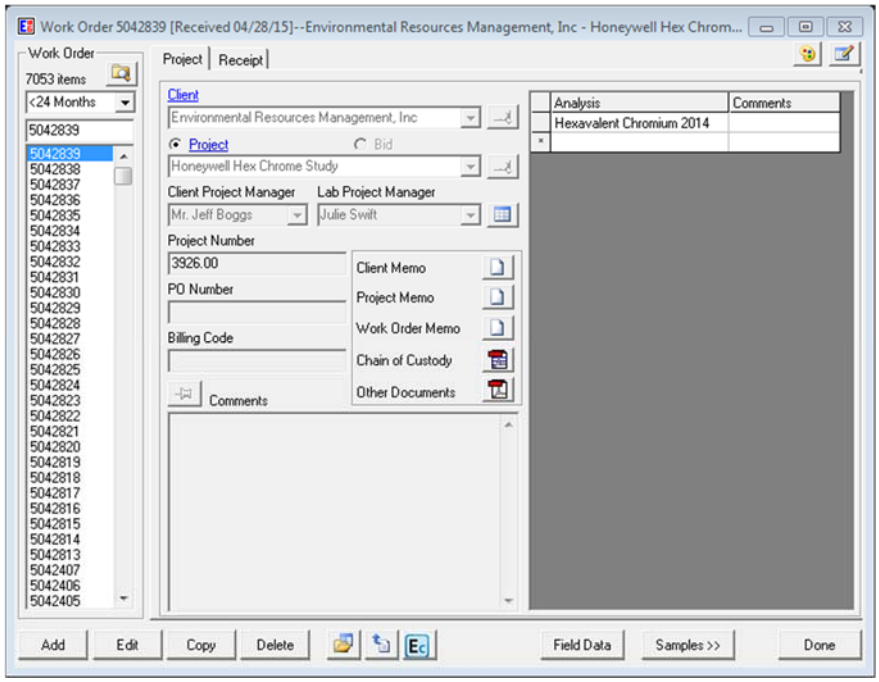
23.0 REFERENCES

Element Datasystem User=s Manual, Promium LLC.

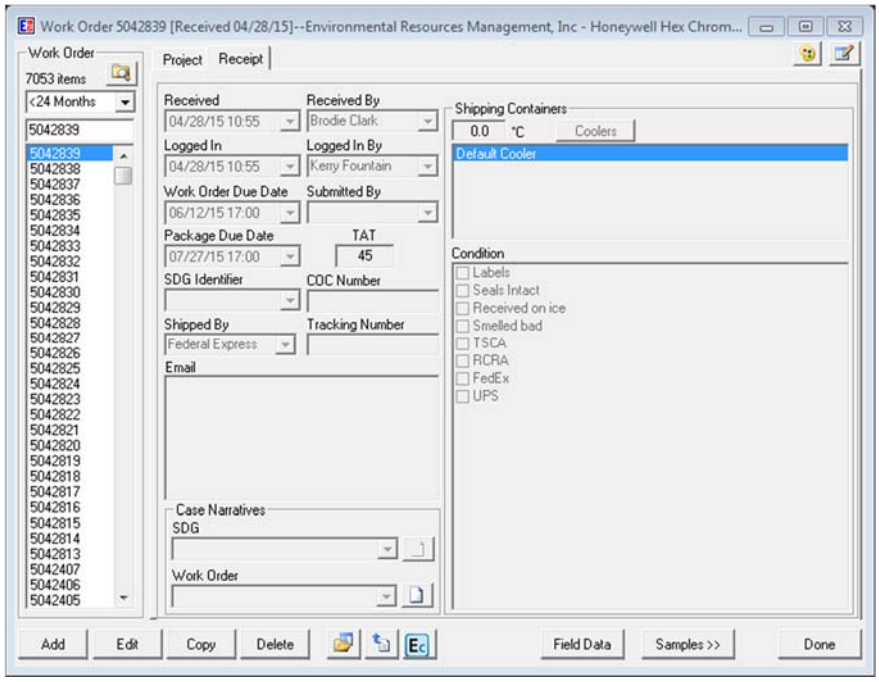
Promium website, www.promium.com

24.0 TABLES, DIAGRAM, FLOWCHARTS, VALIDATION DATA

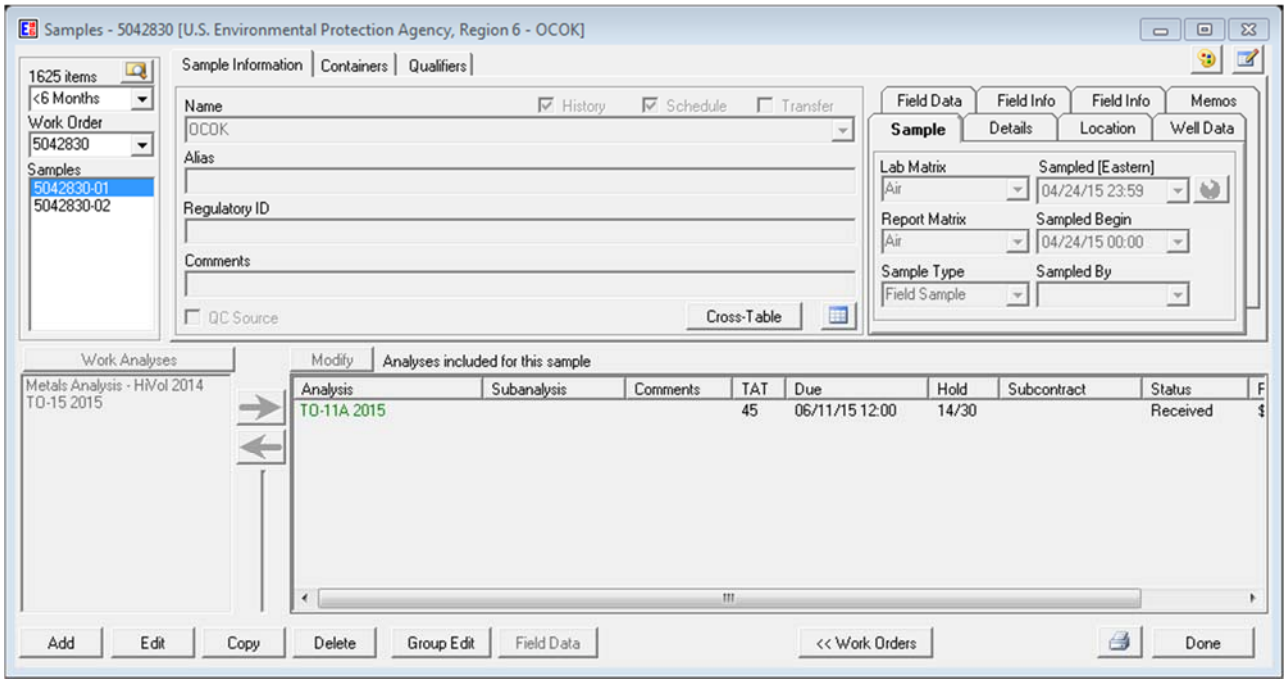
24.1 Work Order window- Project tab:



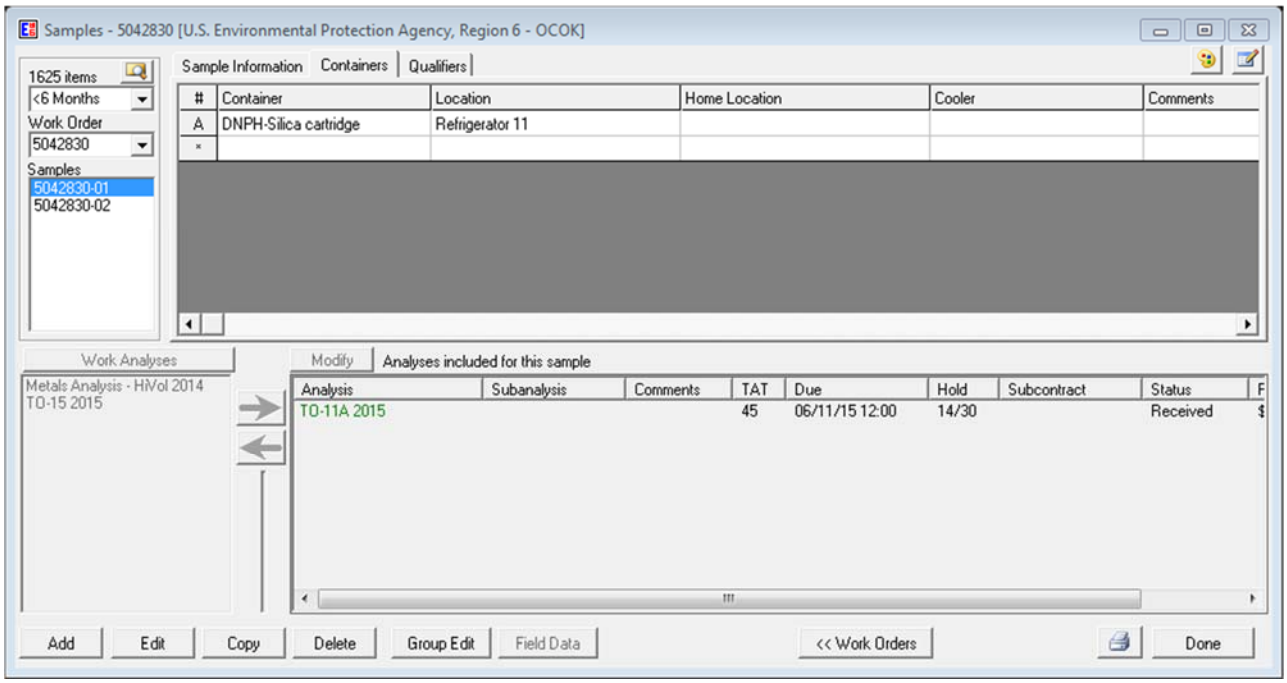
24.2 Work Order window – Receipt tab:



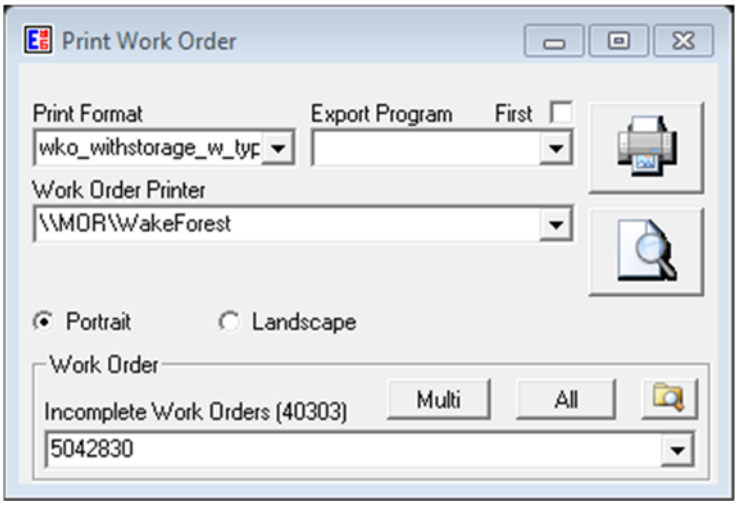
24.3 Sample window – Sample Information tab:



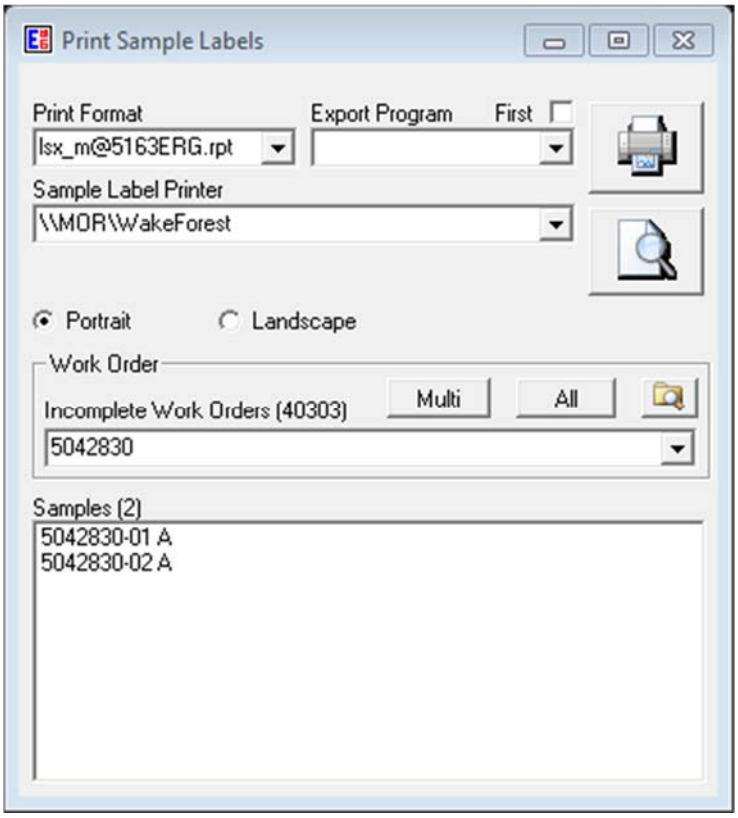
24.4 Sample window – Containers tab:



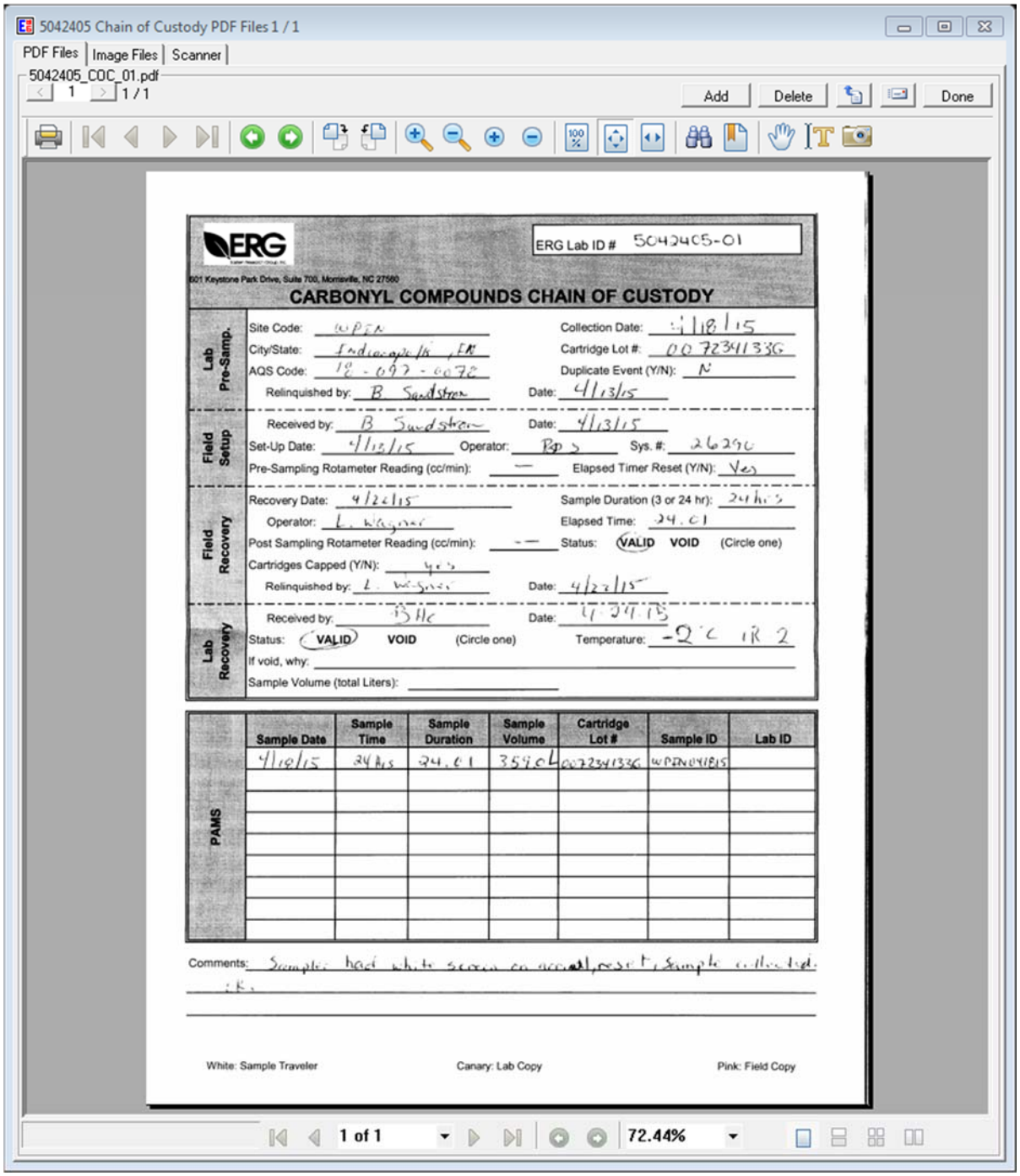
24.5 Print Work Order window:



24.6 Print Sample Label window:



24.7 Chain of Custody PDF window:



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ERG
EASTERN RESEARCH GROUP, INC.

CONFIDENTIAL
Standard Operating Procedure
Procedure Number: ERG-MOR-084
Revision Number: 11
Revision Date: March 31, 2015
Page: 1 of 58

ENGINEERING AND SCIENCE DIVISION

TITLE: Standard Operating Procedure for the Preparation and Analysis of High Volume Quartz Filters for Metals by ICP-MS using Method IO 3.5 and FEM Method EQL-0512-201	EFFECTIVE DATE: APR - 3 2015
REFERENCES: ERG-MOR-031, ERG-MOR-033, ERG-MOR-045, Corporate Quality Management Plan, ERG Health and Safety Manual, Laboratory Quality Systems Manual, 40 CFR, Part 136, Appendix B, EPA Compendium Method IO-3.5, FEM Method "Standard Operating Procedure for the Determination of Lead in TSP by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Hot Block Dilute Acid and Hydrogen Peroxide Filter Extraction" (EQL-0512-201)	
SATELLITE FILES: ICP-MS Laboratory	
REVISIONS: Updated MDLs, Added procedure for extraction record, Removed word duplicate from Collocate/Duplicate listed in Table 24-3 and review checklist	
WRITER/EDITOR: NAME/DATE Jennifer Nash 4/3/15	PROJECT MANAGER/TECHNICAL DIRECTOR: NAME/DATE Janet L. Swift 4/1/15
QUALITY ASSURANCE COORDINATOR: NAME/DATE Donna Tedder 4/1/15	NEXT SCHEDULED REVIEW: 1/31/2016

1.0 IDENTIFICATION AND PURPOSE

This standard operating procedure (SOP) provides the sample preparation and analysis procedures for suspended particulate matter collected on quartz filters (PM10 or TSP) for total metals determination by Inductively Coupled Plasma - Mass Spectrometer (ICP-MS).

2.0 MATRIX OR MATRICES

This procedure applies to the preparation and analysis of ambient particulate matter samples collected with quartz filters (PM10 or TSP) for total metals.

3.0 METHOD DETECTION LIMIT

3.1 Method Detection Limits (MDL)

3.1.1 The method detection limit (MDL) for each isotope is calculated according to Appendix D: DQ FAC Single Laboratory Procedure v2.4, 8/30/2007. MDL values are determined from historic method blank (BLK2) data following the procedures outlined in the document above.

3.1.2 The y-intercept for each linear calibration must be set to zero.

3.1.3 Use the same internal standards, calibration standards, instrument method and settings (sweeps and dwell) for the MDL study and field sample analysis.

3.1.4 The MDL determination should be reported in ng/L, ng/filter and ng/m^3 (assuming 2000 m^3 per sample). Refer to Table 24-1.

3.1.5 The MDL study should be repeated once per year and whenever a significant change in background or instrument response is expected (e.g., detector change).

4.0 SCOPE AND APPLICATION

4.1 Scope

This procedure details the acid extraction and trace elemental analysis of ambient air samples using an inductively coupled plasma-mass spectrometer (ICP-MS). The extraction procedures are suitable for high-volume ambient air samples collected on quartz membrane filters, sized 8 x 10". The procedure is applicable, but not limited to the metals listed in Table 24-1.

4.2 Applicability

This SOP is applicable to the analysis of suspended particulate matter collected with quartz filters. Acid digestion and filtration is required prior to analysis of quartz filter extracts. Analytes for which ERG has demonstrated the acceptability of this method are listed below. See Table 24-7 for a list of isotopes used for quantitation and monitoring.

Element	Symbol	CASRN
Aluminum*	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium*	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium*	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper*	Cu	7440-50-8
Iron*	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Mercury	Hg	7439-97-6
Molybdenum*	Mo	7439-98-7
Nickel	Ni	7440-02-0
Rubidium*	Rb	7440-17-7
Selenium	Se	7782-49-2
Strontium*	Sr	7440-24-6
Thallium*	Tl	7440-28-0
Thorium*	Th	7440-29-1
Uranium*	U	7440-61-1
Zinc*	Zn	7440-66-6

* Elements not on our standard EPA UATMP/NATTS analysis list of elements.

5.0 METHOD SUMMARY

This SOP describes the multi-elemental determination of total metals by ICP-MS in ambient air samples collected on quartz filters following guidelines in EPA method IO-3.5 and EPA FEM Method "Standard Operating Procedure for the Determination of Lead in TSP by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Hot Block Dilute Acid and Hydrogen Peroxide Filter Extraction" (EQL-0512-201). An 8"x 1" portion is cut from the exposed filter. The filter is digested in a HotBlock™ for 2.5 hours using an extraction fluid containing 1.5% hydrochloric acid (HCl), 5.55% nitric acid (HNO_3) and 25 mg/L Au for mercury stabilization. Two aliquots of hydrogen peroxide (H_2O_2) are added after 1.5 hours and 2.0 hours of extraction and are allowed to effervesce. The extract is filtered and analyzed by ICP-MS. Data are collected using the manufacturer's software.

6.0 DEFINITIONS AND ABBREVIATIONS

6.1 Definitions

6.1.1 **Analytical Duplicate (DUP).** A second aliquot of a sample extract that is analyzed from the original sample in order to determine the precision of the method. This sample is also referred to as a replicate. See Section 16.4 for further elaboration on duplicates.

6.1.2 **Blank (BLK).** An analytical sample designed to assess specific sources of contamination. In this method there are two BLKs, the Laboratory Reagent Blank (LRB), which is always reported as BLK1 and the Method Blank (MB), which is always reported as BLK2.

6.1.3 **Blank Spike (BS).** A quality control sample (QCS) that contains a quartz filter strip spiked with a known quantity of analytes that is carried through the entire extraction process. This sample is synonymous with the Laboratory Control Sample (LCS).

6.1.4 **Calibration Blank.** A blank solution containing all of the reagents in the same concentration as those used in the analytical sample preparation when brought to final volume. This blank is not subject to the preparation method but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

6.1.5 **Calibration Standards.** A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions are not subject to the preparation method but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

6.1.6 **Continuing Calibration Blank (CCB).** A volume of ASTM Type I water acidified with the same acid matrix as is present in the calibration standards used to verify after every CCV that the instrument blank checks are reading ≤ MDL.

6.1.7 **Continuing Calibration Verification (CCV).** A multi-element standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV is the original calibration standard, whose concentration is at the midpoint of the calibration curve that is re-analyzed as a quality control (QC) sample.

6.1.8 **Field Blank (FB).** This is any sample that is submitted from the field and is identified as a blank. This also includes trip blanks.

6.1.9 **High Standard Verification (HSV).** The HSV is the highest calibration standard that is reanalyzed after the ICB and before the analysis of samples to verify the accuracy of the calibration curve at that concentration.

6.1.10 **Initial Calibration Blank (ICB).** The ICB is a re-analysis of the calibration blank, which is analyzed after the Initial Calibration Verification (ICV) and used to verify that the instrument blank checks read ≤ MDL.

6.1.11 **Initial Calibration Verification (ICV).** A solution prepared from a stock standard solution obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration.

6.1.12 **Interference Check Standard (ICS).** A solution that may contain only interfering elements (ICSA) or both interfering elements and analytes of interest (ICSAB) in known concentrations that can be used to verify background and interference correction equations.

6.1.13 **Interferents.** Substances (atoms, ions, polyatomic ions, etc.) which may affect the analytical result for the element of interest.

6.1.14 **Internal Standard (ISTD).** A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

6.1.15 **Laboratory Control Sample (LCS).** A spiked aliquot of LRB with a blank quartz filter used as a QCS that is prepared and brought through the entire digestion/extraction and analytical process to demonstrate spike recoveries. This sample is synonymous with the BS.

6.1.16 **Laboratory Reagent Blank (LRB).** An aliquot of ASTM Type I water that is treated exactly as a sample including exposure to all labware, equipment, solvents, reagents and internal standards that are used with other samples that is always reported as BLK1. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or apparatus.

6.1.17 **Limit of Quantitation (LOQ).** The lowest concentration of an analyte that produces a signal/response that is sufficiently greater than the signal/response of lab reagent blanks to enable reliable detection and quantification during routine lab operating conditions. Statistically defined, this is the concentration of analyte in sample matrix that produces an instrument signal/response that is 10 times the standard deviation above the LRB (at 99% confidence, n-1 degree freedom; see Table 24-2).

6.1.18 **Linear Dynamic Range (LDR).** The concentration range over which the analytical working curve generated from the calibration standards is proven to remain linear. See Section 13.8 for more information on the LDR.

6.1.19 **Lower Limit of Quantitation Check (LLQC).** A check sample that is used to both establish and confirm the lower limit of quantitation and is prepared by spiking a low concentration of analyte into reagent water and carrying the solution through the entire preparation and analytical procedure.

6.1.20 **Lower Limit of Quantitation Limit (LLQL).** The lower limit of quantitation is considered the lowest reliable laboratory reporting concentrations and should be established from the lower limit of quantitation check sample and then confirmed using the lowest calibration point and/or from a low level calibration check standard.

6.1.21 **Low Level Calibration Verification (LCV).** A stock standard solution prepared using the same source as the calibration standards that is analyzed to verify the LLQL. The standard is prepared at the same concentration as the LLQL. An LCV is analyzed at the beginning, typically just before or after CCV1, and at the end of every analysis just before or after the final CCV.

6.1.22 **Matrix Interference/Effect.** In general, the interference and/or effect that particular matrix constituents may cause during sample processing and/or analysis. Matrix effects may be determined to exist from the careful interpretation of QC samples and criteria. Examples of observed effects include but are not limited to poor recoveries of spikes/ISTD and poor percent differences.

6.1.23 **Matrix Spike and Matrix Spike Duplicate (MS/MSD).** A sample chosen in a batch where additional filter strips are spiked prior to digestion/extraction with known quantities of specific analytes and carried through the entire analytical process to demonstrate their spike recoveries and precision.

6.1.24 **Method Blank (MB).** An aliquot of LRB with a blank quartz filter strip that is carried through the entire preparation and extraction process to demonstrate background contamination contribution from the filter and process and is always reported as BLK2.

6.1.25 **Method Detection Limit (MDL).** The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

6.1.26 **Performance Evaluation (PE) Sample.** A sample of known composition provided by a source outside the laboratory for analysis that evaluates the laboratory's analytical performance.

6.1.27 **Post Digestion Spike (PDS).** A spiked aliquot of an already digested sample used to demonstrate spike recoveries in the sample matrix when MS/MSD samples cannot be made. The analytical results of this spike may also be used to verify matrix interference in conjunction with the SRD results when the MS/MSD fail to meet criteria.

6.1.28 **Quality Control Sample (QCS).** A solution containing known concentrations of method analytes that is used to fortify an aliquot of LRB matrix. The QCS is prepared from a source(s) external to the laboratory and is used to verify the laboratory's analytical performance.

6.1.29 **Serial Dilution (SRD).** The dilution of a sample by a factor of five. If the undiluted parent sample concentration is minimally a factor of 50 above the MDL, the diluted sample should agree with the parent sample concentration within ± 10% when corrected by the dilution factor. The SRD may indicate the influence of interferents.

6.1.30 **Standard Reference Material (SRM).** A prepared standard material that has certified metals concentrations, for example the NIST SRM 1648a (Urban particulate matter with certified concentrations of lead at 0.655 ± 0.033 mass fraction (in %)). The SRM is used to verify the extraction procedure.

6.1.31 **Stock Standard Solution.** A commercially prepared standard solution (traceable to NIST or other certified standard sources), which can be diluted to derive other standards.

6.1.32 **Tuning Solution.** A solution used to determine acceptable instrument performance prior to calibration and sample analyses. This solution is used for mass calibration, nebulizer optimization, auto lens optimization, and daily performance reports.

6.2 Abbreviations

amu	Atomic Mass Units
ASTM	American Society for Testing and Materials
CASRN	Chemical Abstract Services Registry Number
COC	Chain of Custody
cps	Counts Per Second
DI	Deionized
DQO	Data Quality Objective
HNO_3	Nitric Acid
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
kW	Kilowatts
L	Liter(s)
LDPE	Low Density Polyethelyene
LIMS	Laboratory Information Management System
MΩ	Megohm
MCA	Multichannel Analyzer
MQO	Method Quality Objectives
m	Meter(s)
m^3	Cubic Meter(s)
mg	Milligram(s)
mg/L	Milligram(s) per liter(s)
min	Minute(s)
mL	Milliliter(s)
mm	Millimeter(s)
ms	Millisecond(s)
ng	Nanogram(s)
ng/L	Nanogram(s) per liter(s)
ng/mL	Nanogram(s) per milliliter(s)
NIST	National Institute of Standards and Technology
QC	Quality Control
RSD	Relative Standard Deviation
RPD	Relative Percent Difference
SD	Standard Deviation
SOP	Standard Operating Procedure
Std.	Standard
µg/L	Microgram(s) per liter
$\mu g/m^3$	Microgram(s) per cubic meter(s)
µg/mL	Microgram(s) per milliliter(s)
µL	Microliter (s)
µm	Micrometer
v/v	Volume per volume ratio

7.0 INTERFERENCES

Note: The background level of metals on a given lot of quartz filters can vary. Any background levels found on blanks should be documented for all the filters from the corresponding lot when available. It is recommended to consult 40 CFR Part 50, Section 6.1 Appendix G for guidance.

7.1 Laboratory Interferences

7.1.1 Wear powder-free nitrile or neoprene gloves when handling unexposed or exposed filters.

7.1.2 Clean all equipment used in the sample preparation and analysis in a manner consistent with good laboratory practices for metals analysis (See Section 20.3 in this SOP and Section 14.3.1 in SOP ERG-MOR-031).

7.1.3 Use ASTM Type I DI water or equivalent, with a resistivity greater than 17.3 MΩ, for sample extraction and standard preparation. Record the water resistivity prior to use.

7.2 Chemical Interferences

Pay close attention to the nature of solutions introduced to the ICP-MS.

7.2.1 Nitric acid must be less than 2% (v/v) for ICP-MS analysis to minimize the damage to the interface and to minimize isobaric molecular interferences. The use of platinum cones and other acid-resistant sample introduction components can be used for more aggressive acid matrices.

7.2.2 If higher acid extractions are required, dilute final digestate to 2% HNO_3.

7.2.3 The final dilutions of sample extracts must match the acid content of the calibration standards in order to match potential interferences.

7.2.4 The concentrations of dissolved solids in analysis solutions should be less than 2% to protect the sample interface on the instrument and prevent signal suppression. Higher concentrations may plug the sample and/or skimmer cone orifices.

Note: Protect the channel electron multiplier from high chemical concentrations (high ion currents). The channel electron multiplier suffers from fatigue after being exposed to high ion currents. This fatigue can last from several seconds to hours depending on the extent of exposure. During this period, response factors are constantly changing, which causes

instrument instability that invalidates the calibration curve, and thereby, invalidates all associated sample results. A sodium bicarbonate ($NaHCO_3$) sample matrix is known to cause this problem.

7.3 Instrument Interferences

7.3.1 Isobaric molecular and doubly charged ion interferences are caused by polyatomic ions (e.g., the contribution of ArCl on the 75As signal) or more than one charge (example, MoO+ ions on Cd isotopes).

7.3.2 Spectral interferences result from the presence of other isotopes or ions that have the same atomic weight or mass number as the analyte.

7.3.3 Transport interferences are a specific physical interference associated with the sample nebulization and transport process through the instrument. These usually result from sample matrix components that influence the aerosol formation or cause a change in the surface tension or viscosity. Changes in the matrix composition can cause observed signal suppression or enhancement.

7.3.4 Matrix interferences may be caused by elemental chemical and physical properties in the samples. For matrices of known composition, match the composition of the calibration and QC standards to that of the samples. For matrices of unknown composition, use an ISTD that has been matched to the analytes' chemical and physical properties (i.e., ionization potential, ±50 amu) so that the ISTD and element of interest behave similarly during the analytical process.

7.3.5 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sample and skimmer cones, spray chamber, peristaltic pump tubing and the type of nebulizer all affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interferences.

7.3.6 Lead values are reported from isotope 208; however, all three isotopes must be used to quantitate lead to allow for the variability of lead isotopes in nature. The following correction equation must be applied to isotope 208:

$$(1.000)\ (^{206}Pb) + (1.000)\ (^{207}Pb) + (1.000)\ (^{208}Pb)$$

8.0 SAFETY

8.1 Personal protection should be used for all work performed in the inorganic laboratory, (e.g., gloves, safety glasses, laboratory coats, etc.).

8.2 The compressed gas cylinders must be stored and handled according to relevant safety codes outlined in the corporate health and safety manual. In use, the cylinders must be secured to an immovable structure and moved using a gas cylinder cart.

8.3 Make sure that sample vials are kept capped and in racks to prevent spills.

8.4 All personnel should be trained in the handling, extraction and analysis of acid samples for inorganic analysis.

8.5 Strong acids must not be stored with organic solvents or samples.

8.6 Follow normal laboratory safety procedures as outlined in the ERG Health and Safety Manual and the site-specific laboratory SOP.

9.0 EQUIPMENT

9.1 ICP-MS

The PerkinElmer SCIEX™ ELAN® 9000 ICP-MS consists of an inductively coupled plasma source, ion optics, a quadrupole mass spectrometer, a computer that controls the instrument, data acquisition and data handling software (ELAN® Software SCIEX™, Version 3.4), a printer, an autosampler (AS-93plus) and a recirculator. The quadrupole mass spectrometer has a mass range of 2 to 270 amu. Typical operating conditions are listed below.

Typical Operating Conditions

Plasma forward power	1.3 kW
Plasma/Coolant argon flow rate	13.8 L/min
Auxiliary argon flow rate	1.2 L/min
Nebulizer flow rate	0.9 L/min
Solution uptake rate	1.0 mL/min
Spray chamber temperature	Room Temperature
Detector mode	Dual (Pulse counting/Analog)
Replicate integrations	3
Mass range	6 - 240 amu
Dwell time	50 ms
Number of MCA channels	1

Number of scan sweeps	20
Total acquisition time	4.2 min/sample

9.2 Digestion System

Environmental Express HotBlock™ Digestion System or equivalent system capable of maintaining a temperature of 95°C within ± 2˚C. This temperature will heat the samples to a temperature of ~85°C (±5°C).

10.0 MATERIALS

10.1 Graduated polypropylene sample vials with screw caps, 50 mL volume (certified to be within ± 0.2mL).

10.2 Branson 8510, sonication bath with heating capability.

10.3 Pipetters with adjustable volumes ranging from 0.5 µL to 10 mL and disposable tips. Mechanical pipettes must be verified for accuracy quarterly (or every three months). Repeatable, mechanical pipettes, such as Eppendorf Research®, may be used and their accuracy should be verified on a quarterly basis to be within the manufacturer's specifications. If a pipette's accuracy exceeds the manufacturer's specifications its use should be discontinued and it should be replaced or sent in for repair.

10.4 Reflux caps and FilterMate™ 2 µm Teflon® filters with plungers.

10.5 Miscellaneous: powder-free nitrile or neoprene gloves; disposable laboratory wipes; self adhesive labels.

10.6 Volumetric flasks. Teflon®, Class A: 50, 100, 250 and 500 mL capacities.

10.7 Storage bottles. Wide and narrow mouth, Teflon® FEP (fluorinated ethylene propylene) with Tefzel ETFE (ethylene tetraflourethylene) screw closure: 50, 100, 250, 500, 1,000 and 2,000 mL capacities.

10.8 Wash bottles made of LDPE and Teflon® having 500 mL and 1 L capacities.

10.9 Plastic or Teflon® coated tweezers and Teflon stirring rods.

10.10 Filter cutting apparatus with 2 part Plexiglas board and plastic rotary cutter.

11.0 CHEMICALS, REAGENTS, STANDARDS AND THEIR PREPARATION

Note: In general, chemicals, reagents and commercial stock standards expire when specified by the manufacturer. If the manufacturer does not provide an expiration date then they shall expire one year from the opened date. Standards and other solutions prepared in-house expire as specified throughout the SOP. Proper disposal of hazardous wastes are discussed in detail in the Solid and Hazardous Wastes SOP (ERG-MOR-033).

11.1 High Purity Acids - ultrapure and concentrated stored in Teflon® Bottles. These reagents are used for the preparation of sample extraction fluid and all standards.

Note: Concentrated high purity reagents are not 100% of the specified reagent. It should be understood that all percentages in this SOP are expressed in terms of volume per volume (v/v) rather than true percentages of reagent in solution.

11.1.1 Nitric Acid (HNO_3), 60-70%

11.1.2 Hydrochloric Acid (HCl), 32-35%

11.1.3 Extraction fluid (1.5% (v/v) HCl, 5.55% (v/v) HNO_3, and 25 mg/L Au).

11.1.4 Standard solvent (0.3% (v/v) HCl, 1.11% (v/v) HNO_3 with 5 mg/L Au)

11.2 Hydrogen Peroxide (H_2O_2) - ultrapure and concentrated (30-32%) stored in Teflon® bottles. This reagent is used in the extraction procedure.

11.3 Nitric Acid-Trace Metal Grade in 2.5 L glass for rinse blank and labware cleaning.

11.3.1 Rinse blank (2% (v/v) HNO_3, 0.5% (v/v) HCl with 5 mg/L Au)

11.3.2 10% (v/v) HNO_3 acid bath for labware cleaning

Preparation: The acid bath solution is prepared by adding 2.5L of concentrated trace metal grade HNO_3 to 22.5 L of ASTM Type I DI water in a clean 42 L polypropylene acid bath tank. The acid bath should be stored in a fume hood.

11.4 ASTM Type I deionized water - with a resistivity greater than 17.3 MΩ.

11.5 Argon gas - purity > 99.996%, Oxygen < 5 mg/L, Hydrogen < 1 mg/L, Nitrogen < 20 mg/L and Water < 4 mg/L.

11.6 Secondary Source Control Standards - A commercially prepared single- or multi-element standard from a secondary source (different manufacturer from the multi-element calibration standard). These NIST traceable calibration standards are used to produce the ICV, which is run as a verification of the instrument's calibration for accuracy and precision.

11.7 Single-Element Stock Standard Solutions - Commercially prepared NIST traceable standards from ultra high-purity grade chemicals or metals (99.99 – 99.999% pure) designed for use with ICP-MS instruments (e.g., Mercury Std.).

11.8 Multi-Element Stock Standard Solutions - Commercially prepared NIST traceable standards from ultra high-purity grade chemicals or metals (99.99 – 99.999% pure) designed for use with ICP-MS instruments (e.g., ISTD solution).

11.9 Interference Check Standard - Commercially prepared standard that is diluted to prepare ICSA and ICSAB interferent checks.

11.10 Smart Tune Solution - Although custom tuning solutions may be used, the tuning solution for this SOP may be purchased through the manufacturer (Perkin Elmer #N8125040) or is prepared in-house using single-element standards to contain 10 µg/L of Be, Mg, Co, Rh, In, Ba, Ce, Pb, and U in 1% (v/v) HNO_3.

To prepare 1 L of this solution, add 10 mL of Ultrex Nitric Acid to ~ 900 mL of ASTM Type I deionized water and add 10 µL of each 1,000 µg/mL single-element standard, then bring to volume. This solution may be stored in LDPE bottles but ideally in Teflon®. The expiration date is either that specified by the manufacturer or if prepared in-house, no later than the earliest expiration date of any standard used for preparation.

11.11 Dual Detector Cross Calibration Solution - Refer to Section 13.3 for the purpose and final concentration of this solution. Although custom cross calibration solutions can be used, it may be purchased through the manufacturer (Perkin Elmer #8125032) or prepared in-house using single-element standards.

11.12 Multi-element ISTD stock standard – Commercially prepared standard that is used in conjunction with the single-element standards for Sc, Ga, and Li to prepare the internal standard.

11.13 Internal Standard Spike Solution - Prepared standard used to manually spike all calibration and QC standards as well as all samples that are analyzed by the ICP-MS.

11.14 Matrix Spike (MS) Standard - The MS standard is used to spike the BS/LCS and MS/MSD (See Section 16.3.2). Prepare the MS standard according to Table 24-6 with a final volume of 50 mL.

11.15 Post Digestion Spike (PDS) Standard - The PDS standard is used to spike the PDS source sample. This post digestion spike is used to help determine if poor matrix spike recoveries are due to interferents. Spike 1 µL per 1 mL of sample analyzed.

11.16 Second Source Working Standard - The second source working standard is used to create the ICV.

11.17 Citranox® Acid Cleaner and Detergent - Prepare a 5% solution by adding 500 mL of Citranox® to 9.5 L of warm – hot tap water for labware cleaning and decontamination. This detergent bath should be changed about once every month, depending on use.

11.18 Standard Reference Material (SRM) - The standard reference material used to prepare the SRM samples. NIST SRM 1648a (Urban particulate matter with certified concentrations of lead at 0.655 ± 0.033 mass fraction (in %) is used to verify the extraction procedure.

11.19 Lower Limit of Quantitation Check (LLQC) - This quality control sample is used to determine the LLQL, and is prepared by creating a spike solution that will create a final concentration of analyte in the sample matrix that produces and instrument signal/response that is 10x the standard deviation above the lab reagent blank (at 99% confidence; n-1 degrees of freedom). Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within ± 30% of their true value.

12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

12.1 Collection and Handling of Filters

Whenever the filter is handled use clean disposable nitrile or neoprene gloves and if necessary clean Teflon® coated (no exposed metal surfaces) or plastic tweezers. Never touch the particulate laden surface of the filter and take care not to puncture or damage the filter with the tweezers. The filter should be folded in half lengthwise to prevent loss of particulate. See Section 16.1 for more information about filter condition.

12.2 Preservation and Storage of Filters

Samples do not have a preservative and can be stored for up to 180 days in ambient conditions. Upon retrieval from field sampling during humid or rainy

conditions be certain the filter is not moisture-laden. Damp filters may stick to the sample transport container causing damage and thereby invalidating the sample. If the sample is found to be moisture-laden allow the membrane to dry before enclosing in the transport container. Once the filter arrives at the laboratory, a unique LIMS identification number is assigned and placed on the outside of the COC, sample envelope and/or container for tracking and storage purposes. Extraction hold time is 180 days from the sample date.

12.3 Shipment of Filters

When filters are shipped to or from the laboratory follow proper handling instructions in Section 12.1 and take proper precautions when packing such that filters are not exposed to contaminants or damage during shipment.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Daily Optimization Procedures

Daily optimization is performed through the software's Smart Tune Wizard. Refer to Tables 24-4 and 24-5 for Optimization Procedures and Performance Specifications respectively. For more detailed information, the PerkinElmer Elan Version 3.4 Software Reference Guide is also available for reference as a PDF file on the desktop of the instrument computer.

13.2 Mass Calibration and Resolution

Before performing any kind of calibration or optimization, allow a period of not less than 30 minutes (preferably 1 hour) for instrument warm-up. After the warm-up, the mass calibration and resolution may be optimized using the tuning solution (see Section 11.11) by running a mass calibration and resolution optimization through the Smart Tune Wizard. Resolution at low mass is indicated by magnesium isotopes 24, 25, and 26. Resolution at high mass is indicated by lead isotopes 206, 207, and 208. These peaks may be viewed in the Interactive graphics window. For optimal performance, adjust spectrometer mass calibration to ± 0.05 amu and the resolution to produce a peak width of 0.70 ± 0.1 amu at 5% peak height. Repeat mass calibration and resolution optimization if it has shifted by more than ± 0.05 or ±0.1 amu respectively.

13.3 Dual Detector Cross Calibration

The dual detector cross calibration is used to provide a smooth transition from the pulse counting mode to the analog mode, which extends the linear dynamic range of the detector. Typically a 2% (v/v) nitric acid solution containing 200 µg/L of Mg, Cu, Rh, Cd, and Pb as well as 2000 µg/L of Be is used for this calibration

although custom solutions may be used. This calibration must be performed daily before each analysis to report values above the HCV.

13.4 Daily Performance

13.4.1 The daily performance report must be generated daily or before each analysis, whichever is more frequent.

13.4.2 Instrument stability must be demonstrated by running a daily performance check using the tuning solution. A minimum of five replicates with resulting relative standard deviations of absolute signals for all analytes of less than 3% is required prior to calibration. Performance specifications for the daily performance are listed in Table 24-5.

13.5 Calibration

13.5.1 Prior to initial calibration, set up proper instrument software routines for quantitative analysis (i.e., autosampler table, QC sample names etc.). The instrument must be calibrated using a minimum of a calibration blank and four non-zero calibration standards. Consideration should be give to adding more standards, particularly lower concentrations, in order to better define the LDR and quantitation limit (QL). A minimum of three replicate integrations with an RSD < 10.0% are required for data acquisition. RSDs > 10.0% are allowed for the first non-zero calibration standard (CAL2). Use the average of the integrations for instrument calibration and data reporting. To be considered acceptable, the calibration curve should have a correlation coefficient ≥ 0.998.

