Quality Assurance Project Plan for the Alaska Lead Monitoring Program

2011

State of Alaska Department of Environmental Conservation

Division of Air Quality

Air Monitoring and Quality Assurance Section

619 East Ship Creek Suite 249

Anchorage, AK 99501

A Project Management Elements

1 QA Plan Identification and Approval

Title: Quality Assurance Project Plan for the Alaska Lead Study

Quality Assurance Project plan for the Alaska Lead Study is hereby recommended for approval and commits the Program to follow the elements within.

Alaska Department of Environmental Conservation

Signature _____ Date 6/8/2011

Barbara Trost, AMQA Program Manager

Signature _____ Date 6/8/2011

Dan Fremgen, Quality Assurance Officer

EPA

Signature _____ Date 6/14/11

Chris Hall, Quality Assurance Officer

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4 Distribution List

A copy of Quality Assurance Project Plan for the Alaska Lead Study will be distributed to the individuals listed in Table 3.1. The Air Monitoring Section for the Alaska Department of Environmental Conservation (DEC) will post the quality assurance project plan and associated standard operating procedures on the DEC Air Quality Division website site.

Table 3.1: Contact List

NAME	POSITION	AGENCY	Contact Information
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5 Project Organization

5.1 Roles and Responsibilities

Federal, state, regional and tribal agencies have important roles in developing and implementing air quality assessment and monitoring programs. EPA is responsible for developing National Ambient Air

Quality Standards (NAAQS), defining the quality of the data necessary to make comparisons to the NAAQS and identifying a minimum set of quality control (QC) samples from which to judge data quality. The state and local organizations are responsible for taking this information and developing and implementing an air quality assessment and monitoring project that will meet the data quality requirements. It is the combined responsibility of EPA, the state and local organizations to assess the quality of the data and take the appropriate actions to assure compliance with the NAAQS monitoring requirements.

With the promulgation of the revised lead rule, a monitoring requirement was established for sources emitting more than one ton of lead per year. The Red Dog Mine in northwest Alaska is the only source of lead emissions meeting the monitoring criteria in the state of Alaska for potential emission greater than one ton per year. Implementation of the lead rule will be by the Alaska DEC with oversight by the EPA. The field monitoring will be contracted out to members of the community of Noatak, near the mine. The organizational structure is presented in Figure 4-1. The responsibilities of each organization follow.

5.2 EPA Region 10

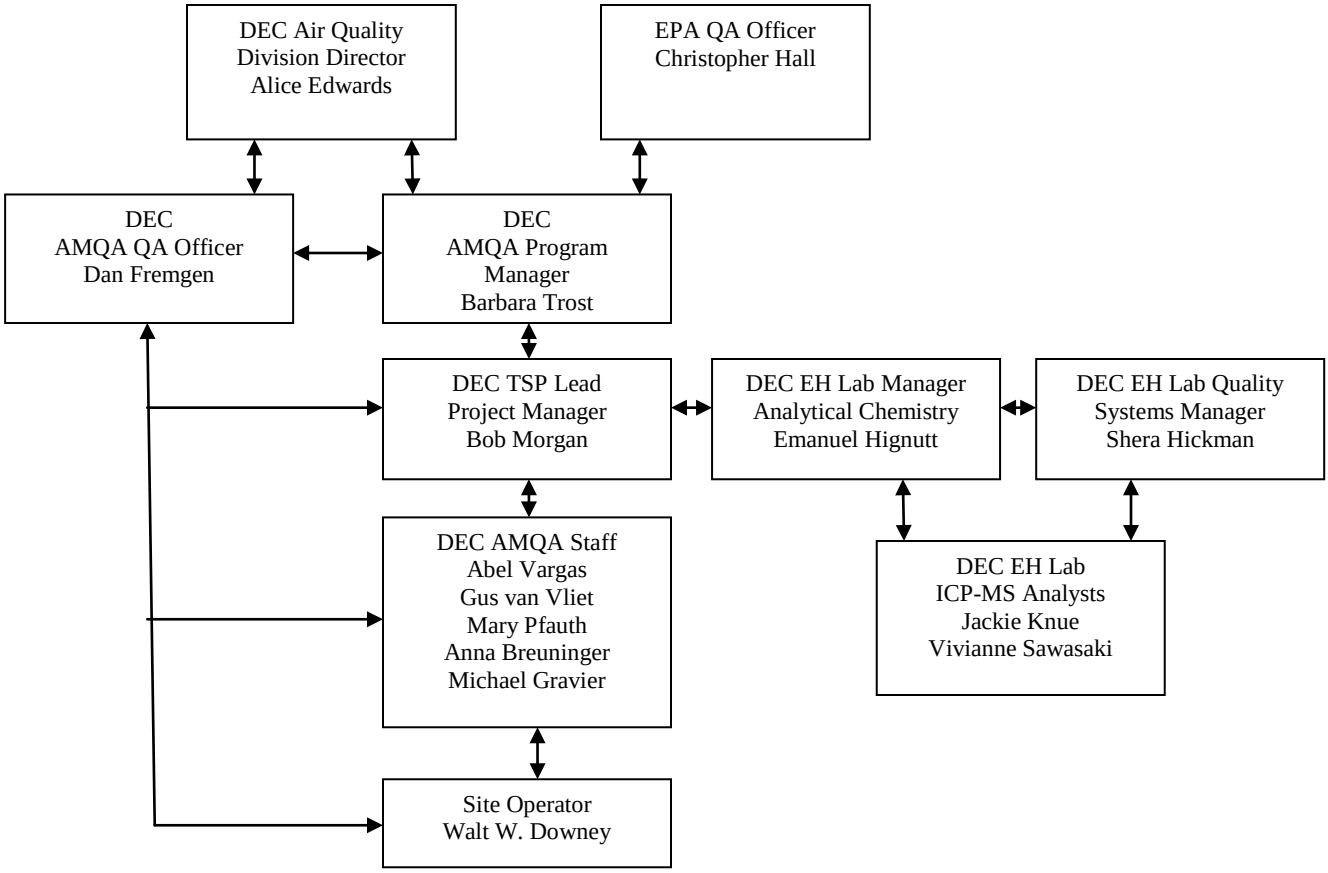
The major responsibilities of EPA's Region 10 Office are the coordination of quality assurance matters at the regional levels. This is accomplished by the designation of EPA Regional Project Officers who are responsible for the technical aspects of the program.

5.3 Alaska Department of Environmental Conservation

The Division of Air Quality of the Department of Environmental Conservation (DEC) contains the Air Monitoring and Quality Assurance (AMQA) Program, which is responsible for coordinating all aspects of air quality studies.

The major responsibility of the department's AMQA section is the design and implementation of the monitoring project and the quality assurance program. This includes oversight responsibilities in all phases of the monitoring operation, including field sampling, laboratory analysis, data processing, and reporting. Air sampling activities will be directed by DEC staff with support from the local site operators. Analytical sample preparation, digestion, and analysis, and all other analytical quality assurance/quality control (QA/QC) will be the responsibility of the DEC Environmental Health (EH) Laboratory located in Anchorage.

Figure 4-1 Alaska Lead Monitoring Program – Organizational Scheme



The title and responsibilities of key project personnel are:

DEC Air Quality Division Director

- Responsible for overall management of the Air Division
- Coordinates with the Department Commissioner to establish Division goals and objectives
- Testifies before the State Legislature on funding issues and coordinates with staff on legislative initiatives
- Coordinates with the EPA to meet regulatory priorities as established by the Clean Air Act and facilitate implementation of the Alaska SIP
- Coordinates with the EPA on grant funds
- Set Division priorities, coordinates funding, and approves budgets among the Division programs
- Provides general supervisor to Division program managers

DEC AMQA Program Manager

- Responsible for overall management of the Air Division AMQA program
- Facilitates funding, reviews and approves of program expenditures
- Sets monitoring program priorities
- Coordinates with the AMQA QA Officer to review audit results and data validation issues
- Coordinates with the AMQA Monitoring Manager to direct all monitoring efforts
- Communication with EPA project officers and EPA QA personnel
- Network evaluation, design, and site selection
- Project planning and implementation

DEC AMQA Quality Assurance Officer

- Supervises or directs all quality assurance activities within the AMQA section
- Review and approval of all QA documents (i.e. QAPP and associated SOPs)
- Conduct performance and system audits
- Review and verify traceability of all quality control standards
- Assure the lead monitoring program is compliant with NAAQS data quality objectives
- Prepares quarterly QA audit reports for submission to DEC AMQA Program Manager and AMQA TSP Lead Project Manager
- Coordinates with the AMQA program manager and monitoring manager on issues concerning data validation and corrective actions

DEC AMQA Project Manager

- Overall technical management of the lead monitoring program to assure compliance with NAAQS monitoring requirements
- Air monitoring equipment procurement
- Site and utility contracts
- Station installation and operation coordination

- Development and implementation of QAPP and SOPs, and work plans
- 3rd tier data review and validation of monitoring data and report approval
- Directs all corrective actions to assure data quality
- Oversees agreements and facilitates payment to tribal entities for site operators

DEC AMQA Technical Support Staff

- Assure strict adherence to all standard operating procedures (SOPs) for: field sampling and data collection, recordkeeping and shipping; data processing; and reporting
- Coordinate equipment mobilization, transport, installation, and initial calibration of on-site monitoring sites
- Oversee on-site training of contract site operators
- Provide on-going technical oversight of all field operations including assistance to site operators, and facilitate logistics for shipment of supplies, and equipment
- Schedule and conduct routine site visits for on-going review and training of site operators and perform non-routine site maintenance
- Receive and log all sample and data deliveries from site operators
- Document chain-of-custody and facilitate transfer of filter samples to the DEC EH laboratory
- Enter field data with analytical data to calculate preliminary sample results
- Compile sample results, QA/QC data, and generate preliminary report
- Conduct an initial data review to identify, flag, and document all suspect data
- Once the preliminary report is compiled and reviewed by the project coordinator, the data is transfer to the senior staff for 2nd and 3rd tier review
- Report finalized data to the EPA Air Quality System (AQS)

DEC EH Lab Manager Analytical Chemistry

- Coordinate with the AMQA Project Manager to develop and receive approval of an analytical SOP which meets EPA Federal Equivalent Method (FEM) requirements
- Provide direct technical oversight all laboratory operations
- Assure strict adherence to laboratory SOPs for ICP/MS analyses including receipt of samples, chain of custody, sample preparation, chemical digestion, instrumental analysis, and QA/QC
- Perform supervisory review of analytical data, chemical calculations, and verify results
- Coordinate with Laboratory QA/QC Manger on validated results
- Prepare and submit final report with analytical results and associated laboratory QA/QC to the AMQA Project Manager

DEC EH Lab Quality Systems Manager

- Review and approves quality assurance documents for all analytical methods and procedures
- Assure strict adherence to analytical SOPs
- Coordinate four-tier quality review process among laboratory staff including technical review by the analyst, senior peer review, supervisory review, and an administrative review for completeness.

DEC EH Lab Analyst

- Perform all sample preparation, chemical digestion, instrumental analysis, data compilation, and analysis in strict adherence with the analytical SOP.
- Conduct initial data quality review and coordinate with senior staff for peer review and supervisory review

5.4 On-Site Operation Contractors

The DEC Air quality Division will establish a contract with a local resident to operate and maintain the monitoring site equipment and perform on-site sampling. The on-site operator will be independent contractor. DEC AMQA will provide payment for labor and expenses involved in the monitoring program.

Responsibilities for the on-site operator(s) include:

- Train with DEC AMQA staff
- Perform duties associated with sampling, data collection, recordkeeping, equipment calibration, maintenance and repair in accordance with the TSP field sampling SOP
- To insure data quality and maximize data capture, communicate weekly with the DEC AMQA coordinator to discuss ongoing monitoring operations
- The first business day of the month, fax or email a copy of the previous months field data sheets to the DEC AMQA coordinator along with a monthly invoice of the hours worked and a description of the monitoring activities performed
- Perform duties associated with sample shipping and data transfer in accordance with the SOP
- Participate in ongoing training provided by the DEC AMQA coordinator during routine site visits
- Participate and provide technical assistance during quarterly quality assurance audits

The on-site operator insures good data quality by strictly following SOP requirements and clearly communicating information or questions to DEC AMQA coordinator concerning the on-site monitoring activities.

6 Problem Definition and Background

6.1 Problem Statement and Background

The EPA established a national ambient air quality standard (NAAQS) for lead in 1978 at 1.5 micrograms per cubic meter ($\mu g/m^3$). Between 1978 and now, more than 6000 studies have bolstered the underpinnings of the study of lead's deleterious health effects. Of primary importance is the finding that lead can cause neurological defects and learning disabilities in children at lower levels than previously thought. Low levels of lead can result in decreases in IQ and memory, slower learning and changes in behavior. For children and infants there is no known lead concentration that is safe. The EPA projects

that the revised standard will result in $3.7 to $6.9 billion in health benefits. Based on this new evidence, on October 15, 2008, the EPA lowered the primary (health based) standard for lead from 1.5 $\mu g/m^3$ to 0.15 $\mu g/m^3$. This represents a reduction of the NAAQS by a factor of 10 times.

6.2 Overall Objectives

The overall objective of the monitoring program is to determine Alaska's compliance status with the revised NAAQS for lead and if not, to develop and implement control strategies to attain compliance with the NAAQS. To fulfill the monitoring requirements of the revised NAAQS, the EPA and DEC have agreed to install a lead monitoring site located in a village close to the Teck Alaska Red Dog Mine. The site will meet criteria in Title 40 CFR Part 53. The monitoring program will continue for a period of, at least, three years to determine attainment status with the NAAQS for lead.

7 Project Description

7.1 Sampling Site Location

The Red Dog Mine is the only entity in the state of Alaska that emits over a ton of lead per year and is therefore required to monitor for airborne lead. The mine is located in a remote area of northwestern Alaska in the Northwest Arctic Borough which has an area about the size of the state of Indiana, or 40,762 square mile. The borough has a population of 7,523 mostly living in Kotzebue, the borough seat with a population of 3,201. The remainder of the population is spread among 11 scattered villages. The mine is located about 90 miles north of Kotzebue and about 55 miles east of the Chukchi Sea. In 1999, the State agreed to extend the mine's ambient air quality boundary as part of a "public access control plan." The agreement was made through revisions to the mine's existing Construction Permit 9932-AC005. The extended ambient air boundary is well beyond the mine's operational area where the terrain is extremely rugged and has no road access. This makes source-oriented monitoring at the mine site prohibitively expensive in an area with no significant risk of human exposure. The State and EPA have agreed to conduct lead monitoring in one of the communities closest to the mine. Monitoring in the community will provide a better assessment of human exposure in the regional area. After communications with the tribal government, DEC agreed to monitor lead in the nearby village of Noatak, 30 miles south of Red Dog mine. Figure 7-1 shows the location of Noatak in relation to the Red Dog Mine site and other area villages.

7.2 Work to be Performed

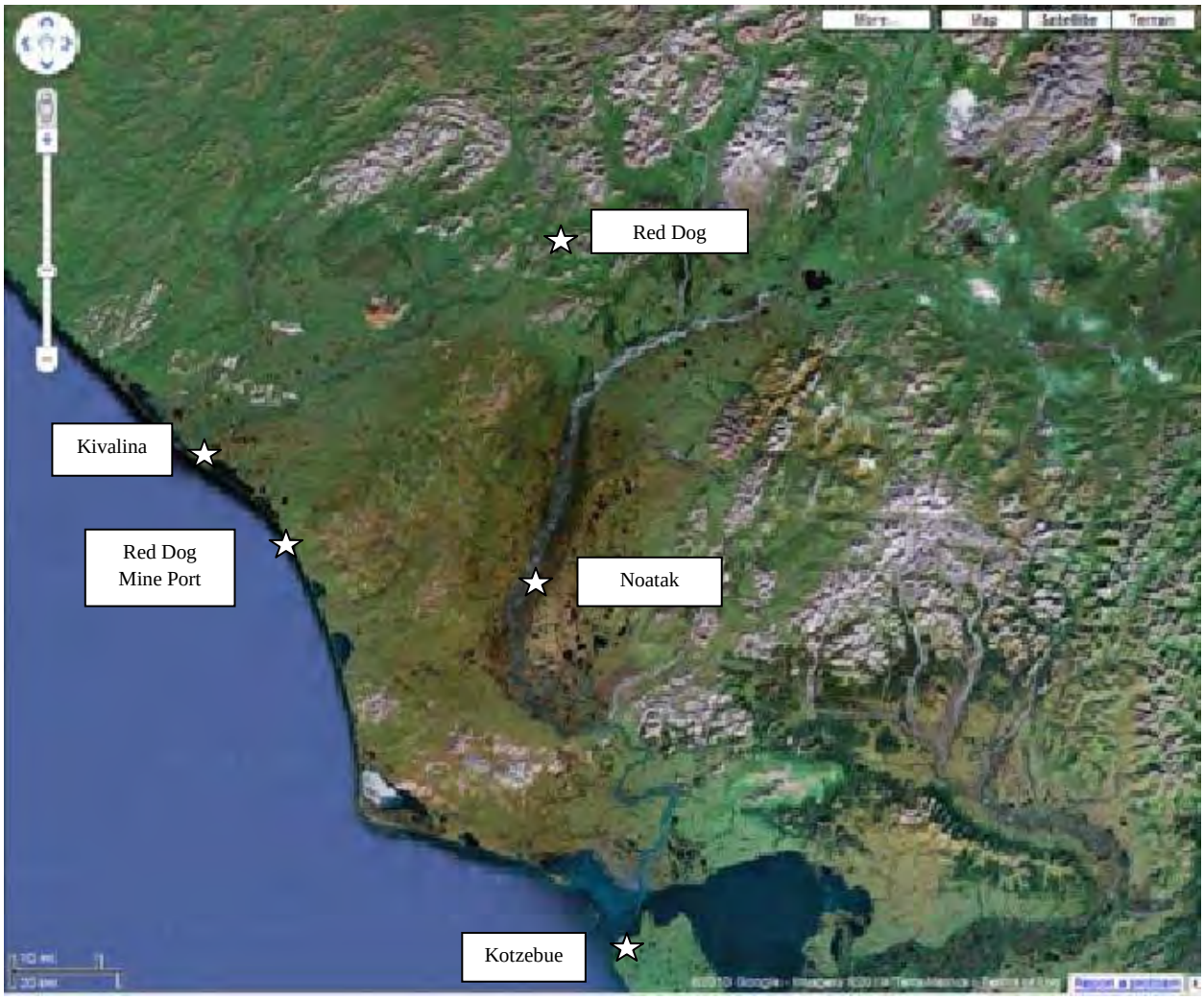
Installed in January 2010, the Noatak monitoring site consists of two collocated TSP (total suspended particulate) high volume (Hi-Vol) samplers. Both samplers will run on a 1 in 6 day sampling schedule specified by the EPA. Startup and commencement of sampling delayed to mid-January 2010 by weather and staff scheduling. Work plan schedules are presented in Table 7.1.

Table 7.1: Schedule of Activities

Activities	**Start Dates**	**Completion Dates**	**Responsible Party**	**Comments**
Work Plan development	6/09	11/09	DEC AMQA Staff	
QAPP Preparation, Review and Approval	9/09	Estimated 6/10	DEC AMQA Staff with EPA Approval-EPA	QA/QC procedures in place, awaiting OAQPS ICP/MS FEM approval for analytical SOP review & approval
Site Selection	8/09	10/09	DEC AMQA Staff	Used previous site
Site Installation	11/09	1/10	DEC AMQA Staff	platform to be extended 6/10
Operator Training	11/09	Initial training completed 1/10	DEC AMQA Staff	Refreshers during routine site visits
Sampling	1/15/10	Through 12/13*	Site Operators	1 in 6 day schedule
Collocated Samples	1/10	Through 12/13*	Site Operators	1 in 12 day schedule (minimum)
QA Audits	1/10	Through 12/13*	DEC AMQA QA Officer	Semi-annually {minimum)
Laboratory Analysis	5/10	Through 12/13*	DEC Anchorage EH laboratory	See comment on QAPP ICP/MS SOP
Data Processing and Validation	Quarterly	Through 12/13*	DEC AMQA Staff	Upon completion of the quarter
Report Preparation and upload to AQS	Quarterly	Through 3/14*	DEC AMQA Staff	3 months after completion of the quarter

* Completion date pending demonstration of attainment status with Lead NAAQS

Figure 7-1: Map of the Kotzebue area within Northwest Alaska includes Noatak, Kivalina, Kotzebue, the Teck Alaska Red Dog Mine, and the Port.



8 Quality Objectives and Criteria for Measurement Data

8.1 Data Quality Objectives (DQOs)

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

- Clarify the monitoring objectives.

- Define the appropriate type of data to be collected.
- Specify the tolerable levels of decision errors for the monitoring program.

By applying the DQO Process to the development of a quality system the Air Quality Program guards against committing resources to data collection efforts that do not support a defensible decision.

8.2 Define Appropriate Type of Data

In order to accomplish the monitoring objectives, the appropriate data needed is defined by the NAAQS. For criteria pollutants, compliance with the NAAQS is determined by specific measurement requirements. The sampling will follow the Hi-Vol method for the TSP sampler. The measurement system is designed to produce criteria pollutant concentration data of the appropriate quantity and quality necessary to determine compliance with these standards.

8.3 Specify Tolerable Levels of Decision Errors for the Monitoring Plan

DQOs for criteria pollutant monitoring are based on data requirements of the EPA. Regarding the quality of the measurement system, the objective is to control precision and bias in order to reduce the probability of decision errors.

8.4 Measurement Quality Objectives (MQOs)

Once a DQO is established, the quality of the data must be evaluated and controlled to ensure that it is maintained within the established acceptance criteria. MQOs are designed to evaluate and control various phases (sampling, preparation, and analysis) of the measurement process to ensure that total measurement uncertainty is within the range prescribed by the DQOs. MQOs can be defined in terms of precision, bias, representativeness, detectability, completeness and comparability.

Precision - Precision is a measure of mutual agreement among individual measurements of the same property usually under prescribed similar conditions, or how well side-by-side measurements of the same parameter agree with each other. It is important that the measurements be as similar as possible, using the same, or comparable, equipment. Precision represents the random component of uncertainty. Precision is estimated by various statistical techniques using the standard deviation or, if you only have two measurements, the percent difference.

Bias – Bias (accuracy) is the systematic or persistent distortion of a measurement process that causes uncertainty in one direction. (e.g., results are either higher than or lower than they should be). It is estimated by evaluating the instrument measured result against a known standard used as the "true" value. It is expressed as a positive or negative percentage of the "true" value. Bias is measured by conducting independent performance audit(s) of the monitoring equipment used to measure and report data. The

audit standards used as well as the audit personnel must be completely independent from standards used to calibrate the monitoring equipment and the personnel responsible for site operations.

Representativeness - Representativeness is defined as a measure of the degree which data really represent some characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. The representativeness of measurements made in this program is ensured by following EPA siting guidelines.

Detectability – Detectability is defined as the lowest value that a method procedure can reliably discern a measured response above background noise; in other words, that level below which the instrument cannot discriminate from zero. Because there is always variation in any measurement process (precision uncertainty), the level of detectability depends on how much precision error is in the process. Detection limits for ADEC-M&QA air quality instruments are consistent with the requirements listed in 40 CFR 53. For Federal Reference Methods (FRM) and Federal Equivalent Methods (FEM), the detection limits are specified with the respective EPA FRM/FEM designation.

Completeness - Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions. Data completeness requirements are included in the reference methods (40 CFR 50) and 40 CFR 58 Appendix A.

Comparability – Comparability is a measure of confidence with which one set of data can be compared to another. Comparability is important so that data sets within one part or area of the country can be compared with another area or data from another year.

Various parts of 40 CFR have identified acceptance criteria for some of these attributes as well as U.S. EPA Quality Assurance Guidance Documents and additional DEC ambient air regulatory monitoring methods. These Ambient Air Quality parameter MQOs are listed in Table 8-1. More detailed descriptions of these MQOs and how they will be used to control and assess measurement uncertainty are described in method specific data validation tables.