For a linear calibration to be considered acceptable, the calibration curve should have a correlation coefficient ≥ 0.998. The resulting curve should then be verified with mid-level and low-level calibration standards as described in Section 16.6.

Preparation: Non-blank calibration standards are prepared by diluting the calibration working standard to appropriate levels using the standard solvent.

13.5.2 For matrices of known composition, match the composition of the calibration and QC standards to that of the samples. For matrices of unknown composition, use an ISTD that has been matched to the analytes chemical and physical properties (i.e., ionization potential, ±50 amu) so that the ISTD and element of interest behave similarly during the analytical process.

13.5.3 The rinse blank should flush the system between solution changes for blanks, standards, and samples. Allow sufficient rinse time (≥ 1 min) to remove traces of the previous sample. Solutions should aspirate for at least 30 seconds prior to the acquisition of data to establish equilibrium.

13.5.4 Refer to Section 11.1.4 for the preparation of standard solvent. Once prepared, all calibration standards must be stored in Teflon® bottles/flasks.

Note: Commercial stock standards used to prepare calibration standards and other quality control standards must be used within their expiration date. Calibration blanks/standards and other QC standards made from the stock standards may be set to expire no later than the earliest expiration date of any standard used for preparation.

13.5.5 Refer to the quality control requirements presented in Table 24-3 for calibration acceptance criteria.

13.6 Internal Standardization

13.6.1 Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. ISTD quality control requirements as described in Section 16.7 must be followed.

13.6.2 Internal standards (ISTD) for this method are ^{45}Sc, ^{69}Ga, ^{89}Y, ^{115}In, and ^{209}Bi for analytes beginning with mass 6 and ending with mass 238. The ISTDs ^{6}Li, ^{72}Ge, ^{103}Rh, ^{159}Tb & ^{165}Ho may also be used if necessary. Internal standards must be manually added to each calibration standard **after** they are brought to volume in the proportion of 2.0 µL for every 1 mL. For example, add 200 µL of ISTD solution to a 100 mL standard.

13.6.3 Concentrations of the internal standards for this method are determined by the concentration of each element that will produce an intensity that is sufficiently stable. Typical intensities are between 200,000 and 500,000 cps; however, ideal intensities may be as high as 1,000,000 cps.

13.6.4 The concentration of the internal standard must be added equally and in the same manner to the calibration blank/standards, QC standards and samples.

13.6.5 Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. However, be aware that internal standards themselves may be responsible for polyatomic and/or doubly charged interferences.

13.7 Instrument Performance

13.7.1 After instrument calibration, an ICV and ICB must be analyzed for initial verification of the calibration curve. Refer to Sections 16.6.1, 16.2.2, and Table 24-3 for specific QC criteria.

13.7.2 To verify that the instrument is properly calibrated on a continuing basis, analyze a CCV and CCB before the analysis of samples and after every 10 samples.

13.8 Linear Dynamic Range (LDR)

The LDR study is performed every 6 months to determine the maximum concentration level at which the initial calibration is linear. The recovery criteria for the LDR are 90-110%. Dilutions must be performed for elements with concentrations over 90% of the established LDR. If an LDR study has not been performed within 6 months, any analytes with concentrations over the highest calibration concentration must be diluted.

13.9 Lower Limit of Quantitation Limit (LLQL)

The LLQL study is performed every 6 months to determine the lowest concentration level at which data may be reported. This is done by extracting and analyzing a lower limit quantitation check (LLQC). The recovery criterion for the LLQC is 70-130%. Any results reported below the LLQL must be qualified as an estimated value.

14.0 PROCEDURE

14.1 LIMS Batch Procedure

Note: Please perform the following procedure the same day that you plan to begin the extraction. If something happens and the extraction cannot be performed, edit the bench sheet with the correct extraction date, both in LIMS and on the hardcopy bench sheets.

14.1.1 Log into Element.

14.1.2 From the “Laboratory” menu, select “Batch.”

14.1.3 With the Inorganics department selected from the drop-down menu at the left of the screen, click “Add” at the bottom, left-hand corner. A blank bench sheet will appear. The following information should be input into

the bench sheet using the drop-down menus: 1) Preparation Method: ICP-MS Extraction and 2) Batch Matrix: Air

14.1.4 Select the appropriate inorganic analysis ("Metals Analysis – HiVol 20xx") from the "Available" analysis box and press the right-facing arrow button.

Note: Options: 1.) Additional information may be added in the "comments" box for batches that are different from standard samples, for example samples that are for the Midlothian or Schools projects. Reagent lot numbers used in the extraction are also added in the comments section. 2.) You may choose to press "Copy" instead of "Add" from Step3. Use caution here, as both the analysis and comments from the copied batch will be included in the new batch and this information may need to be changed.

14.1.5 Press the save button. The new LIMS-created batch number will be visible in the box to the left of the screen.

14.1.6 Select the "Bench Sheet" button at the bottom of the screen. This is where you will include sample information and appropriate batch QC.

14.1.7 Click "Edit" at the bottom of the new screen.

14.1.8 At the top of the screen, press "Add" and select "Sample by Container." Select the samples from the list that you would like to include in the batch. For each sample, the following information needs to be included: 1) Initial (m^3): This is the total flow through the filter as it was being sampled in the field. This information can be found or calculated from the information provided on the sample chain of custodies. Field blank samples are assigned the same volume as the primary sample collected on the same day. A field blank volume may also be an average of all the filter volumes for a given month. 2) Comments: Include two spaces followed by the sample ID for each sample in the list. This includes any additional designations such as C1, C2, FB, etc.

Note: A maximum of twenty samples may be selected for any given batch. If more than 20 samples are selected, additional batch QC must also be added to the bench sheet and extracted to meet the requirements described in Table 24-3.

14.1.9 Each batch requires QC to be prepared/extracted with each batch of twenty samples. This QC is included in the bottom box on the screen. Required QC includes:

i. Duplicate Samples: One duplicate QC sample is added per batch. Click the "Add" button at the top of the screen and select "Duplicate." Right click on a duplicate sample to assign its source sample and initial volume. This volume will be the same as the source sample. Duplicate samples must also be added for each collocated (C1/C2) or duplicate (D1/D2) spam type to be extracted.

ii. Blank Samples: Each batch must include one reagent blank and one method blank per twenty samples.

a.) One blank (BLK) sample is already included. To add another, click the "Add" button at the top of the screen and select "Blank."

b.) Change the sample name by right-clicking on each blank and selecting "Name." BLK1 should be changed to "Reagent Blank" and BLK2 should be changed to "Method Blank."

c.) Include the blank filter number in the comments section of the Method Blank.

iii. Spiked Samples: Each batch must include two spiked samples per twenty samples.

a.) One matrix spike (MS) and one matrix spike dup (MSD) are included for Hi-Vol extractions. Click on "Add" and select "Matrix Spike" and "Matrix Spike Dup" to include these QC. For each of these QC samples, assign the same source sample. The volume will be the same as that source sample.

b.) Identifying spikes: For both matrix spikes and laboratory control samples, the appropriate spike ID, type, and volume will need to be assigned in the same manner. Right click on the sample QC and select "Spike 1 ID." Select the appropriate standard from the list. Edit the "Spike 1 Type" to read "Pre-prep" and the "Spike 1 Volume" to read 500uL (or whatever volume is being used.)

iv. Post Spike (PS): Each batch must include one post spike (PS) sample per twenty samples.

a.) One post spike is automatically included for Hi-Vol extractions. Assign the appropriate spike ID, type, and volume using the instructions provided above. The correct spike amount is 50 µL and the spike type is "Post-prep."

v. Standard Reference Material: Each batch must include one SRM per twenty samples.

a.) Click on "Add" and select "Reference" to add the SRM to the bench sheet. Add the SRM identification number as assigned during weighing in the comments section. Identify the reference standard used by right-clicking on "Spike 1 ID" and selecting the appropriate standard. Indicate the weight of SRM digested (in mg) in the Spike 1 Volume. "Spike 1 Type" should be listed as "Pre-prep."

14.1.10 Print two copies of the bench sheet in landscape format. Both will need to be signed on the "Extraction Reviewed by" line and dated once the extraction is complete. One copy is to be three-hole punched and placed in the Extraction Notebook, while the other is to be kept bound to the corresponding samples in the cabinet in the lab.

14.2 Filter Extraction Procedure

14.2.1 Prior to sample processing, be sure to turn on the HotBlock ™ and select the appropriate program and initiate to allow it to warm to extraction temperature. Be sure to allow the HotBlock™ interface establish connection with the block itself (shown on the screen as "Please Wait") prior to beginning any extraction method. Failure to do this will cause the HotBlock™ to heat to higher than intended temperatures.

14.2.2 Wipe the filter-cutting apparatus and Teflon® rotary-cutter with a Kimwipe® prior to use.

14.2.3 If the filter is not already folded in half, carefully fold the filter in half along the 10" length with the side containing the particulates facing on the inside of the fold. Using the filter-cutting apparatus, align the 10" folded edge of the filter along the dashed line. Cut a 4" by 1" section of the folded filter using the Teflon® rotary-cutter.

14.2.4 Carefully fold the 4" by 1" section end over end into a size just larger than the mouth of the extraction vial. Place the folded filter into the labeled extraction vial and push to the bottom using a pre-cleaned Teflon® stirring rod for each sample to be sure the filter is below the 10 mL line.

Note: Do not place the filter flush with the bottom of the sample tube, as this will prevent proper acid flow through the tube during extraction. It is only necessary to make sure the filter is below the 10 mL line.

14.2.5 Add 0.5 mL of Matrix Spike Solution to the LCS, MS/MSD samples. Add 10 mL of extraction fluid (1.5% HCl (v/v), 5.55% HNO_3 (v/v) and 25 mg/L Au) to each extraction tube with a mechanical pipette.

14.2.6 The SRM sample is prepared for extraction by adding a blank 8x1" filter strip to a clean sample vial as described in Section 14.2.4. The loaded vial is then weighed, zeroed, and then re-weighed after an aliquot of SRM standard of approximately 2-3 mg is added to the vial. Add 10 mL extraction fluid to the extraction tube.

Note: It may be more efficient to weigh several SRM standard vials for extraction at one time. The weight information should be recorded in the balance room notebook. Individual standards should be numbered with the date the vial was created (i.e. "01012013-SRM01"). The vial number should be recorded in the LIMS in the SRM sample comments in the bench sheet.

14.2.7 Print out a copy of the Quartz Extraction Record from L:\Metals Lab (an example is shown in Table 24-10). Record the batch ID and extraction date. Record the temperature of the HotBlock™ as indicated by the calibrated thermometer, as well as the temperature indicated on the HotBlock UI.

14.2.8 Place all samples in a plastic HotBlock™ rack and place in HotBlock™. Add a reflux cap to each sample tube. Record the time samples were placed in the HotBlock on the table printed above. Samples will be extracted for a total time of 2.5 hours at a HotBlock™ temperature of 95ºC and an approximate sample temperature of 85ºC.

Note: Monitor sample temperature with a thermometer in an extraction tube with a reflux cap and 10 mL of extraction fluid.

14.2.9 After 1.5 hours of extraction, add 1.8 mL of hydrogen peroxide (H_2O_2) and allow to effervesce for 0.5 hours. Add an additional 1.8 mL of H_2O_2 after 2 hours of extraction and allow to effervesce again for 0.5 hours. This completes the extraction process. Record the time both aliquots of H2O2 were added on the Quartz Extraction Record.

14.2.10 After extraction is complete, remove the rack of samples and allow them to cool to room temperature. Record the time samples were removed, as well as the temperatures indicated by both the certified thermometer and HotBlock™ UI on the Quartz Extraction Record. Add 30 mL of DI water, cap, shake vigorously and allow to stand for 0.5 hours. This critical step must not be omitted; it allows the acid to diffuse from the filter into the

rinse. Record the time the DI water was added to the samples on the Quartz Extraction Record.

14.2.11 After this time period bring the sample volume to the 50 mL line on the vial with a wash bottle filled with ASTM Type I DI water prior to filtering. Record the time the samples were brought to final volume on the Quartz Extraction Record.

Note: If the final volume exceeds 50 mL measure the amount exceeded using an appropriate pipette and (usually the 1 or 5 mL) and record the final sample volume. Using the pipettor, pull sample from the vial in varying increments until enough liquid is removed so that the sample line is level with the 50 mL line on the vial. Add the amount of liquid removed to 50 mL and record that as the final volume. This information must be entered into the LIMS so that the final sample concentration is calculated and reported correctly. This overfilling may be avoided by using fine streamed wash bottles while adding the D.I. water.

14.2.12 Carefully filter the sample extract with a 2 µm FilterMate™ filter.

Note: When using a FilterMate™, slowly plunge the filter through the sample and be sure to get the filter all the way to the bottom. Do not force the filter or you will rupture the FilterMate™ filter. Ruptured filters that caused a loss of sample extract result in the re-extraction of that sample.

14.2.13 Homogenize the sample by inverting three times and then the extract is ready to be analyzed. The extracted filter and FilterMate™ remain in the sample vial with the extract to be analyzed. This does not interfere with sample recoveries or analysis.

14.2.14 Record the sample slot the certified thermometer was placed in during the sample extraction on the Quartz Extraction Record. After initialing the sheet, attach the Quartz Extraction Record to the batch paperwork.

14.3 LIMS Sequence Procedure

14.3.1 Log into LIMS Element Software.

14.3.2 From the “Laboratory” menu, select “Sequence.”

14.3.3 Click “Add” in the top right corner and select “Randy” as the Template ID then click done. This will automatically add all of the calibration standards and QC samples for a typical analysis sequence.

14.3.4 Click the pull-down menu for "Source Batch" at the upper middle part of the screen and select the batch you are going to analyze. Then click the "Add" button and select "Batch QC Sample." When the list of QC samples appears click the first sample and holding the shift button double-click the last sample and it will insert them into the sequence. Then click "Add" again and select "Batch Sample." Add all of the samples listed as you did for the Batch QC Samples.

14.3.5 Arrange all of the samples according to the example below, being sure to follow the requirements summarized in Table 24-3:

Autosampler Position	Sample Name
1	BTB0001-CAL1
2	BTB0001-CAL2
3	BTB0001-CAL3
4	BTB0001-CAL4
5	BTB0001-CAL5
6	BTB0001-ICV1
1	BTB0001-ICB1
5	BTB0001-HCV1
7	BTB0001-IFA1
8	BTB0001-IFB1
4	BTB0001-CCV1
2	BTB0001-LCV1
1	BTB0001-CCB1
9	B0B1707-BLK1
10	B0B1707-BLK2
11	B0B1707-SRM1
12	B0B1707-BS1
13	0020993-01
14	B0B1707-DUP1
15	B0B1707-MS1
16	B0B1707-MSD1
17	B0B1707-SRD1
18	B0B1707-PS1
4	BTB0001-CCV2
1	BTB0001-CCB2
7	BTB0001-IFA2
8	BTB0001-IFB2
4	BTB0001-CCV3
2	BTB0001-LCV2
1	BTB0001-CCB3

14.3.6 Right-click the SRD sample and choose the appropriate source sample from your batch.

Note: Make sure that the source sample for the SRD is updated to the current batch SRD sample or your data will not be saved.

14.3.7 Using the shift key select all of the samples in the sequence and right-click to choose "Internal Standard ID" then select the ISTD you are using for this sequence.

14.3.8 Right-click each calibration standard and QC sample and select "Standard ID" to set the current standard being used for each solution.

14.3.9 Click "Save" and print a double-sided copy in landscape format to use as the cover of the data package and to help enter the sequence in the Elan software.

14.4 Filter Analysis Procedure

14.4.1 Prior to analyzing samples, check the instrument performance by analyzing the tuning solution using the Daily Performance Check in the Smart Tune Wizard. The performance specifications that must be met are in Table 24-5. Also refer to Section 13.1 for other criteria. If the performance check fails, follow the optimization procedures in Table 24-4.

14.4.2 Before starting the calibration, be sure to flush the sample introduction system with sufficient rinse blank and be certain the rinse blank bottle has enough solution for the analysis. Enter all sequence information (sample and QC sample names) into the autosampler and QC tables in the Elan software. The autosampler table should be named as the month, day and year (e.g., 01012010.sam) and saved. Create the file name for the collected data with the numeric month, day and year (e.g., 01012010.rep) in the Method window under the "Report" tab (upper right) in the "Report Filename" field. Any changes made to the method (i.e., QC tables) must be saved before you exit that screen or they will be lost.

14.4.3 Pour off the calibration blank/standards (Blank (CAL1), LOQ (CAL2), CAL3, CAL4 and CAL5) and initial/continuing QC standards (ICV, ICB, ICSA, ICSAB, HSV, CCV, CCB) spiked with ISTD in the appropriate autosampler positions.

14.4.4 To start the analysis, highlight all samples in the autosampler table and click "Build Run." Then click "Run" in the following screen. Once the calibration has been completed and reviewed, save the calibration file with the same month, day and year as the .sam and .rep files in Section 14.4.2

and print the Quantitative Analysis Report to PDF to be included with the sequence files..

14.4.5 Label autosampler tubes with a black marker. Batch samples may be prepared by adding 20 µL of ISTD into the autosampler tube and then adding 10 mL of sample. Mix the sample well and place in appropriate autosampler location for analysis.

14.4.6 The PDS sample is prepared by adding 1.0 uL of spike solution to 1.0 mL of sample to be analyzed (i.e., 12 uL of PDS to 12 mL of sample).

14.4.7 Samples with analyte concentrations greater than 90% of the current LDR must be diluted and re-analyzed.

14.5 Unexpected Instrument Shutdown

In the event that the ICP-MS shuts down during an analysis the proper procedure to be followed by the analyst is:

14.5.1 Restart the instrument and allow it to warm-up for a minimum of 30 minutes but preferably one hour, especially if the instrument has been inoperable overnight and is at room temperature. If the analyst was present during the loss of the plasma and the instrument has not significantly cooled then a shorter time period for warm-up may be sufficient.

14.5.2 After the instrument has been thermally stabilized a new daily performance report (DPR) should be analyzed with the operating conditions being used for the analysis.

14.5.3 If the DPR passes the analysts must check the calibration by analyzing a continuing calibration verification (CCV) and a continuing calibration blank (CCB) to be sure the calibration is still valid. If the CCV & CCB passes the analyst may proceed with where the analysis left off. Any samples that didn't complete their analysis should be repeated.

14.5.4 If the DPR, CCV or CCB do not pass the analysis must be terminated and any samples not bracketed by valid CCV & CCB checks must be reanalyzed with a new analysis/calibration.

14.5.5 The DPR, CCV and CCB checks should be kept for documentation. The DPR may be placed in the DPR binder and the CCV & CCB checks must be included with the data package. The analyst should document the event briefly in the sequence narrative so the reviewer is aware of the instrument shutdown.

14.6 LIMS Data Upload Procedure

14.6.1 When a data package is complete, the analyst will transfer the data from the instrument computer to network server BART.

14.6.2 To begin LIMS upload, open Element. Go to the laboratory menu and click on "Data Entry/Review" In this window, select "Sequence" in the top left corner, making sure that "Inorganics" is selected from the drop-down menu. Highlight the correct sequence and click on "Create" in the Data Entry box in the top right corner. Once the spreadsheet is created in LIMS, select "DataTool" in the Data Entry box and save the file as the sequence name in the UserFiles folder of your harddrive (C:\ELMNT\UserFiles).

14.6.3 The DataTool interface should open to the "Select Data System Files" window. In this window, check to make sure the correct file information is selected:

1) File Type: PE ELAN_REP(*.rep)
2) Drives: y:\Bart

14.6.4 In the box below Drives: select the folder the data is stored in for the sequence. In the Bart drive, select the ICP-MS DATA folder, then the corresponding year, and finally the folder for the data the sequence was run.

14.6.5 All of the data files for that sequence will appear in the lower right-hand box. Double-click on the appropriate data file and click "Auto Select" for each file that needs to be included in the sequence. (*Note: Only undiluted samples and sequence QC should be included here.)

14.6.6 Click "Done" when all sample and QC files have been selected to return to the main window. Click "Merge Files" at the bottom of the window. DataTool will merge the files and show the data in the Data Transfer window.

Note: Review the content of the top windows in the Data Transfer window for red text. If there is any, the DataTool cross table requires editing. Seek the advice of the LIMS administrator to correct this.

14.6.7 Click "Save" and save the spreadsheet in the UserFiles folder of your hard drive. Close DataTool.

14.6.9 In Element, go back to the Data Entry/Review window. The newly merged data should appear in the window. Click "Save" to save the files to Element and then "Query" in the Data Review box. Element will perform all necessary calculations at this point.

14.6.10 In the Data Entry/Review window, samples and QC can be reviewed for pass/fails. Any data that does not pass its assigned criteria will have red text. Use appropriate data qualifiers to flag data that does not meet criteria.

14.7 LIMS Dilution Data Upload Procedure

Note: Dilution data is not uploaded with sequence QC, as this data is typically only required for one or two analytes in a given sample. Therefore, the data is hand-entered into Element.

14.7.1 Open Element and go to the Data Entry/Review window. Select the sequence that the diluted sample was originally run with. Click on "Query."

14.7.2 Scroll down to the needed sample information. Click "Edit" in the Data Review box. Right click on the sample & analyte that has dilution information and select "Qualifiers" -> "Quick Analyte Qualifiers." Select qualifier flag D-01 for dilutions.

14.7.3 In the IResult column, type the new dilution data. Make sure the new data has been corrected for the dilution factor (i.e. results for a 5x dilution should be multiplied by 5 if the instrument software did not make the correction). In the Diln column, type the dilution factor for the sample.

14.7.4 Repeat steps 2 & 3 for any additional dilution data that needs to be input for the sequence. If any QC data was altered (for example, Dups) then you will click on "Re-calc" and "Save." If only sample information was altered, click "Save."

14.7.5 Re-run the query to verify all dilution data was saved to the sequence.

Note: In LIMS, the dilution factor is applied to the associated MDL as well as to the sample concentration.

14.8 Data Review

All instrument data should be first reviewed by the analyst and then a secondary reviewer, usually the project task lead for metals analysis. Both the analyst and secondary review must use the "Quality Control Requirements for Metals

Analysis" checklist to complete data review (see Figure 24-8). Reviewers must initial and date each parameter check on the review form to verify that each meets the established acceptance criteria.

14.8.1 Initial Calibration

In addition to the requirements outlined in Figure 24-8, the analyst and secondary reviewer must also verify that the intensities measured for reportable analytes in the calibration blank are acceptable and will not interfere with the sensitivity. A review of previously analyzed calibration blanks can demonstrate acceptable intensity values. The intensities of the internal standards in the calibration standard should be monitored relative to the intensities seen in the calibration blank.

14.8.2 Internal Standards

Internal standards must be monitored for each sample throughout a sequence; the measured intensities must stay between 60 and 125% of the measured intensity of internal standard in the calibration blank. See Section 16.7 for corrective actions to remedy internal standard intensities that are measured outside of this range.

14.8.3 Relative Standard Deviation's RSD's

Follow the prescribed acceptance criteria for RSD's of calibration standards as listed in Table 24-3. Sample RSD's should also be monitored throughout analysis. High RSD's (greater than 20%) for concentrations above the MDL can indicate memory interference from previous samples, as well as other instrumentation issues that may need to be corrected before analysis can be continued.

14.8.4 Element/LIMS Data

The analyst and second reviewer must verify quantities imported into the LIMS reflect the raw data. This can be accomplished by checking a few analytes for random client samples and QC samples. Hand entered data (i.e., dilution) should be verified by the second reviewer to be certain the values, dilution factors and flags are properly inserted. The LIMS calculated final value should also be checked to ensure the system is correctly using the method's custom equation.

14.8.5 Multiple Isotopes

If an element has more than 1 monitored isotope, examine the concentration calculated for each isotope, or isotope ratios, to detect a possible spectral interference. Consider both primary and secondary isotopes when evaluating the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences that the primary recommended isotopes; therefore, differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes.

14.8.6 Reprocessing Data

Reprocessed data must include reprocessing of all calibration standards and QC samples associated with the reprocessed sample results. Original data shall be kept with the reprocessed data and annotated with the reason for reprocessing. It is imperative that the calibration file be saved as a separate file from the original calibration file. The reprocessed calibration file shall be renamed with the added distinction of "_reprocessed" (i.e. 03052010_reprocessed). All other samples including QC are automatically saved to the dataset file.

15.0 CALCULATIONS

15.1 Analyte Concentration

Metal concentration in the air sample should be calculated as follows:

$$C = \frac{[C_i \times V_f \times 9]}{V_{std}}$$

Where:

C = concentration, ng metal/m3

C_i = metal concentration determined from Section 14.4, ng metal/L.

V_f = total sample extraction volume from extraction procedure (i.e., 0.05 L).

9 = [Usable filter area (8"×9")] / [Exposed area of one strip ((4"×1") × 2 (representing a strip that has been folded)]

V_{std} = standard air volume pulled through the filter, m3

15.2 Method Detection Limits

The MDL is calculated as follows for the CFR MDL calculation method:

$$MDL = (K) \times (SD)$$

Where:

K = K Value as prescribed in Appendix D: DQ FAC Single Laboratory Procedure v2.4, 08/30/2007
SD = standard deviation of the historical BLK2 analysis.

15.3 Relative Percent Difference (RPD)

The RPD is calculated as follows:

$$RPD = \frac{R_1 - R_2}{(R_1 + R_2)/2} \times 100$$

Where:

R_1, R_2 = values that are being compared (i.e., duplicate and replicate analysis data)

15.4 Percent Recovery

Percent Recovery is calculated as follows:

$$Percent\ Recovery = \frac{Analytical\ Result}{Theoretical\ Result} \times 100$$

15.5 Relative Standard Deviation (RSD)

RSD is calculated as follows:

$$RSD = \frac{Standard\ Deviation}{Average} \times 100$$

16.0 QUALITY CONTROL

The analyst must perform the quality control checks listed in Table 24-3 and meet the requirements in this section. Data Quality Objectives (DQO) and data assessment criteria are determined from the results of the quality control samples. The DQO criteria are summarized in Table 24-3.

16.1 Sample Collection Quality Control

16.1.1 Filters which are dropped or become contaminated with any foreign matter (i.e., dirt, finger marks, ink, liquids, etc.) are invalid.

16.1.2 Filters with tears or pinholes that occurred before or during sampling are invalid.

16.1.3 A power failure during a field sample collection event invalidates the sample collected during that event. See SOP ERG-MOR-045 for more information about the sample acceptance criteria.

16.2 Blanks

16.2.1 The Rinse Blank must be used to flush the system between standards and samples. Refer to Section 11.3.1 for preparation details.

16.2.2 Initial Calibration Blank (ICB) is analyzed immediately following the initial calibration verification. The absolute value of the instrument response should be less than the method detection limit. If the ICB fails it may be re-analyzed twice. If the ICB still does not pass, the analysis should be terminated, the problem corrected and the ICV and ICB must be verified again before the analysis can continue. If the ICV or ICB still fail, recalibrate the instrument and verify the ICV and ICB again before continuing. If recalibration fails to correct the problem, all QC and samples included in the sequence must be flagged.

16.2.3 Continuing Calibration Blanks (CCB) are analyzed following each continuing calibration verification sample. The acceptance criteria are the same as the ICB. If the first CCB analyzed fails it may be reanalyzed twice. If it fails again the analysis should be terminated, the instrument recalibrated and the initial QC repeated. If the CCB fails later in the analysis it may also be reanalyzed twice. If it continues to fail the affected analyte must be flagged. If multiple elements fail any samples analyzed before or after the failing CCB must be reanalyzed.

16.2.4 Laboratory Reagent Blank (LRB/BLK1) consists of all reagents (extraction fluid) used to process samples and is carried through the entire preparation and extraction process to determine the background levels, if any, from the extraction fluid and process. If the absolute value of the analyte concentration of the LRB is less than or equal to the MDL (See Table 24-1), no further action is required. If the BLK1 fails it may be reanalyzed once. If it fails again and the concentration is greater than the MDL, but less than 5 times the MDL, the BLK1 and all associated

samples are flagged. If the concentration of a reported analyte is greater than 5 times the MDL all associated samples must have that analyte result blank subtracted and flagged as such.

16.2.5 The Method Blank (MB/BLK2) is prepared as an LRB but with the addition of a blank quartz filter strip and analyzed as a sample to determine the background levels, if any, from the blank filter and extraction process. Resulting data is recorded and used to determine FAC MDL values. While the absolute value of resulting values should be less than the established MDL, there is no corrective action procedure should the BLK2 fail.

16.3 Spikes

16.3.1 Laboratory Control Sample (LCS/BS): An LCS is prepared and carried through the entire sample digestion process. At least one LCS is analyzed with each sample batch. The LCS is essentially a MB with a spike added and is referred to as a BS. The results of the spike recovery must be within 80 -120% of actual values, with the exception of Al and Sb. Mercury may recover within 75-125% of actual value. In cases where background contamination is present from the filter media, these criteria may fail; however, it is appropriate to subtract the known background concentration found in the method blank but if it still fails the LCS must be flagged appropriately.

16.3.2 Standard Reference Material (SRM) is prepared and carried through the entire sample digestion process. At least one SRM is analyzed with each batch. The SRM is a NIST Urban dust that is prepared with a blank filter and can be referred to as a SRM in the LIMS. The results of the SRM Pb recovery must be within 80 – 120% of the certified value. If not, the sample may be reanalyzed once. If the SRM still fails to meet criteria, all associated samples must be flagged.

16.3.3 Matrix Spike and Matrix Spike Duplicate (MS/MSD): A sample from an extraction is chosen to be the source sample for the MS/MSD sample. Two extra filter strips are cut from the source sample filter for this purpose. They are prepared and carried through the entire preparation and extraction procedure as described in Section 14.2. These samples should recover within 75% to 125% of the true value. If not, the MS and/or MSD may be reanalyzed once each. If they still do not pass, examine the result of the PDS and SRD for possible matrix interference. If matrix interference is confirmed, the MS and/or MSD sample must be flagged as such. If the PDS and SRD are within their acceptance criteria and a matrix interference is not suspected, re-extraction of the samples should be

considered. Otherwise, all related samples may be flagged to indicate MS DQO failure.

Note: If the parent sample concentration is greater than 4 times the spike concentration, the recovery criteria does not apply and the sample should be flagged appropriately.

16.3.4 Post Digestion Spike (PDS, also known as PS): The PDS is an analyzed aliquot of an extracted sample that is spiked with the PDS standard (See Section 11.16). Preferably, the same sample that is used for the MS/MSD is also spiked for the PDS. The PDS spike addition should produce a minimum level of 10 times and a maximum of 100 times the QL. The spike recovery should be within ± 25%, or 75% to 125%, of the true spike value. If the PDS fails, the analyst should assess the SRD. If both the PDS and SRD fail for the same element it is an indication that matrix interference has occurred and any analytes that have failed should be flagged as possible matrix interference. In the event that the parent sample used for the PDS requires a dilution, the PDS should be prepared by diluting the sample and then spike with the PDS standard.

16.4 Duplicates

There are three types of duplicates found in this procedure:

16.4.1 Matrix spike duplicate (MSD): A true spiked laboratory duplicate of a separate strip of the parent (also called source) sample filter used to prepare the matrix spike. The RPD for this duplicate is ± 20%.

16.4.2 Laboratory duplicate: This duplicate is also prepared with an additional strip of the parent (or source) sample filter. An RPD of ± 20% from the parent sample values is required for values greater than or equal to 10 times the MDL.

16.4.3 Analytical duplicate (or replicate): A second aliquot of an extracted sample analyzed using the same analytical method as the first, or primary aliquot. These are performed on duplicate and collocated samples collected in the field. The RPD for analytical duplicates (replicates) shall be less than or equal to ± 10% for values greater than or equal to 10 times the MDL. If a duplicate/collocated sample fails to meet the established criteria, the duplicate or collocated sample must be reanalyzed. If initial sample results are confirmed then the replicate or collocated sample must be flagged.

16.5 Performance Evaluation (PE) Samples

Performance evaluation samples should be obtained as available from independent sources and analyzed as a routine sample. PE samples are prepared and analyzed in the same way as field samples and should be analyzed in replicate to verify results.

16.6 Standard Checks

16.6.1 Immediately after the initial calibration, the ICV is analyzed. The measured concentrations should be within ±10%, or 90-110%, of the actual concentration. If the criteria are not met, reanalyze the standard. If the criteria are still not met, a fresh standard may be prepared and analyzed or repeat the initial calibration and ICV.

16.6.2 The HSV must be analyzed after the ICB and prior to analysis of samples. The measured concentration should be within 95% to 105% of the actual concentration. If the HSV fails it may be reanalyzed twice. If the HSV still fails, a fresh standard may be prepared and analyzed. If it continues to fail, the instrument must be recalibrated and all initial QC must also be reanalyzed.

16.6.3 Before the analysis of samples and after every 10 samples during a batch analysis, the calibration must be verified using a CCV. Results must be within ±10%, or 90% - 110%, of the target value for each analyte to verify that the calibration is valid. If a standard check exceeds the limit, the analysis must be stopped and the check standard must be reanalyzed. If the target value exceeds the limit again, a fresh standard may be prepared and analyzed or the instrument must be recalibrated. Any samples analyzed before or after an invalid CCV must be reanalyzed, minimally for the failing element.

16.6.4 Following the first and last CCV of each analysis, an LCV must be analyzed. The measured Pb concentration should be within ± 30% of the true concentration. If the criteria are not met for the first LCV, reanalyze the standard once. If criteria still are not met, a fresh standard may be prepared and analyzed or terminate the analysis, correct any issues, and repeat the initial calibration, and reanalyze all initial QC. Should the second LCV fail for Pb, the analysis should be reanalyzed for Pb only.

16.6.5 Document each standard check value as a percent ratio of the actual value over the target value.

16.7 Internal Standards

The intensities of all ISTDs must be monitored for every analysis (see Table 24-7 for isotopes). When the intensity of any ISTD fails to register between 60 to 125% of the intensity of that ISTD in the calibration blank, the following procedure is implemented:

16.7.1 If the intensities are too high as a result of the internal standard being present in the sample, the sample must be diluted and reanalyzed with the addition of appropriate amounts of ISTD.

16.7.2 Repeat and increase the dilution until the internal standard intensities fall within the prescribed window.

16.7.3 If the intensities are determined to be a result of instrument drift, stop the analysis, find and correct the problem, recalibrate if needed and reanalyze the affected samples since the last acceptable ISTD recoveries.

16.8 Interferences

16.8.1 The interference check standards (ICS) are analyzed at the beginning and end of the run and for every 8 hours of continuous operation. They consist of two different standards, the ICSA and ICSAB. The interference check standards obtained from suppliers are never contaminant free. The certificate of analysis documents the levels found in each lot obtained. Therefore, the presence of target elements in the ICSA and ICSAB are expected and may vary from lot to lot of ICS solution used to make these standards. In some cases these contaminants may cause the ICSA or ICSAB to fail the QC requirements. It is acceptable to use the concentrations from the certificate of analysis to correct for these observed contaminants. The known concentrations of contaminants in the ICSA or ICSAB solutions may be subtracted from the experimental values and the established QC criteria must be met or the associated samples are flagged. Samples containing levels of the interferents above the levels in the ICS should be considered for dilution.

16.8.2 The first interference check standard (ICSA) contains only the interference analytes of interest. Any components of the ICS that are calibrated by the instrument must recover within 20% of the expected value. Values obtained for reported analytes not present in the standard should be within ±3 times the QL values (See Table 24-3). If the ICSA fails, it may be reanalyzed. If it fails again, a fresh standard may be prepared and analyzed. If it continues to fail, terminate the analysis and correct the

problem. .Any samples analyzed before or after a failing ICSA sample must be reanalyzed.

16.8.3 The second interference check standard (ICSAB) contains the same concentration of interference analytes in the ICSA as well as a known concentration of reported analytes. The concentration of known analytes should be near the middle of the calibration curve. The values obtained for reported analytes should be within 80% to 120% of the known concentration. If the ICSAB fails it may be reanalyzed. If it fails again a fresh standard may be prepared and analyzed. If it continues to fail terminate the analysis and correct the problem. Any samples analyzed before or after a failing ICSAB sample must be reanalyzed.

16.8.4 Failing ICS are commonly the result of correction equations that need to be adjusted for new instrument conditions. If this is suspected, calculate the new interference equations and reprocess the analytical sequences as described in Section 14.8.6.

16.9 Dilutions

16.9.1 Serial Dilution

The SRD analysis must be performed on one sample per batch, preferably on the parent sample chosen for the MS/MSD as guidance for poor recoveries in the spiked samples that may be due to matrix interference. The same sample matrix used to extract the parent sample must be used as the diluent. After the dilution is applied to the SRD results, the analyte concentration should be within 90% and 110% of the undiluted sample results if the parent sample analyte concentration is minimally a factor of 50 above the MDL in the original sample. If the SRD, MS/MSD, and PDS all fail for the same analyte, a matrix interference must be suspected and the QC data must be flagged for all affected elements. If matrix interference is not suspected, the SRD should be reprepared and analyzed a second time.

16.9.2 Sample Dilution

Any samples needing dilution due to concentrations exceeding the LDR or the presence of ISTD in the sample shall be diluted in an appropriate manner to bring the diluted concentration within the calibration curve and preferably near the mid-point. See Section 16.7 for dilutions required due to high ISTD recoveries. If the parent sample of a DUP/MS/MSD/PDS sample needs to be diluted, the dilution should be performed on each of these QC samples (See also Section 16.3.3). If the SRD was performed on

the parent sample of a DUP or PDS the results from the SRD may be used to report values of that parent sample if the 5 times dilution was appropriate.

Note: Dilution also increases the associated MDL by the dilution factor, so care must be taken not to dilute a sample so that the corrected concentration value is less than increased MDL.

16.10 Initial Demonstration of Capability

Each analyst must demonstrate initial proficiency for sample preparation and analysis by generating data of acceptable accuracy and precision for four LCSs. For demonstration of proficiency, acceptable accuracy and precision is defined as having both RSD ≤20% and percent recovery of 75-125%. This demonstration is repeated whenever new staff receives training or significant changes in extraction procedure or instrumentation are made. The applicable LCS recoveries are collected and maintained in the staff training files.

16.11 Decontaminating/Cleaning Labware

Procedures for proper cleaning and removal of trace metals from labware are found in Section 14.3.1 in SOP ERG-MOR-031. Detergent bath preparation instructions can be found in Section 11.19. Transport/dip baths of DI water ("To" and "From" acid baths) should be labeled with the date they are filled with fresh DI and changed about once every month to minimize contaminants being brought to the acid bath and from being reintroduced to clean labware. This will not only extend the life of the acid bath but it will help reduce hazardous waste production. To be certain that the acid bath will effectively clean and not contaminate labware a 5x dilution should be analyzed to determine the background about once a month.

17.0 PREVENTION

When possible, minimize the amount of chemicals used in the preparation and analysis of the metals filters to reduce waste.

18.0 DATA REVIEW AND CORRECTIVE ACTION

18.1 Data Review Documentation

Project files including at a minimum the information required in Section 22 are assembled and maintained by the performing analyst. Documentation for metals analysis by ICP/Mass Spectrometry will be reviewed for completeness and

meeting acceptance criteria by the Task Lead or secondary reviewer associated with the project or program requiring the analysis as described in this section.

A second review of the data is performed by the Task leader or designated secondary reviewer using the QC review checklist (checklist) shown in Figure 24-8 to confirm that quality requirements have been met. Corrections and flags are added to the data consistent with the corrective action required for each review finding. Second level reviewers must complete, initial and date the checklist.

The completed check list is included as part of the data package. Data not meeting SOP requirements are flagged and brought to the attention of the Project Manager for resolution.

18.2 Quality Staff Review

A minimum of 10% of the data is reviewed by ERG Quality Staff. Quality staff review checks that all SOP required quality parameters have been met and that data reviewers have completed their review checklists. Additional items may also be reviewed at the discretion of the data reviewer. Quality staff reviews are documented on the review form initiated in Section 18.1 by the primary data reviewer. Comments or issues with data identified by the Quality Staff reviewer are brought to the attention of the Project Manager for resolution. Quality Staff will use the review process as an indication of episodic or systematic quality program issues that may require improvements to the ERG laboratory quality system and or additional training for ERG staff.

As an option, Quality Staff may request an additional review of 1% of the data from this method for a project. The one percent (1%) review will follow the guidance in Section18.2.

Corrective action for metals by ICP/Mass Spectrometry analysis data quality issues are presented in Table 24-3.

If required, a corrective action form is filled out as described in the ERG Laboratory Quality Systems Manual.

19.0 WASTE MANAGEMENT

Hazardous waste disposal is discussed in detail in SOP ERG-MOR-033. Sample digestate, including the extracted filter and FilterMate™ filter, is retained in its original tube for a period of at least 6 months from the sample date. After this time, sample digestate is disposed of by pouring it off in the satellite waste containers located in the laboratory. Used sample vials are disposed of in the laboratory waste bin.

20.0 MAINTENANCE

The ICP-MS system is not maintained under a service contract. The preventative and routine maintenance are performed by the analyst(s). In the event a maintenance issue cannot be addressed by the analyst a service call is placed with the manufacturer and when appropriate a service engineer will perform any necessary maintenance. All maintenance activities are documented in the instrument maintenance logs.

20.1 The following maintenance procedures need to be addressed daily.

20.1.1 Check sample waste container level.

20.1.2 Inspect liquid argon cylinder supply and its pressure to the instrument.

20.1.3 Inspect chiller coolant level and connections for possible leaks.

20.1.4 Inspect torch and aerosol injector tubes.

20.1.5 Inspect nebulizer for clogs.

20.1.6 Inspect sample capillary tubing to be sure it is clean and in good condition.

20.1.7 Check peristaltic pump tubing before operation.

20.1.8 At the end of each analysis, flush system for 5 minutes with the plasma on with a maximum of 2% nitric acid, followed by deionized water.

20.1.9 Inspect vacuum pump oil level and replace as needed.

20.1.10 Inspect sample and skimmer cones for excessive salt build-up.

20.2 The following maintenance procedures need to be addressed quarterly (or more frequently if instrument performance indicates maintenance is needed).

20.2.1 Clean torch components and replace any worn O-rings on the torch assembly.

20.2.2 Inspect and clean the RF coil.

20.2.3 Inspect nebulizer spray pattern. Clean and replace gem tips and O-rings as necessary.

20.2.4 Check nebulizer components and replace worn O-ring on the transducer face.

20.2.5 Check spray chamber drain fitting for leaks.

20.2.6 Check that pump rollers are clean and remove and clean pump head as necessary.

20.2.7 Clean skimmer and sampling cones (See Section 20.3) and inspect orifices for damage or corrosion. Replace cones and/or O-rings as needed.

20.2.8 Replace interface roughing and turbo backing vacuum pump oil.

20.2.9 Inspect autosampler rinse pump rollers and clean or replace as necessary.

20.3 Cleaning Sample Introduction Components

20.3.1 Fill small plastic tank for sonicating components with warm to hot 2% Citranox® solution. Place torch, injector, spray chamber, gem tips, and transfer tube (remove all o-rings) into the tank. Sonicate all components for 15 minutes.

20.3.2 After removing the o-rings from the cones, **gently** insert each cone into a separate beaker with enough detergent solution to cover completely and allow to soak for ~ 1.75 hours. Care must be taken to only handle cones by the base – any contact with either cone orifice can easily cause irreparable damage.

20.3.3 Rinse all components thoroughly with tap water followed by a thorough rinse with DI water.

20.3.4 Place all components back in the cleaned tank/beakers with DI water and sonicate for another 15 minutes. Cones should be contained in a separate container from the other sample introduction components.

20.3.5 Carefully remove sample introduction components and rinse thoroughly with DI water.

20.3.6 Inspect cones under dissecting microscope to be sure that the edges of the orifices are not damaged and that the surfaces inside and out of the cone orifice area have been cleaned well. If the cones still appear to be dirty, place in 2% HNO_3 and sonicate for **no more than** 2 minutes (more than 2 minutes of acid exposure will damage the cones) then rinse thoroughly with DI water and reinspect. If cones are needed immediately they may be dried by spraying Dust Off® over the entire surface, taking care not to make physical contact with the cones.

All other sample components that need further cleaning should be sonicated for 2 or more minutes in 2% HNO_3. Place in Class 100 hood or leave to air dry on a dust free cloth (e.g., Technicloth®) or Kimwipes® may be used.

20.3.7 For cones that are still dirty after the procedure described in 20.3.6 and believed to be usable, refer to the Spectron Cone Cleaning Guide for additional procedures. Cones that are no longer usable may be returned to the manufacturer for recycling and platinum cones can be sent in for refurbishing.

21.0 SHORTHAND PROCEDURE

The flow chart shown in Figure 24-9 shows the procedural steps and sequence for analysis of inorganic samples.

22.0 DOCUMENTATION AND DOCUMENT CONTROL

22.1 All information concerning sample preparation, standard preparation, instrument conditions, etc., must be documented in the appropriate binders (i.e., Extraction Log, Daily Performance Reports, Standards Log etc.) and/or electronically in either the LIMS or the local instrument computer.

22.2 All calculations and the type of method for determining concentration must be recorded in the analyst's notebook. Any unusual problems or conditions must also be noted.

22.3 Record all maintenance performed on the instrument in the maintenance logbook for this particular instrument.

22.4 Record all sample analyses, including quality control samples, performed by the instrument in the ICP-MS run logbook for this particular instrument.

22.5 Reviewer must sign laboratory notebook weekly.

22.6 Any hard copies of instrument data should be filed chronologically. Electronic copies of instrument data are maintained on the L: drive in the “Metals Lab” folder.

22.7 It is imperative the project documentation be updated following each analysis. Analysts will copy raw instrument and QC files to a designated corporate network shared drive at the completion of each analysis sequence or batch. Primary data reviewers will use the data on the shared network drive for their data review

process. The completed data packages ready for upload into the ERG LIMS system will be retained on the network drive as the backup for this data.

22.8 All processed data are archived in the LIMS on the shared network drive. Data is archived monthly to compact disc (CD) or digital versatile disc (DVD), verified on the system where the data originated and stored for at least five years in the laboratory. An archive copy of a data package is retained for at least five years in the laboratory data storage. The data backup should include enough information to manually generate the numbers used for reporting.

22.9 Reporting

22.9.1 Sample results are uploaded into the LIMS in ng/L as analyzed. Any dilutions performed must be accounted for in the instrument software. The internal standard recoveries must be included with the result calculation. Final results should be reported in ng/m^3 to three significant figures as shown in Section 12.1. If required by the sampling organization, results can also be reported in $\mu g/m^3$ by multiplying the ng/m^3 results by 1,000.

22.9.2 Sample results should not be corrected based on analyte results from the laboratory blanks, field, trip, or filter lot blanks provided by sampling agencies, unless specifically requested. However, samples are blank-subtracted if any analyte is detected in the LRB at greater than 5 times the MDL as described in Section 16.2.4.

22.9.3 As stated in Section 11.2.6, samples with metal concentrations greater than 90% of the current LDR must be diluted and re-analyzed. The diluted value will be reported to the sampling agency.

22.9.4 Data should meet all specifications as presented in Table 24-3. If data does not meet specifications, corrective reporting actions listed must be followed (flag or invalidate data).

23.0 REFERENCES

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24.0 TABLES, DIAGRAMS, FLOWCHARTS, VALIDATION DATA

Table 24-1. 2015 Method Detection Limits (MDLs) for Metals Hi-Vol Filters

Element	2015 MDL by FAC[1] (ng/L)	2015 MDL by FAC[2] (ng/filter)	2015 MDL by FAC[2,3] (ng/m^3)
Aluminum *	88512	39830	19.9
Antimony	55.1	24.8	0.012
Arsenic	252.8	114	0.057
Barium *	17901	8055	4.03
Beryllium	9.468	4.26	0.002
Cadmium	25	11.24	0.006
Calcium *	873889	393250	197
Chromium	11376	5119	2.56
Cobalt	189.6	85.3	0.043
Copper *	2302	1036	0.518
Iron *	112437	50596	25.3
Lead	503	226	0.113
Magnesium *	202561	91152	45.6
Manganese	966.8	435	0.218
Mercury	28.5	12.82	0.006
Molybdenum *	628.3	283	0.141
Nickel	2542	1144	0.572
Rubidium *	69.2	31.1	0.016
Selenium	137.2	61.8	0.031
Strontium *	1721	774	0.387
Thallium *	1.57	0.705	0.0004
Thorium *	15.3	6.86	0.003
Uranium *	25.6	11.5	0.006
Zinc *	34975	15739	7.87

* Elements not on our standard analysis list of elements.
† Total Chromium.

Table 24-2. 2015 Limit of Quantitation (LOQ) for Metals

Element	ng/L
Aluminum*	78015
Antimony	100
Arsenic	742
Barium*	32676
Beryllium	25.6
Cadmium	58.2
Calcium*	817139
Chromium†	11396
Cobalt	467
Copper*	3672
Iron*	205994
Lead	982
Magnesium*	264057
Manganese	1460
Mercury	59.2
Molybdenum*	553
Nickel	3935
Rubidium*	129
Selenium	428
Strontium*	2839
Thallium*	2.95
Thorium*	33.0
Uranium*	20.6
Zinc*	56292

* Elements not on our standard analysis list of elements
† Total Chromium.

Note: This calculation assumes a total volume of 2000 m^3.

Table 24-3. Summary of Quality Control Procedures for Metals Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Daily Performance Check (DPR)	Daily, prior to samples	Mg-24 > 40,000 cps, < 3% RSD In > 300,000 cps, < 3% RSD Pb-208 >100,000, <3% RSD Ba/Ba++ and Ce/CeO < 0.03 Bkgd < 30 cps at Mass 220	1)Repeat analysis of DPR 2)Re-optimize instrument tuning parameters 3) Reprepare DPR standard 4) Perform instrument maintenance
Initial Calibration Standards (IC)	Daily, at least 5 calibration points	Correlation coefficient ≥ 0.998 & %RSD ≤ 10. RSDs >10 are acceptable for the CAL2 standard.	1) Repeat analysis of calibration standards 2) Reprepare calibration standards and reanalyze
Initial Calibration Verification (ICV)	Immediately after calibration	Recovery 90-110%, with the exception of Al	1) Repeat analysis of ICV 2)) Reprepare ICV standard 3Recalibrate and reanalyze
Initial Calibration Blank (ICB)	Immediately after ICV	Absolute value must be ≤ MDL	1) Locate and resolve contamination problems before continuing 2) Reanalyze, recalibrate or flag failing elements for the entire analysis when appropriate
High standard verification (HSV)	After ICB and before ICS	Recovery from 95-105% with the exception of Al	1) Repeat analysis of HSV 2) Reprepare HSV
Interference Check Standard (ICSA/IFA)	Following the HSV, every 8 hours and at the end of each run	Within ±3 times LOQ from zero or from the standard background contamination when present	1) Repeat analysis of ICSA 2) Reprepare ICSA and analyze 3) Adjust correction equation(s) and reprocess entire analysis
Interference Check Standard (ICSAB/IFB)	Following each ICSA	Recovery 80-120% of true value plus standard background contamination when present	1) Repeat analysis of ICSAB 2) Reprepare ICSAB and analyze 3) Adjust correction equation(s) and reprocess entire analysis
Continuing Calibration Verification (CCV)	Analyze before samples, after every 10 samples, and at the end of each run	Recovery 90-110%, with the exception of Al	1) Reanalyze CCV 2) Reprepare CCV 3) Recalibrate and reanalyze samples since last acceptable CCV
Low Calibration Verification (LCV)	At the beginning and end of each analysis, between the CCV and CCB	Recovery 70-130% for Pb only	1) Reanalyze LCV 2) Reprepare LCV 3) Recalibrate and reanalyze samples since last acceptable LCV
Continuing Calibration Blanks (CCB)	Analyzed after each CCV	Absolute value must be ≤ MDL	1) Reanalyze CCB 2) Reanalyze samples since last acceptable CCB
Laboratory Reagent Blank (LRB/BLK1)	1 per 20 samples, a minimum of 1 per batch	Absolute value must be ≤ MDL	1) Reanalyze 2) If > MDL, but < 5x MDL, sample results for that element must be flagged for the entire analysis 3) If > 5x the MDL then sample results for that element must be blank subtracted
Method Blank (MB/BLK2)	1 per 20 samples, a minimum of 1 per batch	Absolute value must be ≤ MDL. Note: The MB is used only for the purpose of MDL generation	This standard is not required by the method and there is no corrective action

Table 24-3 (Cont'd) Summary of Quality Control Procedures for Metals Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Standard Reference Material (SRM)	1 per 20 samples, a minimum of 1 per batch	Recovery 80-120% for Pb only	1) Reanalyze 2) Flag sample data 3) Re-extract batch
Laboratory Control Sample (LCS/BS)	1 per 20 samples, a minimum of 1 per batch	Recovery 80-120%, with the exception of Al and Sb	1) Reanalyze 2) Flag data if recovery for only one or two elements fail criteria 3) Re-prepare sample batch if recovery for most elements fail criteria
Replicates (DUP) (Replicate Analysis)	1 per 20 samples, a minimum of 1 per batch	≤ ±20% RPD for sample and duplicate values ≥ 10 times the MDL	1) Check for matrix interference in the cased of DUP1. 2) Repeat replicate analysis 3) Flag data
Collocated Samples (C1/C2)	10% of samples annually	≤ ±20% RPD for sample and collocate values ≥ 10 times the MDL	1) Flag C2 data if associated replicate analysis are within criteria 2) Repeat analysis if replicate analysis fail.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD) for 8x10" Quart filters only	1 per 20 samples per sample batch	Recovery 75-125%, with the exception of Al and Sb, when the parent sample concentration is less than 4 times the spike concentration	1) Flag data if recovery for only one or two elements fail criteria, or when a matrix interference is confirmed by SRD and/or PDS results 2) Reanalyze 3) Reprepare sample batch if recovery for most elements fail criteria or contamination is evident.
Post Digestion Spike (PDS)	1 per 20 samples, minimum of 1 per batch	Recovery 75%-125%	1) Flag failed elements for parent sample and PDS 2) Reprepare PDS if preparation issue is suspected reason for failure.
Serial Dilution (SRD)	1 per batch	Recovery 90-110% of undiluted sample if the element concentration is minimally a factor of 50 above the MDL in the original sample	1) Re-prepare dilution if preparation issue is suspected reason for failure. 2) Flag failed analytes
Internal Standards (ISTD)	Every Calibration, QC and Field Sample	Recovery 60-125% of the measured intensity of the calibration blank	1) If drift suspected, stop analysis and determine cause, recalibrate if necessary 2) Reprepare sample 3) If recovery > 125% due to inherent ISTD, dilute sample and reanalyze

Table 24-4. Optimization Procedures

Procedure	When to Perform
Nebulizer Gas Optimization	Daily.
Ion Lens Voltage Optimization	Daily.
Auto Lens Optimization	Daily, when Auto Lens is used in the acquisition method.
Dual Detector Cross Calibration	Cross calibration is necessary if you have selected Dual Mode as your Processing Method. This would only be done when you require extended dynamic range (above 2 million cps). **Note:** This must be performed before each analysis for this method.
Instrument Performance Check	Daily.
X-Y Adjustment	Whenever the cones have been cleaned or replaced, or after any torch maintenance procedure.
Detector Optimization: Pulse Stage Voltage Analog Stage Voltage	When sensitivity cannot be recovered through other cleaning or optimization methods or when the detector is replaced.
Deadtime Correction	This procedure should only be performed if a detector has been replaced, and after the new detector has been optimized.

Table 24-5. Instrument Performance Specifications

24Mg Sensitivity	> 40,000 cps	< 3% RSD
In Sensitivity	> 300,000 cps	< 3% RSD
208Pb Sensitivity	> 100,000 cps	< 3% RSD
CeO/Ce	<0.03	N/A
Ba++/Ba+	<0.03	N/A
Background	< 30 cps @ Mass 220	N/A

Table 24-6 Matrix Spike Standard Preparation

Element	Stock Std. Concentration (µg/mL)	Added Volume (mL)
Al	1000	1.5
Sb	100	0.25
As	100	1.0
Ba	1000	0.5
Be	100	0.25
Cd	100	0.25
Ca	1000	1.25
Cr	100	2.5
Co	100	0.25
Cu	1000	0.5
Fe	1000	0.5
Pb	100	0.25
Mg	1000	0.5
Mn	100	0.5
Hg	10	0.5
Mo	100	0.25
Ni	100	0.5
Rb	1000	0.025
Se	100	1.0
Sr	100	0.25
Tl	10	0.5
Th	10	0.5
U	10	0.5
Zn	1000	0.5

Table 24-7. Analytical Isotopes for Quantitation and Monitoring of Reported Elements

Element	Quantitation Isotope(s)	Monitored/Confirmation Isotope(s)
Aluminum	27	NA
Antimony	121	123
Arsenic	75	NA
Beryllium	9	NA
Barium	137	135
Bismuth (ISTD)	209	NA
Cadmium	111	106, 108, 114
Calcium	43	NA
Chromium	52	53
Cobalt	59	NA
Copper	63	65
Iron	57	54
Gallium (ISTD)	71	NA
Indium (ISTD)	115	NA
Lead	208	206, 207
Lithium (ISTD)	6	NA
Magnesium	25	24
Manganese	55	NA
Mercury	201	200, 202
Molybdenum	98	92, 94, 95, 97
Nickel	60	62
Rubidium	85	NA
Scandium (ISTD)	45	NA
Selenium	82	77, 78
Strontium	88	NA
Thallium	205	203
Thorium	232	NA
Uranium	238	NA
Yttrium (ISTD)	89	NA
Zinc	66	67, 68

NA = Not applicable/none.

Figure 24-8. Quality Control Review Form (Page 1)

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Quality Control Review Form Metals Analysis -- 8x10" Quartz Filters 2015-1

Sequence ID: _____ **Instrument:** _____ **Batch:** _____
Cal Curve (Method): _____ **Analyst:** _____ **Date:** _____
10% Review Sample IDs: _____ **Reviewer:** _____ **Date:** _____
Optional 1% Review Sample IDs: _____ **Reviewer:** _____ **Date:** _____

Parameter	Acceptance Criteria	Analyst Check (Initials and Date)	Task Lead/Data (Initials and Date)	10% QA Review (Initials and Date)	1% Optional QA Review (Initials and Date)	Comments
Instrument QC						
Daily Performance Report	Mg-24 > 40,000 cps, < 3% RSD In > 300,000 cps, < 3% RSD Pb-208 >100,000, < 3% RSD Ba/Ba++ and Ce/CeO < 0.03 Bkgd < 30 cps at Mass 220					
Initial Calibration Standards (IC)	≥0.998□ correlation coefficient & RSD ≤10. RSDs >10 are acceptable for the CAL2 standard.					
Initial Calibration Verification (ICV)	Recovery 90-110%.					
Initial Calibration Blank (ICB)	Absolute value must be ≤ MDL.					
High Calibration Verification (HCV)	Recovery 95-105%.					
Interference Check Standard (ISCA/IFA)	ICSA: ±3 times QL from zero or from the standard background contamination when present					
Interference Check Standard (ICSAB/IFB)	ICSAB: Recovery 80-120% of true value plus standard background contamination when present					
Continuing Calibration Verification (CCV)	Recovery 90-110%.					
Low Calibration Verification (LCV)	Recovery 70-130% for Pb; must be analyzed at the beginning and end of each analysis.					

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Figure 24-8. Quality Control Review Form (Page 2)

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Quality Control Review Form Metals Analysis -- 8x10" Quartz Filters 2015-1 (Continued)

Parameter	Acceptance Criteria	Analyst Check (Initials and Date)	Task Lead/Data (Initials and Date)	10% QA Review (Initials and Date)	1% Optional QA Review (Initials and Date)	Comments
Continuing Calibration Blanks (CCB)	Absolute value must be ≤ MDL.					
Internal Standard Response	Recovery must be between 60 and 125% of the measured intensity of the calibration blank					
Linear Dynamic Range Check	All sample values must less than 90% of the established linear dynamic range.					
Extraction QC						
Check Sample Volume	Check COC or filter envelope against Bench Sheet to make sure sample volumes are correct.					
Laboratory Reagent Blank (LRB/BLK1)	Absolute value must be ≤ MDL.					
Method Blank (MB/BLK2)	Absolute value must be ≤ MDL.					
Standard Reference Material (SRM)	Recovery 80-120% for Pb.					
Laboratory Control Sample (LCS/BS)	Recovery 80-120%, with the exception of Sb (and Al).					
Collocated Samples (C1/C2)	±20% RPD when concentration of either sample is ≥ 10x the MDL					
Replicate Analyses (DUP)	For DUP1: ±20% RPD when concentration of the parent sample is ≥ 10x the MDL, all other DUPs ±10% RPD when concentration of the parent sample is ≥10x the MDL					
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	Recovery 75-125%, with the exception of Sb (and Al).					

Figure 24-8. Quality Control Review Form (Page 3)

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Quality Control Review Form Metals Analysis -- 8x10" Quartz Filters 2015-1 (Continued)

Parameter	Acceptance Criteria	Analyst Check (Initials and Date)	Task Lead/Data (Initials and Date)	10% QA Review (Initials and Date)	1% Optional QA Review (Initials and Date)	Comments
Serial Dilution (SRD)	Recovery 90-110% of undiluted sample if the parent sample concentration is > than 50x the MDL.					
Post Digestion Spike (PDS)	Recovery 75-125%					
Reporting Requirements						
Manual Check of Calculations	Hand calculate an equation - a unit conversion equation, a sample concentration equation, a dilution equation, etc. – to verify equation					
Check LIMS Qualifiers	Check to make sure the LIMS data flags are correct					
Negative Sample Values	Negative sample values must be less than the absolute value of the MDL.					

This review check sheet must be completed by primary data reviewer/TL/QA.

Figure 24-9. Flow Diagram for ICP-MS Preparation and Analysis for PM10 or TSP Filters

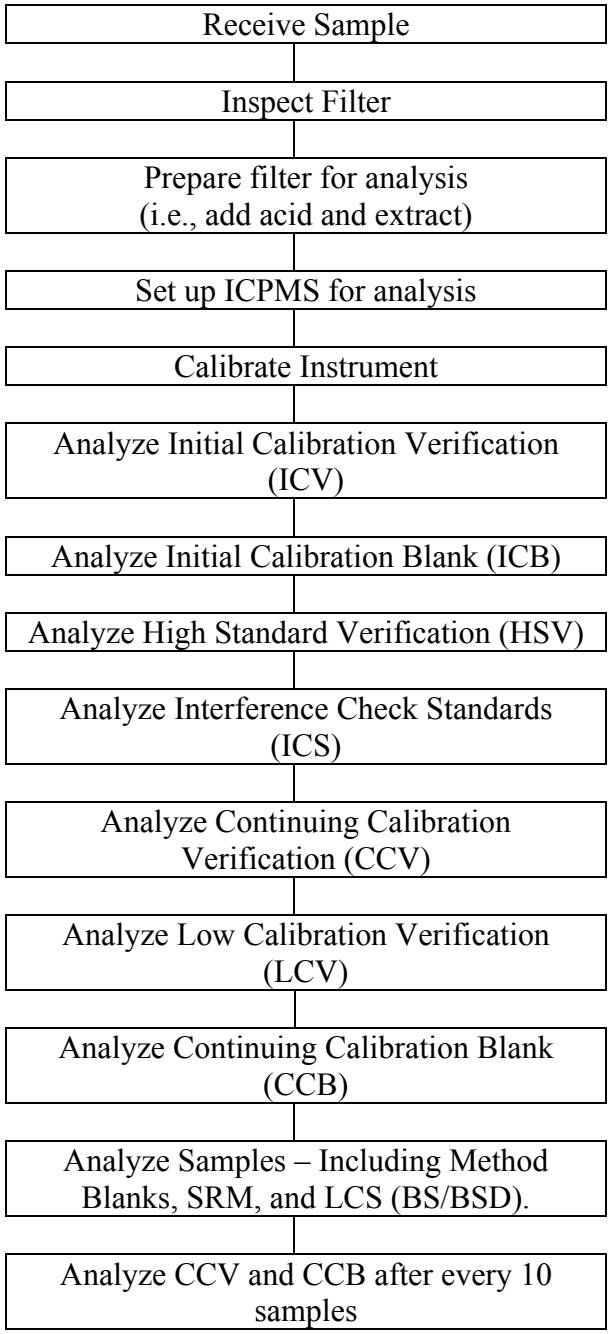

Table 24-10. Quartz Extraction Record

Record for Quartz Extraction Times, Temperatures & H_2O_2 additions 2014-1

Batch: **Date:** **Initials:**_____

Start Time	
Start Block Temperature (°C)*	
Sample Temperature (°C)†/Time	
Time 1st aliquot of H_2O_2 was added	
Time 2nd aliquot of H_2O_2 was added	
Stop Time (Removed from Block)	
Stop Block Temperature (°C)*	
Sample Temperature (°C)†/Time	
Time D.I. H_20 added & shaken/Sample Temp. (°C)	
Time brought to final volume	

*Block temperature is read from the HotBlock™ controller using an internal thermocouple.
†Sample thermometer serial #: 19583, actual thermometer temperature not the corrected temp.;

Thermometer block position # _____

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CONFIDENTIAL
Standard Operating Procedure
Procedure Number: ERG-MOR-085
Revision Number: 10
Revision Date: March 31, 2015
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ENGINEERING AND SCIENCE DIVISION

TITLE: Standard Operating Procedure for the Preparation and Analysis of 47mm Filters for Metals by ICP-MS using Method IO 3.5 and FEM Method EQL-0512-202	EFFECTIVE DATE: APR - 3 2015
REFERENCES: ERG-MOR-031, ERG-MOR-033, ERG-MOR-045, EPA Compendium Method IO-3.5, Corporate Quality Management Plan, ERG Health and Safety Manual, ERG Laboratory Quality Systems Manual, 40 CFR, Part 136, Appendix B, FEM Method "Standard Operating Procedure for the Determination of Lead in PM_{10} by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Hot Block Dilute Acid and Hydrogen Peroxide Filter Extraction" (EQL-0512-202)	
SATELLITE FILES: ICP-MS Laboratory	
REVISIONS: Updated MDLs, Added procedure for extraction record, Removed word duplicate from Collocate/Duplicate listed in Table 24-3 and review checklist, Corrected standard solvent recipe. DST 4/1/15	
WRITER/EDITOR: NAME/DATE Jennifer Nash 4/3/15	PROJECT MANAGER/TECHNICAL DIRECTOR: NAME/DATE Julie C. Swit 4/1/15
QUALITY ASSURANCE COORDINATOR: NAME/DATE Donna Tedder 4/1/15	NEXT SCHEDULED REVIEW: 1/31/2016

1.0 IDENTIFICATION AND PURPOSE

This standard operating procedure (SOP) provides the sample preparation and analysis procedures for suspended particulate matter collected on Teflon® 47mm filters for total metals determination by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS).

2.0 MATRIX OR MATRICES

This procedure applies to the preparation and analysis of ambient particulate matter samples obtained by low-volume sampling on 47mm Teflon® filters for total metals.

3.0 METHOD DETECTION LIMIT

3.1 Method Detection Limits (MDL)

3.1.1 The method detection limit (MDL) for each isotope is calculated according to Appendix D: DQ FAC Single Laboratory Procedure v2.4, 8/30/2007, with the exception of arsenic. MDL values are determined from historic method blank (BLK2) data following the procedure in the document above.

In the case of arsenic, where the FAC MDL calculation method does not provide MDLs lower than an analyte's minimum risk level (MRL), the MDL method described in the CFR is employed instead. This involves spiking ten filters with a known concentration between 3 and 5 times the estimated MDL. The filters are then extracted and analyzed following the entire analytical method.

3.1.2 The y-intercept for each linear calibration must be set to zero.

3.1.3 Use the same internal standards, calibration standards, instrument method and settings (sweeps and dwell) for the MDL study and field sample analysis.

3.1.4 The MDL determination should be reported in ng/L, ng/filter and ng/m3 (assuming 24.04 m^3 per sample). Refer to Table 24-1.

3.1.5 The MDL study should be repeated once per year and whenever a significant change in background or instrument response is expected (e.g., detector change).

4.0 SCOPE AND APPLICATION

4.1 Scope

This procedure details the acid extraction and trace elemental analysis of ambient air samples using an inductively coupled plasma-mass spectrometer (ICP-MS). The extraction procedures are suitable for low-volume ambient air samples collected on Teflon® membrane filters, sized up to 47 millimeters in diameter. The procedure is applicable, but not limited to the metals listed in Table 24-1.

4.2 Applicability

This SOP is applicable to the analysis of suspended particulate matter collected with Teflon® filters. Acid digestion of samples and filtration (if necessary) is

required prior to analysis of Teflon® filter extracts. Analytes for which ERG has demonstrated the acceptability of this method are listed below. See Table 24-7 for a list of isotopes used for quantitation and monitoring.

Element	Symbol	CASRN
Aluminum*	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium*	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium*	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper*	Cu	7440-50-8
Iron*	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium*	Mg	7439-95-4
Manganese	Mn	7439-96-5
Mercury	Hg	7439-97-6
Molybdenum*	Mo	7439-98-7
Nickel	Ni	7440-02-0
Rubidium*	Rb	7440-17-7
Selenium	Se	7782-49-2
Strontium*	Sr	7440-24-6
Thallium*	Tl	7440-28-0
Thorium*	Th	7440-29-1
Uranium*	U	7440-61-1
Zinc*	Zn	7440-66-6

* Elements not on our standard EPA UATMP/NATTS analysis list of elements.

5.0 METHOD SUMMARY

This SOP describes the multi-elemental determination of total metals by ICP-MS in ambient air samples collected on 47mm Teflon® filters following guidelines in EPA method IO-3.5 and EPA FEM Method "Standard Operating Procedure for the Determination of Lead in PM_{10} by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Hot Block Dilute Acid and Hydrogen Peroxide Filter Extraction" (EQL-0512-202). The filters are digested in a HotBlock™ for 2.5 hours using an extraction fluid containing 1.85% nitric acid (HNO_3), 0.5% hydrochloric acid (HCl), and 0.17% hydrofluoric acid (HF) with 8.33 mg/L of gold added for mercury stabilization. One aliquot of hydrogen peroxide (H_2O_2) is added after 1.5 hours of extraction and is allowed

to effervesce. The extract is analyzed by ICP-MS and the data are collected using the manufacturer's software.

6.0 DEFINITIONS AND ABBREVIATIONS

6.1 Definitions

6.1.1 **Analytical Duplicate (DUP).** A second aliquot of a sample extract that is treated the same as the original sample in order to determine the precision of the method. This sample is also referred to as a replicate. Due to the fact that Teflon® filters are not collected as duplicates, the DUP in this method is performed by analyzing a second aliquot of the parent sample digestate as an analytical duplicate. See Section 16.4 for further elaboration on duplicates.

6.1.2 **Blank (BLK).** An analytical sample designed to assess specific sources of contamination. In this method there are two BLKs, the Laboratory Reagent Blank (LRB), which is always reported as BLK1 and the Method Blank (MB), which is always reported as BLK2.

6.1.3 **Blank Spike/Blank Spike Duplicate (BS/BSD).** A spiked aliquot of LRB with a blank Teflon® filter used as a quality control sample (QCS) to demonstrate spike recoveries and precision.

6.1.4 **Calibration Blank.** A volume of ASTM Type I water acidified with the same acid matrix as is present in the calibration standards and sample extracts ready for analysis. This blank is not subject to the extraction procedure but contains the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

6.1.5 **Calibration Standards.** A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions are not subject to the extraction procedure but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample extracts to be analyzed.

6.1.6 **Continuing Calibration Blank (CCB).** The CCB is a re-analysis of the calibration blank after every CCV to verify that the instrument blank checks are reading ≤ MDL.

6.1.7 **Continuing Calibration Verification (CCV).** A multi-element standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV is the original calibration standard

whose concentration is at the midpoint of the calibration curve that is re-analyzed as a quality control (QC) sample.

6.1.8 **Field Blank.** This is any sample that is submitted from the field and is identified as a blank. This also includes trip blanks.

6.1.9 **High Standard Verification (HSV).** The HSV is the highest calibration standard that is reanalyzed to verify the accuracy of the calibration curve at that concentration before the analysis of samples.

6.1.10 **Initial Calibration Blank (ICB).**The ICB is a re-analysis of the calibration blank, which is analyzed after the ICV and used to verify that the instrument blank checks are ≤ MDL.

6.1.11 **Initial Calibration Verification (ICV).** A solution prepared from a stock standard solution obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration.

6.1.12 **Interference Check Standard (ICS).** A solution that may contain only interfering elements (ICSA) or both interfering elements and analytes of interest (ICSAB) in known concentrations that can be used to verify background and interference correction equations.

6.1.13 **Interferents.** Substances (atoms, ions, polyatomic ions, etc.) which may affect the analytical result for the element of interest.

6.1.14 **Internal Standard (ISTD).** A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

6.1.15 **Laboratory Control Sample (LCS).** A spiked aliquot of LRB with a blank Teflon® filter used as a QCS that is prepared and brought through the entire digestion/extraction and analytical process to demonstrate spike recoveries. This sample is synonymous with the BS/BSD.

6.1.16 **Laboratory Reagent Blank (LRB).** An aliquot of ASTM Type I water that is treated exactly as a sample including exposure to all labware, equipment, solvents, reagents and internal standards that are used with other samples that is always reported as BLK1. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or apparatus.

6.1.17 **Limit of Quantitation (LOQ)** – The lowest concentration of an analyte that produces a signal/response that is sufficiently greater than the signal/response of lab reagent blanks to enable reliable detection and quantification during routine lab operating conditions. Statistically defined, this is the concentration of analyte in sample matrix that produces an instrument signal/response that is 10 times the standard deviation above the LRB (at 99% confidence, n-1 degree freedom; see Table 24-2).

6.1.18 **Linear Dynamic Range (LDR).** The concentration range over which the analytical working curve generated from the calibration standards is proven to remain linear. See Section 13.8 for more information on the LDR.

6.1.19 **Lower Limit of Quantitation Check (LLQC)** – A check sample that is used to both establish and confirm the lower limit of quantitation and is prepared by spiking a low concentration of analyte into reagent water and carrying the solution through the entire preparation and analytical procedure.

6.1.20 **Lower Limit of Quantitation Limit (LLQL)** – The lower limit of quantitation is considered the lowest reliable laboratory reporting concentrations and should be established from the lower limit of quantitation check sample and then confirmed using the lowest calibration point and/or from a low level calibration check standard.

6.1.21 **Low Level Calibration Verification (LCV)** – A stock standard solution prepared using the same source as the calibration standards that is analyzed to verify the LLQL. The standard is prepared at the same concentration as the LLQL. An LCV is analyzed at the beginning, typically just before or after CCV1, and at the end of every analysis just before or after the final CCV.

6.1.22 **Matrix Interference/Effect.** In general, the interference and/or effect that particular matrix constituents may cause during sample processing and/or analysis. Matrix effects may be determined to exist from the careful interpretation of QC samples and criteria. Examples of observed effects include but are not limited to poor recoveries of spikes/ISTD and poor percent differences.

6.1.23 **Method Blank (MB).** An aliquot of LRB with a blank Teflon® filter that is carried through the entire preparation and extraction process to demonstrate background contamination contribution from the filter and process and is always reported as BLK2.

6.1.24 **Method Detection Limit (MDL).** The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

6.1.25 **Performance Evaluation (PE) Sample.** A sample of known composition provided by a source outside the laboratory for analysis that evaluates the laboratory's analytical performance.

6.1.26 **Post Digestion Spike (PDS).** A spiked aliquot of an already digested sample used to demonstrate spike recoveries in the sample matrix. The analytical results of this spike may be used to verify matrix interference in conjunction with the SRD results.

6.1.27 **Quality Control Sample (QCS).** A solution containing known concentrations of method analytes that is used to fortify an aliquot of LRB matrix. The QCS is prepared from a source(s) external to the laboratory and is used to verify the laboratory's analytical performance.

6.1.28 **Serial Dilution (SRD).** The dilution of a sample by a factor of five. If the undiluted parent sample concentration is minimally a factor of 50 above the MDL, the diluted sample should agree with the parent sample concentration within ± 10% when corrected by the dilution factor. The SRD may indicate the influence of interferents.

6.1.29 **Standard Reference Material (SRM)** – A prepared standard material that has certified metals concentrations, for example the NIST SRM 1648a (Urban particulate matter with certified concentrations of lead at 0.655 ± 0.033 mass fraction (in %)). The SRM is used to verify the extraction procedure.

6.1.30 **Stock Standard Solution.** A commercially prepared standard solution (traceable to NIST or other certified standard sources), which can be diluted to derive other standards.

6.1.31 **Tuning Solution.** A solution used to determine acceptable instrument performance prior to calibration and sample analyses. This solution is used for mass calibration, nebulizer optimization, auto lens optimization, and daily performance reports.

6.2 Abbreviations

amu	Atomic Mass Units
ASTM	American Society for Testing and Materials
CASRN	Chemical Abstract Services Registry Number

COC	Chain of Custody
cps	Counts Per Second
DI	Deionized
DQO	Data Quality Objective
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
kW	Kilowatt(s)
L	Liter(s)
LDPE	Low Density Polyethelyene
LIMS	Laboratory Information Management System
MΩ	Megohm
MCA	Multichannel Analyzer
MQO	Method Quality Objectives
m	Meter(s)
m^3	Cubic Meter(s)
mg	Milligram(s)
mg/L	Milligram(s) per liter(s)
min	Minute(s)
mL	Milliliter(s)
mm	Millimeter(s)
ms	Millisecond(s)
ng	Nanogram(s)
ng/L	Nanogram(s) per liter(s)
ng/mL	Nanogram(s) per milliliter(s)
NIST	National Institute of Standards and Technology
QC	Quality Control
RSD	Relative Standard Deviation
RPD	Relative Percent Difference
SD	Standard Deviation
SOP	Standard Operating Procedures
Std.	Standard
µg/L	Microgram(s) per liter(s)
$\mu g/m^3$	Microgram(s) per cubic meter(s)
µg/mL	Microgram(s) per milliliter(s)
µL	Microliter
µm	Micrometer
v/v	Volume per volume ratio

7.0 INTERFERENCES

Note: The background level of metals on a given lot of Teflon® filters can vary. Any background levels found on blanks should be documented for all the filters from the corresponding lot when available. It is recommended to consult 40 CFR Part 50, Section 6.1 Appendix G for guidance.

7.1 Laboratory Interferences

7.1.1 Wear powder-free nitrile or neoprene gloves when handling unexposed or exposed filters.

7.1.2 Clean all equipment used in the sample preparation and analysis in a manner consistent with good laboratory practices for metals analysis (See Section 20.3 in this SOP and Section 14.3.1 in SOP ERG-MOR-031).

7.1.3 Use ASTM Type I DI water or equivalent, with a resistivity greater than 17.3 MΩ, for sample extraction and standard preparation. Record the water resistivity prior to use.

7.2 Chemical Interferences

Pay close attention to the nature of solutions introduced to the ICP-MS.

7.2.1 Nitric acid must be less than 2% (v/v) for ICP-MS analysis to minimize the damage to the interface and to minimize isobaric molecular interferences. The use of platinum cones and other acid-resistant sample introduction components can be used for more aggressive acid matrices.

7.2.2 If higher acid extractions are required, dilute final digestate to 2% HNO_3.

7.2.3 The final dilutions of sample extracts must match the acid content of the calibration standards in order to match potential interferences.

7.2.4 The concentrations of dissolved solids in analysis solutions should be less than 2% to protect the sample interface on the instrument and prevent signal suppression. Higher concentrations may plug the sample and/or skimmer cone orifices.

Note: Protect the channel electron multiplier from high chemical concentrations (high ion currents). The channel electron multiplier suffers from fatigue after being exposed to high ion currents. This fatigue can last from several seconds to hours depending on the extent of exposure. During this period, response factors are constantly changing, which causes instrument instability that invalidates the calibration curve, and thereby, invalidates all associated sample results. A sodium bicarbonate ($NaHCO_3$) sample matrix is known to cause this problem.

7.3 Instrument Interferences

7.3.1 Isobaric molecular and doubly charged ion interferences are caused by more than one atom (example, the contribution of ArCl on the 75As signal) or more than one charge (example, MoO+ ions on Cd isotopes).

7.3.2 Spectral interferences result from the presence of other isotopes or ions that have the same atomic weight or mass number as the analyte.

7.3.3 Transport interferences are a specific physical interference associated with the sample nebulization and transport process through the instrument. These usually result from sample matrix components that influence the aerosol formation or cause a change in the surface tension or viscosity. Changes in the matrix composition can cause observed signal suppression or enhancement.

7.3.4 Matrix interferences may be caused by elemental chemical and physical properties in the samples. For matrices of known composition, match the composition of the calibration and QC standards to that of the samples. For matrices of unknown composition, use an ISTD that has been matched to the analytes' chemical and physical properties (i.e., ionization potential, ±50 amu) so that the ISTD and element of interest behave similarly during the analytical process.

7.3.5 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sample and skimmer cones, spray chamber, peristaltic pump tubing and the type of nebulizer all affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interferences.

7.3.6 Lead values are reported from isotope 208; however, all three isotopes must be used to quantitate lead to allow for the variability of lead isotopes in nature. The following correction equation must be applied to isotope 208:

$$(1.000)\ (^{206}\mathrm{Pb}) + (1.000)\ (^{207}\mathrm{Pb}) + (1.000)\ (^{208}\mathrm{Pb})$$

8.0 SAFETY

8.1 Personal protection should be used for all work performed in the inorganic laboratory, (e.g., gloves, safety glasses, laboratory coats, etc.).

8.2 The compressed gas cylinders must be stored and handled according to relevant safety codes outlined in the corporate health and safety manual. In use, the cylinders must be secured to an immovable structure and moved using a gas cylinder cart.

8.3 Make sure that sample vials are kept capped and in racks to prevent spills.

8.4 All personnel should be trained in the handling, extraction and analysis of acid samples for inorganic analysis.

8.5 Strong acids must not be stored with organic solvents or samples.

8.6 Follow normal laboratory safety procedures as outlined in the ERG Health and Safety Manual and the site-specific laboratory SOP.

9.0 EQUIPMENT

9.1 ICP-MS

The PerkinElmer SCIEX™ ELAN® 9000 ICP-MS consists of an inductively coupled plasma source, ion optics, a quadrupole mass spectrometer, a computer that controls the instrument, data acquisition and data handling software (ELAN® Software SCIEX™, Version 3.4), a printer, an autosampler (AS-93plus) and a recirculator. The quadrupole mass spectrometer has a mass range of 2 to 270 amu. Typical operating conditions are listed below.

Typical Operating Conditions

Plasma forward power	1.3 kW
Plasma/Coolant argon flow rate	13.8 L/min
Auxiliary argon flow rate	1.2 L/min
Nebulizer flow rate	0.9 L/min
Solution uptake rate	1.0 mL/min
Spray chamber temperature	Room Temperature
Detector mode(s)	Pulse counting/Analog
Replicate integrations	3
Mass range	6 - 240 amu
Dwell time	50 ms
Number of MCA channels	1
Number of scan sweeps	20
Total acquisition time	4.2 min/sample

9.2 Digestion System

Environmental Express HotBlock™ Digestion System or equivalent system capable of maintaining a temperature of 95°C within ± 2˚C. This temperature will heat the samples to a temperature of ~85°C (±5°C).

10.0 MATERIALS

10.1 Graduated polypropylene sample vials with screw caps, 50 mL volume (certified to be within ± 0.2mL).

10.2 Branson 8510 sonication bath with heating capability.

10.3 Pipetters with adjustable volumes ranging from 0.5 µL to 10 mL and disposable tips. Mechanical pipettes must be verified for accuracy quarterly (or every three months). Repeatable, mechanical pipettes, such as Eppendorf Research®, may be used and their accuracy should be verified on a quarterly basis to be within the manufacturer's specifications. If a pipette's accuracy exceeds the manufacturer's specifications its use should be discontinued and it should be replaced or sent in for repair.

10.4 Miscellaneous: powder-free nitrile or nitrile gloves; disposable laboratory wipes; self adhesive labels.

10.5 Volumetric flasks. Teflon®, Class A, 50, 100, 250 and 500 mL capacities.

10.6 Storage bottles. Wide and narrow mouth, Teflon® FEP (fluorinated ethylene propylene) with Tefzel® ETFE (ethylene tetraflourethylene) screw closure, 50, 100, 250, 500, 1,000 and 2,000 mL capacities.

10.7 Reflux caps and FilterMate™ 2 µm Teflon® filters.

10.8 Wash bottles made of LDPE and Teflon® having 500 mL and 1 L capacities.

10.9 Plastic or Teflon® coated tweezers.

11.0 CHEMICALS, REAGENTS, STANDARDS AND THEIR PREPARATION

Note: In general, chemicals, reagents and commercial stock standards expire when specified by the manufacturer. If the manufacturer does not provide an expiration date then they shall expire one year from the opened date. Standards and other solutions prepared in-house expire as specified throughout the SOP. Proper disposal of hazardous wastes are discussed in detail in the Solid and Hazardous Wastes SOP (ERG-MOR-033).

11.1 High Purity Acids - ultrapure and concentrated stored in Teflon® Bottles. These reagents are used for the preparation of sample extraction fluid and all standards.

Note: Concentrated high purity reagents are not 100% of the specified reagent. It should be understood that all percentages in this SOP are expressed in terms of volume per volume (v/v) rather than true percentages of reagents in solution.

11.1.1 Nitric Acid (HNO_3), 60-70%

11.1.2 Hydrochloric Acid (HCl), 32-35%

11.1.3 Hydrofluoric Acid (HF), 47-51%

11.1.4 Extraction fluid (0.5% (v/v) HCl, 1.85% (v/v) HNO_3, 0.17% (v/v) HF and 8.30 mg/L Au)

11.1.5 Standard solvent (0.30% (v/v) HCl, 1.11% (v/v) HNO_3, 0.10% (v/v) HF with 5 mg/L Au)

11.2 Hydrogen Peroxide (H_2O_2) - ultrapure and concentrated (30-32%) stored in Teflon® bottles. This reagent is used for the extraction procedure.

11.3 Nitric Acid - Trace Metal Grade in 2.5 L glass for rinse blank and labware cleaning.

11.3.1 Rinse blank (2% (v/v) HNO_3, 0.5% (v/v) HCl with 5 mg/L Au)

11.3.2 10% (v/v) HNO_3 acid bath for labware cleaning

Preparation: The acid bath solution is prepared by adding 2.5L of concentrated trace metal grade HNO_3 to 22.5 L of ASTM Type I DI water in a clean 42 L polypropylene acid bath tank. The acid bath should be stored in a fume hood.

11.4 ASTM Type I deionized water - with a resistivity greater than 17.3 MΩ.

11.5 Argon gas - purity > 99.996%, Oxygen < 5 mg/L, Hydrogen < 1 mg/L, Nitrogen < 20 mg/L and Water < 4 mg/L.

11.6 Secondary Source Control Standards - A commercially prepared single- or multi-element secondary source (different manufacturer from the multi-element calibration standard). These NIST traceable calibration standards are used to produce the ICV, which is run as a verification of the instrument's calibration for accuracy and precision.

11.7 Single-Element Stock Standard Solutions - Commercially prepared NIST traceable standards from ultra high-purity grade chemicals or metals (99.99 – 99.999% pure) designed for use with ICP-MS instruments (e.g., Mercury Std.).

11.8 Multi-Element Stock Standard Solutions - Commercially prepared NIST traceable standards from ultra high-purity grade chemicals or metals (99.99 – 99.999% pure) designed for use with ICP-MS instruments (e.g., ISTD solution).

11.9 Interference Check Standard - Commercially prepared standard that is diluted to prepare ICSA and ICSAB interferent checks.

11.10 Smart Tune Solution - Although custom tuning solutions may be used, the tuning solution for this SOP may be purchased through the manufacturer (Perkin Elmer #N8125040) or is prepared in-house using single-element standards to contain 10 µg/L of Be, Mg, Co, Rh, In, Ba, Ce, Pb, and U in 1% (v/v) HNO_3.

To prepare 1 L of this solution, add 10 mL of Ultrex Nitric Acid to ~ 900 mL of ASTM Type I deionized water and add 10 µL of each 1,000 µg/mL single-element standard, then bring to volume. This solution may be stored in LDPE bottles but ideally in Teflon®. The expiration date is either that specified by the manufacturer or if prepared in-house no later than the earliest expiration date of any standard or reagent used for preparation.

11.11 Dual Detector Cross Calibration Solution - Refer to Section 13.3 for the purpose and final concentration of this solution. Although custom cross calibration solutions can be used, it may be purchased through the manufacturer (Perkin Elmer #8125032) or it is prepared in-house using single-element standards.

11.12 Multi-element ISTD stock standard – Commercially prepared standard that is used in conjunction with the single-element standards for Sc, Ga, and Li to prepare the internal standard.

11.13 Internal Standard Spike Solution – Prepared standard used to manually spike all calibration and QC standards as well as all samples that are analyzed by the ICP-MS.

11.14 Blank Spike (BS) Standard – The BS standard is used to spike the BS/BSD (See Section 16.3). Prepare the BS standard according to Table 24-6 with a final volume of 50 mL.

11.15 Post Digestion Spike (PDS) Standard –The PDS standard is used to spike the PDS source sample. This post digestion spike is used to help determine if poor matrix spike recoveries are due to interferents. Spike 1 µL/mL of sample analyzed.

11.16 Second Source Working Standard – The second source working standard is used to create the ICV.

11.17 Citranox® Acid Cleaner and Detergent - Prepare a 5% solution by adding 500 mL of Citranox® to 9.5 L of warm – hot tap water for labware cleaning and decontamination. This detergent bath should be changed about once every month, depending on use.

11.18 Standard Reference Material (SRM) – The standard reference material used to prepare the SRM samples. NIST SRM 1648a (Urban particulate matter with certified concentrations of lead at 0.655 ± 0.033 mass fraction (in %) is used to verify the extraction procedure.

11.19 Lower Limit of Quantitation Check (LLQC) –This quality control sample is used to determine the LLQL, and is prepared by creating a spike solution that will create a final concentration of analyte in the sample matrix that produces and instrument signal/response that is 10x the standard deviation above the lab reagent blank (at 99% confidence; n-1 degrees of freedom). Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within ± 30% of their true value.

12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

12.1 Collection and Handling of Filters

Whenever the filter is handled use clean disposable nitrile or neoprene gloves and if necessary clean Teflon® coated (no exposed metal surfaces) or plastic tweezers. Never touch the membrane of the filter and take care not to puncture or damage the filter with the tweezers. See Section 16.1 for more information about filter condition.