Table 8.1: Critical Criteria Tables for the Lead on FRM/FEM TSP method

Criteria	Frequency	Acceptable Range	Information (CFR or Method 2.2 or 2.8)
CRITICAL CRITERIA- Pb in TSP			
Filter Holding Times			
Sample Recovery	all filters	ASAP	Part 50 App B
Sampling Period	all filters	1440 minutes ± 60 minutes midnight to midnight	Part 50 App B sec 8.15
Sampling Instrument			
Average Flow Rate	every 24 hours of operation	1.1 to 1.7 m^3/min (varies with instrument)	Part 50 App B sec 8.8
Filter			Part 50 App B sec 7.1
Visual Defect Check (unexposed)	all filters	see reference	Part 50 App B sec 8.2
Collection Efficiency	all filters	99 %	Part 50 App B sec 7.1.4
Pressure Drop Range	all filters	42-54 mm Hg	Part 50 App B sec 7.1.5
pH	all filters	6-10	Part 50, App B sec 7.1.6
Pb Content	all filters pre-sampling batch check	<75 µg/filter	Part 50, App G sec 6.1.1 Method 2.8 sec 6.2.1
Verification/Calibration			
One-point Flow Rate Verification	1/3 mo	±7% from design transfer standard ±10% from design	Part 58 App A Method 2.2 sec 2.6
Calibration Reproducibility	Beginning, every 10 samples	± 5% of value predicted by calibration curve	Part 50, App G Sec 9.3

Criteria	Frequency	Acceptable Range	Information (CFR or Method 2.2 or 2.8)
Checks	and end		
Reagent Blank	Every analytical batch	< LDL	recommendation
Daily Calibration	Daily	until good agreement is obtained among replicates	Method 2.8 sec 2.8.5
OPERATIONAL EVALUATIONS TABLE Pb in TSP			
Verification/Calibration			
System Leak Check	During pre-calibration check	Auditory inspection with faceplate blocked	Method 2.2 sec 2.0
FR Multi-point Verification/Calibration	After receipt, after motor maintenance or failure of 1-point check and 1/yr	5 points over range of 1.1 to 1.7 m^3/min within ± 5% limits of linearity	Method 2.2 sec 2.6
Precision			
Collocated Samples	15% of each method code in PQAO Frequency - every 12 days	CV ≤ 20% of samples > 0.02 µg/m^3 (cutoff value)	Part 58 App A sec 3.2.5
Audits			
Semi Annual Flow Rate Audit	2/yr	± 10% of audit standard and design value	Part 58, App A, sec 3.3.3
Lead Strip Analysis	6 strips/quarter 3 strips at low concentration 30-100% of NAAQS 3 strips at high concentration 200-300% of NAAQS	3 10% (percent difference)	Part 58, App A, sec 3.3.3
Blanks			
Field Filter Blank	1/quarter	< LDL	recommendation

Criteria	Frequency	Acceptable Range	Information (CFR or Method 2.2 or 2.8)
Monitor Maintenance			
Inlet cleaning	1/3 mo	Cleaned	recommendation
Motor/housing gaskets	~400 hours	Inspected replaced	Method 2.2 sec 7
Blower motor brushes	400-500	Replace	Method 2.2 sec 7
Manufacturer-Recommended Maintenance	per manufacturers' SOP	per manufacturers' SOP	NA
SYSTEMATIC CRITERIA - Pb Filter Based Hi-Vol			
Data Completeness	quarterly	three -month mean (i.e., the 3-month data capture rate) ≥ 75%	Part 50 App. R, sec. 4
Reporting Units	all filters	µg/m^3 at local temperature and pressure.	Part 50 App R
Rounding Convention			
3-month arithmetic mean	quarterly	Report data to 3 decimal places (data after 3 are truncated.	Part 50 App R
Lower Detectable Limit			
Inductively Coupled Plasma – Mass Spectrometry (ICP-MS)		≤ 0.0075 µg/m3	Part 50 App G sec 2.3
Verification/Calibration Standards and Re-certifications - All standards should have multi-point certifications against NIST Traceable standards			
Flow Rate Transfer Std.	1/yr	Resolution 0.02 m^3/min ±2% reproducibility	Part 50, App B sec 7.8
Field Thermometer	1/yr	2° C resolution	Part 50, App B sec 7.5
Field Barometer	1/yr	±5 mm Hg resolution	Part 50, App B sec 7.6

Criteria	Frequency	Acceptable Range	Information (CFR or Method 2.2 or 2.8)
Analytical Standards			
Reagents (HNO_3 and HCL)		ACS reagent grade	Part 50 App G sec.6.2
Pb nitrate Pb $(NO_3)_2$		ACS reagent grade (99.0% purity)	Part 50 App G sec.6.2
Verification/Calibration			
Clock/timer Verification	4/year	5 min/mo	recommendation
Precision			
Single analyzer	1/3 mo.	Coefficient of variation (CV) $\leq$ 20%	recommendation
Single analyzer	1/ yr	CV $\leq$ 20%	recommendation
Primary Quality Assurance Org.	Annual and 3 year estimates	90% CL of CV $\leq$ 20%	Part 58, App A, sec 4.3.1
Bias			
Performance Evaluation Program (PEP)	5 audits for PQAOs with $\leq$ 5 sites 8 audits for PQAQs with $\geq$ 5 sites	95% CL Absolute bias ±15%	Part 58, App A, Sec 2.3.1

SD = standard deviation
CV = coefficient of variation

9 Training

Air monitoring personnel will receive sufficient training in their appointed duties to contribute to the gathering and reporting of complete and high quality data. DEC staff will train the local site operator.

10 Documentation and Records

Within three months after the end of the quarter is finished, the data will be finalized and entered into AQS. Quality control data such as flow checks and audits will be entered as necessary. Data will be collated into a draft report, validated by an independent member of DEC staff and finalized. The independent reviewer will certify and, if necessary, flag the data for entry into AQS.

The information from this monitoring will be used to evaluate the ambient air in Noatak for compliance with the revised lead NAAQS of 0.15 $\mu g/m^3$.

B Measurement and Data Acquisition

11 Sampling Design

11.1 Sampling Site Description

The site will be located in the same location as the site used by Tech Alaska for monitoring a few years ago. It is next to the old high school kitty corner from the IRA office (Figure 11-1).

Site coordinates are:
Latitude 67 degrees, 34.2 minutes (67.5701), North
Longitude 162 degrees, 58.1 minutes (-162.9680), West

Figure 11-1: Map of Noatak with the site location marked on it.

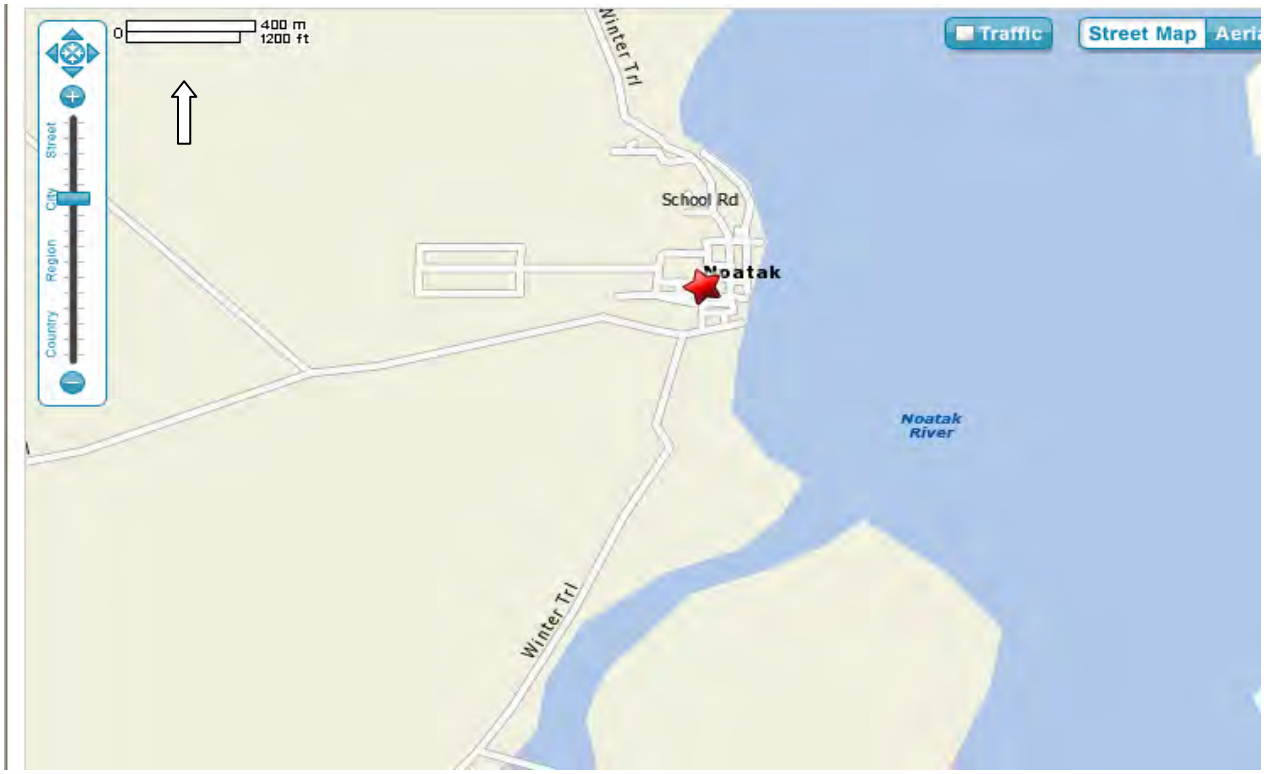


Figure 11-2: Noatak Lead Monitoring, Site Photographs

	
Looking from the North toward the Site	Looking North from the Site

Looking from the East toward the Site

Looking East from the Site

Looking from the South toward the Site

Looking South from the Site

12 Field Method

12.1 Total Suspended Particulate (TSP) High Volume (Hi-Vol) Method

The TSP Hi-Vol method collects a 24-hour sample of total suspended particulate from the atmosphere. The 24-hour sample is then analyzed (see section 14) to determine the lead (Pb) content of the collected particulate matter. The field sampler apparatus specifications and data collection procedures will be in accordance with Title 40 of the Code of Federal Regulations, Part 50, Appendix B. By means of a vacuum motor, the TSP Hi-Vol sampler draws air at a rate of 1.1 to 1.7 m^3 per minute through a glass fiber filter. Particles of 25 to 50 μm aerodynamic diameter collect on the surface of the filter for 24 hours. The sampler is equipped with a calibrated electronic mass flow controller, flow chart recorder and a calibrated elapse time indicator to determine total sample flow. All calibration standards used to assure data quality will be traceable to the National Institute of Standard and Technology (NIST).

13 Sampling Handling and Custody

13.1 Sampling Handling

The DEC EH lab will prepare and ship unexposed filters to the Noatak field operator. DEC will also ship field data sheets, sample labels, envelopes and inserts for sample recovery and return shipment. Sample collection, record keeping and handling will be performed by the Noatak site operator. Collected samples and data sheets will be mailed directly from Noatak to the DEC EH lab in Anchorage where they will be received, logged in and appropriately stored prior to analysis.

13.2 Sample Custody

Sample and sample data integrity will be strictly maintained by tracking samples and sample data throughout the sampling/analytical process: through data reduction, data validation/verification, data reporting and archiving of sample/sample data. Sample handling and custody procedures are addressed in the Standard Operating Procedures (SOP) for leading monitoring using a TSP high volume sampler presented in Appendix A.

14 Analytical Methods

The TSP sampler is a filter-based method. The analysis procedures used to determine the lead content of the particulate matter collected on the filter will include sample preparation by acid digestion and

instrumental analysis by inductively coupled plasma/mass spectrometry (ICP/MS). The lead analysis will be performed in accordance with a Federal Equivalent Method (FEM) for ICP/MS, as approved through the EPA Office of Research and Development (ORD). The DEC EH laboratory located in Anchorage will conduct the lead analysis using Method EQL-0510-191 – *"Determination of Lead in TSP by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Heated Ultrasonic Nitric and Hydrochloric Acid Filter Extraction,"* as approved May 28, 2010 in Federal Register Vol. 75, No. 103, page 30022. The DEC EH laboratory has developed specific standard operating procedures (SOP) for Method EQL-0510-191. The analytical SOP is presented in Appendix B.

15 Quality Control Requirements

Quality Control (QC) is the overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the EPA methods. QC is both corrective and proactive in establishing techniques to prevent the generation of unacceptable data, and so the policy for corrective action should be outlined.

15.1 Field Quality Control

QC for the TSP sampling conducted on-site at the sample location(s) will be performed in accordance with the EPA Federal Reference Methods at presented in the 40 CFR 50 Appendix B and the DEC SOP for lead monitoring using a TSP high-volume sampler.

15.2 Laboratory Quality Control

QC for the lead analyses at the EH lab will be performed in accordance with the EPA approved FEM and the DEC EH laboratory SOP for ICP-MS.

16 Instrument/Equipment Testing, Inspection and Maintenance

This section details the procedures used for procuring, inspecting, testing, and accepting instruments, supplies and consumables that directly or indirectly affect data quality. By having documented inspection and acceptance criteria consistency can be assured.

16.1 Procurement and Acceptance Testing of Equipment

The DEC Project Manager will be responsible for identifying field equipment needs and approving equipment purchases. The following protocol will be used in procurement of air monitoring equipment:

- **Equipment evaluation and selection:** Prior to purchase, the equipment's performance will be evaluated and other users queried in regard to the performance, dependability and ease of operation.
- **Purchase specifications:** The purchase contract will state the performance specifications that insure only equipment of the desired quality is obtained, require a one year warranty, and indicate payment will not be made until the equipment has passed an acceptance test.
- **Acceptance Testing:** Prior to payment, the equipment will be tested to ensure that it meets the requirements listed in the purchase specifications.

16.2 Maintenance of Equipment

Utilizing the specifications in EPA's "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II," preventive and remedial maintenance tasks, schedules, parts and supplies will be maintained by site operators with assistance from DEC. For monitoring systems not covered by EPA guidance, preventive and remedial maintenance tasks, schedules, parts and supplies, will be performed in accordance with manufacturer guidelines.