12.2 Preservation and Storage of Filters

Samples do not have a preservative and can be stored for up to 180 days in ambient conditions. Upon retrieval from field sampling during humid or rainy conditions be certain the filter is not moisture-laden. Damp filters may stick to the sample transport container causing damage and thereby invalidating the sample. If the sample is found to be moisture-laden allow the membrane to dry before enclosing in the transport container. Once the filter arrives at the laboratory, a unique LIMS identification number is assigned and placed on the outside of the COC, sample envelope and/or container for tracking and storage purposes. Extraction hold time is 180 days from the sample date.

12.3 Shipment of Filters

When filters are shipped to or from the laboratory follow proper handling instructions in Section 12.1 and take proper precautions when packing such that filters are not exposed to contaminants or damage during shipment.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Daily Optimization Procedures

Daily optimization is performed through the software's Smart Tune Wizard. Refer to Tables 24-4 and 24-5 for Optimization Procedures and Performance Specifications respectively. For more detailed information, the PerkinElmer Elan Version 3.4 Software Reference Guide is also available for reference as a PDF file on the desktop of the instrument computer.

13.2 Mass Calibration and Resolution

Before performing any kind of calibration or optimization, allow a period of not less than 30 minutes (preferably 1 hour) for instrument warm-up. After the warm-up, the mass calibration and resolution may be optimized using the tuning solution (see Section 11.11) by running a mass calibration and resolution optimization through the Smart Tune Wizard. Resolution at low mass is indicated by magnesium isotopes 24, 25, and 26. Resolution at high mass is indicated by lead isotopes 206, 207, and 208. These peaks may be viewed in the Interactive graphics window. For optimal performance, adjust spectrometer mass calibration to ± 0.05 amu and the resolution to produce a peak width of 0.70 ± 0.1 amu at 5% peak height. Repeat mass calibration and resolution optimization if it has shifted by more than ± 0.05 or ±0.1 amu respectively.

13.3 Dual Detector Cross Calibration

The dual detector cross calibration is used to provide a smooth transition from the pulse counting mode to the analog mode, which extends the linear dynamic range of the detector. Typically a 2% nitric acid solution containing 200 µg/L of Mg, Cu, Rh, Cd, and Pb as well as 2000 µg/L of Be is used for this calibration although custom solutions may be used. This calibration must be performed daily before each analysis to report values above the HCV.

13.4 Daily Performance

13.4.1 The daily performance report must be generated daily or before each analysis, whichever is more frequent.

13.4.2 Instrument stability must be demonstrated by running a daily performance check using the tuning solution. A minimum of five replicates with resulting relative standard deviations of absolute signals for all analytes of less than 3% is required prior to calibration.

13.5 Calibration

13.5.1 Prior to initial calibration, set up proper instrument software routines for quantitative analysis (i.e., autosampler table, QC sample names etc.). The instrument must be calibrated using a minimum of a calibration blank and four non-zero calibration standards. Consideration should be give to adding more standards, particularly lower concentrations, in order to better define the LDR and quantitation limit (QL). A minimum of three replicate integrations are required for data acquisition with an RSD < 10.0%. RSDs > 10.0% are allowed for the first non-zero calibration standard (CAL2). Use the average of the integrations for instrument calibration and data reporting.

For a linear calibration to be considered acceptable, the calibration curve should have a correlation coefficient ≥ 0.998. The resulting curve should then be verified with mid-level and low-level calibration standards as described in Section 16.6.

Preparation: Non-blank calibration standards are prepared by diluting the calibration working standard to appropriate levels using the standard solvent.

13.5.2 For matrices of known composition, match the composition of the calibration and QC standards to that of the samples. For matrices of unknown composition, use an ISTD that has been matched to the analytes chemical and physical properties (i.e., ionization potential, ±50 amu) so that the ISTD and element of interest behave similarly during the analytical process.

13.5.3 The rinse blank should flush the system between solution changes for blanks, standards, and samples. Allow sufficient rinse time (≥ 1 min) to remove traces of the previous sample. Solutions should aspirate for at least 30 seconds prior to the acquisition of data to establish equilibrium.

13.5.4 Refer to Section 11.1.4 for the preparation of standard solvent. Once prepared, all calibration standards must be stored in Teflon® bottles/flasks.

Note: Commercial stock standards used to prepare calibration standards and other quality control standards must be used within their expiration

date. Calibration blanks/standards and other QC standards made from the stock standards may be set to expire no later than the earliest expiration date of any standard used for preparation.

13.5.5 Refer to the quality control requirements presented in Table 24-3 for calibration acceptance criteria.

13.6 Internal Standardization

13.6.1 Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. ISTD quality control requirements as described in Section 16.7 must be followed.

13.6.2 Internal standards (ISTD) for this method are ^{45}Sc ^{69}Ga, ^{89}Y, ^{115}In, and ^{209}Bi for analytes beginning with mass 6 and ending with mass 238. The ISTDs^{6}Li,, ^{72}Ge ^{103}Rh, ^{159}Tb & ^{165}Ho may also be used if necessary. Internal standards must be manually added to each calibration standard **after** they are brought to volume in the proportion of 2.0 µL for every mL. For example, add 200 µL of ISTD solution to a 100 mL standard.

13.6.3 Concentrations of the internal standards for this method are determined by the concentration of each element that will produce an intensity that is sufficiently stable. Typical intensities are between 200,000 and 500,000 cps; however, ideal intensities may be as high as 1,000,000 cps.

13.6.4 The concentration of the internal standard must be added equally and in the same manner to the calibration blank/standards, QC standards and samples.

13.6.5 Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. However, be aware that internal standards themselves may be responsible for polyatomic and/or doubly charged interferences.

13.7 Instrument Performance

13.7.1 After instrument calibration an ICV and ICB must be analyzed for initial verification of the calibration curve. Refer to Sections 16.6.1, 16.2.2, and Table 24-3 for specific QC criteria.

13.7.2 To verify that the instrument is properly calibrated on a continuing basis, analyze a CCV and CCB before the analysis of samples and after every 10 samples.

13.8 Linear Dynamic Range (LDR)

The LDR study is performed every 6 months to determine the maximum concentration level at which the initial calibration is linear. The recovery criterion for the LDR are 90-110%. Dilutions must be performed for elements with concentrations over 90% of the established LDR. If the LDR study has not been performed within 6 months, any elements with concentrations over the highest calibration concentration must be diluted.

13.9 Lower Limit of Quantitation Limit (LLQL)

The LLQL study is performed every 6 months to determine the lowest concentration level at which data may be reported. This is done by extracting and analyzing a lower limit quantitation check (LLQC). The recovery criterion for the LLQC is 70-130%. Any results reported below the LLQL must be qualified as an estimated value.

14.0 PROCEDURE

14.1 LIMS Batch Procedure

Note: Please perform the following procedure the same day that you plan to begin the extraction. If something happens and the extraction cannot be performed, edit the bench sheet with the correct extraction date, both in LIMS and on the hardcopy bench sheets.

14.1.1 Log into Element.

14.1.2 From the "Laboratory" menu, select "Batch."

14.1.3 With the Inorganics department selected from the drop-down menu at the left of the screen, click "Add" at the bottom, left-hand corner. A blank bench sheet will appear. The following information should be input into the bench sheet using the drop-down menus: 1) Preparation Method: ICP-MS Extraction and 2) Batch Matrix: Air

14.1.4 Select the appropriate inorganic analysis ("Metals Analysis – 47mm 20xx") from the "Available" analysis box and press the right-facing arrow button. **Note:** Options: 1.) Additional information may be added in the "comments" box for batches that are different from standard samples, for example samples that are for the Midlothian or Schools projects. Reagent lot numbers used in the extraction are also added in the comment section. 2.) You may choose to press "Copy" instead of "Add" from Step3. Use

caution here as both the analysis and comments from the copied batch will be included in the new batch and this information may need to be changed.

14.1.5 Press the save button. The new LIMS-created batch number will be visible in the box to the left of the screen.

14.1.6 Select the "Bench Sheet" button at the bottom of the screen. This is where you will include sample information and appropriate batch QC.

14.1.7 Click "Edit" at the bottom of the new screen.

14.1.8 At the top of the screen, press "Add" and select "Sample by Container." Select the samples from the list that you would like to include in the batch. For each sample, the following information needs to be included: 1) Initial (m^3): This is the total flow through the filter as it was being sampled in the field. This information can be found or calculated from the information provided on the sample chain of custodies. Field blank samples are assigned the same volume as the primary sample collected on the same day. A field blank volume may also be an average of all the filter volumes for a given month. 2) Comments: Include two spaces followed by the sample ID for each sample in the list. This includes any additional designations such as C1, C2, FB, etc.

Note: A maximum of twenty samples may be selected for any given batch. If more than 20 samples are selected, additional batch QC must also be added to the bench sheet and extracted to meet the requirements described in Table 24-3.

14.1.9 Each batch requires QC to be prepared/extracted with each batch of twenty samples. This QC is included in the bottom box on the screen. Required QC includes:

i. Duplicate Samples: One duplicate QC sample is added per batch. Click the "Add" button at the top of the screen and select "Duplicate." Right click on a duplicate sample to assign its source sample and initial volume. This volume will be the same as the source sample. Duplicate samples must also be added for each collocated (C1/C2) or duplicate (D1/D2) sample type to be extracted.

ii. Blank Samples: Each batch must include one reagent blank and one method blank per twenty samples.

a.) One blank (BLK) sample is already included. To add another, click the "Add" button at the top of the screen and select "Blank."

b.) Change the sample name by right-clicking on each blank and selecting "Name." BLK1 should be changed to "Reagent Blank" and BLK2 should be changed to "Method Blank."

c.) Include the filter lot number in the comments section of the Method Blank.

iii. Spiked Samples: Each batch must include two spiked samples per twenty samples.

a.) One laboratory control sample (BS) and one laboratory control sample dup (BSD) are included for 47mm extractions. The BS should already be included, so click on "Add" and select "Laboratory Control Sample Dup" to add the second BS to the list. For each of these QC samples, include the Teflon filter lot blank in the comments section.

b.) Identifying spikes: For laboratory control samples, the appropriate spike ID, type, and volume will need to be assigned in the same manner. Right click on the sample QC and select "Spike 1 ID." Select the appropriate standard from the list. Edit the "Spike 1 Type" to read "Pre-prep" and the "Spike 1 Volume" to read 500uL (or whatever volume is being used.)

iv. Post Spike (PS): Each batch must include one post spike (PS) sample per twenty samples.

a.) One post spike is automatically included for 47mm extractions. Assign the appropriate spike ID, type, and volume using the instructions provided above. The correct spike amount is 50 µL and the spike type is "Post-prep."

v. Standard Reference Material: Each batch must include one SRM per twenty samples.

a.) Click on "Add" and select "Reference" to add the SRM to the bench sheet. Add the SRM identification number as assigned during weighing in the comments section. Identify the reference standard used by right-clicking on "Spike 1 ID" and selecting the appropriate standard. Indicate the weight of SRM digested (in mg) in the Spike 1 Volume. "Spike 1 Type" should be listed as "Pre-prep."

14.1.10 Print two copies of the bench sheet in landscape format. Both will need to be signed on the "Extraction Reviewed by" line and dated once the extraction is complete. One copy is to be three-hole punched and placed in the Extraction Notebook, while the other is to be kept bound to the corresponding samples in the cabinet in the lab.

14.2 Filter Extraction Procedure

14.2.1 Prior to sample processing, be sure to turn on the HotBlock ™ and select the appropriate program and initiate to allow it to warm to extraction temperature. Be sure to allow the HotBlock™ interface establish connection with the block itself (shown on the screen as "Please Wait") prior to beginning any extraction method. Failure to do this will cause the HotBlock™ to heat to higher than intended temperatures.

14.2.2 Place filter in a labeled extraction tube as far down as possible. This is done by gently gripping the Teflon® support ring with gloved fingers and bending the filter so that it fits in the tube. Use the tip of a gloved finger to slide the filter to the bottom of the tube.

14.2.3 Add 0.5 mL of Matrix Spike to BS/BSD samples. Add 30 mL of extraction fluid (0.5% (v/v) HCl, 1.85% (v/v) HNO_3, 0.17% (v/v) HF and 8.33 mg/L Au) to the extraction tube with the filter.

14.2.4 The SRM sample is prepared for extraction by adding a blank Teflon filter to a clean sample vial as described in Section 14.2.2. The loaded vial is then weighed, zeroed, and then re-weighed after an aliquot of SRM standard of approximately 2-3 mg is added to the vial. Add 30 mL extraction fluid to the extraction tube.

Note: It may be more efficient to weigh several SRM standard vials for extraction at one time. The weight information should be recorded in the balance room notebook. Individual standards should be numbered with the date the vial was created (i.e. "01012013-SRM01"). The vial number should be recorded in the LIMS in the SRM sample comments in the bench sheet.

14.2.5 Print out a copy of the Teflon Extraction Record from L:\Metals Lab (an example is shown in Table 24-10). Record the batch ID and extraction date. Record the temperature of the HotBlock™ as indicated by the calibrated thermometer, as well as the temperature indicated on the HotBlock UI.

14.2.6 Place all samples in plastic HotBlock™ rack and place in HotBlock™. Add a reflux cap to each sample tube. Record the time samples were placed in the HotBlock on the table printed above. Samples will be extracted for a total time of 2.5 hours at a HotBlock™ temperature of 95ºC and an approximate sample temperature of 85ºC.

Note: Monitor sample temperature with a thermometer in an extraction tube with a reflux cap and 30 mL of extraction fluid.

14.2.7 After 1.5 hours of extraction, add 1.8 mL of hydrogen peroxide (H_2O_2) and allow to effervesce for 0.5 hours. Record the time the aliquot of H2O2 was added on the Teflon Extraction Record.

14.2.8 Check the filters occasionally during extraction. If a filter floats out of the acid, use acid-cleaned teflon stirring rods to push the filter below the extraction fluid.

14.2.9 After extraction, samples must be removed from the HotBlock™ as soon as possible so they are not allowed to heat to dryness. Allow the samples to cool to room temperature. Record the time samples were removed, as well as the temperatures indicated by both the certified thermometer and HotBlock™ UI on the Teflon Extraction Record.

14.2.10 Leaving the Teflon filter in the sample vial, bring the sample volume to the 50 mL line on the vial with a wash bottle filled with DI water prior to filtering. Record the time the samples were brought to final volume on the Teflon Extraction Record.

Note: If the final volume exceeds 50 mL, measure the amount exceeded using an appropriate pipette (usually the 1 or 5 mL) and record the final sample volume. Using the pipettor, pull sample from the vial in varying increments until enough liquid is removed so that the sample line is level with the 50 mL line on the vial. Add the amount of liquid removed to 50 mL and record that as the final volume. This information must be entered into the LIMS so that the final sample concentration is calculated and reported correctly. This overfilling may be avoided by using fine streamed wash bottles while adding the D.I. water.

14.2.11 Homogenize the sample by inverting three times and then the extract is ready to be analyzed. Should the sample contain noticeable particulate material, an aliquot of sample may be poured off and filtered with the use of a FilterMate™, however; this is not typically necessary. Alternatively, the sample may be allowed to rest until all debris settle. Care should be taken to not disturb the sample when transferring sample extract to an

autosampler tube. For samples with no noticeable particulate matter, it is not necessary to allow the sample to settle prior to analysis.

14.2.12 Record the sample slot the certified thermometer was placed in during the sample extraction on the Teflon Extraction Record. After initialing the sheet, attach the Teflon Extraction Record to the batch paperwork.

14.3 LIMS Sequence Procedure

14.3.1 Log into LIMS Element Software.

14.3.2 From the "Laboratory" menu, select "Sequence."

14.3.3 Click "Add" in the top right corner and select "Randy" as the Template ID then click done. This will automatically add all of the calibration standards and QC samples for a typical analysis sequence.

14.3.4 Click the pull-down menu for "Source Batch" at the upper middle part of the screen and select the batch you are going to analyze. Then click the "Add" button and select "Batch QC Sample." When the list of QC samples appears click the first sample and holding the shift button double-click the last sample and it will insert them into the sequence. Then click "Add" again and select "Batch Sample." Add all of the samples listed as you did for the Batch QC Samples.

14.3.5 Arrange all of the samples according to the example below, being sure to follow the requirements summarized in Table 24-3:

Autosampler Position	Sample Name
1	BTB0001-CAL1
2	BTB0001-CAL2
3	BTB0001-CAL3
4	BTB0001-CAL4
5	BTB0001-CAL5
6	BTB0001-ICV1
1	BTB0001-ICB1
5	BTB0001-HCV1
7	BTB0001-IFA1
8	BTB0001-IFB1
4	BTB0001-CCV1
2	BTB0001-LCV1
1	BTB0001-CCB1
9	B0B1707-BLK1
10	B0B1707-BLK2

Autosampler Position	Sample Name
11	B0B1707-SRM1
12	B0B1707-BS1
13	B0B1707-BSD1
14	0020993-01
15	B0B1707-DUP1
16	B0B1707-SRD1
17	B0B1707-PS1
18	0020993-02
4	BTB0001-CCV2
1	BTB0001-CCB2
7	BTB0001-IFA2
8	BTB0001-IFB2
4	BTB0001-CCV3
2	BTB0001-LCV2
1	BTB0001-CCB3

14.3.6 Right-click the SRD sample and choose the appropriate source sample from your batch.

Note: Make sure that the source sample for the SRD is updated to the current batch sample SRD sample or your data will not be saved.

14.3.7 Using the shift key select all of the samples in the sequence and right-click to choose “Internal Standard ID” then select the ISTD you are using for this sequence.

14.3.8 Right-click each calibration standard and QC sample and select “Standard ID” to set the current standard being used for each solution.

14.3.9 Click “Save” and print a double-sided copy in landscape format to use as the cover of the data package and to help enter the sequence in the Elan software.

14.4 Filter Analysis Procedure

14.4.1 Prior to analyzing samples, check the instrument performance by analyzing the tuning solution using the Daily Performance Check in the Smart Tune Wizard. The performance specifications that must be met are in Table 24-5. Also refer to Section 13.1 for other criteria. If the performance check fails, follow the optimization procedures in Table 24-4.

14.4.2 Before starting the calibration be sure to flush the sample introduction system with enough rinse blank and be certain the rinse blank bottle has enough solution for the analysis. Enter all sequence information (sample and QC sample names) into the autosampler and QC tables (see Section 14.3.5). The autosampler table should be named as the month, day and year (e.g., 01012010.sam) and saved. Create the file name for the collected data with the numeric month, day and year (e.g., 01012010.rep) in the Method window under the "Report" tab (upper right) in the "Report Filename" field. Any changes made to the method (i.e., QC tables) must be saved before you exit that screen or they will be lost.

14.4.3 Pour off the calibration blank/standards (Blank (CAL1), LOQ (CAL2), CAL3, CAL4 and CAL5) and initial/continuing QC standards (ICV, ICB, ICSA, ICSAB, HSV, CCV, CCB) spiked with ISTD in the appropriate autosampler positions.

14.4.4 To start the analysis, highlight all samples in the autosampler table and click "Build Run." Then click "Run" in the following screen. Once the calibration has been completed and reviewed, save the calibration file with the same month, day and year as the .sam and .rep files in Section 14.4.2 and print the Quantitative Analysis Report to PDF to be included with the sequence files.

14.4.5 Label autosampler tubes with a black marker. Batch samples may be prepared by adding 20 µl of ISTD into the autosampler tube and then adding 10 mL of sample. Mix the sample well and place in appropriate autosampler location for analysis.

14.4.6 The PDS sample is prepared by adding 1.0 uL of spike solution to 1.0 mL of sample to be analyzed (i.e., 10 uL of PDS to 10 mL of sample).

14.4.7 Samples with analyte concentrations greater than 90% of the current LDR must be diluted and re-analyzed.

14.5 Unexpected Instrument Shutdown

In the event that the ICP-MS shuts down during an analysis the proper procedure to be followed by the analyst is:

14.5.1 Restart the instrument and allow it to warm-up for a minimum of 30 minutes but preferably one hour, especially if the instrument has been inoperable overnight and is at room temperature. If the analyst was present during the loss of the plasma and the instrument has not significantly cooled then a shorter time period for warm-up may be sufficient.

14.5.2 After the instrument has been thermally stabilized a new daily performance report (DPR) should be analyzed with the operating conditions being used for the analysis.

14.5.3 If the DPR passes the analyst must check the calibration by analyzing a continuing calibration verification (CCV) and a continuing calibration blank (CCB) to be sure the calibration is still valid. If the CCV & CCB passes the analyst may proceed with where the analysis left off. Any samples that didn't complete their analysis should be repeated.

14.5.4 If the DPR, CCV or CCB do not pass the analysis must be terminated and any samples not bracketed by valid CCV & CCB checks must be reanalyzed with a new analysis/calibration.

14.5.5 The DPR, CCV and CCB checks should be kept for documentation. The DPR may be place in the DPR binder and the CCV/CCB checks must be included with the data package. The analyst should document the event briefly in the sequence narrative so the reviewer is aware of the instrument shutdown.

14.6 LIMS Data Upload Procedure

14.6.1 When a data package is complete, the analyst will transfer the data from the instrument computer to BART.

14.6.2 To begin LIMS upload, open Element. Go to the laboratory menu and click on "Data Entry/Review" In this window, select "Sequence" in the top left corner, making sure that "Inorganics" is selected from the drop-down menu. Highlight the correct sequence and click on "Create" in the Data Entry box in the top right corner. Once the spreadsheet is created in LIMS, select "DataTool" in the Data Entry box and save the file as the sequence name in the UserFiles folder of your harddrive (C:\ELMNT\UserFiles).

14.6.3 The DataTool interface should open to the "Select Data System Files" window. In this window, check to make sure the correct file information is selected:

1) File Type: PE ELAN_REP(*.rep)
2) Drives: y:\Bart

14.6.4 In the box below Drives: select the folder the data is stored in for the sequence. In the Bart drive, select the ICP-MS DATA folder, then the

corresponding year, and finally the folder for the data the sequence was run.

14.6.5 All of the data files for that sequence will appear in the lower right-hand box. Double-click on the appropriate data file and click "Auto Select" for each file that needs to be included in the sequence. (*Note: Only undiluted samples and sequence QC should be included here.)

14.6.6 Click "Done" when all sample and QC files have been selected to return to the main window. Click "Merge Files" at the bottom of the window. DataTool will merge the files and show the data in the Data Transfer window.

Note: Review the content of the top windows in the Data Transfer window for red text. If there is any, the DataTool cross table requires editing. Seek the advice of the LIMS administrator to correct this.

14.6.7 Click "Save" and save the spreadsheet in the UserFiles folder of your hard drive. Close DataTool.

14.6.8 In Element, go back to the Data Entry/Review window. The newly merged data should appear in the window. Click "Save" to save the files to Element and then "Query" in the Data Review box. Element will perform all necessary calculations at this point.

14.6.9 In the Data Entry/Review window, samples and QC can be reviewed for pass/fails. Any data that does not pass its assigned criteria will have red text. Use appropriate data qualifiers to flag data that does not meet criteria.

14.7 LIMS Dilution Data Upload Procedure

Note: Dilution data is not uploaded with sequence QC, as this data is typically only required for one or two analytes in a given sample. Therefore, the data is hand-entered into Element.

14.7.1 Open Element and go to the Data Entry/Review window. Select the sequence that the diluted sample was originally run with. Click on "Query."

14.7.2 Scroll down to the needed sample information. Click "Edit" in the Data Review box. Right click on the sample & analyte that has dilution information and select "Qualifiers" -> "Quick Analyte Qualifiers." Select qualifier flag D-01 for dilutions.

14.7.3 In the IResult column, type the new dilution data. Make sure the new data has been corrected for the dilution factor (i.e. results for a 5x dilution should be multiplied by 5 if the instrument software did not make the correction). In the Diln column, type the dilution factor for the sample.

14.7.4 Repeat steps 2 & 3 for any additional dilution data that needs to be input for the sequence. If any QC data was altered (for example, Dups) then you will click on "Re-calc" and "Save." If only sample information was altered, click "Save."

14.7.5 Re-run the query to verify all dilution data was saved to the sequence.

Note: In LIMS, the dilution factor is applied to the associated MDL as well as to the sample concentration.

14.8 Data Review

All instrument data should be first reviewed by the analyst and then a secondary reviewer, usually the project task lead for metals analysis. Both the analyst and secondary review must use the "Quality Control Requirements for Metals Analysis" checklist to complete data review (see Figure 24-8). Reviewers must initial and date each parameter check on the review form to verify that each meets the established acceptance criteria.

14.8.1 Initial Calibration

In addition to the requirements outlined in Table 24-3, the analyst and secondary reviewer must also verify that the intensities measured for reportable analytes in the calibration blank are acceptable and will not interfere with the sensitivity. A review of previously analyzed calibration blanks can demonstrate acceptable intensity values. The intensities of the internal standards in the calibration standard should be monitored relative to the intensities seen in the calibration blank.

14.8.2 Internal Standards

Internal standards must be monitored for each sample throughout a sequence; the measured intensities must stay between 60 and 125% of the measured intensity of internal standards in the calibration blank. See Section 16.7 for corrective actions to remedy internal standard intensities that are measured outside of this range.

14.8.3 Relative Standard Deviation's

Follow the prescribed acceptance criteria for RSD's of calibration standards as listed in Table 24-3. Sample RSD's should also be monitored throughout analysis. High RSD's (greater than 20%) for concentrations above the MDL can indicate memory interference from previous samples, as well as other instrumentation issues that may need to be corrected before analysis can be continued.

14.8.4 Element/LIMS Data

The analyst and second reviewer must verify quantities imported into the LIMS reflect the raw data. This can be accomplished by checking a few analytes for random client samples and QC samples. Hand entered data (i.e., dilution) should be verified by the second reviewer to be certain the values, dilution factors and flags are properly inserted. The LIMS calculated final value should also be checked to ensure the system is using the correct equation.

14.8.5 Multiple Isotopes

If an element has more than 1 monitored isotope, examine the concentration calculated for each isotope, or isotope ratios, to detect a possible spectral interference. Consider both primary and secondary isotopes when evaluating the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences that the primary recommended isotopes; therefore, differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes.

14.8.6 Reprocessing Data

Reprocessed data must include reprocessing of all calibration standards and QC samples associated with the reprocessed sample results. Original data shall be kept with the reprocessed data and annotated with the reason for reprocessing. It is imperative that the calibration file be saved as a separate file from the original calibration file. The reprocessed calibration file shall be renamed with the added distinction of "_reprocessed" (i.e. 03052010_reprocessed). All other samples including QC are automatically saved to the dataset file.

15.0 CALCULATIONS

15.1 Analyte Concentration

Metal concentration in the air sample should be calculated as follows:

$$C = \frac{(C_i \times V_f)}{V_{std}}$$

Where:

C = concentration, ng metal/m^3
C_i = metal concentration determined from Section 14.4, ng metal/L.
V_f = total sample extraction volume from extraction procedure (i.e., 0.05 L).
V_{std} = standard air volume pulled through the filter, m^3

15.2 Method Detection Limits

The MDL is calculated as follows for the CFR MDL calculation method:

$$MDL = (t) \times (SD)$$

Where:

t = Student's t value for a 99% confidence level and a standard deviation estimate with n - 1 degrees of freedom [t = 3.14 for seven replicates]
SD= standard deviation of the replicate analysis.

The MDL is calculated as follows for the FAC MDL calculation method:

$$MDL = (K) \times (SD)$$

Where:

K = Value as prescribed in Appendix D: DQ FAC Single Laboratory Procedure v2.4, 08/30/2007
SD= standard deviation of the historical BLK2 analysis.

15.3 Relative Percent Difference (RPD)

The RPD is calculated as follows:

$$RPD = \frac{R_1 - R_2}{(R_1 + R_2)/2} \times 100$$

Where:

R_1, R_2 = values that are being compared (i.e., duplicate and replicate analysis data)

15.4 Percent Recovery

Percent Recovery is calculated as follows:

$$Percent\ Recovery = \frac{Analytical\ Result}{Theoretical\ Result} \times 100$$

15.5 Relative Standard Deviation (RSD)

RSD is calculated as follows:

$$RSD = \frac{Standard\ Deviation}{Average} \times 100$$

16.0 QUALITY CONTROL

The analyst must perform the quality control checks listed in Table 24-3 and meet the requirements in this section. Data Quality Objectives (DQO) and data assessment criteria are determined from the results of the quality control samples. The DQO criteria are summarized in Table 24-3.

16.1 Sample Collection Quality Control

16.1.1 Filters which are dropped or become contaminated with any foreign matter (i.e., dirt, finger marks, ink, liquids, etc.) are invalid.

16.1.2 Filters with tears or pinholes that occurred before or during sampling are invalid.

16.1.3 A power failure during a field sample collection event invalidates the sample collected during that event. See SOP ERG-MOR-045 for more information about the sample acceptance criteria.

16.2 Blanks

16.2.1 The Rinse Blank must be used to flush the system between standards and samples. Refer to Section 11.3.1 for preparation details.

16.2.2 Initial Calibration Blank (ICB) is analyzed immediately following the initial calibration verification. The absolute value of the instrument response should be less than the method detection limit. If the ICB fails it may be re-analyzed once. If the ICB still does not pass, the analysis should be terminated, the problem corrected and, the ICV and ICB must be verified again before the analysis can continue. If the ICV or ICB still fail, recalibrate the instrument and verify the ICV and ICB again before continuing. If recalibration fails to correct the problem, all QC and samples included in the sequence must be flagged.

16.2.3 Continuing Calibration Blanks (CCB) are analyzed following each continuing calibration verification sample. The acceptance criteria are the same as the ICB. If the first CCB analyzed fails it may be reanalyzed once. If it fails again the analysis should be terminated, the instrument recalibrated and the initial QC repeated. If the CCB fails later in the analysis it may also be reanalyzed twice. If it continues to fail again the affected analyte must be flagged. If multiple elements fail any samples analyzed before or after the failing CCB must be reanalyzed.

16.2.4 Laboratory Reagent Blank (LRB/BLK1) consists of all reagents (extraction fluid) used to process samples and is carried through the entire preparation and extraction process to determine the background levels, if any, from the extraction fluid and process. If the absolute value of the analyte concentration of the LRB is less than or equal to the MDL (See Table 24-1), no further action is required. If the BLK1 fails it may be reanalyzed once. If it fails again and the concentration is greater than the MDL, but less than 5 times the MDL, the BLK1 and all associated samples are flagged. If the concentration of a reported analyte is greater than 5 times the MDL all associated samples must have that analyte result blank subtracted and flagged as such.

16.2.5 The Method Blank (MB/BLK2) is prepared as an LRB but with the addition of a blank Teflon® filter and analyzed as a sample to determine the background levels, if any, from the blank filter and extraction process. Resulting data is recorded and used to determine FACA MDL values. While the absolute value of resulting values should be less than the established MDL, there is no corrective action procedure should the BLK2 fail.

16.3 Spikes

16.3.1 Laboratory Control Sample (LCS): An LCS is prepared and carried through the entire sample digestion process. At least two LCS are analyzed with each sample batch. The LCS is represented by the BS/BSD

samples. They are prepared by spiking blank Teflon® filters and carrying them through the entire preparation and extraction procedure. The results must be within 80 -120% of actual values, with the exception of Al. Mercury may recover within 75-125% of actual value. If not, the BS and/or BSD may be reanalyzed once each. If criteria are still not met the batch of samples associated with the BS/BSD must be flagged for any analytes that failed in either or both. A re-extraction of the samples in this case is not possible because the entire available sample has been used.

16.3.2 Standard Reference Material (SRM) is prepared and carried through the entire sample digestion process. At least one SRM is analyzed with each batch. The SRM is a NIST Urban dust that is prepared with a blank filter and can be referred to as a SRM in the LIMS. The results of the SRM Pb recovery must be within 80 – 120% of the certified value. If not, the sample may be reanalyzed once. If the SRM still fails to meet criteria, all associated samples must be flagged.

16.3.3 Matrix Spike and Matrix Spike Duplicate (MS/MSD): Due to the lack of actual sample duplicates for Teflon® filter sampling events, the traditional MS/MSD cannot be prepared. In place of the MS/MSD, a Blank Spike and Blank Spike Duplicate (BS/BSD) are prepared. See Section 16.3.1 for more detailed information.

16.3.4 Post Digestion Spike (PDS): In order to demonstrate spike recovery in an actual sample matrix at least one PDS sample is analyzed with every batch. The PDS is an analyzed aliquot of an extracted sample that is spiked with the PDS standard (See Section 11.16). The PDS spike addition should produce a minimum level of 10 times and a maximum of 100 times the QL. The spike recovery should be within ± 25% or 75% to 125% of the true spike value. If the PDS fails, the analyst should assess the SRD. If both the PDS and SRD fail for the same element it is an indication that matrix interference has occurred and any analytes that have failed should be flagged as possible matrix interference. In the event that the parent sample used for the PDS requires a dilution, the PDS should be prepared by diluting the sample and then spike with the PDS standard.

16.4 Duplicates

There are two types of duplicates found in this procedure:

16.4.1 Blank spike duplicate (BSD): A true laboratory duplicate of the blank spike prepared using a separate blank Teflon filter. The RPD for this duplicate is ± 20%.

16.4.2 Analytical duplicate (or a replicate): A second aliquot of an extracted sample analyzed using the same analytical method as the first, or primary aliquot. These are performed on duplicate and collocated samples collected in the field. The RPD for analytical duplicates (replicates) shall be less than or equal to ± 10% for values greater than or equal to 10 times the MDL. If a duplicate/collocated sample fails to meet the established criteria, the duplicate or collocated sample must be reanalyzed. If initial sample results are confirmed then the replicate or collocated sample must be flagged.

16.5 Performance Evaluation (PE) Samples

Performance evaluation samples should be obtained as available from independent sources and analyzed as a routine sample. PE samples are prepared and analyzed in the same way as field samples and should be analyzed in replicate to verify results.

16.6 Standard Checks

16.6.1 Immediately after the initial calibration, the ICV is analyzed. The measured concentrations should be within ±10%, or 90-110%, of the actual concentration. If the criteria are not met, reanalyze the standard. If the criteria are still not met, a fresh standard may be prepared and analyzed or repeat the initial calibration and ICV.

16.6.2 The HSV must be analyzed after the ICB and prior to analysis of samples. The measured concentration should be within 95% to 105% of the actual concentration. If the HSV fails it may be reanalyzed once. If the HSV still fails, a fresh standard may be prepared and analyzed. If it continues to fail, the instrument must be recalibrated and all initial QC must also be reanalyzed.

16.6.3 Before the analysis of samples and after every 10 samples during a batch analysis, the calibration must be verified using the CCV. Results must be within ±10% or 90% to 110% of the target value for each analyte to verify that the calibration is valid. If a standard check exceeds the limit, the analysis must be stopped and the check standard must be reanalyzed. If the target value exceeds the limit again, a fresh standard may be prepared and analyzed or the instrument must be recalibrated. Any samples analyzed before or after an invalid CCV must be reanalyzed, minimally for the failing element.

16.6.4 Following the first and last CCV of each analysis an LCV must be analyzed. The measured Pb concentration should be within ± 30% of the

true concentration. If the criteria are not met for the first LCV, reanalyze the standard once. If criteria still are not met, a fresh standard may be prepared and analyzed or terminate the analysis, correct any issues, and repeat the initial calibration, and reanalyze all initial QC. Should the second LCV fail for Pb, the analysis should be reanalyzed for Pb only.

16.6.5 Document each standard check value as a percent ratio of the actual value over the target value.

16.7 Internal Standards

The intensities of all ISTDs must be monitored for every analysis (see Table 24-7 for isotopes). When the intensity of any ISTD fails to register between 60 to 125% of the intensity of that ISTD in the calibration blank, the following procedure is implemented:

16.7.1 If the intensities are too high as a result of the ISTD being present in the sample, the sample must be diluted and reanalyzed with the addition of appropriate amounts of ISTD.

16.7.2 Repeat and increase the dilution until the internal standard intensities fall within the prescribed window.

16.7.3 If the intensities are determined to be a result of instrument drift, stop the analysis, find and correct the problem, recalibrate if needed and reanalyze the affected samples since the last acceptable ISTD recoveries.

16.8 Interferences

16.8.1 The interference check standards (ICS) are analyzed at the beginning and end of the run and for every 8 hours of continuous operation. They consist of two different standards the ICSA and ICSAB. The interference check standards obtained from suppliers are never contaminant free. The certificate of analysis documents the levels found in each lot obtained. Therefore, the presence of target elements in the ICSA and ICSAB are expected and may vary from lot to lot of ICS solution used to make these standards. In some cases these contaminants may cause the ICSA or ICSAB to fail the QC requirements. It is acceptable to use the concentrations from the certificate of analysis to correct for these observed contaminants. The known concentrations of contaminants in the ICSA or ICSAB solutions may be subtracted from the experimental values and the established QC criteria must be met or the associated samples are flagged. Samples containing levels of the interferents above the levels in the ICS should be considered for dilution.

16.8.2 The first interference check standard (ICSA) contains only the interference analytes of interest. Any components of the ICS that are calibrated by the instrument must recover within 20% of the expected value. Values obtained for reported analytes not present in the standard should be within ±3 times the LOQ values (See Table 24-2). If the ICSA fails it may be reanalyzed. If it fails again a fresh standard may be prepared and analyzed. If it continues to fail, terminate the analysis and correct the problem. Any samples analyzed before or after an invalid ICSA sample must be reanalyzed.

16.8.3 The second interference check standard (ICSAB) contains the same concentration of interference analytes in the ICSA as well as a known concentration of reported analytes. The concentration of known analytes should be near the middle of the calibration curve. The values obtained for reported analytes should be within 80% to 120% of the known concentration. If the ICSAB fails it may be reanalyzed. If it fails again a fresh standard may be prepared and analyzed. If it continues to fail terminate the analysis and correct the problem. Any samples analyzed before or after an invalid ICSAB sample must be reanalyzed.

16.8.4 Failing ICS are commonly the result of correction equations that need to be adjusted for new instrument conditions. If this is suspected, calculate the new interference equations and reprocess the analytical sequences as described in Section 14.8.6.

16.9 Dilutions

16.9.1 Serial Dilution

The SRD analysis must be performed on one sample per batch, preferably with the parent sample chosen for the analytical duplicate and PDS. The same sample matrix used to extract the parent sample must be used as the diluent. After the dilution is applied to the SRD results, the analyte concentration should be within 90% and 110% of the undiluted sample results if the parent sample analyte concentration is minimally a factor of 50 above the MDL in the original sample. If the SRD and PDS fail for the same analyte, a matrix interference must be suspected and the data flagged for all affected elements. If matrix interference is not suspected, the SRD should be reprepared and analyzed a second time.

16.9.2 Sample Dilution

Any samples needing dilution due to concentrations exceeding the LDR or the presence of ISTD in the sample shall be diluted in an appropriate manner to bring the diluted concentration within the calibration curve and preferably near the mid-point. (See Section 16.7 for dilutions required due to high ISTD recoveries. If the parent sample of a DUP/PDS sample needs to be diluted, the dilution should be performed on each of these QC samples (See also Section 16.3.3). If the SRD was performed on the parent sample of a DUP or PDS the results from the SRD may be used to report values of that parent sample if the 5 times dilution was appropriate.

Note: Dilution also increases the associated MDL by the dilution factor, so care must be taken not to dilute a sample so that the corrected concentration value is less than increased MDL.

16.10 Initial Demonstration of Capability

Each analyst must demonstrate initial proficiency for sample preparation and analysis by generating data of acceptable accuracy and precision for four LCSs. For demonstration of proficiency, acceptable accuracy and precision is defined as having both RSD $\leq$ 20% and percent recovery of 75%-125%. This demonstration is repeated whenever new staff receives training or significant changes in extraction procedure or instrumentation are made. The associated LCS recoveries are collected and maintained in the staff training files.

16.11 Decontaminating/Cleaning Labware

Procedures for proper cleaning and removal of trace metals from labware are found in Section 14.3.1 in SOP ERG-MOR-031. Detergent bath preparation instructions can be found in Section 11.19. Transport/dip baths of DI water ("To" and "From" acid baths) should be labeled with the date they are filled with fresh DI and changed about once every month to minimize contaminants being brought to the acid bath and from being reintroduced to clean labware. This will not only extend the life of the acid bath but it will help reduce hazardous waste production. To be certain that the acid bath will effectively clean and not contaminate labware a 5x dilution should be analyzed to determine the background about once a month.

17.0 PREVENTION

When possible, minimize the amount of chemicals used in the preparation and analysis of the metals filters to reduce waste.

18.0 DATA REVIEW AND CORRECTIVE ACTION

18.1 Data Review Documentation

Project files including at a minimum the information required in Section 22 are assembled and maintained by the performing analyst. Documentation for metals analysis by ICP/Mass Spectrometry will be reviewed for completeness and meeting acceptance criteria by the Task Lead or secondary reviewer associated with the project or program requiring the analysis as described in this section.

A second review of the data is performed by the Task leader or designated secondary reviewer using the QC review checklist (checklist) shown in Figure 24-8 to confirm that quality requirements have been met. Corrections and flags are added to the data consistent with the corrective action required for each review finding. Second level reviewers must complete, initial and date the checklist.

The completed check list is included as part of the data package. Data not meeting SOP requirements are flagged and brought to the attention of the Project Manager for resolution.

18.2 Quality Staff Review

A minimum of 10% of the data is reviewed by ERG Quality Staff. Quality staff review checks that all SOP required quality parameters have been met and that data reviewers have completed their review checklists. Additional items may also be reviewed at the discretion of the data reviewer. Quality staff reviews are documented on the review form initiated in Section 18.1 by the primary data reviewer. Comments or issues with data identified by the Quality Staff reviewer are brought to the attention of the Project Manager for resolution. Quality Staff will use the review process as an indication of episodic or systematic quality program issues that may require improvements to the ERG laboratory quality system and or additional training for ERG staff. As an option, Quality Staff may request a secondary review of 1% of the data from this method for a project. One percent (1%) review will follow the guidance in this section.

Corrective action for metals by ICP/Mass Spectrometry analysis data quality issues are presented in Table 24-3.

If required, a corrective action form is filled out as described in the ERG Laboratory Quality Systems Manual.

19.0 WASTE MANAGEMENT

Hazardous waste disposal is discussed in detail in SOP ERG-MOR-033. Sample digestate, including the extracted filter, is retained in its original tube for a period of at least 6 months from the sample date. After this time, sample digestate is disposed of by pouring it off in the satellite waste containers located in the laboratory. Used sample vials are disposed of in the laboratory waste bin.

20.0 MAINTENANCE

The ICP-MS system is not maintained under a service contract. The preventative and routine maintenance are performed by the analyst(s). In the event a maintenance issue cannot be addressed by the analyst a service call is placed with the manufacturer and when appropriate a service engineer will perform any necessary maintenance. All maintenance activities are documented in the instrument maintenance log.

20.1 The following maintenance procedures need to be addressed daily.

20.1.1 Check sample waste container level.

20.1.2 Inspect argon tank supply and its pressure to the instrument.

20.1.3 Inspect chiller coolant level and connections for possible leaks.

20.1.4 Inspect torch and aerosol injector tubes.

20.1.5 Inspect nebulizer for clogs.

20.1.6 Inspect sample capillary tubing to be sure it is clean and in good condition.

20.1.7 Check peristaltic pump tubing before operation.

20.1.8 At the end of each analysis, flush system for 5 minutes with the plasma on with a maximum of 2% nitric acid, followed by deionized water.

20.1.9 Inspect vacuum pump oil and replace as needed.

20.1.10 Inspect sample and skimmer cones for excessive salt build-up.

20.2 The following maintenance procedures need to be addressed quarterly (or more frequently if instrument performance indicates maintenance is needed).

20.2.1 Clean torch components and replace any worn O-rings on the torch assembly.

20.2.2 Inspect and clean the RF coil.

20.2.3 Inspect nebulizer spray pattern. Clean and replace gem tips and O-rings as necessary.

20.2.4 Check nebulizer components and replace worn O-ring on the transducer face.

20.2.5 Check spray chamber drain fitting for leaks.

20.2.6 Check that pump rollers are clean and remove and clean pump head as necessary.

20.2.7 Clean skimmer and sampling cones (See Section 20.3) and inspect orifices for damage or corrosion. Replace cones and/or O-rings as needed.

20.2.8 Replace interface roughing and turbo backing vacuum pump oil.

20.2.9 Inspect autosampler rinse pump rollers and clean or replace as necessary.

20.3 Cleaning Sample Introduction Components

20.3.1 Fill small plastic tank for sonicating components with warm to hot 2% Citranox® solution.Place torch, injector, spray chamber, gem tips, and transfer tube (remove all o-rings) into the tank. Sonicate all components for 15 minutes.

20.3.2 After removing the o-rings from the cones, **gently** insert each cone into a separate beaker with enough detergent solution to cover completely and allow to soak for ~ 1.75 hours. Care must be taken to only handle cones by the base – any contact with either cone orifice can easily cause irreparable damage.

20.3.3 Rinse all components thoroughly with tap water followed by a thorough rinse with DI water.

20.3.4 Place all components back in the cleaned tank/beakers with DI water and sonicate for another 15 minutes. Cones should be contained in a separate container from the other sample introduction components.

20.3.5 Carefully remove sample introduction components and rinse thoroughly with DI water.

20.3.6 Inspect cones under dissecting microscope to be sure that the edges of the orifices are not damaged and that the surfaces inside and out of the cone orifice area has been cleaned well. If the cones still appear to be dirty place in 2% HNO_3 and sonicate for **no more than** 2 minutes (more than 2 minutes of acid exposure will damage the cones) then rinse thoroughly with DI water and reinspect. If cones are needed immediately they may be dried by spraying Dust Off® over the entire surface, taking care not to make physical contact with the cones.

All other sample components that need further cleaning should be sonicated for 2 or more minutes in 2% HNO_3. Place in Class 100 hood or leave to air dry on a dust free cloth (e.g., Technicloth®) or Kimwipes® may be used. If cones are needed immediately they may be dried by spraying Dust Off® over the entire cone.

20.3.7 For cones that are still dirty after the procedure described in 20.3.6, and believed to be usable, refer to the Spectron Cone Cleaning Guide for additional procedures. Cones that are no longer usable may be returned to the manufacturer for recycling and platinum cones can be sent in for refurbishing.

21.0 SHORTHAND PROCEDURE

The flow chart shown in Figure 24-9 shows the procedural steps and sequence for analysis of inorganic samples.

22.0 DOCUMENTATION AND DOCUMENT CONTROL

22.1 All information concerning sample preparation, standard preparation, instrument conditions, etc., must be documented in the appropriate binders (i.e., Extraction Log, Daily Performance Reports, Standards Log etc.) and/or electronically in either the LIMS or the local instrument computer.

22.2 All calculations and the type of method for determining concentration must be recorded in the analyst's notebook. Any unusual problems or conditions must also be noted.

22.3 Record all maintenance performed on the instrument in the maintenance logbook for this particular instrument.

22.4 Record all sample analyses, including quality control samples, performed by the instrument in the ICP-MS run logbook for this particular instrument.

22.5 Reviewer must sign laboratory notebook weekly.

22.6 Any hard copies of instrument data should be filed chronologically. Electronic copies of instrument data are maintained on the L: drive in the "Metals Lab" folder.

22.7 It is imperative the project documentation be updated following each analysis. Analysts will copy raw instrument and QC files to a designated corporate network shared drive at the completion of each analysis sequence or batch. Primary data reviewers will use the data on the shared network drive for their data review process. The completed data packages ready for upload into the ERG LIMS system will be retained on the network drive as the backup for this data.

22.8 All processed data are archived in the LIMS on the shared network drive. Data is archived monthly to compact disc (CD) or digital versatile disc (DVD), verified on the system where the data originated and stored for at least five years in the laboratory. An archive copy of a data package is retained for at least five years in the laboratory data storage. The data backup should include enough information to manually generate the numbers used for reporting.

22.9 Reporting

22.9.1 Sample results are uploaded into the LIMS in ng/L as analyzed. Any dilutions performed must be accounted for in the instrument software. The internal standard recoveries must be included with the result calculation. Final results should be reported in ng/m^3 to three significant figures as shown in Section 12.1. If required by the sampling organization, results can also be reported in µg/m^3 by multiplying the ng/m^3 results by 1,000.

22.9.2 Sample results should not be corrected based on analyte results from the laboratory blanks, field, trip, or filter lot blanks provided by sampling agencies, unless specifically requested. However, samples are blank-subtracted if any analyte is detected in the LRB at greater than 5 times the MDL as described in Section 16.2.4.

22.9.3 As stated in Section 11.2.6, samples with metal concentrations greater than 90% of the current LDR must be diluted and re-analyzed. The diluted value will be reported to the sampling agency.

22.9.4 Data should meet all specifications as presented in Table 24-3. If data does not meet specifications, corrective reporting actions listed must be followed (flag or invalidate data).

23.0 REFERENCES

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24.0 TABLES, DIAGRAMS, FLOWCHARTS, VALIDATION DATA

Table 24-1. 2015 Method Detection Limits (MDLs) for Metals 47 mm Teflon Filters

Element	2015 MDL by FAC[1] (ng/L)	2015 MDL by FAC[2] (ng/filter)	2015 MDL by FAC[2,3] (ng/m^3)
Aluminum *	25779.74	1289	53.6
Antimony	19.647	0.98	0.041
Arsenic	73.69	3.68	0.153
Barium *	132.847	6.64	0.276
Beryllium	8.293	0.415	0.017
Cadmium	5.071	0.254	0.011
Calcium *	50738.52	2537	106
Chromium	5791.16	290	12.0
Cobalt	5.994	0.300	0.012
Copper *	250.362	12.52	0.521
Iron *	7579.5	379	15.8
Lead	18.638	0.932	0.039
Magnesium *	2615.008	131	5.44
Manganese	60.982	3.05	0.127
Mercury	17.111	0.856	0.036
Molybdenum *	42.075	2.10	0.088
Nickel	137.733	6.89	0.286
Rubidium *	3.269	0.163	0.007
Selenium	140.446	7.02	0.292
Strontium *	54.749	2.74	0.114
Thallium *	0.886	0.044	0.002
Thorium *	3.287	0.164	0.007
Uranium *	0.449	0.022	0.001
Zinc *	7055.234	353	14.7

* Elements not on our standard analysis list of elements.
† Total Chromium.
¥ MDL Calculated using the CFR Method.

Table 24-2. 2015 Limit of Quantitation (LOQ) for Metals

Element	ng/L
Aluminum*	23414
Antimony	70.6
Arsenic	261
Barium*	350
Beryllium	28.9
Cadmium	16.3
Calcium*	95407
Chromium†	5291
Cobalt	21.5
Copper*	709
Iron*	19063
Lead	53.4
Magnesium*	5443
Manganese	151
Mercury	38.0
Molybdenum*	151
Nickel	409
Rubidium*	10.7
Selenium	505
Strontium*	125
Thallium*	2.45
Thorium*	11.8
Uranium*	1.46
Zinc*	14896

* Elements not on our standard analysis list of elements.
† Total Chromium.

Note: This calculation assumes a total volume of 24.04 m^3.

Table 24-3. Summary of Quality Control Procedures for Metals Analysis

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Daily Performance Check (DPR)	Daily, prior to samples	Mg-24 > 40,000 cps, < 3% RSD In > 300,000 cps, < 3% RSD Pb-208 >100,000, < 3% RSD Ba/Ba++ and Ce/CeO < 0.03 Bkgd < 30 cps at Mass 220	1)Repeat analysis of DPR 2)Re-optimize instrument tuning parameters 3) Reprepare DPR standard 4) Perform instrument maintenance
Initial Calibration Standards (IC)	Daily, at least 5 calibration points	Correlation coefficient ≥ 0.998 & %RSD ≤ 10. RSDs >10 are acceptable for the CAL2 standard.	1) Repeat analysis of calibration standards 2) Reprepare calibration standards and reanalyze
Initial Calibration Verification (ICV)	Immediately after calibration	Recovery 90-110%, with the exception of Al	1) Repeat analysis of ICV 2) Reprepare ICV standard 3) Recalibrate and reanalyze.
Initial Calibration Blank (ICB)	Immediately after ICV	Absolute value must be ≤ MDL	1) Locate and resolve contamination problems before continuing 2) Reanalyze, recalibrate or flag failing elements for the entire analysis when appropriate
High standard verification (HSV)	After ICB and before ICS	Recovery from 95-105% with the exception of Al	1) Repeat analysis of HSV 2) Reprepare HSV
Interference Check Standard (ICSA/IFA)	Following the HSV, every 8 hours and at the end of each run	Within ±3 times LOQ from zero or from the standard background contamination when present	1) Repeat analysis of ICSA 2) Reprepare ICSA and analyze 3) Adjust correction equation(s) and reprocess entire analysis
Interference Check Standard (ICSAB/IFB)	Following each ICSA	Recovery 80-120% of true value plus standard background contamination when present	1) Repeat analysis of ICSAB 2) Reprepare ICSAB and analyze 3) Adjust correction equation(s) and reprocess entire analysis
Continuing Calibration Verification (CCV)	Analyze before samples, after every 10 samples, and at the end of each run	Recovery 90-110%, with the exception of Al	1) Reanalyze CCV 2) Reprepare CCV 3) Recalibrate and reanalyze samples since last acceptable CCV
Low Calibration Verification (LCV)	At the beginning and end of each analysis, between the CCV and CCB	Recovery 70-130% for Pb only	1) Reanalyze LCV 2) Reprepare LCV 3) Recalibrate and reanalyze samples since last acceptable LCV
Continuing Calibration Blanks (CCB)	Analyzed after each CCV	Absolute value must be ≤ MDL	1) Reanalyze CCB 2) Reanalyze samples since lastacceptable CCB
Laboratory Reagent Blank (LRB/BLK1)	1 per 20 samples, a minimum of 1 per batch	Absolute value must be ≤ MDL	1) Reanalyze 2) If > MDL, but < 5x MDL, sample results for that element must be flagged for the entire analysis 3) If > 5x the MDL then sample results for that element must be blank subtracted
Method Blank (MB/BLK2)	1 per 20 samples, a minimum of 1 per batch	Absolute value must be ≤ MDL. Note: The MB is used only for the purpose of MDL generation	This standard is not required by the method and there is no corrective action

Table 24-3. Summary of Quality Control Procedures for Metals Analysis (cont'd)

Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Standard Reference Material (SRM)	1 per 20 samples, a minimum of 1 per batch	Recovery 80-120% for Pb only	1) Reanalyze 2) Flag sample data 3) Re-extract batch
Laboratory Control Sample (LCS/BS and BSD)	1 per 20 samples, a minimum of 1 per batch	Recovery 80-120%, with the exception of Al	1) Reanalyze 2) Flag data if recovery for only one or two elements fail criteria 3) Reprepare sample batch if recovery for most elements fail criteria
Replicates (DUP) (Replicate Analysis)	1 per 20 samples, a minimum of 1 per batch	≤ ±10% RPD for sample and duplicate values ≥ 10 times the MDL	1) Check for matrix interference in the cased of DUP1. 2) Repeat replicate analysis 3) Flag data
Collocated Samples (C1/C2)	10% of samples annually	≤ ±20% RPD for sample and collocate values ≥ 10 times the MDL	1) Flag C2 data if associated replicate analysis are within criteria 2) Repeat analysis if replicate analysis fails.
Post Digestion Spike (PDS)	1 per 20 samples, minimum of 1 per batch	Recovery 75%-125%	1) Flag failed elements for parent sample and PDS 2) Reprepare PDS if preparation issue is suspected reason for failure.
Serial Dilution (SRD)	1 per batch	Recovery 90-110% of undiluted sample if the element concentration is minimally a factor of 50 above the MDL in the original sample	1) Re-prepare dilution if preparation issue is suspected reason for failure. 2) Flag failed analytes
Internal Standards (ISTD)	Every Calibration, QC and Field Sample	Recovery 60-125% of the measured intensity of the calibration blank	1) If drift suspected, stop analysis and determine cause, recalibrate if necessary 2) Reprepare sample 3) If recovery > 125% due to inherent ISTD, dilute sample and reanalyze

Table 24-4. Optimization Procedures

Procedure	When to Perform
Nebulizer Gas Optimization	Daily.
Ion Lens Voltage Optimization	Daily.
Auto Lens Optimization	Daily, when Auto Lens is used in the acquisition method.
Dual Detector Cross Calibration	Cross calibration is necessary if you have selected Dual Mode as your Processing Method. This would only be done when you require extended dynamic range (above 2 million cps). **Note:** This must be performed before each analysis for this method.
Instrument Performance Check	Daily.
X-Y Adjustment	Whenever the cones have been cleaned or replaced, or after any torch maintenance procedure.
Detector Optimization: Pulse Stage Voltage Analog Stage Voltage	When sensitivity cannot be recovered through other cleaning or optimization methods or when the detector is replaced.
Deadtime Correction	This procedure should only be performed if a detector has been replaced, and after the new detector has been optimized.

Table 24-5. Performance Specifications

24Mg Sensitivity	> 40,000 cps	< 3% RSD
In Sensitivity	> 300,000 cps	< 3% RSD
208Pb Sensitivity	> 100,000 cps	< 3% RSD
CeO/Ce	<0.03	N/A
Ba++/Ba+	<0.03	N/A
Background	< 30 cps @ Mass 220	N/A

Table 24-6 Blank Spike Standard (Used for BS/BSD) Preparation (50 mL)

Element	Stock Std. Concentration (µg/mL)	Added Volume (mL)
Al	1000	1.5
Sb	100	0.25
As	100	1.0
Ba	1000	0.5
Be	100	0.25
Cd	100	0.25
Ca	1000	1.25
Cr	100	2.5
Co	100	0.25
Cu	1000	0.5
Fe	1000	0.5
Pb	100	0.25
Mg	1000	0.5
Mn	100	0.5
Hg	10	0.5
Mo	100	0.25
Ni	100	0.5
Rb	1000	0.025
Se	100	1.0
Sr	100	0.25
Tl	10	0.5
Th	10	0.5
U	10	0.5
Zn	1000	0.5

Table 24-7. Analytical Isotopes for Quantitation and Monitoring of Reported Elements

Element	Quantitation Isotope(s)	Monitored/Confirmation Isotope(s)
Aluminum	27	NA
Antimony	121	123
Arsenic	75	NA
Beryllium	9	NA
Barium	137	135
Bismuth (ISTD)	209	NA
Cadmium	111	106, 108, 114
Calcium	43	NA
Chromium	52	53
Cobalt	59	NA
Copper	63	65
Iron	57	54
Gallium (ISTD)	71	NA
Indium (ISTD)	115	NA
Lead	208	206, 207
Lithium (ISTD)	6	NA
Magnesium	25	24
Manganese	55	NA
Mercury	201	200, 202
Molybdenum	98	92, 94, 95, 97
Nickel	60	62
Rubidium	85	NA
Scandium (ISTD)	45	NA
Selenium	82	77, 78
Strontium	88	NA
Thallium	205	203
Thorium	232	NA
Uranium	238	NA
Yttrium (ISTD)	89	NA
Zinc	66	67, 68

NA = Not applicable/none.

Figure 24-8. Quality Control Review Form (Page 1)

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Quality Control Review Form Metals Analysis -- 47mm Filters 2015-1

Sequence ID: _____ **Instrument:** _____ **Batch:** _____
Cal Curve (Method): _____ **Analyst:** _____ **Date:** _____
10% Review Sample IDs: _____ **Reviewer:** _____ **Date:** _____
Optional 1% Review Sample IDs: _____ **Reviewer:** _____ **Date:** _____

Parameter	Acceptance Criteria	Analyst Check (Initials and Date)	Task Lead/Data (Initials and Date)	10% QA Review (Initials and Date)	1% Optional QA Review (Initials and Date)	Comments
Instrument QC						
Daily Performance Report	Mg-24 > 40,000 cps, < 3% RSD In > 300,000 cps, < 3% RSD Pb-208 >100,000, < 3% RSD Ba/Ba++ and Ce/CeO < 0.03 Bkgd < 30 cps at Mass 220					
Initial Calibration Standards (IC)	≥0.998 correlation coefficient & RSD ≤10. RSDs >10 are acceptable for the CAL2 standard.					
Initial Calibration Verification (ICV)	Recovery 90-110%.					
Initial Calibration Blank (ICB)	Absolute value must be ≤ MDL.					
High Calibration Verification (HCV)	Recovery 95-105%.					
Interference Check Standard (ISCA/IFA)	ICSA: ±3 times QL from zero or from the standard background contamination when present					
Interference Check Standard (ICSAB/IFB)	ICSAB: Recovery 80-120% of true value plus standard background contamination when present					
Continuing Calibration Verification (CCV)	Recovery 90-110%.					
Low Calibration Verification (LCV)	Recovery 70-130% for Pb; must be analyzed at the beginning and end of each analysis.					
Continuing Calibration Blanks (CCB)	Absolute value must be ≤ MDL.					
Internal Standard Response	Recovery must be between 60 and 125% of the measured intensity of the calibration blank					

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Figure 24-8. Quality Control Review Form (Page 2)

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Quality Control Review Form Metals Analysis -- 47mm Filters 2015-1 (Continued)

Parameter	Acceptance Criteria	Analyst Check (Initials and Date)	Task Lead/Data (Initials and Date)	10% QA Review (Initials and Date)	1% Optional QA Review (Initials and Date)	Comments
Linear Dynamic Range Check	All sample values must less than 90% of the established linear dynamic range.					
Extraction QC						
Check Sample Volume	Check COC or filter envelope against Bench Sheet to make sure sample volumes are correct.					
Laboratory Reagent Blank (LRB/BLK1)	Absolute value must be ≤ MDL.					
Method Blank (MB/BLK2)	Absolute value must be ≤ MDL.					
Standard Reference Material (SRM)	Recovery 80-120% for Pb.					
Laboratory Control Sample (LCS/BS/BSD)	Recovery 80-120%, with the exception of Al.					
Collocated/Duplicate Samples (D1/D2, C1/C2)	±20% RPD when concentration of either sample is ≥ 10x the MDL					
Replicate Analyses (DUP)	±10% RPD when concentration of the parent sample is ≥ 10x the MDL					
Serial Dilution (SRD)	Recovery 90-110% of undiluted sample if the parent sample concentration is > than 50x the MDL.					
Post Digestion Spike (PDS)	Recovery 75-125%					
Reporting Requirements						
Manual Check of Calculations	Hand calculate an equation - a unit conversion equation, a sample concentration equation, a dilution equation, etc. – to verify equation					
Check LIMS Qualifiers	Check to make sure the LIMS data flags are correct					
Negative Sample Values	Negative sample values must be less than the absolute value of the MDL.					

This review check sheet must be completed by primary data reviewer/TL/QA.

Figure 24-9. Flow Diagram for ICP-MS Preparation and Analysis

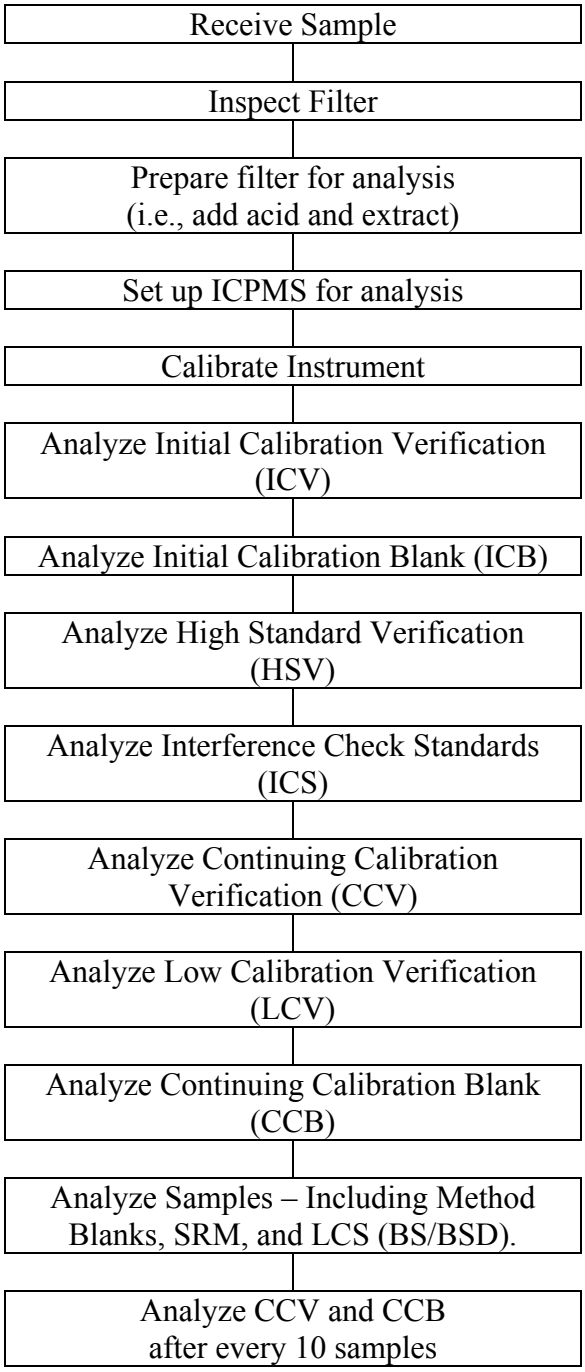

Table 24-10. Teflon Extraction Record

Record for Teflon Extraction Times, Temperatures & H_2O_2 addition 2014-1

Batch: **Date:** **Initials:**_____

Start Time	
Start Block Temperature (°C)*	
Sample Temperature (°C)†/Time	
Time H_2O_2 was added	
Stop Time (Removed from Block)	
Stop Block Temperature (°C)*	
Sample Temperature (°C)†/Time	
Time & Temp. (°C) brought to final volume	

*Block temperature is read from the HotBlock™ controller using an internal thermocouple.
†Sample thermometer serial #: 19587, actual thermometer temperature not the corrected temp.

Thermometer block position # _____

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CONFIDENTIAL
Standard Operating Procedure
Procedure Number: ERG-MOR-098
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ENGINEERING AND SCIENCE DIVISION

TITLE: SOP for the Preparation of Monitoring Data for AQS Upload	EFFECTIVE DATE: OCT - 1 2014
REFERENCES: N/A	
SATELLITE FILES:	
REASON FOR REVISION: Updates	
WRITER: NAME/DATE Jaime Hansen 10-1-14	PROJECT MANAGER: NAME/DATE Julie C. Swift 10/1/14
QUALITY ASSURANCE COORDINATOR: NAME/DATE Donna Tedder 10/1/14	NEXT SCHEDULED REVIEW: 1/31/15

1.0 IDENTIFICATION AND PURPOSE

The purpose of this document is to define, describe, and present the Standard Operating Procedure (SOP) for the preparation and upload of monitoring data into Environmental Protection Agency's (EPA) Air Quality System (AQS) database. This SOP assumes that the user will have knowledge of how to load data into AQS as well as how to use Microsoft Access. This SOP focuses on how to use the various tools described to translate Laboratory Information Management System (LIMS) data into the correct format for AQS upload.

2.0 MATRIX OR MATRICES

N/A

3.0 METHOD DETECTION LIMIT

N/A

4.0 SCOPE AND APPLICATION

This document outlines the step-by-step process of extracting data from the LIMS, importing and coding the data via a Microsoft Access "Macro" database, and loading the data into EPA's AQS database. Although the intent of this document is to provide instructions on how to extract, code, and load data for the National Monitoring Program, these steps may also be replicated and/or adjusted for any given subset of data for which extracting, coding, and/or AQS loading of the data may be necessary. Not every step or query described in Section 14 is applicable in every situation; some queries may need to be altered or may be skipped entirely. However, translated data sets are not affected by running unnecessary queries from the tools described in this SOP.

5.0 METHOD SUMMARY

N/A

6.0 DEFINITIONS

AQS – EPA's "repository of ambient air quality data" and includes ambient air quality data for criteria pollutants and hazardous air pollutants, as well as meteorological data and metadata information on monitoring locations (http://www.epa.gov/ttn/airs/airsaqs/).

"AQS" Database – a Microsoft Access database where the AQS Tables are exported and parsed out based on various client requests.

AQS RB Template – the table in the Macro database where AQS coding is appended for blank (field blank, trip blank, or lot blank) data; this table is directly loaded into AQS.

AQS RD Template – the table in the Macro database where AQS coding is appended for raw (field sample, primary, or collocate) data; this table is directly loaded into AQS.

AQS RP Template – the table in the Macro database where AQS coding is appended for precision (duplicates and/or replicates) data; this table is directly loaded into AQS.

LIMS – Laboratory Information Management System; the lab's repository of concentration data.

"Macro" Database – a Microsoft Access database where queries are run to convert the data into AQS format; queries are also run to perform QA on the AQS Tables that are generated and loaded into AQS.

POC – Parameter Occurrence Code, used in AQS to distinguish data from different sources for the same site, date, and pollutant.

QCData Template – a table in the Macro database where the QC (or precision) data are appended after they have been quality assured; all queries in the Macro database run on this version of the QCData table because the names are formatted in a specific manner.

"Raw" Database – a Microsoft Access database where data is initially imported and reviewed for completeness.

Sampdata Template – a table in the Macro database where the Sample data are appended after they have been quality assured; all queries in the Macro database run on this version of the Sampdata table because the names are formatted in a specific manner.

Worklist Spreadsheet – a Microsoft Excel table exported from LIMS that provides information related to individual samples not available with the raw concentration data.

Worklist Template – a table in the Macro database where the Worklist data are appended after they have been quality assured; all queries in the Macro database run on this version of the Worklist table because the names are formatted in a specific manner.

7.0 INTERFERENCES

N/A

8.0 SAFETY

N/A

9.0 EQUIPMENT

N/A

10.0 MATERIALS

N/A

11.0 CHEMICALS, REAGENTS, AND STANDARDS

N/A

12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

N/A

13.0 CALIBRATION AND STANDARDIZATION

N/A

14.0 PROCEDURE

The procedure described in this section produces two results: 1) it transcribes data from LIMS into AQS-coded files with the proper syntax to export and load directly into EPA's AQS database; and 2) it compiles sample data, QC data, and subsequent sample information into a single table in Microsoft Access that is used to perform various data analyses.

It is recommended that notes are taken for each transcription run-through. A log and notes of every data transcription/translation record error or change required to complete the format of the monitoring data shall be kept. A record of the number of records generated from each of the queries in the Macro database (as directed in specific steps in this SOP) shall be kept. It is especially important to note if a sample or its corresponding data are modified for any reason, the reason the change occurred, and how the omission or change was determined.

A detailed, step-by-step list of instructions is provided below.

14.1 Generating a Worklist and Extracting Data from LIMS

Note: Extraction of data from ERG's LIMS requires authorization to access LIMS data and experience using the LIMS system.

14.1.1 In LIMS, go to Laboratory and select Query Analysis Status.

14.1.2 In the "Query For:" drop-down menu, select the appropriate Lab Project Manager (i.e. Julie Swift) and/or work order numbers.

14.1.3 Select the needed sampled date range (i.e. 12/31/12 23:59 through 4/1/13 00:00). Make sure the time for the last date of the range is 00:00 on the next day or data for samples taken on that date may not be included.

14.1.4 In the Status menu, select all status types except "not required."

14.1.5 To the right of the window, order results by project name – analysis – sampled.

14.1.6 Choose Query. When the list appears in the window, select export and save the worklist as an Excel file in the desired C: or L: drive location.

14.1.7 Open the Excel file. Select the Data menu – Filter – Auto Filter.

14.1.8 Sort ascending for sample Status. All statuses marked "Canceled" can be deleted. Sort ascending for Sample Type and delete the following: Audit samples, Filter Blanks, and Passive Blanks.

14.1.9 Review the Sample Type entries to be sure the sample type agrees with the information in the Sample Name and Comments (C1, C2, etc.) entries.

14.1.10 Sort ascending for Status. For samples marked "Invalid," review sample comments and assign AQS null codes using the WorkUser10 column left of the comments. A list of null codes is provided in Table 14-1 (with the most commonly used codes in bold).

14.1.11 With all records showing, sort ascendingly by client (i.e. Region 4) and note the highest and lowest work order number (the first seven digits of the Lab Number) for each client. This will be used to pull the actual concentration data.

14.1.12 To pull the concentration data from LIMS, go to Project Management – Reports.

14.1.13 Choose client (i.e. Region 4).

14.1.14 Select work orders (on right of screen) corresponding to those on the worksheet for this client (find the highest work order noted in Step 14.1.11 and choose, scroll to find the lowest work order, with the shift button held, choose the lowest work order – this will highlight all of the work orders between the two).

14.1.15 Choose destination – C:\ELMT\Userfiles\.

14.1.16 Choose custom – EDD Format – EEDStdExcel_ERG.

14.1.17 Select modified draft – when window opens sort left side of screen by name – then on the right side of the screen, remove (uncheck) everything except the duplicates.

14.1.18 Select continue - report will be generated and saved in the destination chosen.

14.1.19 Email data and worklist Excel files to the person performing the data conversion.

Table 14-1. List of Null Codes

Qualifier Code	Qualifier Description
AA	**Sample Pressure out of Limits**
AB	Technician Unavaliable
AC	Construction/Repairs in Area
AD	Shelter Storm Damage
AE	Shelter Temperature Outside Limits
AF	**Scheduled but not Collected**
AG	**Sample Time out of Limits**
AH	Sample Flow Rate out of Limits
AI	Insufficient Data (cannot 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	Q C Control Points (zero/span)
AZ	Q C Audit
BA	Maintenance/Routine Repairs
BB	Unable to Reach Site
BC	Multi-point Calibration
BD	Auto Calibration
BE	Building/Site Repair
BF	Precision/Zero/Span

Qualifier Code	Qualifier Description
BG	Missing ozone data not likely to exceed level of standard
BH	Interference/co-elution/misidentification
BI	Lost or damaged in transit
BJ	Operator Error
BK	Site computer/data logger down
BL	QA Audit
BM	Accuracy check
BN	Sample Value Exceeds Media Limit
BR	Sample Value Below Acceptable Range
CS	Laboratory Calibration Standard
DA	Aberrant Data (Corrupt Files, Aberrant Chromatography, Spikes, Shifts)
DL	Detection Limit Analyses
FI	Filter Inspection Flag
MB	Method Blank (Analytical)
MC	Module End Cap Missing
SA	Storm Approaching
SC	Sampler Contamination
ST	Calibration Verification Standard
TC	Component Check & Retention Time Standard
TS	Holding Time Or Transport Temperature Is Out Of Specs.
XX	Experimental Data

14.2 Getting Started in Microsoft Access with the “Raw” Database.

14.2.1 Copy the Microsoft Access database entitled “QX 20XX RAW” from L:\Jaime\REFERENCES\ and paste into the designated location. Adjust the name of the database accordingly.

14.2.2 Import the Microsoft Excel Worklist spreadsheet (may need to do some “un-formatting”of the file such as removing autofilters, unfreezing panes, unhiding columns, etc.) into the Microsoft Access “Raw” database as a table named “Worklist”.

14.2.3 In the Raw database, review the Worklist for errors such as multiple samples on a single date, missing null codes, data from outside the date range of interest, etc. Make note of any errors, changes, deletions, etc. Resolve any issues with the Sample Log-in Leader or Project Manager and request that the Sample Log-in Leader make corrections in LIMS (if needed). Note any special flags that may be needed in the SampleUser5

column based on the SAMPLECOMMENTS column and available Qualifier Flags for AQS (http://www.epa.gov/ttn/airs/airsaqs/manuals/codedescs.htm). Also note any sites performing manual sampling, running PAMS samples or other samples of a different duration than 24 hours. This is needed for step 14.3.14.

14.2.4 Run the Query0 queries. The "Null Code" query checks to make sure all invalid samples have a null code and the "Check Void" query ensures that all samples with "VOID" in the sample names have an "Invalid" sample status. Run the "Qualifier Flag" query to confirm that the correct qualifier flag has been added to the SampleUser5 field based on the SampleComments field.

14.2.5 Import each of the tabs (SAMPDATA, QCDATA, LNOTE) in the Microsoft Excel raw data spreadsheet files into the Raw database. Do not let Microsoft Access assign a primary key during import. If data were pulled from LIMS in multiple spreadsheets, compile tabs of the same name together in a single table. Note that if the data have been pulled by Region (or method, etc.), a Region field needs to be added to the LNOTE table and updated after each successive import in the format "Region X". The same flag in LIMS can be used to denote different things among different datasets. For example, A-01 is used for manual entry of comments in the LNOTE or ANOTE columns of the raw data. A sample from a site in Region 4 may have an A-01 flag denoting late arrival, while a sample from Region 2 may have an A-01 flag for surrogate recovery issues. Since both samples are denoted with A-01 in the raw data, another aspect of the data, such as Region in this case, must be used to distinguish to what the flag refers. Smaller data sets (data pulled from LIMS in a single spreadsheet) do not need this.

14.2.6 If data were pulled from LIMS in multiple spreadsheets, run the "Confirm All Regions" queries to ensure that data for all of the regions was appended during the import process (for the Sampdata and LNOTE tables.

14.2.7 Run the "missing data" queries in the Raw database (Query1s). They verify that the samples listed in the Worklist match the SAMPDATA and vice versa. If everything is correct, no records will appear in the results. If records appear, there are records in one table that do not match the other; this must be resolved before moving forward.

14.2.8 Resolve any missing data issues with the Project Manager and/or Sample Log-in Leader. Often, data were not "locked" in LIMS and the

data must be re-pulled from LIMS. Be careful with the LNOTEs if additional data are appended into the existing tables. If these are added to the LNOTE table, they must agree with the original Region (or method) they were loaded with. Refer to 14.2.5 for further explanation.

14.3 Coding Raw Data in the "Macro" Database

14.3.1 Print out the AQS Steps Record Sheet spreadsheet (Figure 24-1) located at L:\Jaime\AQS. This is used to note the results from various queries and steps in the coding process.

14.3.2 Once the data are ready in the Raw database, copy the most recent Access MACRO database from L:\Jaime\REFERENCES and paste into the designated location. Adjust the name accordingly.

14.3.3 Delete the following tables in the Macro database (if they exist): D1s, D2s, any "Data X" tables, Dups from QCDATA, Invalids, LNOTE, QA1 Dup Data, QA2 Worklist Data, QCDATA, Qualifiers, SAMPDATA, SAMPDATA + INVALIDS, SAMPDATA + INVALIDS + R1R2, and Worklist.

14.3.4 Delete data from (while leaving the shell table intact) the following six TEMPLATE tables in the Macro database: AQS RA TEMPLATE, AQS RB TEMPLATE, AQS RP TEMPLATE, AQS RD TEMPLATE, QCDATA TEMPLATE, SAMPDATA TEMPLATE, WORKLIST TEMPLATE.

14.3.5 Compact the Macro database. This "deletes" items in Access' memory and reduces the size of the database.

14.3.6 Import the new Worklist, SAMPDATA, LNOTE, and QCDATA tables from the RAW database used in steps 14.2.

14.3.7 In the Macro database, run queries 00A-00C to append the Worklist, QCDATA, and SAMPDATA to their associated template tables (the template tables have formatted column names and data). Note the number of records in the record sheet. From this point forward, all data manipulations are done on the Template tables. This is useful in case there is a need to go back to the "original" versions of the data.

14.3.8 Run queries 00D1 and 00D2. These update the Units column in the QCDATA and SAMPDATA TEMPLATES from "ng/m^3 Air" or "ug/m3 Air" to "ng/m3" or "ug/m3", respectively, which is the format in the reference (or LOOKUP) tables. Note the number of records changed

in the "UPDATE UNITS" slot on the record sheet. Run Query 00D3 to confirm the update in the SAMPDATA Template and check for any other unexpected units of measure.

14.3.9 Run queries 00E, 00F, and 00G. These update the Analysis column in the Worklist, SAMPDATA and QCData TEMPLATES from "Metals Analysis – 47mm" or "Metals Analysis – HiVol" to just "Metals Analysis", which is the format in the reference tables. Note the number of records changed in the "UPDATE ANALYSIS" slot on the record sheet.

14.3.10 Run 01A and 01B. These are similar checks as in step 14.2.7 above. They verify that the samples listed in the Worklist Template match the SAMPDATA TEMPLATE and vice versa. If everything is correct, no records will appear in the results. If results appear, there are records in one table that do not match the other and this must be resolved before moving forward.

14.3.11 Run query 01C. This query looks for possible mislabeled blanks by comparing the SampleType column and the SampleComments column in the Worklist Template. Any results must be resolved before moving forward.

14.3.12 Run query 01D1 and 01D2. These queries put site information for BOMA and TOOK into the SampleInfo1 column. These two sites (for metals only) are collocated and sometimes the Field Samples do not correspond to the primary (C1) sampler; this affects which POC should be used. Note that site info designated with "A" refers to the primary (C1) and "B" refers to the collocated (C2) sampler. Verify that these ran correctly by running query 01D3; a slight deviation in sample log-in sometimes affects the way these are listed. The SampleInfo3 column should say "TOOK A" or "TOOK B" for TOOK samples and "Roxbury A" or "Roxbury B" for BOMA samples. Should a dataset not contain these sites, the queries will simply not update these fields.

14.3.13 Run query 01E1 and 01E2. Query 01E1 trims the SampleName column entries in the Worklist Template table to match the site id in the Project column. Query 01E2 handles sites with names that deviate from the typical four-letter site code (like INDEM). Verify that these queries ran correctly by running query 01E3. Check each trimmed site name to verify that it is correctly formatted.

14.3.14 Run query 01F1 to update start times and durations for any manual samples. If no manual samples are included, a pop-up screen appears

stating that "no sites currently do manual sampling." This should be noted when reviewing the Worklist in Section 14.2 and any sites doing manual samples added prior to running this query. Run query 01F2a to update the duration codes (from 24 to 3 hours) for any PAMS samples; run query 01F2b to update the duration codes for any 1-hour samples. Review the start times and start dates of all manual samples or PAMS samples to ensure that the proper start date and time has been applied to the SampleDate and StartTime fields using query 01F3 to manually update and confirm by running query 01F4.

14.3.15 Run query 01G1 through 01G3 to trim the year from the end of the Methodcode or Analysis columns in the Worklist, Sampdata, and QCData Template tables.

14.3.16 Run query 01H1 and 01H2. These update the POC column of the Worklist Template with the correct POC for TOOK and BOMA (refer to step 14.3.12 for the reason).

14.3.17 Run query 01I1 to verify that there is a POC in the POC Rules table for every site-method-sample type combination. Any results that are returned must either be added to the POC Rules table or QA'ed to make sure a sample type was not mislabeled (such as a site that usually runs duplicates mislabeled as collocates or vice versa). Run query 01I2 to update the POC column in the Worklist Template with the POCs for all other site-method-sample type combinations. Run query 01I3 to confirm that all rows have been updated in the POC column. Any results must be resolved before moving forward.

14.3.18 Run query 01J to verify that the correct number of pollutants are provided for each sample contained in the SAMPDATA Template. Any results returned need to be resolved before moving forward. SNMOC samples have 82 analytes; TO-15 (VOC) samples have 58 analytes; TO-13 (SVOC or PAH) samples have 22 analytes or 25 for the TO-13 plus Phenols list; TO-11A w/ MEK (carbonyl) samples have 13 analytes; IO-3.5 (metal or inorganic) samples have 11 analytes, or in the case of the extended list, 24; and Methane and hexavalent chromium have only one analyte each. Queries 01J2 through 01J4 may be used to resolve exact dups. Run query 01J2 to confirm that there are exact dups; run query 01J3a to make a table of the dups ("Dups to Resolve from Sampdata Template") and then query 01J3b to delete the dups from the Sampdata Template. Lastly, run query 01J4 to group on the dups and append back into Sampdata Template. After completing this sequence of queries, re-run query 01J to confirm that the dups have been removed.

14.3.19 Run query 01Ka to look for any hexavalent chromium samples that have the HT flag in the SampleComments column of the Worklist Template. This flag is used to identify a sample that was exposed to an ambient temperature above a given level (60ºF) when not picked up the day after sampling and will be flagged with a qualifier code in AQS. Run query 01Kb to update the Qualifier column of the Worklist for any of the samples from query 01Ka. Verify that the number of samples updated in 01Kb matched the results from 01Ka. Run query 01Kc to confirm that the flag has been applied.

14.3.20 Run query 01L, which looks for any duplication in the Worklist Template table. Any results that appear must be reconciled before moving forward.

14.3.21 Run query 01M1 through 01M3 to check for numerical results associated with data flagged with the co-elution LIMS flag in the SAMPDATA Template table. Because data flagged with the co-elution flag could not be resolved by the instrument, any such results need to be reported to the Project Manager and likely changed to ND in the table as well as LIMS. Run query 01M1 to look for any such results. If found, run query 01M2 to make a table of the original data for record-keeping purposes. Run query 01M3 to manually update the numerical results to ND. Re-run query 01M1 to confirm that all of the changes have been made.

14.3.22 Run query 01N to confirm that all TNMOC results are numeric. These results are put into LIMS manually and occasionally uploaded incorrectly. Any results that appear must be reconciled before moving forward.

14.3.23 Run query 01O. This updates the SAMPDATA Template with all the information from the Worklist Template. Note the number of records in the record sheet in the 01 slot. This number should match the number from the Import SAMP slot (assuming there have been no data changes such as the removal of dups).

14.3.24 Run query 02a1. This query links the QCDATA Template and the Worklist Template and makes a new table “Dups from QCData”. This query is run to retrieve only duplicate sample data for the samples we’re loading into AQS and not all the other types of QC data that could be in the QCDATA Template table (such as LCS, spikes, etc). Note the number of records in the record sheet in the 02 slot. Run query 02a2 to look for dups in the new Dups from QCData tables; if any are identified, run query 02a3 to make a table of the dups (“Data with Dups in

Precision") and resolve these with the Project Manager. Run query 02a4 to append the correct results back into the Dups from QCData table. After completing this sequence of queries, re-run query 02a2 to confirm that the dups have been resolved.

14.3.25 Run query 02b. This query verifies that the correct number of pollutants is provided for each sample contained in the Dups from QC Data table, similar to step 14.3.18.

14.3.26 Because invalid samples are loaded into AQS but no data is generated from LIMS for invalid samples, records must be created for invalid samples (records with no results for each applicable analyte per method). Run query 03a to ensure all invalid samples have been assigned null codes in the Worklist. Any results that appear must be reconciled before moving forward. Run query 03b to make an Invalids table for non-metal samples, then run query 03c to append in records for metals samples. Note that query 03b must be run first as it sets up the template of the table. Note the number of records from query 03b and 03c in the record sheet in the 03 slot.

14.3.27 Run query 04a to make a new table from the SAMPDATA TEMPLATE called SAMPDATA + INVALIDS. Note the number of records in the record sheet in the 04 slot. Run query 04b to append in records from the Invalids table. The number of records from the 03 slot on the record sheet plus the number of records from the 04 slot should equal the total number of records now in the SAMPDATA + INVALIDS table. Note this total on the record sheet in the "Total in Samp+" slot.

14.3.28 Go into the design of the SAMPDATA + INVALIDS table. Scroll to the bottom and change the "DL-NUMBER" and "RESULT-NUMBER" data types from binary to number and change the field size to double. Run query 04c to update these columns in the SAMPDATA + INVALIDS table. These columns will be used later to assign quantitation flags based on the MDLs and Results. (An error message may pop up when query 04c is run. This is because the query is trying to append "ND"s to a numerical field. Click OK, which will result in a null or blank in the RESULT (or DL)-NUMBER columns.)

14.3.29 Run query 04D to ensure that none of the results are in scientific notation. If results are found, these need to be converted to standard format before moving on.

14.3.30 Skip query 04E and run query 04F. These queries look for results that appear too low based on a few marker analytes. Investigate any results

and resolve with Project Manager. Skip query 04g and run query 04h to check on the number of NDs in a sample. This can be a marker for a mislabeled field blank. Investigate any results and resolve with Project Manager.

14.3.31 Run query 05. This formats the date, in case any unformatted dates get through. Note this in the record sheet under the 05 slot.

14.3.32 Run query 06A1. This puts the Region in the Client column of the Worklist Template by trimming the client name to Region X (this relates back to step 14.2.6). Run query 06A2 to verify that query 06A1 ran properly and resolve any records that appear.

14.3.33 Run query 06b1 to update the LNOTE-ANOTE MERGED column in the SAMPDATA + INVALIDS table. Skip query 06b2 and run query 06b3 to append any instances of multiple flags into the LNOTE table. In the case of multiple flags, manually update the Description column in the LNOTE table to account for both flags. Often, the second flag is just the U flag (Under Detection Limit). Make sure that this description is always listed second in the Description field. Note that these are Region-specific, particularly the A-01x flags, in the case of large data sets.

14.3.34 Run query 06c to verify that all flag descriptions in the LNOTE table appear in the LNOTE Descriptions reference table. If there are no results, move to the next step. If results appear, add the missing description from the LNOTE table to the LNOTE Descriptions table and assign the proper AQS flag, if needed. The Project Manager may need to be consulted to identify the proper description.

14.3.35 The LIMS flag D-F is used to denote any precision results with greater than a method-specific percent difference between a primary and a duplicate/collocate. This flag is applied to the duplicate or the collocate record in LIMS. Because the duplicate data are loaded into AQS in the precision data table (RP or QA), and this table does not accept flags, the flag is essentially missed; thus, the flag needs to be moved to the primary sample for coding purposes in the case of duplicates. (Because both the primary and collocate are loaded into the Raw Data table (RD) in AQS, this is not a problem for collocated pairs). Run query 06d1 to check for D2 results with D-F flags and run query 06d2 to apply the flag to the D1s. Confirm that the number of records is the same for both queries. Run query 06d3 to confirm the flag has been added correctly. Confirm that this combination of Regions and LNOTEs is in the LNOTE and LNOTE Description tables using query 06d4.

14.3.36 Run query 06e1 to count how many results have a D flag applied but the dilution factor is 1. Run query 06e2 to confirm that samples with dilutions greater than 1 have a D flag applied. Run query 06e3 to confirm the total. Resolve any issues with the D flag with the Project Manager.

14.3.37 Run query 06f1 to get a preliminary count on how many records need qualifier codes. Run query 06f2 to create the table Qualifiers. (Note, if this is a small data set, remove the link between the Region field of the LNOTE table and the LNOTE-ANOTE Merged field of the SAMP+INV table in the design of the query.) If the number of records is not the same for query 06f1 and 06f2, run query 06f3 to see where the discrepancies lie and resolve them before moving on. Run query 06f4 to update the Client field in the SAMP + INV table then run query 06f5 to count how many records should have qualifiers as a second QA step. The number of qualifiers from query 06f5 should match the number in the Qualifier table. Note the number of records in the Qualifier table on the record sheet.