The site operators will maintain maintenance log sheets and immediately inform DEC staff of any irregularities at the site.

Major maintenance and repairs will be performed by the DEC AMQA technical staff.

17 Instrument/Equipment Calibration and Frequency

17.1 TSP Calibration

DEC staff will train the on-site contract operators to perform full calibrations of the TSP samplers. DEC staff will initially calibrate the TSP samplers upon installation and annually thereafter. The on-site contract operators will perform calibrations as necessary whenever the samplers malfunction and require repair or if they fail a monthly flow check or an audit check.

17.2 Laboratory Instrument Calibrations

Instrument and laboratory equipment procurement and acceptance testing is the responsibility of the DEC EH Laboratory and is detailed in laboratory quality assurance program documents. Calibration of laboratory instrumentation is the responsibility of the DEC EH lab and will follow EPA Method requirements and the laboratory SOPs for the ICP-MS determinations.

18 Inspection /Acceptance of Supplies and Consumables

Supplies and consumables will meet minimum specifications required by the specific method. Upon receipt, supplies and consumables will be inspected per method specifications prior to use.

19 Indirect Measurements

19.1 Maps

Maps are used to aid DEC in decisions regarding monitoring, including community selection and monitoring site collection. Maps selected should be the most current available for the community.

20 Data Management

20.1 Field Data Recording

The site operators will maintain site logbooks documenting operational and maintenance activities at the sampling site. The logbook will be identified with the site name, date, time, operator, and sampler identification. The log book will be used to document all site activity including quality control checks (time, precision, calibration, temperature, pressure, flow, etc.), maintenance, audits, equipment changes (vacuum motors, mass flow controllers, flow recorders, pens, charts, etc), detail any site conditions that may affect data quality and explain any missing, suspect, or invalid data. The site operators will also complete the Hi-Vol sampler field operator forms that record specific information for each sample collected including, filter number, operator initials, date installed and recovered, sample run date, elapsed time indicator start/stop, and flow indicator start/stop.

20.2 Data Transmittal, Processing and Reporting

Data transmittal occurs when data are transferred from one person or location to another or when data are copied from one form to another. Copies of logbook records and field operator forms will be emailed by the site operators to the AMQA staff on a monthly basis and reviewed by the Project Coordinator. The Project Coordinator will enter the data from field operator data forms into a sample calculation Microsoft (MS) spreadsheet. Analytical data for the gravimetric and ICP/MS determinations will be submitted from the DEC EH Lab to AMQA staff in an MS Excel spreadsheet format accompanied by a separate QA/QC report for the batch analyses. The analytical data will be logged and uploaded to the AMQA sample calculation spreadsheet to determine the final ambient air concentration for each sample. All data is logged, reviewed, edited (if warranted) and validated. The final report is presented in an electronic format (MS Excel workbook) and includes the sample calculation spreadsheet with documentation of all QA/QC standards and reference devices along with the data for all flow calibrations, monthly flow checks, and audits. The report workbook also contains a comment section which identifies and provides an explanation for any missing, suspect, or invalidated data. Method specific acceptance criteria are annotated on the spreadsheets for each QA/QC check.

20.3 Records Retention and Storage

Complete records of the monitoring project will be maintained at the DEC Air Quality Division office in Anchorage. Complete electronic files of all validated data and final reports will maintain on the DEC Air Quality Division server located in the Anchorage office. Electronic records on the server are backed up on a daily basis. Hardcopy and electronic records (including field/sampling records, laboratory analytical reports, QA/QC records, etc) will be maintained for a minimum of 5 years, following the determination of NAAQS attainment status.

C Assessments and Oversight

21 Assessments and Oversight

An assessment, for this plan, is defined as an evaluation process used to measure the performance or effectiveness of the quality system, the establishment of the monitoring network and sites, and various measurement phases of the data operation.

21.1 Performance Audits

During this project, utilizing the procedures and calculations specified in 40 CFR 58, Appendix A, “Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS)”, the DEC QA Officer or another qualified person that is not the site operator will audit each ambient air quality monitor

at least once per quarter. Results of audit findings will be reported to the Director of Air Quality with copies to the project manager and AMQA program manager.

21.2 Systems Audit

The systems audit is an on-site review and inspection of the entire ambient air monitoring project to assess its compliance with the approved Quality Assurance Project Plan. A limited systems audit will be performed once during the first year of this project to ensure proper instrument siting and monitoring operations. To provide uniformity in the evaluation, the criteria and procedures identified in EPA's Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Section 2.0.11 are recommended. Results of audit findings will be reported to the Director of Air Quality with copies to the project manager and AMQA program manager.

21.3 Corrective Action and Response

Should audit findings identify a problem(s) with the monitoring project, the QA officer will notify the AMQA project officer and appropriate on-hand operations staff of the problem and recommend appropriate corrective actions. It is incumbent on the project manager to ensure corrective actions are taken in a timely manner and notify the QA officer in writing the following info:

- identify the problem,
- specific actions taken to correct the problem,
- procedures implemented to limit problem recurring, and
- flag/invalidate affected data.

The Quality Assurance Officer will make the final recommendation whether to accept or reject the data.

22 Report to Management

The lead monitoring program is expected to operate for at least three years, pending demonstration of attainment status. Within a given year, data quality objectives will be met at the monitoring site if samples are collected on a 1 in 6 day sampling schedule with at least a 75% quarterly completeness. Monitoring data should be submitted to the Air Quality System (AQS) within 90 days of completing a data collection quarter.

D Data Validation and Usability

23 Validation and Verification Methods

The following data review, validation and verification processes will provide for data that meets the Project's quality assurance criteria.

Data review and validation for this project is a three (3) tier process. The first tier review is with the contract site operators by their strict adherence with SOPs for field sampling, equipment repair/maintenance, record keeping, sample handling/shipping, and data transfer. The site contractors are responsible for noting any conditions or equipment function issues which may affect data quality and communicating with the project coordinator on corrective actions.

The DEC lead monitoring project coordinator will be responsible for the second tier of data review and validation. The project coordinator will compile and enter the field and analytical monitoring data to calculate initial results. The project coordinator will then generate an initial report including the data values, quality control flow check results, calibration results, standard reference device traceability, and QA audit results. The project coordinator will note any non - conformance with QA/QC requirements and flag the data with a comment of explanation.

The DEC project manager or other senior AMQA staff as designated by the project manager will be responsible for the third tier of data validation. They will verify that samples were collected according to the method, required documentation was recorded, QC checks were conducted, etc. The DEC monitoring manager will collaborate with the project coordinator and the QA officer to reconcile any suspect data or data recommended for invalidation. The DEC project manager will direct final revisions, and approve the data for uploading to the AQS data base.

In order for the data to be considered valid the following conditions must be satisfied:

- The air monitoring instrumentation must be calibrated and operated according to standard operating procedures that have been approved by the Quality Assurance Officer.
- All QA/QC checks performed in support of field and analytical measurements must conform to criteria as specified in Table 8-1 of this plan.
- The data must be accompanied by back up documentation which meet the specifications outlined in Sections 12 - 14 of this Plan, and be identified with respect to station name, station number, date, time, operator, instrument identification, parameter, scale and units.
- The data must be bracketed by documented quality control which substantiate that it meets the criteria in Table 8-1 of this plan.

Data which is reviewed and found to satisfy these criteria will be considered valid. Data that does not, will be flagged in accordance with AQS quality assurance qualifier codes.

23.1 Data Processing and Reporting

Data processing and reporting begins with receipt of data from the field. Upon receipt, the project coordinator will review and scan the field data forms into electronic memory for file storage. At the end of the quarter, the project coordinator will manually enter the field data into the MS Excel sample calculation workbook and upload the analytical results to the workbook from the laboratory reports. As part of quality review, spreadsheet calculations are routinely spot checked to assure the integrity of the equations. After all the data is logged, reviewed, edited and validated, the project coordinator will append the workbook with additional spreadsheets including all the supporting quarterly QA/QC data. Once the report workbook is completed by the project coordinator, it is forwarded to the AMQA monitoring manager or his/her designee for the third tier review and validation. The workbook spreadsheets are annotated with relevant QA/QC criteria to facilitate easy comparison of the data with method requirements.

Once the final revision of the report is validated and approved by the AMQA monitoring manger, the report is saved as a write-protected document on the Anchorage AQ server. The project coordinator uploads the data to the U.S. EPA's Air Quality System (AQS) Database (http://www.epa.gov/ttn/airs/airsaqs) within 90 days of the end of the quarter.

24 Reconciliation with User Requirements

The Quality Assurance Officer will prepare a project Air Monitoring Data Quality Assessment Report that describes data quality in terms of precision, accuracy and data completeness.

25 References Cited

Code of Federal Regulation, Title 40, Parts 50, 53, and 58

Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, EPA-454/B-08-003, December, 2008

"Determination of Lead Concentration in TSP by Inductively Coupled Plasma Mass Spectrometry with Heated Ultrasonic Nitric and Hydrochloric Acid Filter Extraction,", Federal Equivalent Method (FEM) EQL-0510-191, http://www.epa.gov/ttn/amtic/files/ambient/pb/EQL-0510-191.pdf , and as approved in Federal Register, vol. 75, No. 103, page 30022, May 28, 2010.

Appendix A – Standard Operating Procedures (SOP) for Lead Monitoring Using a TSP High Volume Sampler

Standard Operating Procedures
for
Lead Monitoring Using a TSP High-Volume Sampler

State of Alaska Department of Environmental Conservation
Division of Air Quality
Air Monitoring and Quality Assurance Section
619 East Ship Creek Ave., Suite 249
Anchorage, AK 99501

	Alaska Department of Environmental Conservation Division of Air Quality Air Monitoring & Quality Assurance 619 East Ship Creek Ave. Suite 249 Anchorage, Alaska 99501	
	Title: Standard Operating Procedures for Lead Monitoring Using a TSP High-Volume Sampler	
	Version: 1.0	Revision Dated: June 2011

Procedure Plan Acknowledge and Approval

Title: Standard Operating Procedures for Lead Monitoring Using a TSP High-Volume Sampler

This manual documents the standard operating procedures of the Alaska Department of Environmental Conservation (ADEC), Division of Air Quality for the determination of lead in ambient air quality samples of total suspended particulate matter. This manual for lead monitoring is hereby recommended for approval and commits the ADEC Air Monitoring and Quality Assurance (AMQA) section to follow the procedures described within.

Alaska Department of Environmental Conservation
Air Quality Division
Air Monitoring and Quality Assurance Section:

Barbara Trost, Program Manager - AMQA **Date:** 6/8/2011

Daniel Fremgen, Quality Assurance Officer - AMQA **Date:** 6/8/2011

Robert L. Morgan, Project Manager - AMQA **Date:** 6/8/2011

	Alaska Department of Environmental Conservation Division of Air Quality Air Monitoring & Quality Assurance 619 East Ship Creek Ave. Suite 249 Anchorage, Alaska 99501	
	Title: Standard Operating Procedures for Lead Monitoring Using a TSP High-Volume Sampler	
	Version: 1.0	Revision Dated: June 8, 2011

Contents

1 General Information

1.1 Introduction

To comply with the November 12, 2008 revision to the National Ambient Air Quality Standard (NAAQS) for lead, the Alaska Department of Environmental Conservation (ADEC), Division of Air Quality, Air Monitoring & Quality Assurance Section established a lead monitoring program. The purpose of this Standard Operating Procedure (SOP) document is to describe the field procedures used to sample for lead in Total Suspended Particulate (TSP) that is particulate matter which is suspended in ambient air and has a mean aerodynamic diameter of up to 25 to 50 micrometers. The objective of this SOP is to formalize procedures to insure the consistent collection of samples and data that meet the data quality required by the revised NAAQS for lead. This SOP manual will encompass all aspects of field operations associated with the collection of the TSP samples. 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.

The procedures used to analyze TSP samples for the presence of lead will be documented in a separate SOP prepared by the ADEC Environmental Health (EH) Laboratory.

1.2 Principles of Operation

An electrical blower motor draws ambient air into the air 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:

- a shelter housing which is constructed of aluminum;
- a rectangular gabled roof inlet that directs air flow onto an 8-inch by 10-inch glass-fiber filter,
- a filter cassette that holds the filter in place on a support that draws air through the filter down and through an attached blower motor;
- a flow controller which electronic senses the flow and controls the motor speed to maintain a constant volumetric flow rate;
- a chart recorder to document any variation in flow rate,
- a mechanical day timer to automatically start and stop sampler operation; and
- an elapse timer to document the sampling period.

The flow controller and blower motor are calibrated with a certified critical orifice flow device (a flow transfer standard) to insure that the required volumetric flow rate is maintained between 1.1 to 1.7 cubic meters of air per minute (m^3/min). In English units the flow rate is from 39 to 60 cubic feet per minute (ft^3/min). The calibrated elapse time indicator provides an accurate time in minutes for each sample run. The flow data, elapsed time, and data for temperature and pressure are recorded for each sample run day and the data are later used to calculate the total volume of air sampled. Samples are collected according to an EPA schedule and 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 average.

1.3 Safety Precautions

Only properly trained personnel should perform the TSP Hi Vol filter changes, installation, testing, operation, maintenance or calibration. As with all monitoring equipment, precautions should be taken when working around electricity, power tools and elevated platforms. Repair should be done by properly trained service personnel. Proper protective clothing is also essential when working in wintertime Arctic conditions.

1.4 Interferences/Limitations

The absolute accuracy of the method is undefined because of the complex nature of atmospheric particulate matter and the difficulty in determining the "true" particulate matter concentration. The TSP sampler does not have as sophisticated of a mechanism for distinguishing the largest particle size measured as the Hi-Vol sampler used to measure particulate matter equal to or less than 10 micrometers (PM10).

2 Installation

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, and other items as discussed in Section 6
- 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 for work in wintertime Arctic conditions

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 ADEC project 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 the Alaska Lead Monitoring Program.

Samplers will be mounted on a safe, suitable monitoring platform according to the following guidelines:

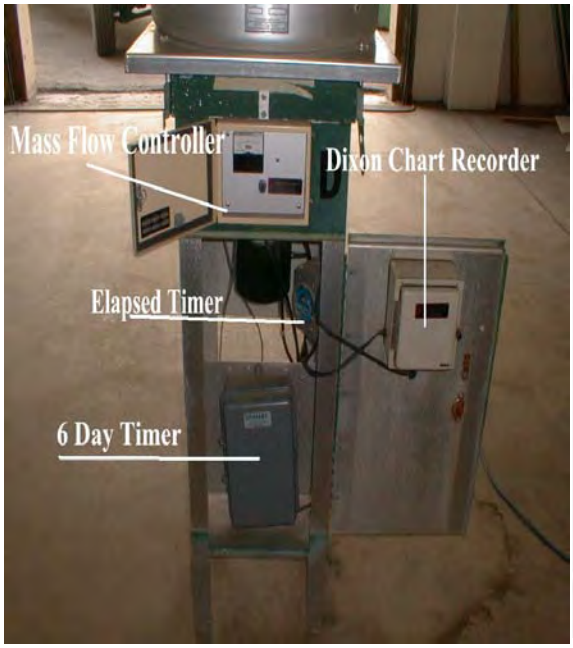
- TSP samplers must be exposed to unobstructed airflow in all directions.
- The sampler inlet must be vertically placed between 2 and 15 meters (m) above ground level.
- If a sampler is collocated with other samplers, the minimum spacing between sampler inlets is as follows:
    - If collocated with other PM10 or PM2.5 or other low-volume samplers (flow rate < 16.7 L/min), maintain a minimum distance between sampler inlets of 2 m.
    - If collocated with other total suspended particulate samplers (TSP) or other high-volume samplers (flow > 1.13 m^3/min), maintain a minimum distance between sampler inlets of 2 m.
- The site security is to be maintained with a locked fence.

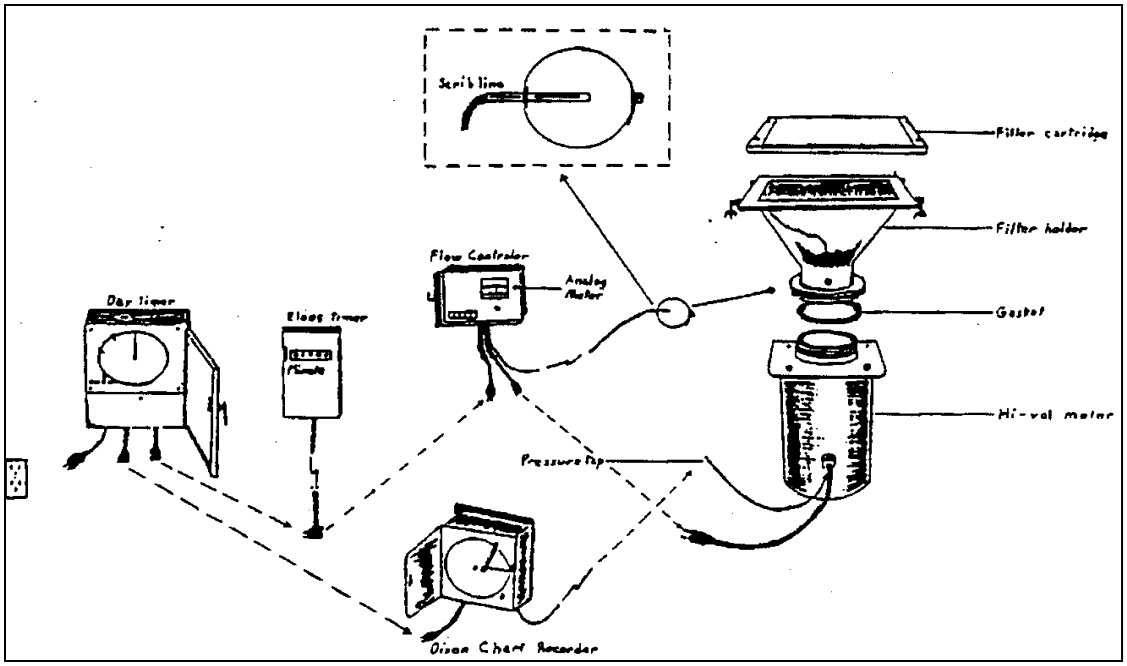
2.4 Sampler Installation

The location of the Hi-Vol sampler will have already been identified as part of the monitoring site selection and is discussed in the 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 that sure the shelter housing and the platform are sufficiently secured to withstand high wind conditions.

2.5 **Sampler Assembly**

Figure 2-1 provides photographs and an illustration of the sampler components and electrical configuration.





Peripheral Equipment

Hi-Vol systems may be operated in several different configurations. The following describes component function and typical configuration used in Hi-Vol Sampler systems. See the illustration in Figure 2-1 for the typical connection sequence.

Day timer: Alaska uses six day or seven-day mechanical timers. These timing devices have been reliable in a wide range of weather conditions. The day timer is the first piece of equipment installed in the Hi-Vol sampler (see figure 2-1) and, providing electrical power to the other sampler components and the timer automatically starts and stops sampler operation. Unless otherwise documented, the day-timer is installed and operated following the manufacturer's operating instructions. *NOTE: The electrical GUARD plate provides a safeguard against electrical shock. If the plate is to be removed, unplug the unit. Upon completion of maintenance, always re-install the safety guard plate.*

Elapsed timer: The elapsed timer is the indicating device that accurately records the time period that electrical power is provided to the sampler components. Unless otherwise documented, the timer is installed and operated according to the manufacturer's instructions. The elapse timer plugs inline between the day-timer and the flow controller. *NOTE: A mechanical or electrical failure down-line of the elapsed time indicator will have no effect on the elapsed time indicator. This can result in inaccurate time measurements and inaccurate daily particle loading calculations.*

Flow Controller: Alaska monitoring network uses flow controllers, which senses the air flowing through the sampler and controls the voltage to the blower motor. The flow controller is calibrated with critical orifice flow device which will allow for calculation of the air flow during the sample run. The flow controller consists of a meter display which indicates air flow and a screw to adjust air flow by varying the voltage to the blower-motor. Installation and operation, unless otherwise documented, will follow the manufacturer's operating instructions. *NOTE: When installing a flow controller probe into the filter holder, make sure the probe scribe mark is facing up as shown in the Figure 2-1 illustration.*

Blower-Motor & Housing: The blower-motor is the driving force of the sampling system. The blower-motor 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 throat of the filter support assembly and attached to the blower-motor housing. The air flow passes through the blow-motor and out an orifice plate in the bottom of the blower-motor housing. The housing has a pressure tap to determine differential pressure which is a measurement used in the calibration of the flow controller and blower-motor to calculate volumetric air flow.

Flow indicators: The Dickson chart recorder provides a visual record of the air flow (differential pressure) through the Hi-Vol sampler during the sample period that is a pen trace on a circular chart for period of operation. The chart trace indicates if the sample flow rate was consistent and uninterrupted, showing if the power failed or it the blower- motor malfunctioned during the 24-hour sample run. The Dickson chart recorder is connected to the pressure tap on blower-motor housing with rubber tubing. The chart movement is electrical and plugs into the day timer.

To assemble, connect the filter support to the Hi-Vol blower motor assembly and ensure the gasket is in place for proper seal. Place the Hi-Vol motor assembly in the Hi-Vol shelter. In most cases, the peripheral equipment is mounted in the Hi-Vol shelter at the factory. If, however, an update is required, the second photograph in Figure 2-1 shows a typical configuration. Attach the electric connections and pressure connections as configured in the Figure 2-1 illustration.

3 TSP Calibration Procedures

Calibrations are to be performed: when the sampler is initially installed at the sampling location prior to initial operation; when the sampler malfunctions or the blower motor fails; and when the sampler fails any monthly flow check or audit flow check. Sections 3.1 through 3.3 describe procedures multipoint flows calibrations and single point flow verification checks.

Following established EPA methods and procedures, all calibration devices (i.e. temperature, pressure and flow) must be certified against NIST-traceable standards at least once per year.

Calibration of a Hi-Vol sampler refers to establishing a mathematical relationship between the sampler's flow rate as indicated by the meter on the flow controller and a known flow as determined by a critical orifice flow device. This calibration provides an accurate measurement of the sampler flow rate during a sample run.

3.1 List of Tools, Equipment and Materials

- Logbooks , TSP-Field Calibration form (Figure 3-1), TSP-Monthly 1-Point Flow Check form (Figure 3-3), and ink pens
- Flow calibration orifice capable of measuring volumetric flow rates from 1.0 to 1.8 m^3/min, which is certified as traceable to the National Institute of Standards and Technology (NIST)
- Digital manometer with traceability to NIST and a measurement range of (at least) 0 to 12 inches H_2O inches to the nearest ±0.05 inches *Note: Digital manometers are typically certified for accurate operation to a minimum temperature of degrees Celsius (°C).* For cold weather conditions (< 0 °C) use

a liquid U-tube manometer with a measurement range of (at least) 0-12 inches H_2O inches to the nearest ± 0.1 inches. The manometer fluid is a 50/50 mixed solution of water and antifreeze which will not freeze until approximately -40°C.

- Leak-free flexible tubing of sufficient length to connect the pressure tap on the flow calibrator to the manometer
- A thermometer capable of measuring temperature over the range of -40 to 50°C to the nearest ± 2°C and traceability to NIST. *Note: In rural village locations, temperature data from the local National Weather Service airport flight station may be used.*
- A barometer capable of measuring barometric pressure over the range of 500 to 800 millimeters (mm) of mercury (Hg) ± 5 mm and traceable to NIST. *Note: In rural village locations, barometric pressure (altimeter) data from the local National Weather Service airport flight station will be used.*
- Personal computer with MS Excel® flow calculation spreadsheet (Figure 3-2) or hand calculator
- Personal protective equipment and winter clothing/footwear for work in wintertime Arctic conditions.

3.2 Leak check

1. Take the critical orifice flow calibrator out of its black case and attach the flow calibrator onto the filter cassette screen. Be sure that the flow calibrator is centered over the filter so the gaskets will properly seal and tighten the screws sealing the flow calibrator onto the filter support screen.
2. Turn on the blower motor via the switch on the mechanical day-timer and allow 5 minutes for warm up of the blower motor. Using duct tape, prepare a patch to place over the inlet to the flow transfer standard. Place the duct tape patch over the inlet of the flow calibrator and make sure the inlet is completely sealed preventing any air flow through the flow calibrator and into the blower motor. Place your thumb over the pressure tap where the manometer tubing attaches to the flow calibrator. With the other hand, check the bottom of the blower-motor housing to check for any air flow passing out of the orifice in the bottom of the housing.
3. If there is a slight leak, tighten the screw knobs which hold the flow calibrator 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.
4. Turn off the sampler. The leak check is now completed.

Caution: Running the sampler for too long (1 – 2 minutes) 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.