14.3.38 Run query 07AA to append non-metal non-detects to the AQS RD TEMPLATE for AQS Raw Data (RD) coding. Note the number of records in the record sheet under the 07A slot. Run query 07AB to append metal non-detects to the AQS RD TEMPLATE. Also note this in the record sheet under the 07A slot.

14.3.39 Run query 07BA to append non-metal detects (and invalids) to the AQS RD TEMPLATE. Note the number of records in the record sheet under the 07B slot. Run query 07BB to append metal detects (and invalids) to the AQS RD TEMPLATE. Also note this in the record sheet under the 07B slot. On the record sheet, sum the four values under 07AA-07BB and enter into the “Total in AQS” slot.

14.3.40 Run query 07C and enter the value in the Total Excluded row of the record sheet. This value represents precision and blank data not converted yet to AQS code as well as excluded internal standards data that are not coded. This value plus the value in the “Total in AQS” slot should equal the value in the “Total in SAMP+” slot. If it does not, there is an error somewhere. Queries have been created to find some common mistakes: Run query 07D (and 07D2) to make sure the pollutants match properly. Run query 07E to verify all site-method-sample type combinations have been assigned a POC in the POC Rules table. Run query 07F to verify if there are any new sites not found in the LOOKUP – Site Info reference table. If there are still errors after these are run, further review is needed.

14.3.41 Run queries 08A and 08B to create tables for D1/C1 data and D2/C2 data, respectively. Note the numbers of records in the 08A and 08B slots on the record sheet. The number of records in each table should be similar, but may not be the same.

14.3.42 Run query 09A1 through 09A3 and note the number of records from each query on the record sheet in slot 09A. Query 09A1 puts D2 data (with a precision id of 1) in the AQS RP TEMPLATE. Queries 09A2 and 09A3 add precision records for C1 and C2 (also with a precision id of 1) for non-metals and metals, respectively.

14.3.43 Run query 09B1 (for nonmetals) and 09B2 (for metals) and note each on the record sheet in slot 09B1/2. These put R1 data into the AQS RP TEMPLATE. R1 data are available for both duplicate and collocated data (and uses a precision id of 2). Run query 09B3 and 09B4 to append replicate data for metals and nonmetals, respectively, to the AQS RP TEMPLATE for precision results for sites that do not run duplicate or collocated events. Note the number of records in the 09B3/4 slot on the record sheet.

14.3.44 Run query 09C and note the number of records on the record sheet in slot 09C. This puts R2 data (with a precision id of 3) in the AQS RP TEMPLATE. These precision data correspond to duplicate data only.

14.3.45 Run query 09D1 (for nonmetals) and 09D2 (for metals) and note each on the record sheet in slot 09D1/2. This puts R2 data into the AQS RP TEMPLATE. These precision data correspond to collocate data only (and uses a precision id of 2).

14.3.46 Total the number of records on the record sheet from slots 09A, B, C, and D and enter into the Total in RP slot.

14.3.47 Run query 10A. This creates a copy of SAMPDATA + INVALIDS (minus blanks) and pastes into SAMPDATA + INVALIDS + R1R2. Note the number of records in the 10A slot on the record sheet. Run queries 10B and 10C. These append the replicate data (for all duplicates and collocates) into the SAMPDATA + INVALIDS + R1R2 table. Note the number of records in the 10B and 10C slots on the record sheet. These should be similar (or the same) as the number of records in the 08A and 08B slots. Run query 10D to append the replicates run on Field Samples, as described in 14.3.43. Note the number of records in the 10D slot on the record sheet. Total the number of records from 10A, B, C, and D and note in the Total in UATMP slot on the record sheet.

14.3.48 Run queries 11A and 11B. These append blank data for non-detect and detect non-metals into the AQS RB TEMPLATE table. Note the number of records in the 11A/B slot on the record sheet.
Run queries 11C and 11D. These append blank data for non-detect and detect metals into the AQS RB TEMPLATE table. Note the number of records in the 11C/D slot on the record sheet. Total the number on the record sheet from slots 11A/B and 11C/D and note in the Total in RB slot. This number may match the number in the Total Excluded column on the record sheet (if the dataset didn't contain any precision data, for example).

14.3.49 If an analyte co-elutes with another, the lab practice is to report it as "ND". However, this is not entirely accurate. All co-eluters have been flagged with a BH in the Qualifier-3 column of the AQS TEMPLATE table. Run query 12A to see if there are any co-eluters in the AQS RD Template table. Because BH is not a flag, but is a null code, AQS will not accept this coding. Run query 12B to 1) move the BH from the Qualifier-3 column to the null code column, 2) remove the 0 representing the ND, and 3) remove the ND flag. Note the number of records updated in the 12 slot on the record sheet and verify that the number of records from 12A match 12B.

14.3.50 Run query 13 to check for any instances of multiple qualifier codes and resolve any results by manually splitting the codes into different Qualifier columns. Note the number of records in the 13 slot on the record sheet.

14.3.51 Run query 14A and 14B to do the same thing to the AQS RB Template table for co-eluters as step 14.3.50. Note the number of records updated in the 14 slot on the record sheet and verify that the number of records from 14A match 14B.

14.3.52 Run query 15a, 15b, and 15c to create data tables for acrolein, dichloromethane, and select GHGs as requested by the Project Manager. These tables need to be exported to Microsoft Excel and emailed to the Project Manager for a separate can study. Note the number of records on the record sheet for the 15 slot.

14.3.53 Run query 16 to add the LK flag to the Oklahoma sites' acetonitrile data. This was requested by the state as they recognize that their acetonitrile data periodically exhibits contamination issues.

14.4 Quality Assurance of the Raw Data in the "Macro" Database

14.4.1 To begin QA of the entire process, perform a manual QA of the data. Select data from the SAMP + INV + R1R2 table and write it down for a representative number of samples. Obtain the appropriate AQS codes for the method, parameter, etc., from the reference tables then use Queries QA 00a-c to visually verify that the coding is correct. These queries put in the site code, which makes it easier to find data by site. Pay special attention to any flags of interest that may have been needed for the dataset. Additionally, Query QA 00 can be used to specifically query blank sample data in the SAMP + INV table.

14.4.2 Skip Queries QA 01-02a/b and run Query QA 04 and 05 (01 through 02 are run by running Query 04-05). If the right two columns are fully populated for Query QA 04 and no results appear in 05, move to step 14.4.3. Otherwise, there is a discrepancy between the number of samples in the WORKLIST TEMPLATE and SAMP + INV + R1R2 tables that must be investigated before proceeding.

14.4.3 Run Queries QA 06-10. These queries make two tables, QA1 Dup Data and QA2 Worklist Data. QA2 is essentially a worklist that includes replicate sample information (WORKLIST TEMPLATE does not). Run Query QA 13 and 16, skipping the ones in between. These compare the number of samples in the new table QA2 Worklist Data to the number of samples in SAMP + INV + R1R2, including replicates. The number of samples for each site/method should match. If they do not, this must be reconciled, and Queries QA 16a-c may be used to try to locate any differences.

14.4.4 Run Query QA 17 and 18. These compare the number of samples in the WORKLIST TEMPLATE to the number of samples in AQS RD TEMPLATE for primary raw data only. The number for each site/method should match. If they do not, this must be reconciled, and Queries QA 16a-c may be used to locate any differences. Note Query QA 18 selects one pollutant from each method to "represent" a sample for each method.

14.4.5 Run Query QA 19 and 20a or 20b. These compare the number of precision samples in the new table, QA2 Worklist Data, to the number of precision samples in AQS RP TEMPLATE. Query 20a includes each individual replicate dates while Query QA 20b provides a count of dates. The number for each site/method should match. If they do not, this must be reconciled; Queries QA 16a-c may be used to locate any differences. Note: Because non-detect results are not included in precision data,

many hex chrome replicates do not go in AQS RP TEMPLATE. Use Query QA 16b to confirm.

14.4.6 Run through Queries QA 21a-e. These queries look for duplicate records in the SAMP + INV + R1R2, AQS RD TEMPLATE, AQS RP TEMPATE, Worklist Template, and AQS RB TEMPLATE tables. Any duplicates must be reconciled before moving forward.

14.4.7 Run Queries QA 22 and 23 or 23b. These compare the number of blank samples in the WORKLIST TEMPLATE to the number of blank samples in AQS RB TEMPLATE. Query QA23 has separate queries, the first pulls all blanks and dates, while query QA 23b performs a count per site and method, similar to step 14.4.5. The number for each site/method should match. If they do not, this must be reconciled, and Queries QA 16a-c may be used to try to locate any differences.

14.4.8 Run Query QA 24. This query verifies that all invalid samples have a null code. Results that appear in the results of the query must be given a null code.

14.4.9 Run Query QA 25a. This query confirms whether the correct MDLs are applied to the VOC and SNMOC samples, which is occasionally a problem at the beginning of a new calendar year. MDLs that are different due to dilutions do not need to be corrected. Query QA 25b can be used to review data for certain pollutants. If changes are needed, run Query QA 25c, which creates a table of all MDLs for these methods. Query QA 25e1 is used to check for samples that need updating and Query 25e2 is used to update the erroneous MDLs to the correct MDLs in the SAMP + INV + R1R2 table. Query QA 25f1 and 25f2 perform similar queries on the AQS RD TEMPLATE table. The counts of these two queries should be identical. Run Query 25g, which is identical to Query 25a, and confirms that there is only one MDL listed per pollutant.

14.4.10 Run Query QA 26a to look for any R2s associated with invalid D2s and valid D1s. These should not be loaded into AQS. Run Query QA 26b to confirm that they were not added to the AQS RP TEMPLATE. If coded properly, no records should appear.

14.4.11 Run Query QA 27 to look for instances of multiple AQS flags in a single Qualifier flag column. Separate any flags that appear by manually adding the second flag to a new Qualifier flag column (such as Qualifier-4, if empty).

14.4.12 Run Query QA 28 to check for results in scientific notation in the Sampdata + Inv + R1R2 table. Step 14.3.29 runs a similar query (Query 04D) on the Sampdata + Invalids table.

14.4.13 Ask another AQS coder to review the files to visually inspect that each file type matches the transaction type (RD, RP, etc). This reviewer shall compare at least one non-ND result for every method contained in each AQS file to the individual reports sent to the clients by the Project Manager to serve as an outside review, per Section 16.0. Both the primary AQS coder and the secondary reviewer should fill out and sign the AQC Quality Control Review Checklist (shown in Figure 24-2) in the AQS binder.

14.5 Creating and Loading Audit Results into AQS

14.5.1 Obtain ERG's quarterly NAAQS FEM Audit Results data in spreadsheet format from the QA Coordinator. Copy and paste the audit results into a new tab. Remove any excess spacing. Inset header information into new columns per the example shown in Figure 24-3.

14.5.2 In the Macro database, delete any data in the AQS RA Template table and the Audit Results table. Import the data from the new tab in the audit spreadsheet into the Audit Results table.

14.5.3 Run Query ZAudit 01 to append the results from the Audit Results table into the AQS RA Template table.

14.5.4 Copy the high-level results from the 1st Actual Value and 1st Indicated Value columns and paste next to the low-level results in the 2nd Actual Value and 2nd Indicated columns.

14.5.5 Run Query ZAudit 02 to delete the high-level result rows that are no longer needed.

14.5.6 Run Query ZAudit 03 to reduce the Indicated results down to three decimal places. Confirm that trailing zeros were not removed; add back in if needed.

14.5.7 Export to text and ask a reviewer to QA the text file.

14.5.8 Load the text files into AQS via the Exchange Network Services Center. Correct any load errors necessary. Update the Black Lab Notebook for AQS.

14.6 Exporting and Loading Raw Data into AQS

14.6.1 Compare and Compact Macro database.

14.6.2 Create a new "QX 20XX AQS" database.

14.6.3 Export the AQS TEMPLATE tables to this database from the Macro database.

14.6.4 Design Make Table and Delete queries to remove any data that doesn't need AQS upload. Design Make Table queries for any sites that want data sent to them directly. There's an electronic copy identifying such data at L:\Jaime\AQS\AQS Special Requests List.xls (Figure 24-4). These queries may be imported from other AQS databases and rerun.

14.6.5 After approval from the secondary review (per Step 14.4.13, export each RD, RP, RB, and/or RA TEMPLATE table to text by right-clicking on each table, selecting EXPORT; select TEXT as the file type and adjust file name as necessary; click on OK; select DELIMITED and then NEXT; choose OTHER and add the "|" character and chose NONE for the TEXT QUALIFIER then select NEXT; then hit FINISH. Review each file.

14.6.6 Load the text files into AQS via the Exchange Network Services Center. Correct any load errors until the data are approved in AQS and post the files for public access.

14.6.7 Update the Black Lab Notebook for AQS (notebook # 00329 as of this revision). Append the WORKLIST TEMPLATE and SAMP + INV + R1R2 tables from the MACRO database into the compiled database for the appropriate NMP reporting year.

15.0 CALCULATIONS

N/A

16.0 QUALITY CONTROL

Greater than 50 queries are located inside the Microsoft Access "Macro" database and are designed to provide a check of common errors (such as duplicate records) that may be encountered during the coding process. The AQS coder shall complete the AQS Quality Control Review Checklist upon completion of the procedures in Section 14.

A reviewer shall inspect the text files to be loaded into AQS by comparing the text files to the original reports sent to the clients by the Project Manager. The reviewer shall inspect one concentration above the detection limit per method per AQS text file and complete an AQS Quality Control Review Checklist after review, as specified in step 14.4.13.

A hardcopy of the AQS Quality Control Review Checklist shall be maintained per the instructions in Section 22.0.

17.0 PREVENTION

N/A

18.0 CORRECTIVE ACTION

N/A

19.0 WASTE MANAGEMENT

N/A

20.0 MAINTENANCE

N/A

21.0 SHORTHAND PROCEDURE

N/A

22.0 DOCUMENTATION AND DOCUMENT CONTROL

The latest version of the Macro and POC Table (for the reference tables) databases shall be posted on the L drive after every update. The current versions are posted at L:\Jaime\REFERENCES. The AQS Special Requests and AQS Record Sheet spreadsheets are also posted and kept up-to-date at L:\Jaime\AQS.

The black AQS laboratory notebook (# 00329) shall be updated whenever data is uploaded into AQS or changes are made to existing data (and associated metadata) in AQS. This notebook shall be maintained per the laboratory's record-keeping procedures.

Hard copies of the AQS Quality Control Review Checklist are maintained in the white AQS binder for a minimum of one year. Afterwards, these hard copies shall be archived per the laboratories archival procedures.

23.0 REFERENCES

Information about EPA's AQS database may be found at the following web address: http://www.epa.gov/ttn/airs/airsaqs/basic_info.htm

24.0 TABLES, DIAGRAMS, FLOWCHARTS, VALIDATION DATA

DATASET						
IMPORT QC						
IMPORT SAMP						
IMPORT WORK						
UPDATE UNITS						
UPDATE ANALYSIS						
01						
02						
03						
04						
TOTAL IN SAMP+						
05						
06						
07A						
07B						
TOTAL IN AQS						
TOTAL EXCLUDED						
08A						
08B						
09A D2/C2						
09B1/2 R1						
09B3/4 R1 for FS						
09C R2 for D2						
09D1/2 R2 for C2						
TOTAL IN RP						
10A						
10B						
10C						
10D						
TOTAL IN UATMP						
11A/B						
11C/D						
TOTAL IN RB						
12						
13						
14						
15						
16						

Figure 24-1. AQS Steps Record Sheet

Data set: _____

Date Coded: _____

AQS Coder: _____

Reviewer: _____

Method/Pollutant Group	Acceptance Criteria	Coder Initials and Date	Reviewer Initials and Date	Comments/Corrections Needed
IO-3.5 (Metals)	Results above detection limit in AQS text file must agree with result reported to the client.*			
Hexavalent Chromium	Results above detection limit in AQS text file must agree with result reported to the client.*			
TO-11A (Carbonyls)	Results above detection limit in AQS text file must agree with result reported to the client.*			
TO-15 (VOC)	Results above detection limit in AQS text file must agree with result reported to the client.*			
TO-13 (SVOC)	Results above detection limit in AQS text file must agree with result reported to the client.*			
SNMOC	Results above detection limit in AQS text file must agree with result reported to the client.*			
Other (Phenols, Methane, Audit Results, etc)	Results above detection limit in AQS text file must agree with result reported to the client.*			

N/A = not applicable to this load

*Reviewer checks 1 concentration per method per upload batch

Figure 24-2. AQS Quality Control Review Checklist

Audit Sample ID	Filter ID	Analyte	ERG Result	Assigned Value	%Recovery	%Diff	Audit	ERG ID	Date Analyzed	Analyst	Result#
RTI-2013-03-001	T2667962	Lead	2.837	2.963	95.8	-4.34	Q1 2014	3022208-10	3/11/2014	RM	1
RTI-2013-03-001	T2667953	Lead	2.703	2.963	91.2	-9.20	Q1 2014	3022208-11	3/11/2014	RM	2
RTI-2013-03-001	T2667955	Lead	2.875	2.963	97.0	-3.01	Q1 2014	3022208-12	3/11/2014	RM	3
RTI-2013-04-001	T2667982	Lead	8.801	8.994	97.8	-2.17	Q1 2014	3022208-22	3/11/2014	RM	1
RTI-2013-04-001	T2667994	Lead	8.894	8.994	98.9	-1.12	Q1 2014	3022208-23	3/11/2014	RM	2
RTI-2013-04-001	T2667996	Lead	8.691	8.994	96.6	-3.42	Q1 2014	3022208-24	3/11/2014	RM	3

Figure 24-3. Sample Lead Audit Header Information

Project	Site	Instructions	Deletion	Contacted or Sent	Separate Load or Performed
Nat'l Program	BTUT	Do not load metals			
Nat'l Program	DEMI	MI loads their data themselves. Export and email the coded text files for VOC, Carbs, SVOC to Debbie Sherrod at SHERRODD@michigan.gov			
Nat'l Program	UNVT	Wait for Robert to approve metals and PAHs data prior to loading, email when completed			
TSAT	MBNV	Code, but hold off on loading for now			
Nat'l Program	PRRI	Load their data then email Melinda Viera at Melinda.Viera@health.ri.gov			
Nat'l Program	BMCO	Waiting on site entry for BMCO; set aside for now			
Nat'l Program	WPFL	Del, Waiting for site to get QAPP completed prior to loading			
Nat'l Program	DECO/PVCO	Delete ERG's "collocated" data (POC E1)			
Nat'l Program	Jaime	Append to compiled database			
Nat'l Program	Julie	Send Acrolein&MeCl& GHG data			

Figure 24-4. AQS Special Requests List

UNCONTROLLED ELECTRONIC COPY EFFECTIVE 5-2015

ERG
EASTERN RESEARCH GROUP, INC.

CONFIDENTIAL
Standard Operating Procedure
Procedure Number: ERG-MOR-099
Revision Number: 2
Revision Date: May 4, 2015
Page: 1 of 14

ENGINEERING AND SCIENCE DIVISION

TITLE: SOP for use of the Laboratory Information Management System and Data Progression and Data Reporting	EFFECTIVE DATE: MAY - 6 2015
REFERENCES: ERG-MOR-010, ERG-MOR-022 , ERG-MOR-079, Element Datasystem User's Manual, Promium, LLC., Promium website, www.promium.com	
SATELLITE FILES: Chromatography, Inorganic, Prep, VOC, Mass Spec, Shipping/Receiving and Can Cleaning	
REVISIONS: Added statement about adding QC samples; Updated the sequence naming convention; Added more detail on periodic updates; Added a data qualifier to the qualifier table	
WRITER/EDITOR: NAME/DATE Laura Krnavek 5/5/2015	PROJECT MANAGER/TECHNICAL DIRECTOR: NAME/DATE Julie A. Swift 5/6/15
QUALITY ASSURANCE MANAGER: NAME/DATE Donna Tedder 5/5/15	NEXT SCHEDULED REVIEW: 1/31/17

1.0 IDENTIFICATION AND PURPOSE

The purpose of this document is to describe the procedures for using the Laboratory Information Management System (LIMS) to record all sample data, information pertaining to each sample, and data reporting. The LIMS allows historical reconstruction of laboratory activities since the history of each sample is readily available. The LIMS also aids in the verification that the data meets the project needs with pre-set control limits, which allows for straightforward qualification of potential quality impacts in the report.

2.0 MATRIX OR MATRICES

N/A

3.0 METHOD DETECTION LIMIT

The LIMS system DataTool has a template called *MDL Study* that can be used to determine MDLs based on the same method used in ERG-MOR-010. Independent of how the MDLs are determined, the annual MDLs must be entered into LIMS from the QA Admin>Analysis Analyte screen. The entered MDLs are reviewed by QA staff prior to reporting.

The MDL in LIMS will be multiplied by the dilution factor which is entered into the LIMS from the instrument file. If a sample is diluted, the MDL is multiplied by the dilution factor to obtain the actual MDL of the sample, since the concentration of the minimum detectable amount will become higher with each dilution.

4.0 SCOPE AND APPLICATION

All samples at the ERG laboratory are logged, tracked, batched, data uploaded, and reported using the LIMS. Samples received at the laboratory are logged into the LIMS following the procedures in the SOP for Sample Login to the Laboratory Information Management System, ERG-MOR-079. Not every step described in this SOP is applicable to every analytical method.

User access to the LIMS is graduated. People are granted permission only to the tasks they need.

5.0 METHOD SUMMARY

Samples at the ERG laboratory are logged, tracked, batched, data uploaded, and reported using the LIMS. The system records all sample data, information pertaining to each sample, and allows for data reporting. The LIMS also aids in the verification that the data meets the project needs with pre-set control limits.

6.0 DEFINITIONS

COA	Certificate of Analysis
ERG	Eastern Research Group
ID	Identification
LIMS	Laboratory Information Management System
MDL	Minimum Detection Limit
NMP	National Monitoring Program
PM	Project Manager
QA	Quality Assurance
QAPP	Quality Assurance Project Plan

QC	Quality Control
RPD	Relative Percent Difference
SOP	Standard Operating Procedure
TL	Task Lead

7.0 INTERFERENCES

N/A

8.0 SAFETY

N/A

9.0 EQUIPMENT

Promium® Element Laboratory Management Systems (LIMS) for commercial and public sector soil, water, air testing laboratories.

10.0 MATERIALS

N/A

11.0 CHEMICALS, REAGENTS, AND STANDARDS

Information for prepared or commercially-bought standards can be entered and tracked by a LIMS-generated standard ID number in the **Standards** section of the **Laboratory** menu. Vendor certificates of analysis (COA) can be scanned, imported, and linked with the appropriate standards (SOP ERG-MOR-022).

12.0 COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

Collection, preservation, shipment, and storage information for samples received at the laboratory are logged into the LIMS following the procedures in the SOP for Sample Login to the Laboratory Information Management System, ERG-MOR-079.

13.0 CALIBRATION AND STANDARDIZATION

Calibrations can be entered into **Calibration** under the **Laboratory** menu; however, the individual chromatographic software is considered to be more useful than the LIMS calibration section at this time.

14.0 PROCEDURE

Not every step described in this section is applicable to every analysis. With periodic updates from Promium®, this section may not address every possible variation of the LIMS menus. Figure 14-1 shows the general LIMS sample data progression and statuses.

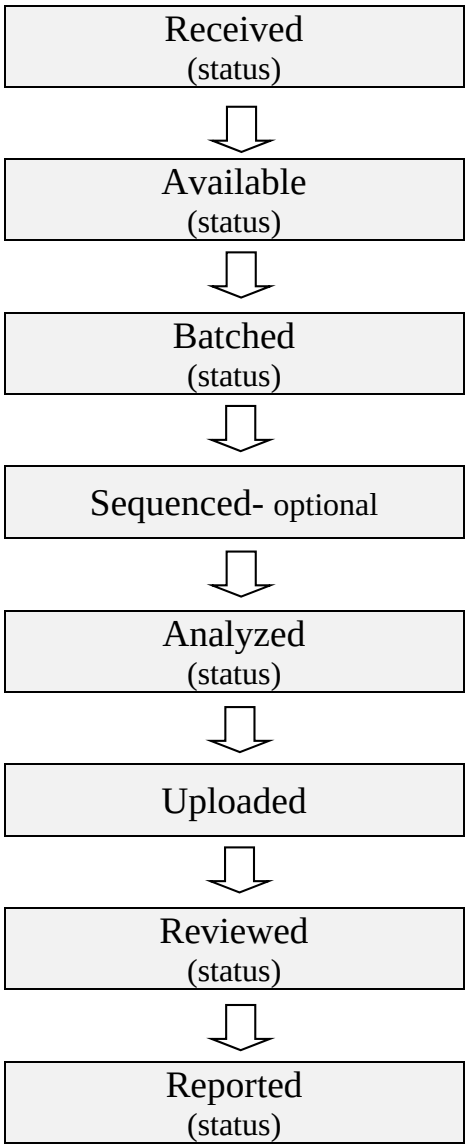

Figure 14-1. LIMS Server and Status Path for Analyses

14.1 LIMS Batch Procedure

14.1.1 Log into Element.

14.1.2 If more than one batch of samples is "Available" use the **Query Analysis Status** tool under the **Laboratory** menu. Query for available samples under the desired analysis, start by clicking **Order Results By** choosing **Sampled** and **LabNumber**. Select the desired number of samples choosing the oldest ones first unless otherwise specified.

14.1.3 From the **Laboratory** menu, select **Batch**. With the desired department selected from the drop-down menu at the left of the screen, click **Add** at the bottom, left-hand corner. A blank batch sheet will appear. (You may choose to press **Copy** instead of **Add**. Use caution here, as the analysis, surrogates, and comments from the copied batch will be included in the new batch and this information may need to be changed.)

14.1.4 Using the drop down menus, fill in or confirm the **Department**, **Preparation Method**, **Batch Matrix**, and any **Surrogates** used. Fill in the **Comments** field to include any additional information required.

14.1.5 Select the appropriate analysis from the **Available** analysis box and press the right-facing arrow button. Be sure to select the analysis denoting the correct year when there are multiple choices, such as "TO-15 yyyy", where yyyy is the year.

14.1.6 Press the **Save** button. The new LIMS-created batch number will be visible in the box to the left of the screen. The numbering scheme BYMDDNN is as follows, the first field is the letter "B". The second field is a number for the year, starting with "1" for 2011 and so on. The third field is a letter for the month of the year starting with "A" for January, the next two fields (fourth and fifth) are for day of the month, and lastly the last two fields (sixth and seventh) are for the batch # on that day.

14.1.7 Select **Bench Sheet** at the bottom of the screen or from the **Laboratory** menu. To make modifications begin by clicking the **Edit** button at bottom of the window.

14.1.8 At the top of the screen, press **Add** and select **Sample by Container**. Select the samples from the list that you would like to include in the batch. Take care to keep all primary and field blank sample pairs as well as duplicate pairs together in the same batch. **Note:** For some analyses, a maximum of twenty samples may be selected for any given batch. If more than 20 samples are selected, additional batch QC must also be added to the bench sheet and extracted to meet the method requirements.

14.1.9 Right click on a sample to bring up a menu, and select **Initial**. Fill in the value for the total collection volume. This will be listed on the sample's corresponding chain of custody, or calculated from the given flow and time values. Repeat this for each sample in the batch. Field blank samples are assigned the same volume as the primary sample collected on the same day.

14.1.10 From the right click menu choose **Comments** and fill in the site code for each sample as it appears on the bench sheet including any C1, C2, or FB designations.

Note: For some analyses two spaces are required before entering comments for formatting reasons.

14.1.11 Each batch will need a number of QC samples included. The exact requirements for batch QC will vary between analyses, but there are three common types that will be used. Each type of QC can be added to a batch using the **Add** command, but the most commonly used QC will be included in the batch automatically after samples are included for most analyses. QC samples will be named by the Batch ID followed by an identifying suffix with a numeral(x).

i. Blank Samples: Each batch will contain at least one Blank Sample (-BLKx). Be sure to confirm the presence of any surrogates used in the analysis using the right click menu.

ii. Spiked Samples: Various types of spiked samples are used in different analyses including Laboratory Control Spikes (-BSx), Matrix Spikes (-MSx), Matrix Spike Duplicates (-MSDx), and Post Spikes (-PSx). As with blank samples confirm any surrogates with the right click menu, but also include the **Spike ID**, **Type** and **Amount** for each spiking solution used.

iii. Duplicate Samples: Include one Duplicate (-DUPx) QC sample for each duplicate or collocated sample in the batch. Using the right click menu assign a **Source** sample to each Duplicate, and change the **Initial** volume to match that of the source sample.

14.1.12 Once all field sample, QC samples have been added and the required information has been updated, print any necessary copies of the bench sheet. If an extraction log is kept, store a copy of the bench sheet in it. A copy of the bench sheet is also kept with the chain of custodies for that batch.

14.2 LIMS Sequence Procedure

14.2.1 From the **Laboratory** menu, select **Sequence**.

14.2.2 Click **Add** at the top right, window labeled **Template ID** will appear. Select the template for the desired method or choose cancel to continue without a template.

14.2.3 Click the pull-down menu for **Source Batch** and select a batch for analysis. Next click **Add** and first select **Batch QC Sample**. This shows a list of all blanks, spikes, and duplicates. Select all the QC samples that will be run in the sequence and double click to add them to the sequence. Repeat this choosing **Add** again but selecting **Batch Sample** to add all desired LIMS samples. It is necessary to keep all primary/field blank pairs and duplicate sample pairs with all corresponding QC samples together in the same sequence.

14.2.4 In addition to Batch QC, Sequence QC must also be included. If using a template the most common sequence QC will be added automatically but any QC sample can be manually added from the Add command.

14.2.5 Once all samples and QC are placed in a sequence they must be placed in the desired run order as per method requirements, typically beginning with QC checks and then sets of samples broken up by additional running QC, and ending with any final QC.

14.2.6 After establishing a desired run order right click on any QC or calibration standards and choose **Standard ID,** then double click on the LIMS standard used in the selected sample. If an internal standard is used select all samples in the sequence and select the correct **Internal Standard ID** using the right click menu. Any sample

prepared from an existing sample (such as PS, BS, or SRD) also needs the **Source Sample** defined using the right click menu.

14.2.7 Once completed, review the sequence for any omissions or errors, make necessary corrections, and click the green **Save** button.

14.2.8 Print a copy of the sequence. File the sequence print out in the appropriate location. The numbering scheme YYMMXXX is as follows, the first field is "YY" for the last two digits of the year. The second field is "MM" for the month number. The third field is "XXX" for the number of sequence in the order they are created starting with 001.

14.3 LIMS Data Upload Procedure

14.3.1 The sample data generated on the instruments is reduced by the analyst via instrument software. When data reduction is complete for a batch or sequence of samples, the analyst will transfer the data from the instrument computer to network server 'bart' (Y:). This server is backed-up daily.

14.3.2 To begin data upload onto LIMS, open **Element**. Go to the **Laboratory** menu and click on **Data Entry/Review.** First choose the department of the analysis being uploaded from the drop down menu on the left, and then in the top left corner choose **Sequence** or **Batch** depending on the analysis. Select the desired sequence or batch from the list and click on **Create** in the **Data Entry** tab in the top right corner. Next click **DataTool**. In DataTool, select the appropriate **File Type** at the top right and choose y: [\\bart\airtoxics] under **Drives**. In the box below choose the folder containing the desired instrument data files.

14.3.5 All of the data files for that sequence will appear in the lower right-hand box. Double-click on the appropriate data file/s or click **Auto Select** for each file that needs to be included in the sequence.

Note: Only undiluted samples and sequence QC should be included here.

14.3.6 Press **Done** when all sample and QC files have been selected to return to the main window. Click **Merge Files** at the bottom of the window.

Data Tool will merge the files and show the data in the Data Transfer window.

Note: Review the content of the top windows in the Data Transfer window for red text indicating unmatched data. If there is any red text, the DataTool cross table requires editing. If possible, match the unmatched data and proceed. If needed, seek the advice of the TL or the LIMS administrator to correct this.

14.3.7 Press **Save** and save this new spreadsheet with any filename.

14.3.8 In Element, at the **Data Entry/Review** window, the merged data will automatically be imported into the data entry table. Press **Save** to save the data to Element and then **Query** in the **Data Review** box. Element will perform all necessary calculations at this point.

14.3.9 In the **Data Entry/Review** window, samples and QC can be reviewed for pass/fails. Any data that does not pass its assigned criteria will have red text. Use the *Summary of Quality Control Procedures* tables in the individual analytical SOPs or in Section 4.0 of the NMP QAPP to decide if criteria have been met and the appropriate corrective action (flagging data, voiding sample, etc.) have been applied. The possible data qualifiers are listed in Table 24-1. Change the Status to "Reviewed".

14.4 After data is uploaded into LIMS, the data package is reviewed by TL using the QA review checklist from the individual analytical SOPs. After which, 10% is reviewed by the QA staff using the same checklist. After the QA staff review, the data is ready for reporting.

14.5 Data reporting is to be done by the Project Manager (PM). The QA staff will notify the PM when their review is complete. Samples marked "Reviewed" are available for final reporting.

14.6 LIMS Data Reporting Procedure

14.6.1 Go to the **Project Management** menu and click on **Reports.**

14.6.2 First choose the client and project of the data being reported from the drop down menu on the top.

14.6.3 Choose a standard **Format** for the report and electronic data deliverable (EDD) or choose a **Custom** format for the report or the EDD.

14.6.4 Choose a **Destination** for the EDD and a printer for the report (PDF995).

14.6.5 On the left side of the screen, select the desired work orders.

14.6.6 Check the appropriate analysis box in the middle screen.

14.6.7 Next click **Modified Final**, if a note to mark the work order as "Closed" comes up, press **No.**

14.6.8 Now choose the samples (and QC, if needed) to be reported in the window that opens up and click **Continue**. The PDF report opens up. Save all but the last page of the report to the desired destination. The EDD, which is an Excel file, is generated at the same time and located in the destination chosen in a previous step.

14.6.9 Invalid samples are listed in the report letter to the client or as a separate excel table.

The reporting formats are templates and have been set up with certain required elements. These reports contain the data qualifiers, footnotes, MDLs, and final units assigned to the reported data. Data reporting is typically done monthly, with a month's worth of samples reported for an individual site. A comprehensive annual NMP data report is prepared each year and includes data statistics, data characterization, and program-wide quality discussion.

15.0 CALCULATIONS

When necessary, standardized procedures for transforming raw data from measured units to final concentrations use calculations listed in the individual analytical SOPs. These calculations were set up in the LIMS and verified annually by QA staff. The TO-15 and SNMOC initial and final units are the same, ppbv and ppbC, respectively. The TO-11A initial units are µg/mL and final units are ppbv. TO-13A has initial units of ng/µL and final units of ng/m^3. The metals analysis has initial units of ng/L and final units of ng/m^3. Hexavalent chromium has initial units of ng/mL and final units of ng/m^3.

Promium® provides a LIMS worksheet to validate algorithms that are utilized in the LIMS system to perform calculations, such the final results from the initial results, standard percent recovery, and RPD between replicate analyses. In the **Print** window,

press **Analysis Info** and choose the template "axi-validation". These calculations are verified with hand calculations biannually by QA staff and documented.

16.0 QUALITY CONTROL

16.1 Each section in LIMS has an "Audit" button. Clicking this button will open a new window that displays the changes made in the applicable section. Element keeps track of the who, when, and what during the editing of information in the LIMS. Right-clicking the mouse in the audit window allows the information to be sorted in various ways and also allows the audit information to be exported or printed.

16.2 Data quality criteria are addressed in the individual analytical SOPs. Data quality and data reduction is reviewed by the TL and 10% of the data is subsequently reviewed by QA staff.

16.3 Data reports are reviewed by the PM prior to being sent to the client.

Note: If requested by a client, uncertainty values can be entered into the Analysis/Matrix screen.

17.0 PREVENTION

N/A

18.0 CORRECTIVE ACTION

Promium® updates are available for the LIMS software automatically and occur often. See the LIMS administrator for any LIMS issues.

19.0 WASTE MANAGEMENT

N/A

20.0 MAINTENANCE

The LIMS system is under an annual service contract with Promium® maintained by the Office Manager. The LIMS administrator is responsible for supporting the periodic updates initiated by Promium®. The Promium updates are first uploaded to an updates folder, then into the LIMS. The updates are uploaded after the monthly reports go out to give the administrator time to fix any potential issues found during testing. The current version number is located at the top of the dashboard.

21.0 SHORTHAND PROCEDURE

N/A

22.0 DOCUMENTATION AND DOCUMENT CONTROL

N/A

23.0 REFERENCES

Element Datasystem User's Manual, Promium®, LLC
Promium website, www.promium.com; also available on the Help menu in the LIMS system.

24.0 TABLES, DIAGRAM, FLOWCHARTS, VALIDATION DATA

Table 24-1. List of LIMS Data Qualifiers

Qualifier	Description
9	Negative value detected – Zero reported
A-01	(Custom Value)
B	Analyte is found in the associated blank as well as in the sample. (CLP B-flag)
B-01	Value is laboratory reagent-blank subtracted.
B-02	Method Blank value exceeds MDL due to background from filter media. Acceptable LRB analysis data indicates the batch was not contaminated.
B-04	Value is laboratory method blank subtracted.
BS-01	Recovery is biased due to high humidity.
C-02	This result was determined using the calibration from the confirmation column.
CE	Not reportable due to a co-eluting compound.
D	This result obtained by dilution.
D-01	This result obtained by diluting and reanalyzing the sample.
D-F	Duplicate exceeds DQO criteria.
D-R	Duplicate RPD greater than 20%; however, parent sample and duplicate values are less than 10 times the MDL.
E	The concentration indicated for this analyte is an estimated value above the calibration range of the instrument. This value is considered an estimate. (CLP E-flag)
GC-BS	Compound exceeds Blank Spike criteria.
I-01	Due to matrix interference, the sample cannot be accurately quantified. The reported result is qualitative.
I-02	This result is outside of the EPA recommended holding time.

Table 24-1. List of LIMS Data Qualifiers (continued)

Qualifier	Description
I-03	Polyatomic interference in ICS standard. Interferent not present in significant levels in samples.
ICS-01	Interference check exceeds criteria.
INT	Not reportable due to interference.
J	Detected but below the Reporting Limit; therefore, result is an estimated concentration. (CLP J-Flag)
L-01	Analyte exceeds HSV criteria. Sample concentrations below the CCV value are accepted based on acceptable CCV recovery.
LK	Analyte identified; Reported value may be biased high.
LL	Analyte identified; Reported value may be biased low.
MS-01	Non-homogeneous matrix interference. Serial Dilution and /or Post spike verifies parent sample value.
NC	Analyte could not be confirmed using the confirmation column.
O-01	This compound is a common laboratory contaminant.
O-02	Due to matrix interference, the sample cannot be accurately quantitated. The reported result is qualitative.
O-03	The concentration reported is an estimated value above the linear quantitation range. Dilution and reanalysis is being performed and an amended report will follow.
O-04	This sample was analyzed outside the EPA recommended holding time.
O-05	This sample was extracted outside the EPA recommended holding time.
PRELIM	Preliminary result. Revised report to follow.
PS-01	Post spike exceeds DQO criteria.
PS-02	Post spike was outside of the control limits due to matrix interference.
QB-01	Compound fails method blank criteria
QB-02	The method blank contains analyte at a concentration above the MRL; however, concentration is less than 10% of the sample results, which is negligible according to method criteria.
QM-01	The spike recovery for this QC sample is outside of established control limits due to sample matrix interference.
QM-05	The spike recovery was outside acceptable limits for the MS and /or MSD due to matrix interference. The LCS and /or LCSD were within acceptable limits showing that the laboratory is in control and the data is acceptable.
QM-06	Due to noted non-homogeneity of the QC sample matrix, the MS/MSD did not provide reliable results for accuracy and precision. Sample results for the QC batch were accepted based on LCS/LCSD percent recoveries and RPD values.
QM-07	The spike recovery was outside acceptance limits for the MS and/or MSD. The batch was accepted based on acceptable LCS recovery.

Table 24-1. List of LIMS Data Qualifiers (continued)

Qualifier	Description
QM-4X	The MS/MSD recovery exceeds criteria because the parent sample concentration is greater than 4 times the spike concentration. Sample results for the QC batch were accepted based on acceptable BS/BSD recoveries.
R-F	Replicate exceeds DQO criteria.
S-01	The surrogate recovery for this sample is not available due to sample dilution required from high analyte concentration and/or matrix interference.
S-02	The surrogate recovery for this sample cannot be accurately quantified due to interference from coeluting organic compounds present in the sample extract.
S-04	The surrogate recovery for this sample is outside of established control limits due to a sample matrix effect.
S-06	The recovery of this surrogate is outside control limits due to sample dilution required from high analyte concentration and/or matrix interference.
S-DUP	Duplicate analysis confirmed surrogate failure due to matrix effects.
S-FS	These surrogates are not spiked in the method blank(s) or blank spike(s).
S-GC	Surrogate recovery outside of control limits. The data was accepted based on valid recovery of the remaining surrogate.
S-HI	High surrogate recovery was confirmed as a matrix effect by a second analysis.
S-LIM	Surrogate recoveries outside method QC limits. Site matrix effects verified by 10% duplicate analysis (including sample duplicate and MS/MSD analysis).
S-LOW	Low surrogate recovery confirmed as a matrix effect by a second analysis.
S-MS	Surrogate recovery outside of acceptance window confirmed as matrix effect by analysis of MS/MSD on this sample.
S-QC	The Surrogate result exceeded the QC limits; however, the percent recovery was acceptable. Sample results for the QC batch were accepted based on percent recoveries of the QC data.
SRD-01	Serial dilution exceeds the control limits.
S-Rec	Sample received by laboratory outside of initial extraction hold time – 14 days.
U	Under detection limit.
Z-01	(Custom Value)

Appendix B

DRI Standard Operating Procedures

DRI STANDARD OPERATING PROCEDURE

X-Ray Fluorescence (XRF) Analysis of Aerosol Filter Samples (PANalytical Epsilon 5)

DRI SOP #2-209.8
Revised October 3, 2014

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1. GENERAL DISCUSSION

1.1 Purpose of procedure

This standard operating procedure is intended to:

- provide a very basic understanding of the principles of X-Ray Fluorescence (XRF) analysis;
- describe a method for the determination of elemental concentrations from ambient and source aerosol filter samples using the PANanlytical Epsilon 5 XRF analyzer;
- detail the concerns and procedures which will ensure a state-of-the-art XRF analysis measurement process.

This procedure will be followed by all analysts at the Environmental Analysis Facility of the Division of Atmospheric Sciences of the Desert Research Institute.

1.2 Measurement principle

Analysis of aerosol filter samples using the PANalytical Epsilon 5 XRF analyzer is based on energy dispersive x-ray fluorescence of elemental components in a thin film sample. The emissions of x-ray photons from the sample are integrated over time and yield quantitative measurements of elements ranging from aluminum (Al) through uranium (U) and semi-quantitative measurements of sodium (Na) and magnesium (Mg). A spectrum of X-ray counts versus photon energy is acquired and displayed during analysis, with individual peak energies corresponding to each element and peak areas corresponding to elemental concentrations. The advantages of XRF analysis include high sensitivity for a number of elements, the ability to analyze small quantities of sample, and the non-destructive nature of the analysis. In addition, because x-ray fluorescence depends on the quantum absorption and emission of photons at the M, L, and K orbitals, the technique is insensitive to the chemical state of the elements. Disadvantages include the subjection of the sample to a vacuum, resulting in loss of some volatile species such as hydrocarbons, ammonia, nitrate, chlorine, and bromine.

The source of x-rays in the PANalytical Epsilon 5 analyzer is a side window dual anode x-ray tube with both Scandium (Sc) and Tungsten (W) anodes. X-rays are focused on one of 11 secondary targets which in turn emit polarized x-rays used to excite a sample. X-rays from a secondary target are absorbed by the sample, exciting electrons to high level orbitals. As the electrons return to their ground state, photons are emitted which are characteristic of the quantum level jumps made by the electron; the energy of the emitted photons are, therefore, characteristic of the elements contained in the sample. The fluoresced photons are detected in a solid state Germanium X-ray detector. Each photon that enters the detector generates an electrical charge whose magnitude is proportional to the photon's energy. The electrical signals from the detector are sorted into energy channels, counted, and displayed. A sample spectrum consists of characteristic peaks superimposed on a background caused by the scatter of x-rays from the tube into the detector. Spectra are collected for a specified length of time and stored on disk for later processing.

DRI uses seven different analysis conditions during a single analysis run to maximize sensitivity to the full range of elements reported. Each of the analysis conditions, which correspond to different

secondary targets, x-ray tube voltage and current, and energy detection range, is designed for a specific group of elements.

1.3 Measurement interferences and their minimization

The XRF is subject to measurement uncertainties from:

- Too much deposit material. Because DRI's XRF analysis and data processing programs for aerosol samples are designed specifically for thin films, x-ray spectra are subject to distortion if unusually heavy deposits are analyzed. This is due to internal absorption of both incident and emitted x-rays within the samples. Optimum loading is ~150 μg/cm^2 (1 mg/filter for 37 mm filters and 2 mg/filter for 47 mm filters). Adjustments in filter deposit area and sampling time and flow should be made to insure deposits are within this range.

- Too little deposit material. At low concentrations counting statistics and signal noise will dominate the calculations of elemental concentrations. Adjustments in filter deposit area and sampling time and flow should be made to insure deposits are at least 15 μg/cm^2.

- Inhomogeneous deposits. The x-ray beam is focused on an area ~ 9 x 16 mm in the center of the filter. The results are extrapolated to the entire deposit area of the filter during data processing. Therefore, the center of the filter must be representative of the entire deposit. This effect is minimized by using the sample spinner during analysis which rotates the sample cup at 12 rpm.

- Large particles. Absorption of both incident and emitted x-rays occurs in the presence of large particles. This particularly affects the light elements (Na through sulfur [S]). In addition, large particles may be enriched in certain elements and may bias results in the same matter as inhomogeneous deposits.

- Filter thickness. Increased x-ray scattering is caused by increased filter thickness, increasing in turn the spectral background and causing problems for accurate background subtraction and quantification of elements at low concentrations. Additional uncertainties are introduced if filter thickness varies considerably within a lot, again making accurate background subtractions difficult. The solutions to these problems include using filters from a reputable manufacturer (e.g., Pall, Whatman, MTL), including blanks from each manufacturing lot in the formulation of background/blank spectra, and inspecting each filter over a light table before use.

- Background contamination. While small levels of contaminations may be corrected during blank subtraction, such contaminations rarely are at consistent levels, and uncertainties can be relatively high, particularly if the contaminants correspond to elements of interest. Using filters from reputable manufacturers and performing acceptance tests on all manufacturing lots will reduce the affect of contaminations.

- Filter types. The DRI XRF analysis and data processing procedures are primarily oriented toward Teflon membrane filters, which are analytically clean, are thin to reduce scattering, and have known pore size and particle collection efficiencies. Historically, glass or quartz fiber filters have been used for Total Suspended Particulate (TSP) or PM_{10} monitoring. These filters may be analyzed but, due to their composition, render analysis results for light elements

meaningless. Since particles are trapped within the filter matrix of quartz or glass-fiber filters, x-ray absorption within the filter fibers adds additional uncertainty. In addition, blank contamination levels and variations among manufacturers and manufacturing lots can be orders of magnitude higher for glass or quartz-fiber filters than for Teflon. Recommended filters are Teflon membrane filters from Pall or Whatman.

- Damaged filters. Uncertainties in results from analyzing the damaged portion of a filter are obvious. However, the x-ray signal is heavily dependent upon distance of the filter from the x-ray tube and from the detector. If filter is damaged so as to cause sagging or puckering which changes these distances, analysis results will also be affected.

1.4 Ranges and typical values of measurements obtained by this procedure

A wide range of aerosol concentrations can be measured with this method, provided adjustments to deposit area and sampling flow and time are made to insure optimum loading on the filters (~150 $\mu g/cm^2$). Filter loadings between 15 and 1000 $\mu g/cm^2$ may be used, but results for heavily loaded samples may require manual corrections during data processing and results for lightly loaded filters will have concentrations below detection limits for many elements.

1.5 Typical lower quantifiable limits

The lower quantifiable limits (LQLs) of DRI's XRF analysis depend on a number of factors, including type of filter media, manufacturer of filter media, consistency of filters with respect to thickness and background contaminations, analysis counting time, analysis conditions, and element. Typical three sigma minimum detection limits for Teflon membrane filters are presented in Table 1-1.

Precision is determined largely by the homogeneity of the filter deposit, rather than the analyzer itself. DRI specifications call for ±10% on each element or within ±3 times the analytical uncertainties, whichever is larger. The analytical uncertainties are propagated from the counting statistics of the elemental peaks and background spectra.

1.6 Responsibilities of personnel for carrying out portions of this procedure

All analysts in the laboratory should read and understand the entire standard operating procedure prior to performing XRF analysis, which includes system operation, actual analysis, data processing, and immediate review of the QA data as it is produced to correct system problems.

It is the responsibility of the XRF supervisor to ensure the XRF analyses procedures are properly followed, to examine and document all replicate, QA standards, calibration results, and acceptance test data, to designate samples for reanalysis, to arrange for maintenance and repair, to maintain the supplies and gases necessary to insure uninterrupted analysis, and to deliver the analysis results to the project manager within the specified time period.

Table 1-1. MDLs.

	MDL (µg/cm2)		MDL (µg/cm2)		MDL (µg/cm2)
Na	0.204	Cu	0.002	Sb	0.023
Mg	0.176	Zn	0.004	Cs	0.036
Al	0.038	Ga	0.006	Ba	0.042
Si	0.009	As	0.004	La	0.048
P	0.006	Se	0.002	Ce	0.090
S	0.001	Br	0.002	Sm	0.087
Cl	0.003	Rb	0.001	Eu	0.071
K	0.006	Sr	0.002	Tb	0.071
Ca	0.014	Y	0.002	Hf	0.032
Sc	0.057	Zr	0.006	Ta	0.018
Ti	0.007	Nb	0.005	W	0.013
V	0.001	Mo	0.003	Ir	0.006
Cr	0.002	Pd	0.012	Au	0.007
Mn	0.003	Ag	0.006	Hg	0.006
Fe	0.002	Cd	0.005	Tl	0.004
Co	0.002	In	0.009	Pb	0.004
Ni	0.002	Sn	0.013	U	0.005

1.7 Definitions

MLK Lines: A series of x-ray lines corresponding to electron transitions to the M, L. and K orbitals.

Secondary Target: A metal foil upon which the primary x-rays from the x-ray tube are focused. Emission of "secondary" x-rays are in turn focused on the sample to be analyzed.

1.8 Related procedures

- SOP's related to XRF analysis activities which should be reviewed in conjunction with this document are:
- DRI SOP #6-014r1 Laboratory Ethics.
- DRI SOP #6-015r1 Demonstration of Capability.
- Any SOP's dealing with filter handling and shipping in conjunction with the specific sampling method used.
- DRI SOP #6-013r0 Creation, Revision, Distribution, and Archiving of Standard Operating Procedures.

The programming, maintenance, and troubleshooting manuals for the PANalytical Epsilon 5 system.

2. APPARATUS, INSTRUMENTATION, REAGENTS, AND FORMS

2.1 Apparatus and instrumentation

The PANalytical Epsilon 5 EDXRF analyzer contains two main components, the XRF cabinet (Figure 2-1) and the computer workstation. The XRF cabinet includes an integrated robotic sample changer and a sample chamber that can hold up to 51 samples. The large sample chamber allows almost continuous analysis of samples by allowing operators to load and unload samples in the queue while an analysis is taking place in the isolated analysis chamber.

Figure 2-1. PANalytical Epsilon 5 x-ray cabinet.

2.1.1 Characterization

The PANalytical EDXRF analyzer running under DRI's analysis protocol uses seven different excitation conditions to maximize sensitivity to select groups of elements (see Table 2-1). Each sample is placed in the analysis chamber and analyzed under a vacuum.

Table 2-1. Summary of Epsilon 5 analysis conditions.

Z	Element	Condition	Analysis time	Line	ROI (LL) Kev	ROI (UL) Kev	Secondary target	kV	mA
11	Na	@CaF_2	400	Ka	0.983	1.098	CaF_2	25	24
12	Mg	@CaF_2	400	Ka	1.195	1.313	CaF_2	25	24
13	Al	@CaF_2	400	Ka	1.426	1.547	CaF_2	25	24
14	Si	@CaF_2	400	Ka	1.677	1.802	CaF_2	25	24
15	P	@CaF_2	400	Ka	1.945	2.075	CaF_2	25	24
16	S	@CaF_2	400	Ka	2.242	2.376	CaF_2	25	24
17	Cl	@CaF_2	400	Ka	2.552	2.690	CaF_2	25	24
19	K	@CaF_2	400	Ka	3.239	3.386	CaF_2	25	24
20	Ca	@Fe	300	Ka	3.614	3.767	Fe	40	15
21	Sc	@Fe	300	Ka	4.011	4.170	Fe	40	15
22	Ti	@Fe	300	Ka	4.415	4.603	Fe	40	15
23	V	@Fe	300	Ka	4.853	5.046	Fe	40	15
24	Cr	@Fe	300	Ka	5.310	5.510	Fe	40	15
25	Mn	@Ge	300	Ka	5.792	5.998	Ge	75	8
26	Fe	@Ge	300	Ka	6.292	6.505	Ge	75	8
27	Co	@Ge	300	Ka	6.814	7.033	Ge	75	8
28	Ni	@Ge	300	Ka	7.358	7.585	Ge	75	8
29	Cu	@Ge	300	Ka	7.920	8.153	Ge	75	8
30	Zn	@Ge	300	Ka	8.504	8.747	Ge	75	8
31	Ga	@Mo	300	Ka	9.112	9.363	Mo	100	6
33	As	@Mo	300	Ka	10.390	10.661	Mo	100	6
34	Se	@Mo	300	Ka	11.064	11.344	Mo	100	6
35	Br	@Mo	300	Ka	11.755	12.047	Mo	100	6
37	Rb	@Mo	300	Ka	13.208	13.525	Mo	100	6
38	Sr	@Mo	300	Ka	13.967	14.296	Mo	100	6
39	Y	@Mo	300	Ka	14.748	15.092	Mo	100	6
40	Zr	@Ag	300	Ka	15.554	15.912	Ag	100	6
41	Nb	@Ag	300	Ka	16.382	16.755	Ag	100	6
42	Mo	@Ag	300	Ka	17.233	17.623	Ag	100	6
46	Pd	@CeO_2	300	Ka	20.866	21.331	CeO_2	100	6
47	Ag	@CeO_2	300	Ka	21.833	22.320	CeO_2	100	6
48	Cd	@CeO_2	300	Ka	22.824	23.333	CeO_2	100	6
49	In	@CeO_2	300	Ka	23.839	24.373	CeO_2	100	6
50	Sn	@CeO_2	300	Ka	24.878	25.437	CeO_2	100	6
51	Sb	@CeO_2	300	Ka	25.942	26.528	CeO_2	100	6

Table 2-1. Continued.

Z	Element	Condition	Analysis time	Line	ROI (LL) Kev	ROI (UL) Kev	Secondary target	kV	mA
55	Cs	@CeO_2	300	Ka	4.182	4.375	CeO_2	100	6
56	Ba	@Al_2O_3	300	Ka	31.633	32.379	Al_2O_3	100	6
57	La	@Al_2O_3	300	Ka	32.847	33.630	Al_2O_3	100	6
58	Ce	@Al_2O_3	300	Ka	34.089	34.911	Al_2O_3	100	6
62	Sm	@Al_2O_3	300	Ka	39.319	40.322	Al_2O_3	100	6
63	Eu	@Al_2O_3	300	Ka	40.696	41.750	Al_2O_3	100	6
65	Tb	@Al_2O_3	300	Ka	43.532	44.696	Al_2O_3	100	6
72	Hf	@Mo	300	La	7.739	8.006	Mo	100	6
73	Ta	@Mo	300	La	7.981	8.254	Mo	100	6
74	W	@Mo	300	La	8.227	8.507	Mo	100	6
77	Ir	@Mo	300	La	8.987	9.287	Mo	100	6
79	Au	@Mo	300	La	9.514	9.827	Mo	100	6
80	Hg	@Mo	300	La	9.784	10.104	Mo	100	6
81	Tl	@Mo	300	La	10.057	10.386	Mo	100	6
82	Pb	@Mo	300	La	10.332	10.669	Mo	100	6
92	U	@Ag	300	La	13.309	13.743	Ag	100	6

2.1.2 Safety Systems

Chapter 3 of the "Epsilon 5 EDXRF Spectrometer System User's Guide" contains a complete description of all safety precautions that need to be adhered to while operating the XRF analyzer. The most important of these are described below

Exposure to X-rays:

The X-ray cabinet contains two indicators that tell personnel in the laboratory that x-rays are being produced: the yellow "X-ray emitting lamp" on top of the cabinet will be illuminated and the three yellow "X-rays ON" lamps on the control panel will also be illuminated. If any of these lamps fail the x-ray source is automatically shut off. X-ray dosimeters are placed in the XRF laboratory on the instrument cabinet, at the computer workstation and at the entrance door. These dosimeters are collected quarterly by the UNR Radiation Safety Office for analysis. Exposure reports are kept in the XRF laboratory Radiation Safety Manual and can be reviewed at any time.

Exposure to Beryllium:

The spectrometers x-ray detector and x-ray tube contain toxic Beryllium (Be), but during normal operation these units are sealed within the x-ray cabinet and the operator should have no contact with either assembly. If it is necessary to gain access to one of these assemblies contact the XRF laboratory supervisor first.

2.1.3 Maintenance

Routine maintenance of the Epsilon 5 includes:

Filling the liquid Nitrogen dewar:

The Ge X-ray detector is liquid nitrogen cooled to provide stability. The dewar has a capacity of 20 liters, which under normal operating conditions should last two weeks. To prevent the dewar from ever running dry, it should be refilled at least weekly. Common practice is to fill the dewar on Friday. Refer to section 3.3.3 of the System Users Guide for detailed safety information regarding the handling of liquid nitrogen. The detector temperature and LN2 level are monitored by the instrument and can be checked in the software. To check these parameters, open the "Maintenance Spectrometer", menu then click on the detector symbol (see Figure 2-2). This action will open the detector window from which the level and temperature as well as several other detector parameters are displayed.

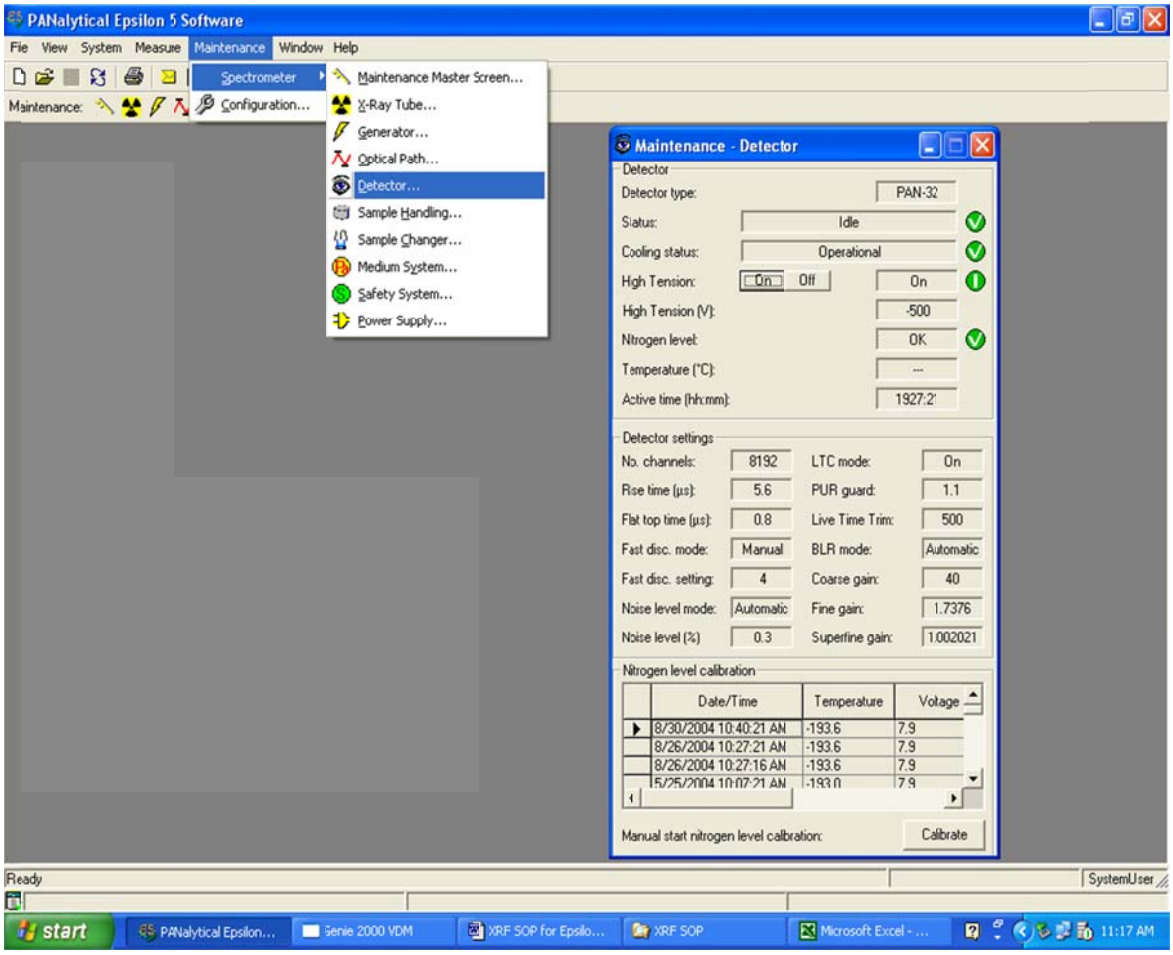


Figure 2-2. Software detector maintenance screen.

Filling the X-ray tube cooling water vessel:

Since the instrument must be shut down prior to filling of the x-ray tube cooling water vessel, this procedure should only be accomplished by the XRF laboratory supervisor. Detailed instructions for this procedure are contained in the Epsilon 5 software Help, under the "User Maintenance", "Routine" menus.

Checking the vacuum pump oil level

Since the instrument must be shut down prior to checking the vacuum pump oil level, this procedure should only be accomplished by the XRF laboratory supervisor. Detailed instructions for this procedure are contained in the Epsilon 5 software Help, under the "User Maintenance", "Routine" menus.

2.1.4 Spare parts list

It is essential that the following spare parts and supplies be kept on hand to insure minimal interruptions in analysis:

- Mylar sheets, 2 X 2" precut squares, 3.6 μm thickness (Somar Spectrofilm, #3615-33).
- Tweezers for handling filters (Millipore flat tipped stainless steel tweezers, #62-000067).
- Kimwipes, large (VWR, #34255) and small (VWR, #34155).
- Copies of current "Epsilon 5 XRF Analysis Logsheet".

2.1.4.1 Reagents

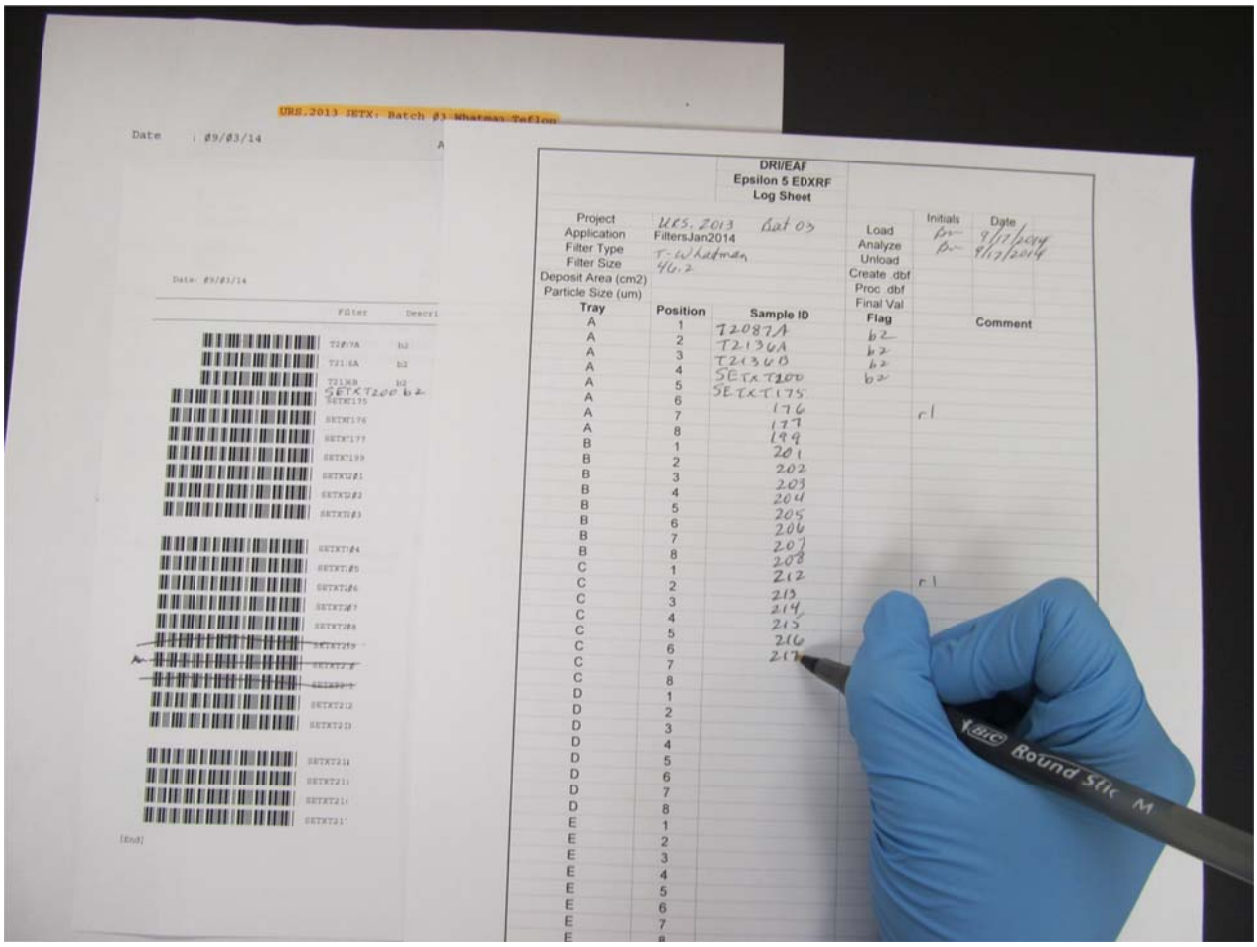
The chemicals required for XRF analysis are:

- methanol in a squeeze bottle for cleaning the sample holders and the filter loading area
- liquid nitrogen for detector cooling

2.1.4.2 Forms

All samples are logged into the EAF LAN upon receipt at the laboratory. A sample analysis list will be prepared by the laboratory or XRF supervisor indicating which samples will be analyzed and any special instructions. Filter IDs from the analysis list are transferred to the XRF analysis log sheet when filters are loaded into the machine for analysis. Figure 2-3 shows an example of analysis list and logsheet.

Figure 2-3. Sample Analysis list.



3. CALIBRATION STANDARDS

3.1 Preparation of working standards, ranges of standard values, and traceability to primary standards

Three types of standards are used with the DRI XRF: elemental thin film standards from µMatter, multiple element thin film standards from µMatter, and NIST certified standards. None of these standards require preparation; they are used as received from the supplier. The µMatter standards are stored in PetriSlides and kept in a cool dark cabinet when not in use to retard oxidation and loss of volatile elements. NIST standards are stored in the XRF room in the sample cabinet at ambient conditions. Certificates of elemental concentrations are provided by the manufacturer and are filed in the XRF lab.

3.2 Use

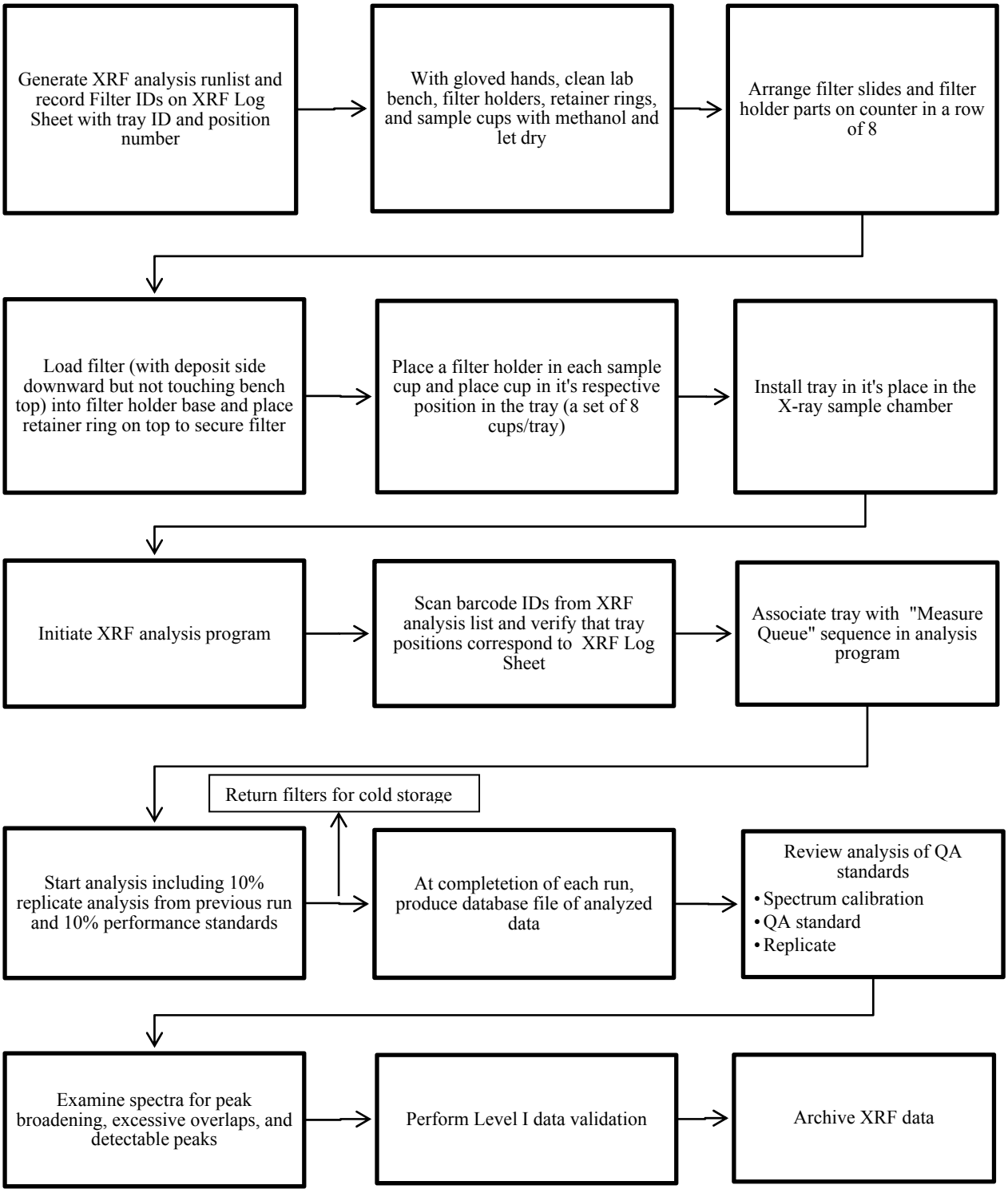
The DRI XRF system is recalibrated approximately every 12 months using the μMatter thin film standards. Recalibration is also performed whenever the QA standard indicated a drift of >±5% in calibration. Standards including elements from Na to U are analyzed in standard 47 mm filter holders under the filter analysis application (Filterxxxyy, where yyy = month and xx = year). Calibration factors in $\mu g/cm^2$ per counts per second per milliamp are calculated for each element using linear regression analysis by the Epsilon 5 software package.

3.3 Typical accuracy of calibration standards

The μMatter standards are accurate to ±5% relative, as stated by the manufacturer.

4. PROCEDURES

The typical flow of samples and data for DRI XRF analysis is depicted in Figure 4-1.


Generate XRF analysis runlist and record Filter IDs on XRF Log Sheet with tray ID and position number
With gloved hands, clean lab bench, filter holders, retainer rings, and sample cups with methanol and let dry
Arrange filter slides and filter holder parts on counter in a row of 8
Load filter (with deposit side downward but not touching bench top) into filter holder base and place retainer ring on top to secure filter
Place a filter holder in each sample cup and place cup in it's respective position in the tray (a set of 8 cups/tray)
Install tray in it's place in the X-ray sample chamber
Initiate XRF analysis program
Scan barcode IDs from XRF analysis list and verify that tray positions correspond to XRF Log Sheet
Associate tray with "Measure Queue" sequence in analysis program
Return filters for cold storage
Start analysis including 10% replicate analysis from previous run and 10% performance standards
At completetion of each run, produce database file of analyzed data
Review analysis of QA standards
• Spectrum calibration
• QA standard
• Replicate
Examine spectra for peak broadening, excessive overlaps, and detectable peaks
Perform Level I data validation
Archive XRF data

4.1 Start-Up

Normally the Epsilon 5 XRF is left running at all times and requires no special startup procedure. If the x-ray cabinet has been turned off, simply press the main power switch on the front panel to the "ON" position (Figure 4-2). If the power has been interrupted the x-ray cabinet will come back on when power is restored, however the computer program will likely require initialization before analysis may begin. To start the Epsilon 5 software, simple double click its icon on the desktop. The software will start and connect automatically to the spectrometer hardware

Figure 4.2. Epsilon 5 front panel display and controls.

4.1.1 Detector Calibration

The detector contains a Digital Signal Processor (DSP) that must be calibrated for the signals coming from the detector in order that they be placed in the proper energy channels. The calibration process consists of repeated measurements of the Tungsten (W) beam stop permanently installed in the Epsilon 5, therefore no standard loading is required. The calibration is performed on a weekly basis, typically on Friday afternoon. From the Epsilon 5 software main screen, choose the system drop down menu, then choose "Detector Calibration", select "Calibrate All" (Figure 4-3). The iterative process will begin and when finished the "Hours since cal" should be near zero for all three detector settings. Simply close the window when this is finished.

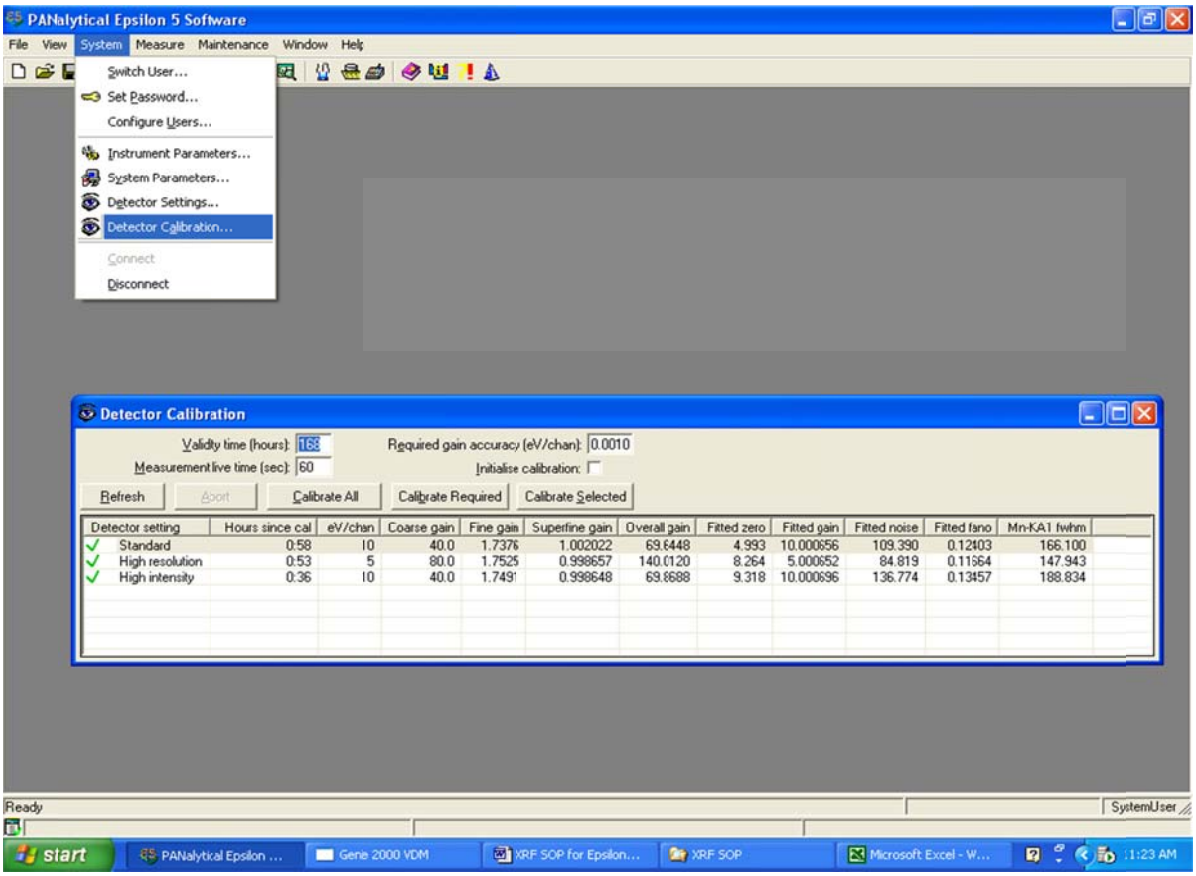


Figure 4-3. Software detector calibration window.

4.1.2 Routine Operation

Figure 4-4a-f shows the typical flow of events that takes place during an XRF analysis.

a)
Computer generated analysis list with barcodes
XRF Log Sheet
b)
Retaining ring
Filter holder
Loaded filter deposit side downward
Petri slide with filter sample
c)
Loaded filter in each metal cup
Metal sleeve
Sample tray of 8 cups
Tray ID
d)
Mechanical arm
X-ray light on during analysis
X-ray sample changer chamber
Sample Tray
e)
Measure Queue
Identification for sample during analysis
Tray ID
Filter sample ID
XRF Protocol
Position ID
Sample tray in queue for analysis
QA Standards
f)
Mechanical arm
Lid of X-ray vacuum chamber
QA Standard loaded by mechanical arm
X-ray Chamber
Additional QA Standard

Figure 4-4. Typical flow of events during an XRF analysis.

4.1.3 Filter Loading

The filter loading procedure depends on the filter media to be analyzed (1) quartz- or glass-fiber filters or (2) Teflon-membrane filters. The procedures are different for these two options. The XRF Supervisor may designate special handling and loading procedure for unusual samples or project requirements. The notes at the top of the analysis list should be read carefully before handling the samples.

Glass-fiber or quartz filters are infrequently analyzed at DRI by XRF for elements heavier than sulfur. Sulfur and lighter elements cannot be measured quantitatively on glass fiber or quartz filters due to high, variable background levels of these elements on these filter media and due to high absorption and backgrounds resulting from the relatively thick filter media.

Glass fiber and quartz filters are most frequently in the form of 8" X 10" sheets used in hi-vol samplers. Sample punches are taken from these large sheets for analysis.

Teflon membrane filters analyzed at DRI are generally 25, 37, or 47 mm diameter ring-mounted filters. The sample holders used in the DRI XRF are custom-designed holders for 25, 37 or 47 mm filters Sample punches of glass fiber or quartz filters using DRI's sample punch are placed into 47 mm holders. Other sizes of filters may be accommodated; refer to the analysis list or XRF Supervisor for additional instructions.

The 25, 37 and 47 mm holders are designed to fit in the solid sample cups in the XRF sample trays. All three styles consist of two parts: a holder base and a friction-fit retainer ring.

The holders are labeled on the side with a number between 1 and 8 which corresponds to a position in the sample tray; the retainer rings are not numbered. Select a complete set of holder bases and retainer rings for positions 1 through 8. Each sample tray can accommodate up to eight sample cups and the sample exchanger can hold up to six sample trays, so a maximum of 48 samples can be loaded at any one time.

Filters are loaded in the following manner:

- Wipe the work area clean with a methanol-dampened Kimwipe.
- Using latex gloves which have been carefully wiped with a methanol-dampened Kimwipe after being slipped on (to remove loose particles from the gloves), carefully wipe each sample holder base and retainer ring with methanol-dampened Kimwipes. Insure that all surfaces are free of particles, particularly the inner lip of the holder bases and the side of the retainer ring which will be contacting the filter. Place the holder bases on the clean Kimwipe in rows of 8 holders each; place the cleaned retainer rings on top of the holder bases. Carefully wipe the surface of eight sample cups with a second methanol-dampened Kimwipe place them in a row above the eight holders. Figure 4-5 shows the 47mm filter holder and the solid sample cup used for the analysis of filters.

- Referring to the XRF analysis list, select filters for analysis. The first two analysis trays for a particular project will include 16 first-time analyses and no replicates. Subsequent trays will include replicates at the rate of 1 per tray. The goal is to run approximately 10% replicates. Final adjustments in the number of replicates run are made in the final tray for a given project.

- Retrieve the samples to be analyzed from the XRF sample cabinet and lay the selected filters evenly across the Kimwipe, in rows of 8 filters each, beneath each filter holder. (The filters are still in their labeled containers at this step).

- Locate a current Epsilon 5 XRF Analysis Logsheet (Figure 2-4). Note: the QA gross counts ranges on the Analysis Logsheets change slightly when the XRF is recalibrated; make sure the latest version of the Analysis Logsheet is used. Complete the project, run ID (as specified in the analysis list), filter type and size, and sample load date and technician's initials sections as shown in Figure 2-4.

- Complete the sample IDs positions with the filter IDs selected for this run, referring to the XRF Analysis List and making sure that the correspondence of holder number and sample ID is the same on the XRF Analysis Logsheet as on the loading table.

- Check the filters for visual defects, large particles, filter damage, or other abnormalities which may affect the quality of the analysis. Note any problems in the comments section of the Analysis Logsheet.

- Teflon Membrane Filter Loading

  The following steps are followed to load ring-mounted membrane filters into the filter holders:

  — After wiping a pair of tweezers clean with a methanol-dampened Kimwipe, remove the filter from the first petri dish or PetriSlide. Examine the deposit closely for defects not previously noted. Remove carefully all large or loose particles which may fall off during analysis.

  — Carefully move the retainer ring from the corresponding holder base (avoid touching the lower surface which will contact the filter). Turn the filter over (face down) and place it into the holder base.

  — Place the retainer ring over the filter, squeeze it slightly to reduce its circumference, and place it over the filter to hold it flat. Insure that the opening in the retainer ring is oriented toward the right. Note: the filters must be completely flat against the inner lip of the holder bases: the x-ray signal is sensitive to distance traveled, and slight changes in the distance between the x-ray tube, the filter, and the detector will have measurable effects.

  — Proceed in a similar manner for the remaining samples.

  — Each loaded filter holder must now be carefully loaded into the corresponding sample cup. The filter holders must lay flat in the sample cup, so take extreme care to ensure that you lower the filter holders straight down into the cup. The sample cups can now be loaded into a sample tray. Each sample tray has a letter designation that corresponds with its position in the XRF sample changer.

— After all samples are loaded, double check that the sample ID on the Logsheet matches the ID on the empty filter container for each position.

— If samples will not be loaded immediately into the XRF sample chamber, place a large, clean Kimwipe over the holders and place the Analysis Logsheet on top.

- Quartz or Glass Fiber Filter Loading

The following steps are followed to load glass fiber or quartz filters into the filter holders:

— Locate the box of Mylar sheets (3.6 μm, 2 X 2 inch squares) in the XRF sample cabinet.

— Open the box of Mylar and carefully remove the top tissue paper; a sheet of Mylar should accompany the tissue paper due to static attraction.

— Carefully move the retainer ring from the corresponding holder base (avoid touching the lower surface which will contact the filter).

— Place the Mylar over the first holder base. Holding one corner of the Mylar with tweezers, remove the tissue paper, leaving the Mylar draped over the filter holder.

— After wiping a pair of tweezers clean with a methanol-dampened Kimwipe, remove the filter from the first petri dish or PetriSlide. Examine the deposit closely for defects not previously noted. Remove carefully all large or loose particles which may fall off during analysis.

— Turn the filter over (face down) and place it into the holder base. Center the punch in holder base opening.

— In a similar manner, place a second Mylar square over the filter punch. Although the two Mylar sheets are not electrostatically attracted to one another, they are attracted to most other objects, including tweezers, fingers, sample holder bases, and the tissue paper. Some caution is necessary to insure both Mylar squares are lying flat.

— Place the retainer ring over the filter, squeeze it slightly to reduce its circumference, and place it over the filter to hold it flat. Note: the bottom Mylar sheet and the filter must be completely flat against the inner lip of the holder bases: the x-ray signal is sensitive to distance traveled, and slight changes in the distance between the x-ray tube, the filter, and the detector will have measurable effects. The bottom Mylar must be as free as possible of wrinkles. Careful tension on alternate corners of the Mylar will generally remove residual wrinkles. Caution: take care that the retainer ring does not pop out as tension is applied to the Mylar.

— Each loaded filter holder must now be carefully loaded into the corresponding sample cup. The filter holders must lay flat in the sample cup, so take extreme care to ensure that you lower the filter holders straight down into the cup. The sample cups can now be loaded into a sample tray. Each sample tray can hold up to eight sample cups and has a letter designation that corresponds with its position in the XRF sample changer.

— Proceed in a similar manner for the remaining samples.

— After all samples are loaded, double check that the sample ID on the Logsheet matches the ID on the empty PetriSlide for each position.

If samples will not be loaded immediately into the XRF sample chamber, place a large, clean Kimwipe over the holders and place the Analysis Logsheet on top.

4.1.4 Loading the sample chamber

Before opening the sample changer cover, check the front panel to ensure that the green "Free to Open" lamp is lit. The cover is interlock protected to prevent the robotic sample changer arm from moving while the cover is open thereby preventing damage to the arm and/or the operator. If the cover is open when the sample changer arm is ready to change the sample, the software will show a warning window on the computer screen requesting that the operator close the sample chamber cover. It is good practice to check the progress of the current analysis, if it is nearly complete, the operator should wait until the sample is finished and the next one is loaded before proceeding with the reloading of the sample chamber. If you are in the middle of loading the sample chamber and the software requests that you close the sample chamber cover, simply finish what you are doing to a convenient point and close the cover to allow the machine to change samples.

Sample trays are loaded into the sample changer with the letter written on the sample tray (A-F) matching the etched letter in the sample chamber (Figure 4-4 d). The trays are keyed to prevent them from being loaded backwards in the sample chamber; this ensures samples one through eight are in the proper order. When all trays have been loaded, close the sample chamber lid.

4.2 Filter Analysis

Once sample trays are loaded into the X-ray cabinet sample chamber, analysis can begin. To start an analysis go to the computer workstation and ensure that the Epsilon 5 software is currently running (it should always be running) and choose the "Sample Changer Measurements" option under the "Measure" menu (Figure 4-5).

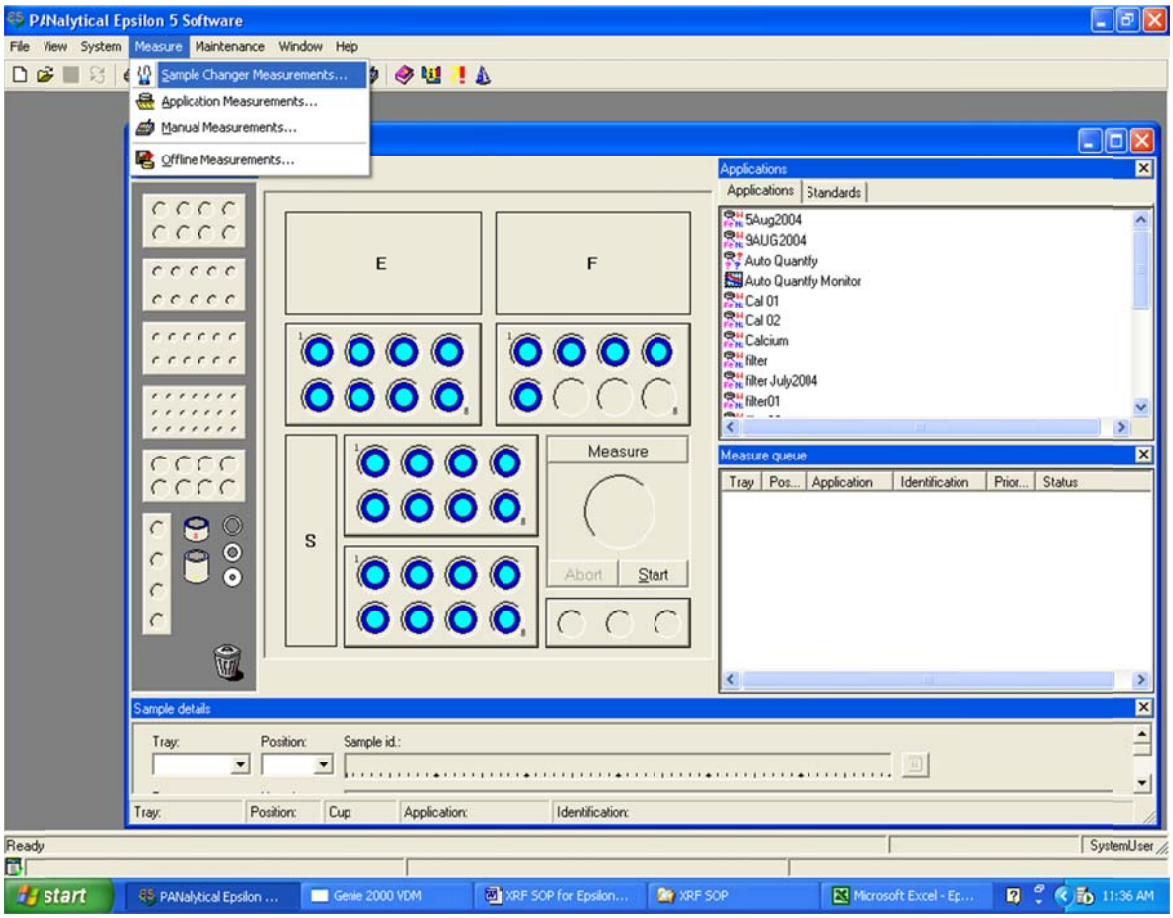


Figure 4-5. Software sample changer measurements window.

From this window sample IDs can be added to the analysis queue by clicking on its position in the tray, this will enable the "Sample id:" window at the bottom of the screen. Once the ID has been entered the sample position color should be blue indicating all is well and analysis can begin. Next the operator simply drags the samples over to the "Measure queue". This action will change the color to yellow. See Figure 4-6 for an explanation of the sample colors.