3.3 **Multi-point calibration**

A multi-point flow calibration must be performed upon initial installation and at least once per year thereafter. In addition, the multi-point calibration must be performed whenever a single-point flow verification check indicates that the sampler flow deviates from the flow calibration device by more than ± 4% or following routine maintenance or repairs. The multi-point calibration is performed as follows:

1. After the leak check is done, leave the flow calibrator on the sampler, removing the duct tape over the inlet. Turn on the digital manometer and re-zero it using the dial between the two ports or zero the liquid manometer.
2. Inspect the tubing for any cracks and unusual wear which may cause a leak. Replace as necessary.
3. Connect one end of the tubing to the negative port of the manometer. Connect the other end to the pressure port of the flow calibrator.
4. Turn on the sampler and allow it to warm up to normal operating temperature (about 5 minutes).
5. While the sampler is warming up, record the following information on the TSP-Field Calibration form: (Figure 3-1)
    a. Date and name of operator doing the calibration
    b. Sampler ID
    c. Ambient temperature (in °C)
    d. Ambient barometric pressure (in inches of mercury, Hg)
    e. Flow calibrator serial number, certification date
6. Once the sampler is properly warmed up, the approximate set point needs to be established.
    Note: The ADEC has established a spreadsheet program in Microsoft (MS) Excel which can calculate set points with some basic information from the user. A laptop computer is necessary while out in the field otherwise the formulas are listed below and in the section titled "Calibration Calculations" for hand calculations. (Figure 3 – 2)
7. To calculate an approximate set point when flow is at 1.3 m^3/min you need to use the following equation:

Equation 3-1

$$\text{Estimated set point, in inches} = \frac{[(1.3 \times m_{act}) + b_{act}]^2 \times P_{amb}}{T_{amb}}$$

Where: m_{act} = slope (from calibration sheet in calibration kit)
b_{act} = intercept (from calibration data sheet in calibration kit)
P_{amb} = ambient pressure, inches Hg converted to mm Hg)
T_{amb} = ambient temperature (degrees Kelvin, °K = °C + 273)

8. Input the data recorded in Step 5 into the calculation spreadsheet.
9. Using a small screwdriver adjust the flow controller screw so the manometer reads what you calculated above in equation 3-1.(e.g. 5.2 inches of water)
10. Record the reading from the flow controller meter which the is "Indicated Flow" (e.g. when the manometer reads 5.2 the meter reading or "Indicated Flow" may read 41).
11. Choose two additional calibration points above and two additional calibration points below "Indicate Flow" from the step above. (e.g. 38, 40, 41, 42, and 44).
12. Adjust the flow rate using the flow controller screw for each of the four addition calibration points recording the manometer readings and the corresponding flow meter reading ("Indicated Flow").
13. In the MS Excel calculation spreadsheet input the manometer reading under the column for "Hd" and the flow meter readings under the column for "Indicated flow". Enter this data for each of the four additional calibration points.
14. The spreadsheet will automatically calculate a linear regression of the flow rate calibration creating a slope and intercept equation (y = mx + b) to represent flow rate of the "Indicated Flow". Regression equations will be generated for flow rates at standard conditions (Q_{std}) for 25° C, 760 mm Hg and at actual site condition (Qact) for ambient temperature and pressure. *Note: The resulting linear regression equation will be specifically for this TSP sampler for this date forward.* The linear regression spreadsheet also creates a graphic plot of the calibration. Check to see that the correlation coefficient is greater than 0.99 and visually to see if any points are questionable. This should be done in the field as the calibration procedure is occurring so adjustments can be made immediately. Save the spreadsheet file identifying the sampler ID and date.
15. Repeat the last two steps for any data points that are questionable on the plot. Running additional calibration points at differing flow rates is encouraged to improve the precision of the calibration.

Figure 3-1 TSP – Field Calibration Form

TSP - Field Calibration Form

Date: _____ Operator Name: _____

Site ID: Noatak Airport Weather Data: Noatak Phone #: 485-2203

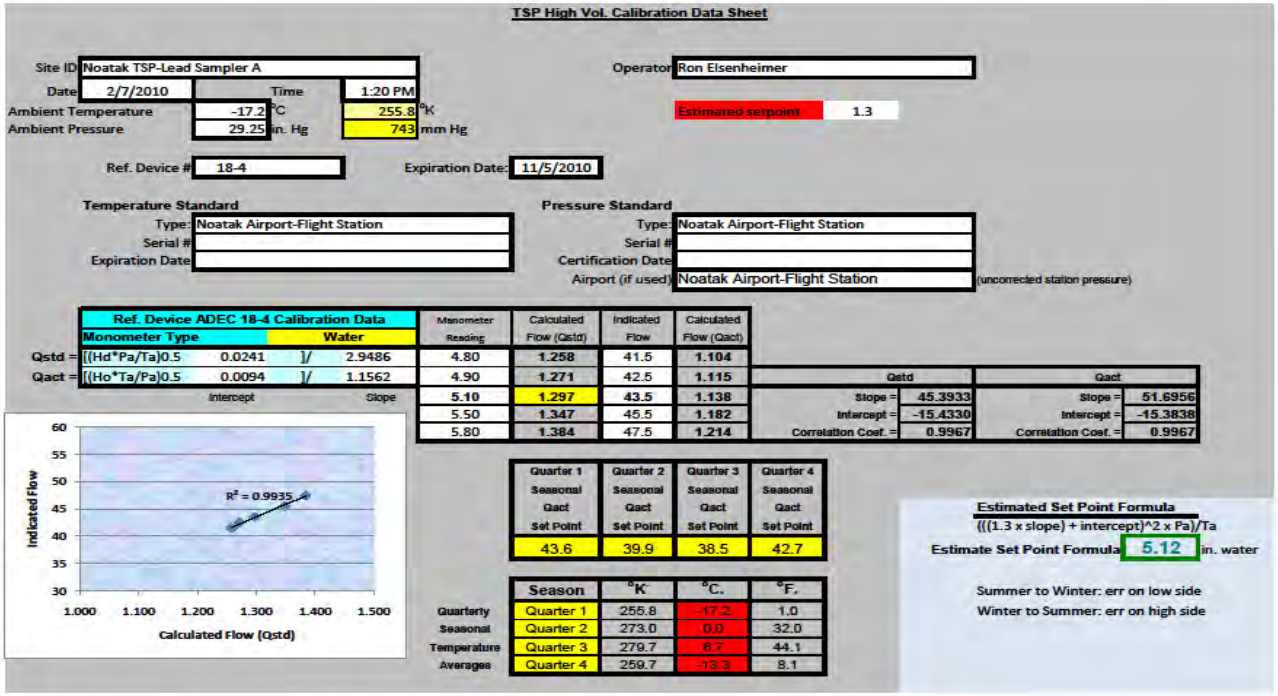
Sampler ID: _____ Ambient Temperature: _____

Ref Device #: _____ Ambient Pressure: _____

Ref Device Certification Date: _____

Flow Checks:	Manometer Readings: Left Side	+	Right Side	=	Total Pressure (inches H2O)	Flow Meter Reading:
Point 1		+		=		
Point 2		+		=		
Point 3		+		=		
Point 4		+		=		
Point 5		+		=		

Figure 3 – 2 TSP Calibration Calculation Spreadsheet



TSP High Vol. Calibration Data Sheet

Site ID: Noatak TSP-Lead Sampler A
Operator: Ron Elsenheimer
Date: 2/7/2010 Time: 1:20 PM
Ambient Temperature: -17.2 °C 255.8 °K
Ambient Pressure: 29.25 in. Hg 743 mm Hg
Estimated setpoint: 1.3
Ref. Device #: 18-4 Expiration Date: 11/5/2010

Temperature Standard
Type: Noatak Airport-Flight Station
Serial #:
Expiration Date:

Pressure Standard
Type: Noatak Airport-Flight Station
Serial #:
Certification Date:
Airport (if used): Noatak Airport-Flight Station (uncorrected station pressure)

Ref. Device ADEC 18-4 Calibration Data
Monometer Type: Water

Qstd = [(Hd*Pa/Ta)0.5 0.0241]/ 2.9486
Qact = [(Ho*Ta/Pa)0.5 0.0094]/ 1.1562
Intercept Slope

Monometer Reading	Calculated Flow (Qstd)	Indicated Flow	Calculated Flow (Qact)
4.80	1.258	41.5	1.104
4.90	1.271	42.5	1.115
5.10	1.297	43.5	1.138
5.50	1.347	45.5	1.182
5.80	1.384	47.5	1.214

Qstd		Qact	
Slope =	45.3933	Slope =	51.6956
Intercept =	-15.4330	Intercept =	-15.3838
Correlation Coef. =	0.9967	Correlation Coef. =	0.9967

Quarter 1 Seasonal Qact Set Point	Quarter 2 Seasonal Qact Set Point	Quarter 3 Seasonal Qact Set Point	Quarter 4 Seasonal Qact Set Point
43.6	39.9	38.5	42.7

Season	°K	°C.	°F.
Quarterly Seasonal Temperature Averages	Quarter 1	255.8	-17.2	1.0
	Quarter 2	273.0	0.0	32.0
	Quarter 3	279.7	6.7	44.1
	Quarter 4	259.7	-13.3	8.1

Estimated Set Point Formula
(((1.3 x slope) + intercept)^2 x Pa)/Ta
Estimate Set Point Formula: 5.12 in. water

Summer to Winter: err on low side
Winter to Summer: err on high side

16. Once you are assured that precision is established, a set point can be determined using the following formula. This calculation is also performed in the spreadsheet by entering quarterly seasonal temperature averages as determined by local meteorological records.

Equation 3-2

Set point = $(m_{Qstd} \times 1.3 \times 760 / T_S \times 298/760) + b_{Qst}$

Where: m_{Qstd} = slope (calculated based on 5 calibration points)
T_S = seasonal temperature in ° K
b_{Qstd} = intercept (calculated based on 5 calibration points

17. Once the set point has been calculated using the formula above, adjust the flow to the seasonal setpoint.
18. Make sure all sections of the calibration sheet are filled out including ambient (or seasonal in some cases) temperature, ambient (or seasonal) barometric pressure, manometer readings, flow recordings, etc…
19. Turn off the sampler. Remove the tubing from the calibration orifice and manometer. Remove the flow calibrator.
Note: The flow calibrator should be stored in its case with the wing nuts as loose as possible otherwise damage to the gasket may occur if the wing nuts are too tight.
20. Set up the monitor for the next sampling run.

3.4 Single-point Flow Verification Check

A single-point flow verification check must be performed at least every month. The monthly flow check is performed as follows:

1. After the leak check is done, leave the calibration orifice on the sampler, removing the duct tape over the inlet. Turn on the digital manometer and re-zero it using the dial between the two ports. If using a liquid manometer leak the manometer and zero the movable scale.
2. Inspect the tubing for any cracks and unusual wear. Replace as necessary.
3. Connect one end of the tubing to the negative port of the manometer. Connect the other end to the pressure port of the calibration orifice.
4. Turn on the sampler and allow it to warm up to normal operating temperature (about 5 minutes).
5. While the sampler is warming up, record the following information on the calibration data sheet:
    a. Location, date, time and name of individual doing the calibration
    b. Sampler serial number, model and designation
    c. Ambient temperature (°C)

d. Ambient barometric pressure (mm Hg)
e. Calibration orifice serial number, calibration, date and actual flow conditions calibration relationship.

6. When the motor is warm, record the reading from the flow controller meter and from the manometer (eg. when the manometer reads 5.2 the meter will read 41). *Note: Take a reading by looking at the meter needle straight on eye level. If the reading is made while looking down on the MFC of at an angle, the reading will not be correct.*
7. Using the calibration regression equation from the most recent valid 5-point calibration calculate the sampler flow rate.
8. Using the calibration regression equation from the calibration orifice calculate the orifice flow rate.
9. Calculate the percent difference between the sampler flow rate as compared to the orifice flow rate. (see equation 3-3) If the percent difference is ≤7% the flow check is within QC criteria and all of the data from the previous flow check to date is valid. If the percent difference is > 7%, then corrective action is required; troubleshoot, repair, and perform a new 5-point calibration.

Equation 3-3

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4 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)
- Blank Dickson (circular) flow chart
- Ink Stamp to label Dickson chart
- Logbook and ink pens
- Filter cassette
- Filter envelops & Filter Mailers

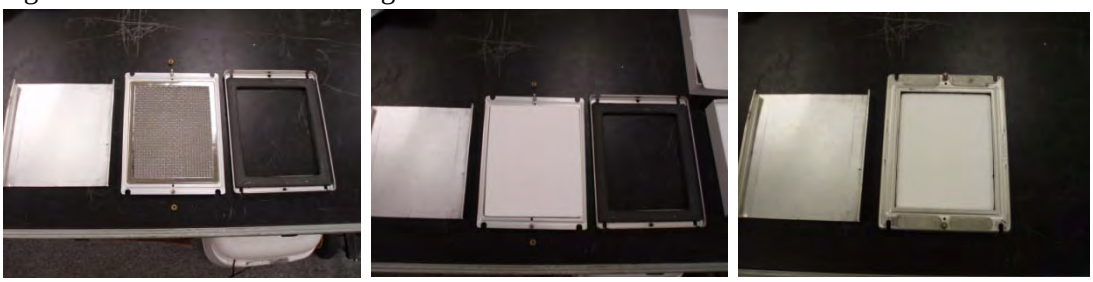
4.2 Preparatory Procedure for Filter Changes

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 and label the Dickson chart in preparation for installing the sample filters on-site.

1. Review the sampling calendar to confirm the run date.
2. Before setting up the samples, clean and dry the working surface on any dust or other contaminants.
3. Label the Dickson (circular) chart so sample data can be recorded. On the back (non-grid) side of the chart, use the provided ink stamp to label the chart. (See Figure 4-1)
4. Open a filter cassette by removing the protective cover and unscrewing the thumbscrew which secure the top of the frame to the screen support. (See Figure 4-2)
5. After clean 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.
6. On the back of the Dickson chart record the sampler ID next to "SAMPLER:" (e.g. Noatak B), filter ID number next to "FILTER#:", and the next sample "RUN DAY:" as confirmed from the EPA sample calendar.
7. Also record the filter number and run date on the log sheet. (See Figure 4-3 TSP Sample-Log Sheet)
8. 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.

Figure 4-1 Dickson Chart

Figure 4-2 Filter Cassette Loading



4.3 On-site Installation and Setup for 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 log sheets.

1. Open the main door to sampler; open the doors to the 6-day timer, the Dickson chart recorder and the mass flow controller
2. Record current time on the log sheet in the comment section. (Note: Statewide air monitoring will be conducted based on Alaska Standard Time year round. The samplers run time will not be adjusted for daylight savings time.)
3. Compare the current time to the time on the 6-day timer, if the time on the 6-day timer is off more than ½ hour you will need to adjust the 6-day timer to the current time. Make a note on the log sheet of any adjustment.
4. Check the Dickson chart – Did the sampler run properly? Did the sampler run for 24 hours (complete red circle)? Remove the chart and note any problems in the log book and call DEC if a problem is noted.
5. Manually turn on the sampler. After the mass flow controller stabilizes (approximately 5 minutes) check the flow meter reading. On the back of the Dickson chart, under the "POST" column, record the current date "DATE" and the flow meter reading next to "FLOW." Record this same information in the log book. Turn sampler off.
6. Record the stop time from the elapse timer in the filter log sheet and on the back of the Dickson chart.
7. Unlatch and open the top of the sampler.
8. 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.
9. 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.
10. Be sure to remove the metal cover from the new filter cassette and use it to cover the used filter cassette.
11. Close and latch the top of the sampler
12. Manually turn the sampler on

13. After the mass flow controller stabilizes (approximately 5 minutes), check flow meter reading. On the back of the new Dickson chart, under the "PRE" column, record the flow meter reading "FLOW" and the current date "DATE." Record this same information on the log sheet.
14. Also record the current time shown on the elapsed timer (sample start time) on the back of the Dickson chart in the "pre" column and on the log sheet.
15. Install the "new" Dickson chart in the Dickson Chart Recorder. (Grid side out with the chart installed so that it runs midnight to midnight). Make sure the little tab in the center is not ripped!
16. Before leaving the monitoring site make sure the 6-day timer is set to "run" on the correct day and close all doors to the sampler and lock the security fence.

Figure 4-3 TSP Lead Sample – Log Sheet

Alaska
Department of Environmental Conservation
Air Quality Division

Lead Monitoring
TSP Sample - Log Sheet

Monitoring Site: Village of Noatak
Sampler A

Sample Run Date:	Filter ID #:	Filter Installation/Removal				Flow Meter Readings		Elapse Timer		
		Installed Date:	Operator Initials:	Removed Date:	Operator Initials:	Pre	Post	Start	Stop	Total Min.
Comments:										
Comments:										
Comments:										
Comments:										
Comments:										
Field Blank						na	na	na	na	na

Page # _____

na= not applicable for monthly field blanks

4.4 Recovery Procedures for Post Sampling

Upon return to work area and before recovering the samples insure the area is still clean and dust free.

1. Review the information recorded on the chart and TSP Sample - Log Sheet. Confirm the information recorded is consistent and correct.
2. Gather the necessary materials to recover and store the samples: clean disposable gloves, protective paper sheets (used to separate the clean filters in the box) and envelops.
3. After cleaning hands and putting on clean disposable gloves remove the protective 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.
4. Remove the filter from the cassette and carefully fold the filter lengthwise so the exposed (dirty) side is folded inward onto itself to prevent loss of particulate matter. 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.
5. Make sure the filter ID number on the chart and log sheet matches the filter number on the filter.
6. Place the filter into the protective insert that comes with the envelop.
7. Insert the filter and protective insert into the envelop.
8. Insert the Dickson chart into the envelop between the protective insert and the inside wall of the envelop.
9. Close the envelop by tucking the flap inside the lip of the envelop. (Do not lick and seal the adhesive.)
10. Label the exterior of the envelop with the site name, sampler ID, filter ID number, and sample run date.
11. Store the sample envelope in a secure dust free place until ready for shipment to DEC.

4.5 Sample Handling and Shipping Procedures

After recovering the final samples of the month, the field site operators will prepare and ship all the filters collected for the month to DEC for processing and laboratory analysis.

1. Inventory the filters by comparing the filter ID numbers and sample run dates shown on the envelops with the information record on the TSP Field – Log Sheet to make sure all the samples are accounted for and ready for shipment.
2. Make copies of the TSP Field – Log Sheets for the month. Include copies of the log sheets with the shipment.
3. The filter samples and log sheet copies will be shipped through the US Postal Service. DEC will provide Flat Rate mailing containers for the shipment.

4. Mail the filter samples to:

   ADEC
   DIVISION OF AIR QUALITY
   619 EAST SHIP CREEK DRIVE STE 249
   ANCHORAGE AK 99501

   ATTN: LEAD MONITORING PROJECT MANAGER

5. Upon arrival at DEC the filters will be logged and inspected for damage.
6. The Dickson charts and field log sheets will be reviewed and filed in preparation for data review and report development.
7. The filter samples information will be entered onto Chain of Custody forms for submittal to the DEC Environmental Health (EH) Laboratory. (see Figure 4-4)
8. Upon transfer of the sample from the Air Quality to the EH laboratory, a copy of the Chain of Custody form will be retained for the lead monitoring file.

5 Sampler Maintenance

The EPA Reference Method for TSP-lead specifies a large number of maintenance items to ensure that the collected samples meet the TSP-lead monitoring program Data Quality Objectives. These maintenance items are listed below, grouped by the frequency of required maintenance.

All maintenance activities should be logged into a station logbook making sure to record on the log sheets the operator ID, specific sites, dates, and times of servicing or maintenance. Log books should be kept in a dry, secure but convenient location.

The cleaning frequencies of the air sampler will vary greatly from site to site. Location will play the biggest part as to how detailed and how often the cleaning process will take place. Keeping the air sampler clean insures the quality of the sample.

The high volume shelter should be routinely inspected and maintained. Power cords should be checked for crimps, cracks, frayed or exposed wiring each sample day. Power cords with cracked cord casings should be taped with electrical tape and replaced as soon as possible. Do not allow power cords to be immersed in water; if necessary raise the power cords above the ground by taping them to the legs of the shelter.

5.1 **Mass Flow Sensor**

The mass flow control sensor should operate without failure. The probe however, should be checked and cleaned using alcohol and canned air. Be extra careful in cleaning this sensor as it will break if mishandled. Make sure when re-installing the flow sensor that the probe is aligned properly. (Figure 5-1)

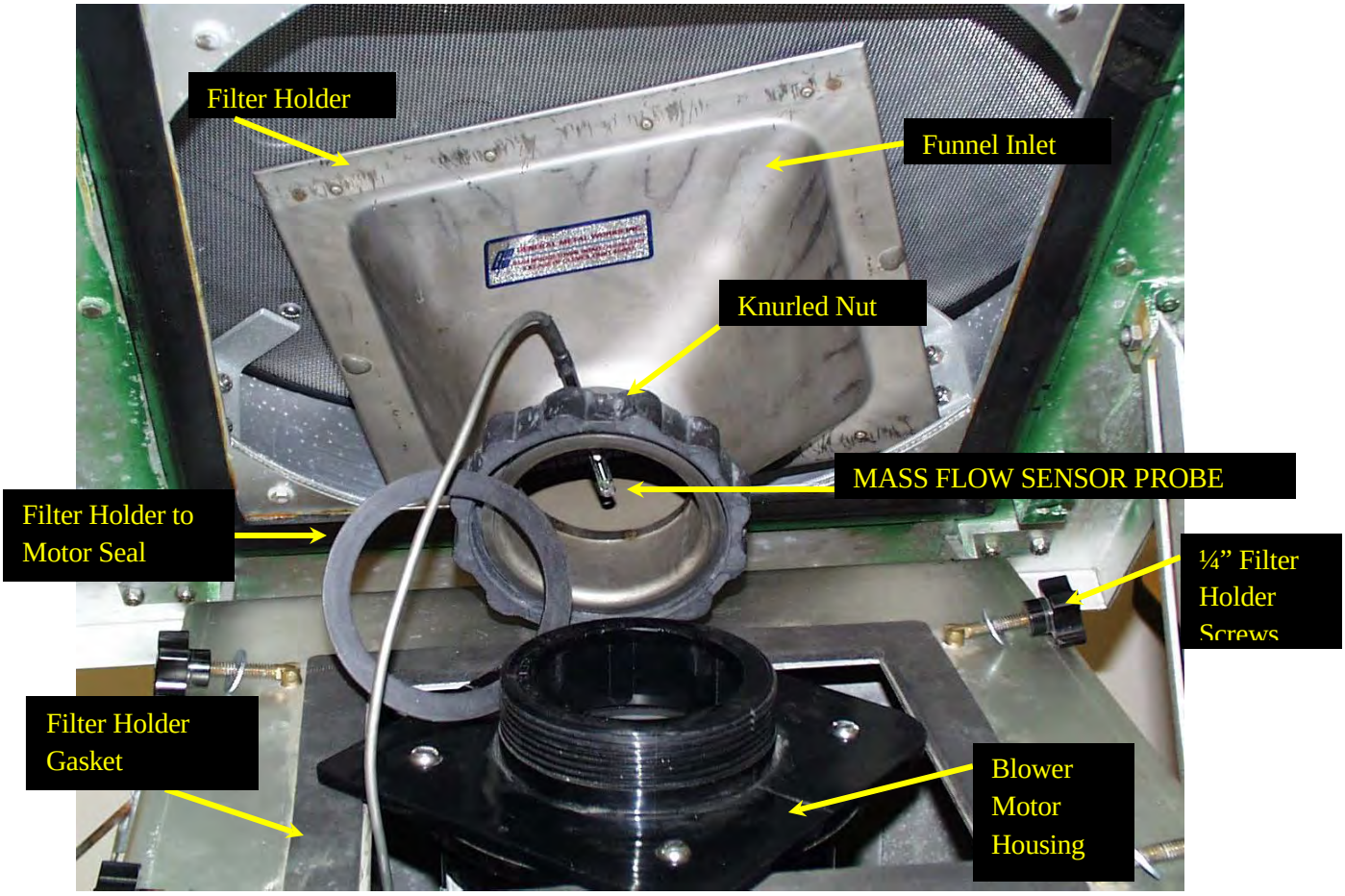


Figure 5-1 Mass Flow Controller Sensor

The filter cartridge or cassette used to support the sample filter and rubber gaskets should be checked for physical damage, compression or degradation each time a filter is installed. Replace the gasket as necessary. (Figure 5-2)

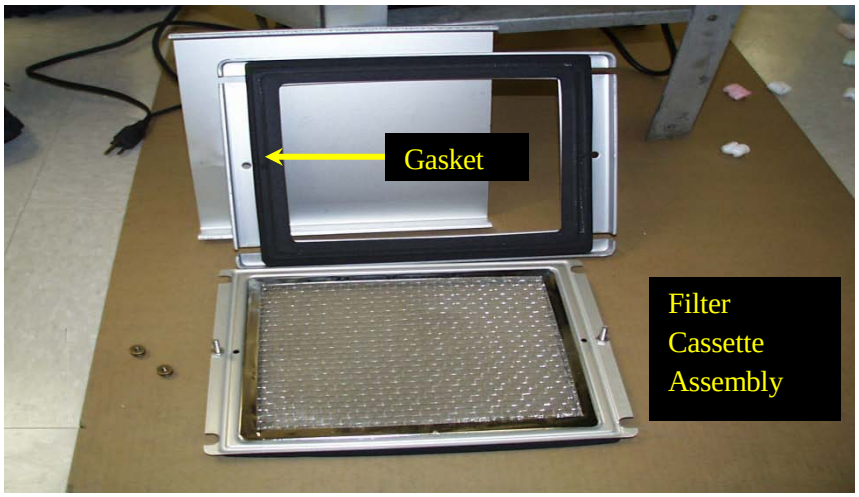


Figure 5-2 Filter Cassette

Look for signs of degradation anywhere in the system. Vibrations can wear holes in the funnel inlet if it is not installed correctly. A/C power cords crack and need replacement over time. Clean upper portion of the sampler to prevent particulate buildup which could eventually be transferred to filters. This is especially important if particulates build up on the stainless steel screen.