If these are the first samples added to the queue the Start button must be selected, otherwise if these samples are being added to a queue that has analysis in progress they will automatically be added to the end of the queue.

For analysis of filters (47mm, 37mm or 25mm) the sample trays used are the "2x4" sample cups (the bottom horizontal tray on the left hand side of Figure 4-5). The application for analysis is chosen from the list of applications, usually the application setup for the measurement of Teflon membrane filters is set as the default application.

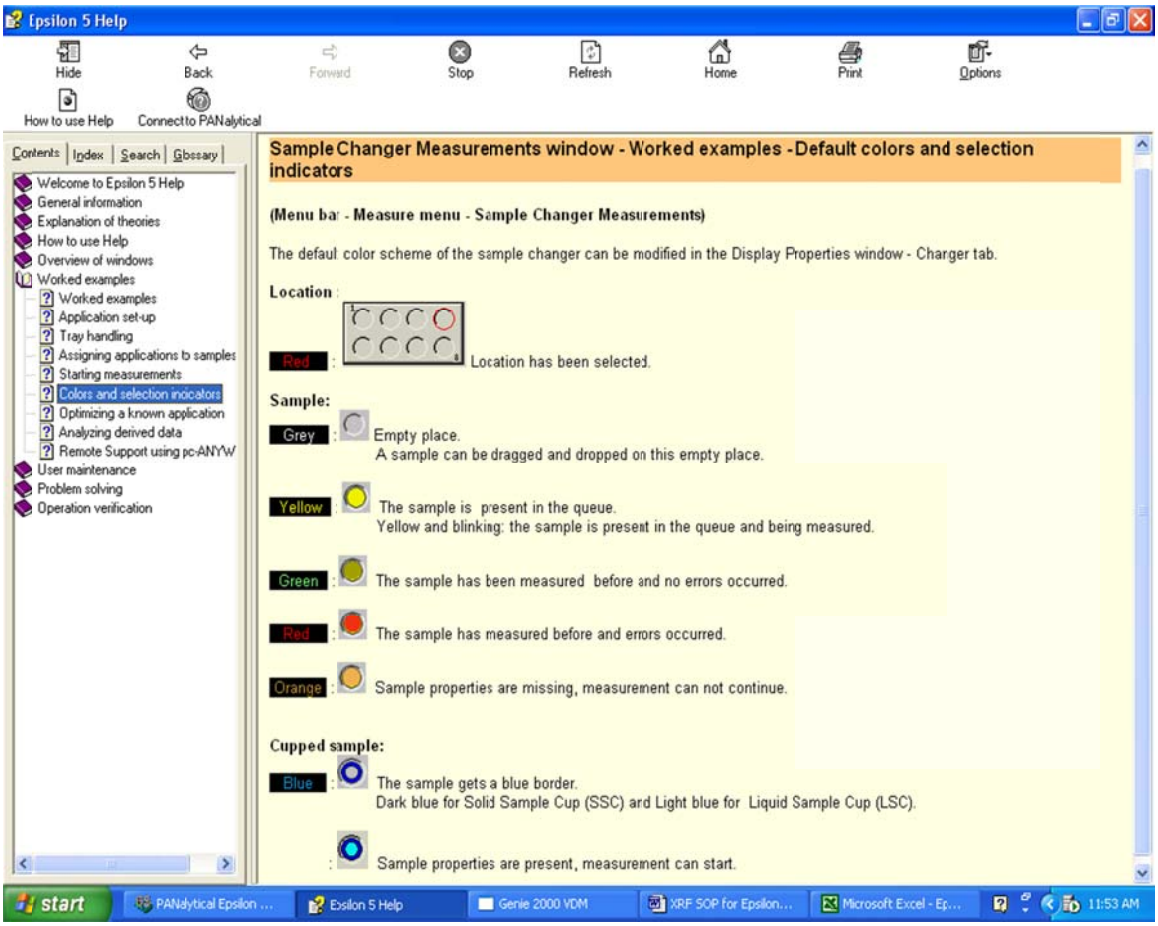


Figure 4-6. Software Help screen defining sample changer window colors.

When the analysis of a sample is successfully completed the sample indicator will be green. If any problems were encountered during analysis the sample will be red. If any samples are red immediately notify the XRF laboratory supervisor.

4.5 Shut-Down

Typically the Epsilon 5 system is left running at all times, but in the event of an emergency the X-ray cabinet can be shut down by simply pressing the "Power On" switch on the control panel to the "Off" position.

When power is removed from the Epsilon 5 or when the software is shut down, immediately notify the XRF laboratory supervisor or his/her designee. The x-ray detector is protected from damage due to liquid nitrogen depletion by the software (the software will shut down the detectors high voltage power supply if the liquid nitrogen runs too low), therefore if the software is shut down and the x-ray cabinet is left on, it is imperative that the liquid nitrogen level be closely monitored.

5. QUANTIFICATION

5.1 Calibration procedures

Calibration of the Epsilon 5 XRF analyzer is achieved by analyzing µMatter thin film standards. The software package calculates a linear regression line for each element using Nuclepore blanks and at least two standards for each element. The calibration line for Cu is shown in Figure 5-1. The results of the calibration for each element are stored with the application and a hardcopy is filed in the XRF laboratory.

For elements where no standards are available a calibration factor is obtained by plotting instrument response versus atomic number and interpolating the factor for the missing element. This operation is performed by the XRF laboratory supervisor using Microsoft Excel spreadsheet software. Electronic copies of the calibration curves for each condition are saved in Excel spreadsheets and posted in the laboratory information management system.

Full calibrations are performed when the QA standard that is analyzed daily falls outside acceptable limits or when any significant repair or parts replacement is performed on the instrument.

5.2 Calculations

The Epsilon 5 software integrates the peaks for each element, performs the deconvolution routine (to account for interelement interferences) and then uses the calibration data for each element to convert the raw counts data to micrograms per square centimeter for reporting. All sample data is stored in the results database on the XRF computer. In order to process batches of filters the data must be saved via the results window to a text file that is converted into a database file for final processing. To select filter results for export, open the results window for the application and highlight the samples, then click the show report button to create the report. To save the data as an exportable text file, print the report to a comma delimited file. This operation is illustrated in Figure 5-2.

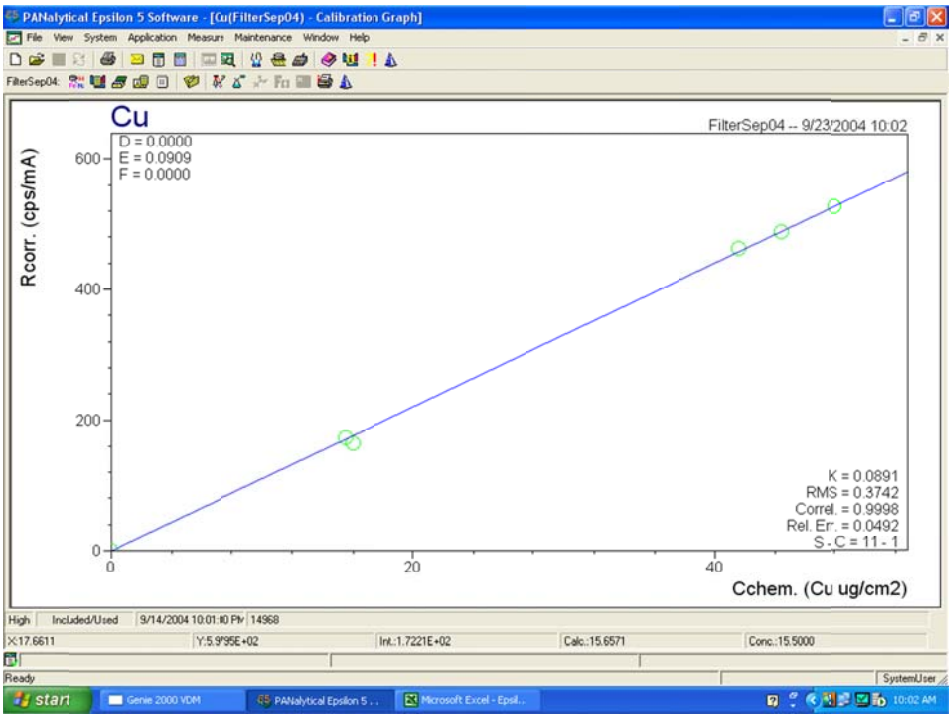


Figure 5-1. Software calibration graph window.

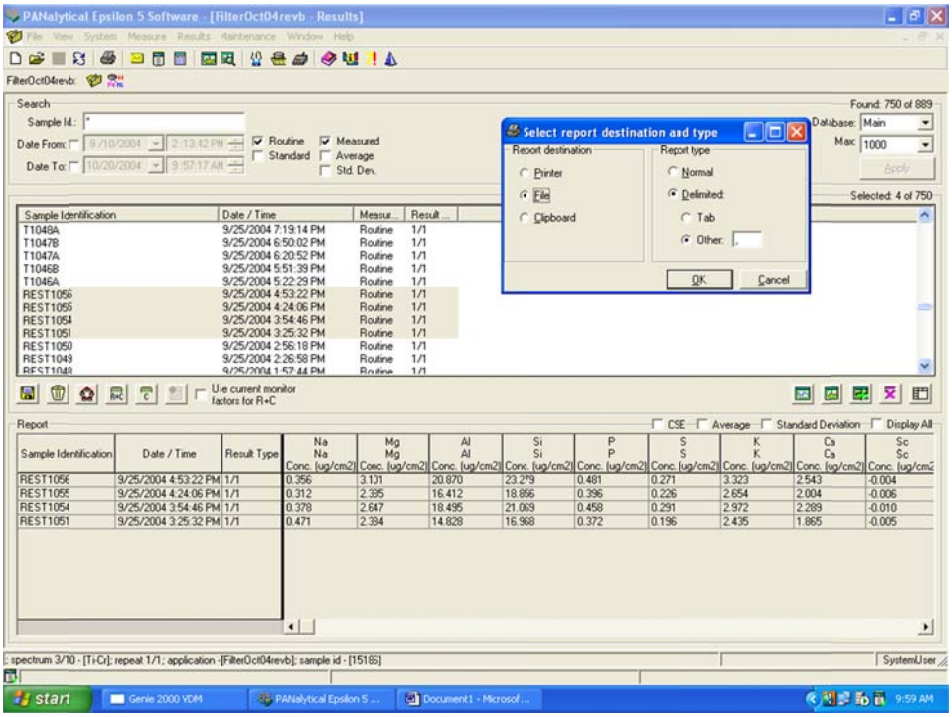


Figure 5-2. Software Analysis Results window.

6. QUALITY CONTROL

Quality control for the XRF analyzer consists of checking the performance of the analyzer against a QA standard which is analyzed every day and looking at replicate analyses.

6.1 Performance testing

A multi-element Micromatter thin film standard is run daily to monitor for instrument drift. Using the Epsilon 5 software the latest results can be displayed while the instrument is still processing the analysis queue. Each day check the results for the QA standard and record the concentrations in micrograms per square centimeter on the XRF Micromatter Multielement QA Standard analysis logsheet. Upper and lower bounds (± 5%) are listed on the logsheet. If the concentration is between these bounds no action is necessary, if it is out of bounds the XRF supervisor must be immediately notified and the problem must be fixed before analysis continues.

6.2 Reproducibility testing

Replicates of analyzed samples are performed at the rate of 10%. This corresponds to one replicate per analysis tray after the first tray. The general pattern to be followed is one tray with two replicates, followed by three trays with one replicate each. This generally corrects for the changing number of original samples which can be analyzed in each batch due to the number of replicates. The total number of replicates is compared to the total number of filters prior to the last run for a given project, and additional replicates are placed in the last tray to insure a 10% replicate rate.

Replicate data is examined during the data validation and spectra review step. In general, the replicate $\mu g/cm^2$ data should be within ± 10% or within 3 times the reported analytical uncertainties. Exceptions to these criteria may be made for such species as chlorine and bromine, which may vaporize under the high vacuum and decrease between the first and second analyses.

6.3 Validation Codes

Flags for all laboratory chemical analysis are defined in Table 6-1. During loading and unloading of the filters from the analyzer the operator should record and flags on the analysis logsheet.

Table 6-1. Validation codes and their meanings.

Flag	Meaning
b	Blank.
b1	Field/dynamic blank.
b2	Laboratory blank.
b3	Distilled-deionized water blank.
b4	Method blank.
b5	Extract/solution blank.
b6	Transport blank.
c	Analysis result reprocessed or recalculated.
c1	XRF spectrum reprocessed using manually adjusted background.
d	Sample dropped.
f	Filter damaged or ripped.
f1	Filter damaged, outside of analysis area.
f2	Filter damaged, within analysis area.
f3	Filter wrinkled.
f4	Filter stuck to PetriSlide.
f5	Teflon membrane separated from support ring.
f6	Pinholes in filter.
g	Filter deposit damaged.
g1	Deposit scratched or scraped, causing a thin line in the deposit.
g2	Deposit smudged, causing a large area of deposit to be displaced.
g3	Filter deposit side down in PetriSlide.
g4	Part of deposit appears to have fallen off; particles on inside of PetriSlide.
g5	Ungloved finger touched filter.
g6	Gloved finger touched filter.
h	Filter holder assembly problem.
h1	Deposit not centered.
h2	Sampled on wrong side of filter.
h4	Filter support grid upside down- deposit has widely spaced stripes or grid pattern.
h5	Two filters in PetriSlide- analyzed separately.
i	Inhomogeneous sample deposit.
i1	Evidence of impaction - deposit heavier in center of filter.
i2	Random areas of darker or lighter deposit on filter.
i3	Light colored deposit with dark specks.

Flag	Meaning
i4	Non-uniform deposit near edge - possible air leak.
m	Analysis results affected by matrix effect.
m1	Organic/elemental carbon split undetermined due to an apparent color change of non-carbon particles during analysis; all measured carbon reported as organic.
m2	Non-white carbon punch after carbon analysis, indicative of mineral particles in deposit.
m3	A non-typical, but valid, laser response was observed during TOR analysis. This phenomena may result in increased uncertainty of the organic/elemental carbon split. Total carbon measurements are likely unaffected.
n	Foreign substance on sample.
n1	Insects on deposit, removed before analysis.
n2	Insects on deposit, not all removed.
n3	Metallic particles observed on deposit.
n4	Many particles on deposit much larger than cut point of inlet.
n5	Fibers or fuzz on filter.
n6	Oily-looking droplets on filter.
n7	Shiny substance on filter.
n8	Particles on back of filter.
n9	Discoloration on deposit.
q	Standard.
q1	Quality control standard.
q2	Externally prepared quality control standard.
q3	Second type of externally prepared quality control standard.
q4	Calibration standard.
r	Replicate analysis.
r1	First replicate analysis on the same analyzer.
r2	Second replicate analysis on the same analyzer.
r3	Third replicate analysis on the same analyzer.
r4	Sample re-analysis.
r5	Replicate on different analyzer.
r6	Sample re-extraction and re-analysis.
r7	Sample re-analyzed with same result, original value used.
s	Suspect analysis result.
v	Invalid (void) analysis result.
v1	Quality control standard check exceeded ± 10% of specified concentration range.

Flag	Meaning
v2	Replicate analysis failed acceptable limit specified in SOP.
v3	Potential contamination.
v4	Concentration out of expected range.
w	Wet Sample.
w1	Deposit spotted from water drops.

Analysis results are categorized as valid, suspect, or invalid. Unflagged samples, or samples with any flag except 's' or 'v' indicate valid results. The 's' flag indicates results of suspect validity. The 'v' flag indicates invalid analysis results. Chemical analysis data validation flags are all lower case.

Data validation feedback

Begin data validation by verifying that all data entry is correct: sample ID's, deposit areas, and flags on the Logsheet and the printouts should match. If there are any discrepancies, note them on the XRF Validation Summary.

Next, examine the results for internal consistency and "reasonableness" of the XRF data. Categories to check are presence of elements whose concentrations are usually below the detection limit for typical ambient air samples, unusual elemental ratios, and replicate analysis results. Since data from the QA standard has already been checked, and since its composition is unlike aerosol samples, do not apply these checks to the QA standard.

The XRF Analysis Logsheet and results data file are reviewed by the XRF Supervisor or his designee. Any samples with incorrect or questionable data are noted on the XRF Validation Summary, record the filter ID, analysis condition, problem, along with steps taken to resolve any problems. Once all data has been validated and any necessary corrections are made in the database, the line labeled "QA Chks" on the XRF Analysis Logsheet is initialed. Hardcopies of all data validation summary sheets are filed in the XRF laboratory.

7. REFERENCES

PANalytical , *Epsilon 5 EDXRF Spectrometer System User's Guide*, First Edition March 2003

8. DOCUMENT CHANGES

7/30/07: r3 – minor formatting changes. Added signature blocks to title page. Note that original SOP started as r1 and not r0.

10/12/09: r4 – Changed to reflect the installation of the new dual anode Sc/W x-ray tube.

12/10/09: r5 Updated MDL table

12/17/10 r6 Updated XRF logbook example

07/09/12: r7 Updated MDL table and conditions table to reflect changes to analysis protocol made during the March 2012 calibration. Deleted section 6.3 regarding control charts.

10/03/14: r8 Updated SOP with pictures and flow chart figures.

DRI STANDARD OPERATING PROCEDURE

General EAF Internal Audit Procedures

DRI SOP #4-117r1
March 17, 2014

Desert Research Institute
Division of Atmospheric Sciences
2215 Raggio Parkway
Reno, NV 89512

(775) 674-7094

Prepared By: _____ **Date:** 3/17/14

Reviewed By: _____ **Date:** 3/17/14

Approved By: _____ **Date:** 3/17/14

1. GENERAL DISCUSSION

1.1 Purpose of Procedure

This procedure outlines the general procedures to be followed when preparing for, conducting, and reporting the results of internal systems audits of Environmental Analysis Facility (EAF) operations.

Internal audits of EAF operations are technical/management systems audits to assess the following:

- Compliance with requirements in QA/QC-related documents
- Documentation of compliance by review of records (forms, log books, etc.)
- Determination of compliance by observing performance of tasks
- Conformance with general quality system practices
- Correction of deficiencies through corrective actions

1.2 Measurement Principle

Depends on the specific EAF operation being audited.

1.3 Measurement Interferences and Their Minimization

Depends on the specific EAF operation being audited.

1.4 Ranges and Typical Values

Depends on the specific EAF operation being audited.

1.5 Typical Lower Quantifiable Limits, Precision, and Accuracy

Depends on the specific EAF operation being audited.

1.6 Personnel Responsibilities

All EAF employees are responsible for adhering to the procedures and documentation requirements contained in the EAF Quality Manual, Quality Management Plans, Quality Assurance Plans, and Standard Operating Procedures. For internal EAF audits, laboratory personnel are responsible for demonstrating procedures as they are normally conducted and responding to auditor questions.

Laboratory supervisors and coordinators are responsible for ensuring that all procedures and documentation are followed properly and that any deviations are quickly resolved. For the internal EAF audits, supervisors/coordinators are responsible for seeing that the audit can be conducted on the agreed date(s), that necessary staff and documents are available, observing the proceeding and responding to questions as needed, reviewing audit findings with the auditor and agreeing to or commenting corrective actions to resolve audit deficiencies.

The QA Officer is responsible for ensuring that all QA/QC requirements for EAF operations are being met. For the internal EAF audits, the QA Officer is responsible for conducting the audit personally or through a designee. The detailed responsibilities of the auditor are given in this SOP.

1.7 Definitions

Audit – A systematic, 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 activities are being conducted as planned and whether these activities effectively achieve quality objectives..

1.8 Related Procedures

Demonstration of Capability – DRI SOP #6-015.

Specific SOPs relevant to the particular operation being audited.

Specific EAF internal audit procedures for particular EAF operational areas.

2. APPARATUS, MATERIALS, AND FORMS

2.1 Apparatus and Instrumentation

Depends on the specific EAF operation being audited.

2.2 Reagents

Depends on the specific EAF operation being audited.

2.2 Forms

Each specific EAF internal audit procedure will have an audit form for that particular operation. Figure 2-1 shows excerpts of an audit form used for an EAF internal audit of $PM_{2.5}$ FRM gravimetry operations. Major deficiencies are tracked through Corrective Action Requests (CARs). An example form is shown in Figure 2-2.

3. CALIBRATION PROCEDURES

Depends on the specific EAF operation being audited.

4. PROCEDURES

4.1 General

Internal EAF audit procedures are to be developed for all EAF operations as time and resources permit based on the following priorities:

1. All operations for which NELAC accreditation is sought and to be retained.
2. Other EAF operations in priority order based on the fraction of the EAF workload that each represents.

DRI Internal Audit of EAF PM2.5 Gravimetry Operations

Auditor(s) Richard Tropp

Date(s) 7/31/09, 8/3/09, 8/4/09, + 8/26/09

Purpose: Conduct a technical/management systems audit to assess the following
- Compliance with requirements contained in documents listed in References
- Documentation of compliance by review of records (forms, log books, etc.)
- Assessment of compliane by observing performance of tasks
- Conformance with general Quality System practices (see References)

References:
1. DRI, SOP #2-114r6, *PM2.5 FRM Gravimetric Analysis* (12/2/08)
2. DRI, SOP #6-015r0, *Demonstration of Capability* (6/13/08)
3. DRI, SOP #6-014r0, *Laboratory Ethics* (6/13/08)
4. DRI, *Quality Assurance Project Plan PM2.5 Filter Laboratory Analysis Program* - Rev. 1 (8/08)
5. EPA, *Quality Assurance Handbook for Air Pollution Measurement Systems - Volume II Ambient Air Monitoring Program*, EPA-454/B-08-003 (12/08)
6. DRI, *Quality Manual - Environmental Analysis Facility (EAF)* - Rev. 1 (6/08)

Personnel Interviewed
Supervisor/Coordinator: Brenda Cristani
Others: Gail Martin

General Information
No. of Technicians: BC + 4
Comments: Observed Gail Martin

Documents Examined

Item	Location	OK (Y/N)
1. Demonstrations of Capability	Weighing Room (PM2.5)	N
2. Laboratory Ethics Agreements	Weighing Room (PM2.5)	Y
3. SOP #2-114r6	Weighing Room (PM2.5)	Y
4. Weighing Logbooks	Weighing Room (PM2.5)	Y see comments
5. Environmental Condition Charts	Weighing Room (PM2.5)	Y
6. Weight Room Audits/Services Book	Weighing Room (PM2.5)	Y
7. Filter Media Acceptance Logsheets	Logistics Prep Room	Y
8. Filter Assignment Logsheets	Weighing Room (PM2.5)	Y

Comments: Item 1 – 4 DOCs, but 3 fr. 6/08 & need to be renewed. Item 21 – looked at random pages from 7/08 on in Current Projects (A-C, D-H, I-P, & Q-Z) & TCEQ Mass (Shipments 110-) Discuss Gridley 11/4/09 w BC & IMP 1/15/09 SERPP 12/12/08 FW; Technicon 8/8/08 FW, & TCEQ 135 5/18/09. Item 4 – Need additional comments/codes to explain unusual items (e.g., unweighed final filters, >30% replicates on final filters). Be careful to indicate where ip sequence T/C taken as go from one sheet to another

Page 1 of 4

Figure 2-1a. Excerpts of EAF Internal Audit Form for $PM_{2.5}$ FRM Gravimetry

DRI Internal Audit of EAF PM2.5 Gravimetry Operations (Continued)

Specific Items Checked

Calibration/Verification/Service/Preventive Maintenance Frequency

Item	Freq.	Criteria	Found	OK (Y/N)
Balance Cal.	1/yr	Manuf.	Both lots 2005-2009	Y
Lab Temp	1/6 mos	± 2 °C	last done in June '09	N
Lab RH	1/6 mos	± 2 %RH	not in book	N
Working Mass Stds (w Primary)	1/3 mos	< ± 25 µg	only 200 mg	N
Primary Mass Stds	1/yr	< ± 25 µg		Y see comment
Calibration Stds	All	NIST-tr C1		Y
Replace P210 Source	1/6 mos	NA	not changed 1/2005	N
Balance Internal Cal	B	NA	not checked	N
Balance Check - Span	B/10/E	≤ ± 3 µg	not check	Y

QC Checks

Item	Freq.	Criteria	Found	OK (Y/N)
Filter Conditioning (Pre-Use)	All filters	> 4 wks		Y
Fiter Conditioning (Pre/Post Wgt)	All filters	> 24 hrs		Y
Env Conds - Tavg	All filters	20-23 °C		Y
Env Conds - Tsd	All filters	± 2 °C		Y
Env Conds - RHavg	All filters	30-40 %RH		Y
Env Conds - RHsd	All filters	± 5 %RH		Y
Lab blanks	2%	± 10 µg diff		Y
Field Blanks	>10%	NA		Y
Replicate Wgts - Pre	100%	± 10 µg diff		Y
Replicate Wgts - Post	30%	± 15 µg diff		Y

Operational

Item	Found	OK (Y/N)
Plastic forceps for std wgts	Yes	Y
Petri-slides open	2 on stab	Y
Time in neutralizer	~ 30 sec	Y
Reading stabilized	3 secs	Y
Record on weight sheet	Y	Y
Replicates done by another tech	Y	Y

General QC

Item	Found	OK (Y/N)
Handwritten records clear & in ink	Not always	N
Errors crossed & corrections near	Usually at bottom sheet	N
Correction maker ID'ed	Usually at top by sheet or bottom	N
Critical data corrections w. reason	Not always	N
Computer correction - audit trail	done by DRI in 3602 db	Y

Comments: Lab Temp & RH - not consistently done every 6 mos
Next scheduled one should be December
Working mass - 1/yr - needs 1/6 mos only 200 mg need to do 50 & 100 mg too
Primary mass - past 1 yrs needs to go back (was due in '09)
P210 source - no available documentation on changeout included in
Service book, Congratulations to do this process.

Page 2 of 4

Figure 2-1b. Excerpts of EAF Internal Audit Form for $PM_{2.5}$ FRM Gravimetry

EAF CORRECTIVE ACTION REQUEST

CAR No. _____ **Date** _____

To **From**
How Identified
Reported (Nonconformance) Conditions **Answer Due Date** **Signature**
Root Cause
Corrective Action (Give Steps & Expected Completion Dates)
Preventive Action
Completed by **Date**
Accepted by **Date**
Follow-up Results **Performed by** **Date**
Closed Out (Y/N) _____ If not closed out on follow-up, issue a new CAR **New CAR No.** **Date**

Figure 2-2. Corrective Action Request (CAR) Form

Separate internal EAF audits (with separate audit forms) may be conducted for particular aspects of a general operation. For example, for $PM_{2.5}$ gravimetry operations, separate audits (and forms) may be performed for gravimetry, shipping and receiving, and cold storage. Internal EAF audits will also review all elements of the quality management system, including corrective actions, calibration and check procedures for balances, sensors, purchasing, and complaints. Usually such audits will be conducted at approximately the same time. However, an audit for one particular portion of a general operation may be conducted separately if only that particular portion of the general operation has changed significantly.

Once initiated, internal EAF audits for a particular operation will be conducted at least annually or within 3 months of a major change in related SOPs. Internal audits will be suspended or stopped if a particular operation is performed rarely (i.e., less than yearly) or has been discontinued.

The following sections describe the general procedures to be used when creating (or revising), conducting, reporting, and following up on an internal EAF audit.

4.2 Creating or Initiating a Specific Internal EAF Audit Procedure

4.2.1 First review relevant reference documents and make sure that they are the most recent approved versions available. Example documents may include:

- EAF Quality Manual (NELAC)
- Quality Management Plans (QMPs)
- Quality Assurance Project Plans (QAPPs)
- EAF Standard Operating Procedures (SOPs)
- EPA (or other agency) requirements and guidance documents such as volume II of the EAP QA Handbook
- The Code of Federal Regulations (CFR), especially 40 CFR Parts 50, 53, and 58
- Specific client contract requirements

4.2.1 Prepare or revise a list of references and applicable requirements. Look for potential conflicts in the requirements. If necessary, resolve conflicts by choosing the most stringent requirement.

4.2.3 Prepare or revise the audit form. The audit form should include the following items:

- Title with area being audited
- Date(s) of the audit
- Purpose of the audit
- References
- Personnel interviewed

- General information
- Documents examined
- Specific items to be checked based on requirements in references
- Comments
- Minor deficiencies
- Major deficiencies
- Follow-up and corrective actions
- Next audit schedule and type
- Closing meeting signoff

If there are revisions to an existing audit procedure document the changes in the audit workbook along with the date and reason.

4.2 Conducting the Audit

4.2.1 Notify the area supervisor/coordinator of the upcoming audit about 1-2 weeks in advance to make sure that the operational tasks to be observed will take place on an agreed date and that laboratory personnel and the supervisor will be present.

4.2.2 Prior to the agreed date of the observational portion of the audit, check the location and contents of documents to be reviewed.

4.2.3 The EAF QA Officer or his designee conducts the audit according to the specific procedures and criteria outlined in the workbook and form for the audit of a particular operational area.

4.2.3.1 The auditor again checks documents in the presence of the supervisor and discusses any related issues.

4.2.3.2 The auditor checks whether or not the audit criteria and requirements listed are being met or not. Clarification may be sought from the supervisor or other laboratory personnel where questions arise. Results are documented on the audit form.

4.2.3.3 The auditor observes the performance of tasks in a specific operational area by laboratory personnel that normally conduct them. The auditor may ask questions or to determine if requirements are met and how certain situations are handled. Results are documented on the form and may be discussed with the laboratory personnel involved and the supervisor.

4.2.3.4 The auditor reviews the results documented on the audit form and then lists minor and major deficiencies.

4.2.3.5 The audit findings are reviewed with the supervisor/coordinator at the end of the audit at which the supervisor's comments are noted for the record. Minor deficiencies or changes that can be implemented immediately are to be implemented within the next few days.

4.2.3.6 Major deficiencies or those that require equipment, software, or facility modifications, purchase, or installation are to be listed in Corrective Action Requests (CARs) with, dates established for their implementation and a follow-up schedule created to check on their timely completion.

4.3 Audit Report and Follow-up Actions

4.3.1 The auditor shall present a written report to the supervisor/coordinator, EAF Executive Director and to the EAF QA Officer (if the audit was performed by a designee).

4.3.2 Archive a hardcopy of the audit report in the audit book and an electronic copy in the SOP section of the LAN.

4.3.3 Schedule, track, and document follow-up progress of the CARs.

4.3.4 Enter a tentative date (typically 1 year later) on the calendar for the next audit.

5. QUANTIFICATION

Depends on the

6. QUALITY CONTROL

Depends on the specific EAF operation being audited. In addition, internal audit reports and CARs are archived in an audit book and on the LAN.

7. QUALITY ASSURANCE

Depends on the specific EAF operation being audited.

8. REFERENCES

Refer to the oven's owner's manual for additional information concerning its operation.

9. DOCUMENT CHANGES

08/09/12: New SOP – r0.

03/17/14: r1 – Replaced CAR Form (Figure 2-2) with updated version.

DRI STANDARD OPERATING PROCEDURE

Demonstration of Capability

DRI SOP #6-0015r1
August 15, 2012

Desert Research Institute
Division of Atmospheric Sciences
2215 Raggio Parkway
Reno, NV 89512

(775) 674-7094

Prepared By: Richard Snopp **Date:** 8/15/12

Reviewed By: John G. Watson **Date:** 8/15/2012

Approved By: Judith Chow **Date:** 8/15/2012

1. GENERAL DISCUSSION

1.1 Purpose of Procedure

This procedure describes the method used for performing, approving, and documenting the initial and continuing demonstration of capability (DOC) for Environmental Analysis Facility (EAF) procedures.

This procedure is performed prior to an employee using a test method or standard operating procedure, any time there is a significant change in instrument type, personnel or test method, or if a new analyte is added to an existing accredited method. In the absence of significant changes, it is repeated annually.

1.2 Measurement Principle

As specified in the SOP for which capability is to be demonstrated.

1.3 Measurement Interferences and Their Minimization

As specified in the SOP for which capability is to be demonstrated.

1.4 Ranges and Typical Values

As specified in the SOP for which capability is to be demonstrated.

1.5 Typical Lower Quantifiable Limits, Precision, and Accuracy

As specified in the SOP for which capability is to be demonstrated.

1.6 Personnel Responsibilities

Technician/Analyst - Person responsible for completing training and demonstration of capability studies.

Laboratory Supervisor – Person responsible for certifying that an employee has demonstrated competence for a certain method/analyte/matrix and for ensuring that training and DOCs are performed when needed, per this SOP. A supervisor who also performs the tasks of a technician analyst is also responsible for a demonstration of his/her capability by his/her supervisor or the QA Officer or designee.

Quality Assurance Officer – Person or designee responsible for ensuring and certifying that employees have documented training and demonstration of capabilities on file, and for annual reviews and updates of DOCs.

1.7 Definitions

Demonstration of Capability (DOC) – A procedure to establish the ability of the analyst/technician to generate acceptable accuracy.

Relative Standard Deviation (RSD) – The absolute value of the coefficient of variation expressed as a percentage.

1.8 Related Procedures

None

2. APPARATUS, MATERIALS, AND FORMS

2.1 Apparatus and Instrumentation

As specified in the SOP for which capability is to be demonstrated.

2.2 Reagents

As specified in the SOP for which capability is to be demonstrated.

2.2 Forms

EAF Demonstration of Capability Form (Figure 2-1).

3. CALIBRATION PROCEDURES

Not applicable

4. PROCEDURES

4.1 General

4.1.1 Upon hire, employees receive initial DRI new employee orientation training. Once completed, the employee then receives EAF orientation and training in specific standard operating procedures.

4.1.2 Prior to approval for the performance of any method, training and a satisfactory demonstration of capability must be performed for that method.

4.1.3 Any time there is a change in instrument type, personnel or test method, a DOC must be performed.

4.1.4 If a method is added to an accreditation or if an analyte is added to an existing accredited method, training and a DOC study must be performed by affected analysts and technicians before routine analysis begins

4.1.5 Demonstration of Capability (DOCs) studies are used as documented evidence of competence. Individual DOCs are recorded on the EAF Demonstration of Capability Form.

4.1.6 DOCs are performed only after proper documented training for the method is complete. The person performing the DOC must have a solid understanding of the content, QA of the method, and of the equipment and/or instrumentation to be used.

EAF DEMONSTRATION OF CAPABILITY
CERTIFICATION STATEMENT

Date:_____

Environmental Analysis Facility (EAF)
Desert Research Institute
2215 Raggio Parkway
Reno, NV 89512

Name: _____

Job Title: _____

Method/Matrix/Analyte(s):

_____

SOP Number and Revision: _____

We, the undersigned, CERTIFY that:

1. The person identified above, using the cited test method(s), which is used at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Initial Demonstration of Capability.
2. The test method was performed by the person identified on this certification.
3. A copy of the laboratory-specific SOPs is available for all personnel on-site.
4. The data associated with the initial demonstration of capability are true, accurate, complete and self-explanatory (1).
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and, the associated information is well organized and available for review by authorized inspectors.

_____	_____	_____
Laboratory Supervisor	Signature	Date
_____	_____	_____
Quality Assurance Officer	Signature	Date

(1)True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

Figure 2-1. EAF Demonstration of Capability Form

4.1.7 In the absence of significant changes, a continuing DOC is to be conducted annually.

4.1.8 Employee training files are annually reviewed and continuing DOC studies may be selected for employees on a random basis.

4.1.9 A continuing DOC may also be performed as part of a corrective action due to suspected sub-standard performance by an individual.

4.2 Performance of Demonstration of Capability (DOC) Studies

4.2.1 DOC studies are not performed with customer samples, instead, proficiency samples or laboratory-spiked samples (prepared from clean matrices) are used.

4.2.2 All DOC studies are considered blind to the analyst, as they are unaware of the content of the sample. All sample preparations and analyses are performed in accordance with the specified method, with no deviations or modifications.

4.2.3 Laboratory samples are prepared from clean matrices, in which no target analytes or interferences are present, and stock standards. The stock standards must be prepared independently from those used in the instrument calibration.

4.2.4 No less than five (5) replicate samples are prepared using the specified method.

4.2.5 For a bulk sample, at least 5 different sample weights will be recorded. For a liquid sample, at least 5 aliquots will be taken and diluted to the specified concentration.

4.2.6 The final sample concentration should be 1-4 times the reporting limit for each analyte.

4.2.7 The 5 preparations and 5 analyses may be prepared and analyzed concurrently, or over a period of days.

4.2.8 When complete, the report (in appropriate reporting units) and raw data (or a copy of the raw data) is reviewed by Laboratory Management and/or QA.

4.2.9 Proficiency studies may be used to satisfy the ongoing DOC requirements.

4.3 Evaluation of DOC Results

4.3.1 Laboratory management and/or QA reviews the laboratory results and the raw data is reviewed to ensure that the results of the QA and calibration standards meet the requirements, as specified by the method.

4.3.2 The analyses results are compared to the certified or spiked values, as appropriate.

4.3.3 Percent recovery and relative standard deviation (RSD) are calculated for each analyte of interest.

4.3.4 To consider a DOC study acceptable using a proficiency sample, the reported results for all 5 analyses (and the average of the 5 results) must be within the published acceptable limits of the proficiency study.

4.3.5 To consider a DOC study acceptable using a laboratory-prepared sample, percent recovery (for each analyte) must be within ±20% of the spiked value for all 5 analyses (and the average of the 5 results) or meet the specifications within the SOP, whichever is more stringent.

4.3.6 The relative standard deviation (RSD) for the 5 results must be <10%, for the DOC to be considered acceptable.

4.3.7 If all parameters meet the acceptance criteria identified in section 4.3, the DOC is considered acceptable, the EAF Demonstration of Capability Form is approved by laboratory management and QA, and analysis of actual customer samples may begin.

4.3.8 If any of the parameters (for one or more of the analytes) do not meet the acceptance criteria, the DOC is considered unacceptable for that parameter and the analysis of actual samples cannot begin. Refer to section 4.4 below for handling of unacceptable DOC results.

4.4 Handling of Unacceptable DOC Results

4.4.1 If any of the tested parameters fail to meet the acceptance criteria, the source of the problem is identified, corrected, and the test is repeated for all failing parameters.

4.4.2 In the case of repeated failure, the steps in Section 4.3 are repeated and the study is conducted with supervision and/or the study is performed concurrently with an approved individual.

4.5 Documentation and Record Control

4.5.1 The EAF Demonstration of Capability Form is used as a record of approval for Demonstration of Capability Studies

4.5.2 DOC forms are signed by the individual (or work cell participants), the department manager and the QA officer.

4.5.3 Training and DOC records are maintained in employee training files.

4.5.4 Training and DOC records are reviewed no less than annually to ensure that they commensurate with the employee's current job description.

4.5.5 Training and DOC records are maintained for no less than 5 years from the date of employee termination or separation.

5. QUANTIFICATION

See section 4.3 and relevant sections of the SOPs for which the DOC is being obtained.

6. QUALITY CONTROL

Signed *EAF Demonstration of Capability* forms are maintained as quality and management records for a minimum of five years from the date of employee separation.

7. QUALITY ASSURANCE

See Section 6, Quality Control.

8. REFERENCES

National Environmental Laboratory Accreditation Conference (NELAC). Quality Systems. Appendix C - Demonstration of Capability. 2003 NELAC Standard. Approved June 5, 2003. Effective July 1, 2005

NELAC Institute. Management and Technical Requirements for Laboratories Performing Environmental Analysis – Module 2: Quality Systems General Requirements. TNI Standard. EL-V1M2-2011. Adopted September 8, 2009. Effective September 1, 2010.

9. DOCUMENT CHANGES

06/13/08: New SOP – r0.
08/15/12: r1 – Clarified that 2) supervisors/coordinators are to perform demonstrations of capability if they perform the tasks of a technician/analyst and 2) that in the absence of significant changes continuing DOCs are performed annually. Also added TNI Standard as reference.

DRI STANDARD OPERATING PROCEDURE

Corrective Action

DRI SOP #6-017r0
August 15, 2012

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1. GENERAL DISCUSSION

1.1 Purpose of Procedure

This procedure outlines the corrective action system and use of the Corrective Action Request (CAR) system within the Environmental Analysis Facility (EAF) to document major changes made to procedures, systems, facilities, instruments, and computer programs occur after the item has been completed and accepted.

1.2 Measurement Principle

Not applicable

1.3 Measurement Interferences and Their Minimization

Not applicable

1.4 Ranges and Typical Values

Not applicable

1.5 Typical Lower Quantifiable Limits, Precision, and Accuracy

Not applicable

1.6 Personnel Responsibilities

All EAF employees are responsible for being aware of the potential for operational and data quality issues and to report any problems to their supervisors, including those that may initiate a CAR.

The supervisor/coordinator for the affected operation is responsible for assigning personnel, assisting in setting completion deadlines, and reviewing and approving the CAR to be acted upon. The EAF Executive Director and QA Officer may need to be included in allocating resources and establishing deadlines, especially when a major, costly effort involving multiple groups may be indicated.

The EAF QA Officer or designee acts as the administrator for CARs, reviews each regularly, provides assistance as needed, tracks the progress of each, and follows up if a CAR's completion is overdue.

1.7 Definitions

Corrective Action Request (CAR) system – A process by which specified operational and data quality issues are addressed, especially if major resources or time or data has been delivered to a client.

1.8 Related Procedures

None

2. APPARATUS, MATERIALS, AND FORMS

2.1 Apparatus and Instrumentation

Not applicable

2.2 Reagents

Not applicable

2.2 Forms

EAF Corrective Action Request (CAR) Form (Figure 2-1).

3. CALIBRATION PROCEDURES

Not applicable

4. PROCEDURES

4.1 Applicability

4.1.1 Typical circumstances in which the CAR process may be used may include the following

- Correcting major bugs in software (especially if the software has been used to deliver data to EAF clients).
- Making major changes to improve procedures and corresponding SOPs or other quality documents (especially when the revisions involve equipment, facility, or software revisions)
- Purchasing, installing, and testing new equipment or software to solve specific performance deficiencies
- Developing new training materials to remedy specific staff performance issues
- Investigating and correcting systematic data quality or quality system deficiencies in the EAF

4.1.2 A CAR must be used if any of the following conditions apply:

- A major effort is needed to solve a specific operational or data quality issue
- Significant costs (e.g., labor hours, equipment, or materials) are involved
- Corrected data needs to be redelivered to the client
- A client is aware of the issue and expects status updates
- The corrective effort will take a relatively long period of time (i.e., more than about two weeks)
- Approved validation and QA criteria are affected
- Information about the corrective action needs to be formally documented

EAF CORRECTIVE ACTION REQUEST

CAR No. _____ **Date** _____

To **From**	
How Identified	
Reported (Nonconformance) Conditions **Answer Due Date**	**Signature**
Root Cause	
Corrective Action (Give Steps & Expected Completion Dates)	
Preventive Action	
Completed by	**Date**
Accepted by	**Date**
Follow-up Results **Performed by**	**Date**
Closed Out (Y/N) _____	If not closed out on follow-up, issue a new CAR
New CAR No.	**Date**

Figure 2-1. EAF Corrective Action Request (CAR) Form

4.1.3 A CAR may be used when none of the circumstances in 4.1.2 apply and the laboratory supervisor/coordinator agrees that the corrective action can be handled within the normal scope of operations. Isolated, limited problems that typically arise during routine operations should not require CARs.

4.2 Procedure

4.2.1 Administration - The CAR forms are issued and logged by the EAF Quality Assurance Officer or a designee. Numbers are assigned to the CARs and are distributed as requested. CARs are logged in the Internal EAF Audit Book, log sheets updated, and forms and (related information) filed.

4.2.2 Initiation – Any EAF personnel may report a problem that initiates a CAR. Often, CARs may arise from internal EAF audit, external audits and ongoing data validation and QA/QC review. The originator of the CAR works with the supervisor/coordinator and EAF QA Officer (or designee) to complete the first two sections of the CAR form.

4.2.3 Assignment and Approval - The supervisor/coordinator for the affected operation is responsible for assigning personnel, working with the EAF QA Officer or designee to set a completion deadline and approving the CAR to be acted upon. CARs requiring significant resources or time to complete must be approved by the EAF Executive Director and the EAF QA officer.

4.2.4 Tracking – Once the CAR has been scheduled and approved, copies of the form are distributed to the initiator, supervisor/coordinator, and any other persons listed at the bottom of the form. The CAR form is maintained by the administrator, who enters due dates into a tracking calendar. The administrator reviews the CAR log, and each CAR regularly, provides input as necessary, and reports delays or concerns to the EAF Executive Director and QA Officer.

4.2.5 Implementation – Assigned personnel and the area supervisor/coordinator are responsible for carrying out the corrective action. The CAR file is updated whenever significant modifications to the approach, costs, resources, or deadlines are necessary.

4.2.6 Completion – When the corrective actions are completed, the successful completion of a CAR should be approved by the area supervisor/coordinator, Quality assurance officer or designee, or the EAF Executive Director.

4.2.7 Archiving – When the corrective action has been completed and approved, the approved CAR form and copies of related material and documentation shall be archived with their location given in the CAR log book.

5. QUANTIFICATION

Not applicable

6. QUALITY CONTROL

Expected due dates and completion are tracked using a schedule calendar.

7. QUALITY ASSURANCE

Not applicable

8. REFERENCES

None

9. DOCUMENT CHANGES

08/15/12: New SOP – r0.