5.2 Dickson Chart Recorder

The circular pen recorder should read zero on the chart paper with the motor turned off. It can be adjusted for zero using the offset adjust on the front panel and by adjusting the level arm length behind the front panel. Motors can be replaced in the event the chart stops turning. Pens typically last for a couple of months and should be changed regularly.

Inspect the pneumatic hoses and connections for cracks or other damage that could cause leaks. Hoses become frozen and brittle; they should generally be replaced once per year. (Figure 5-3)

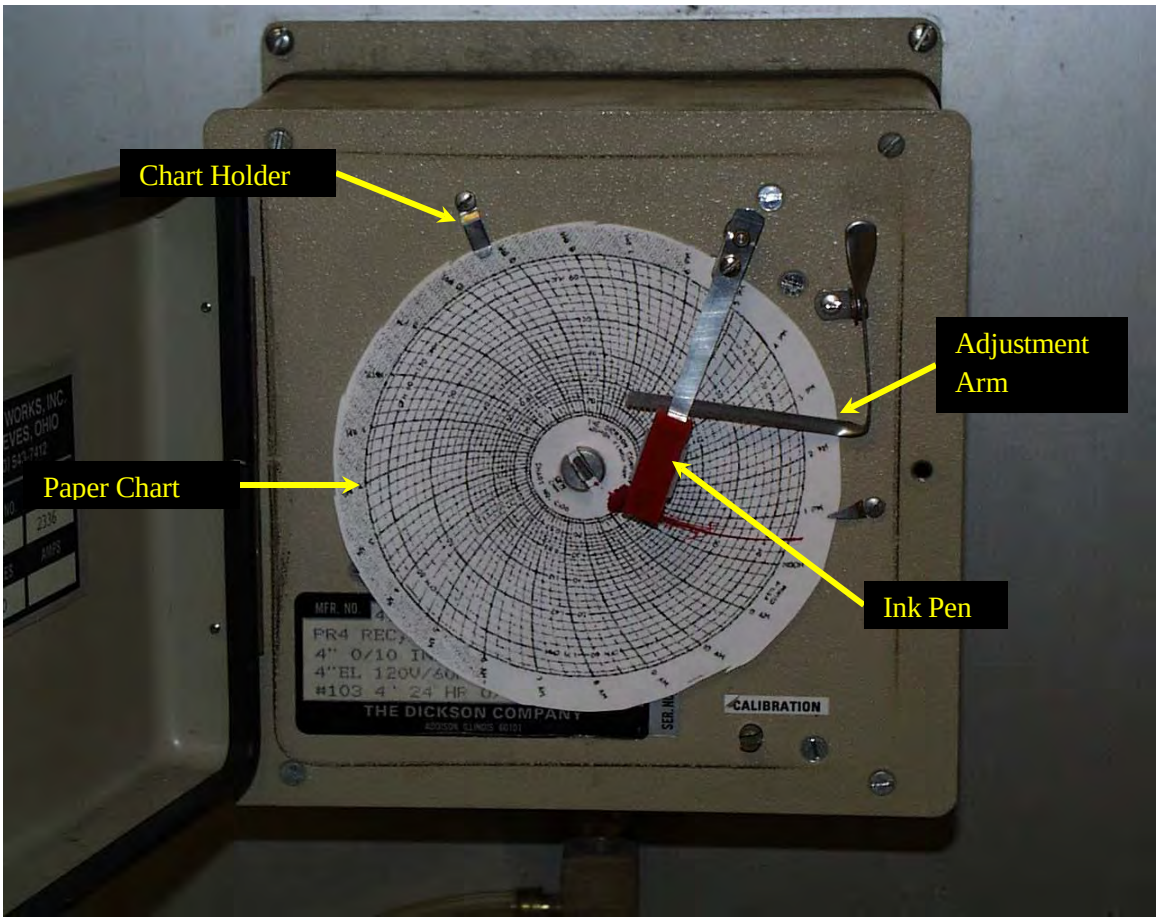


Figure 5-3 Dickson Flow Chart Recorder

All maintenance activities should be logged into a station logbook making sure to record on the log sheets the specific site, dates, and times of servicing or maintenance. Log books should be kept in a convenient, dry, safe location.

5.3 Motor Maintenance

TSP motors are durable and have a long life <u>if properly cared for and maintained.</u> If not maintained they will be a constant problem in the sampling program. The maintenance requirements are; inspecting and replacing the neoprene gaskets routinely, replacing the motor's carbon brushes, cleaning the motor's rotor or bearings.

CAUTION: *The individual technician needs to have a clear understanding of electrical and mechanical applications and his or her ability to perform such maintenance. High voltage is present as well as moving mechanical parts. Possible injury or death could occur if the proper steps are not taken.*

Before starting any maintenance on the air sampler ensure all power is disconnected from its main source of power.

Open the shelter door. Disconnect the rubber hose that connects the motor housing pressure tap to the Dickson chart recorder. (Figure 5-4)

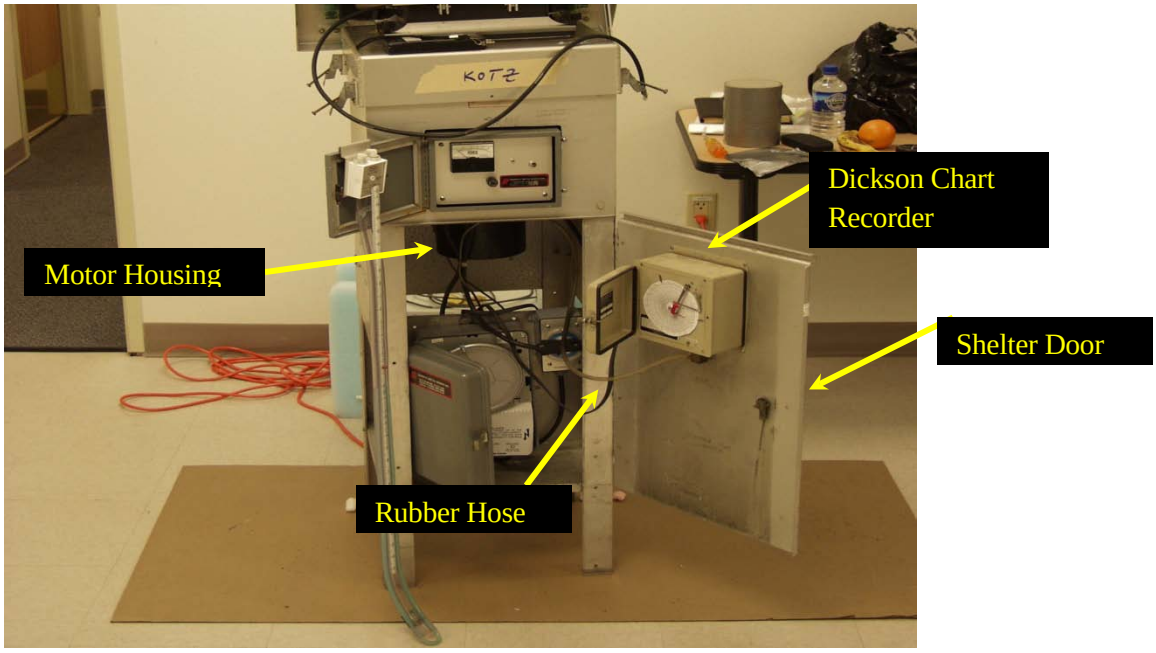


Figure 5-4

Grab the filter screen and carefully lift the motor housing assembly upward through the opening in the top of the base. As the top of the motor housing appears rotate the screen 45 degrees setting the four corners of the motor housing on the sides of the opening. With one hand, loosen the large, black, knurled, threaded nut off the motor housing (a strap wrench helps loosen the nut) and set the funnel inlet out of the way in order to bring the motor housing up through the top of the opening. Make sure the blower motor power cord is disconnected. (Figure 1)

Bring the blower motor assembly up through the opening and place on a working surface. (Figure 5-5 &5-6)

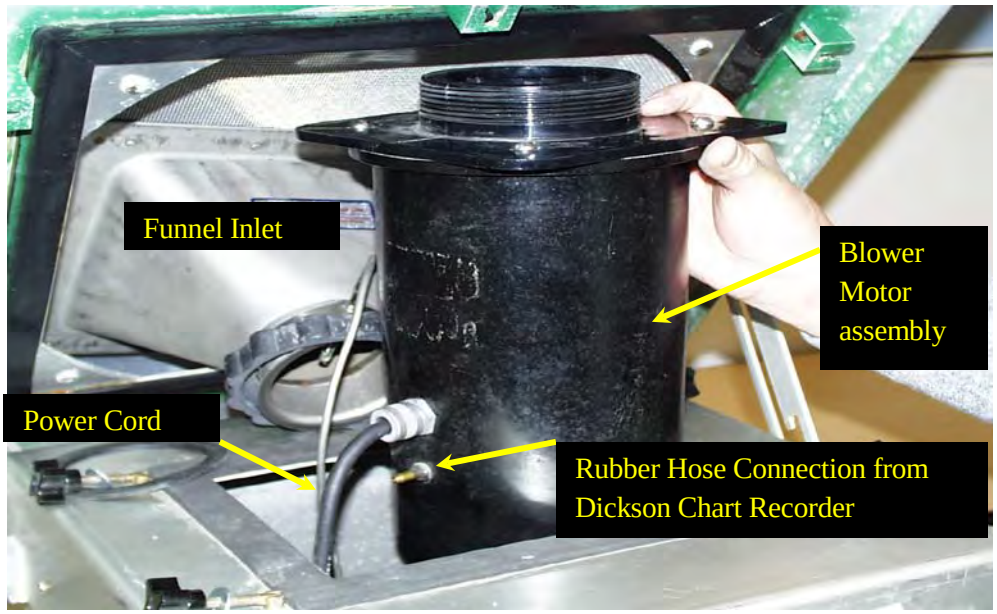


Figure 5-5 Blower Motor Maintenance

Figure 5-6 Blower Motor Maintenance

Remove the four ¼ -inch flathead screws from the top of the blower housing. (Figure 5-7 & 5-8)

Figure 5-7 Blower Motor Maintenance

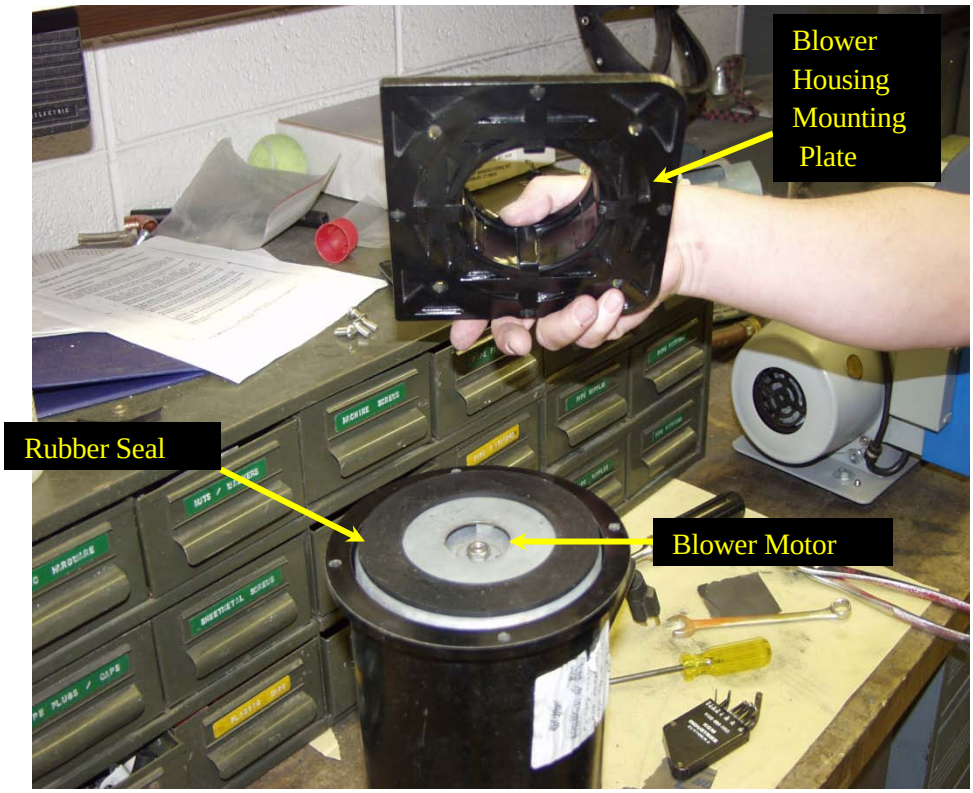


Figure 5-8 Blower Motor Maintenance

Loosen the power cord tightening nut off of the housing to allow the blower motor to be taken out of the housing. Bring motor up out of the housing and disconnect the wiring nuts from the motor. Check rubber seal and replace if needed. (Figures 5-9 through 5-11)

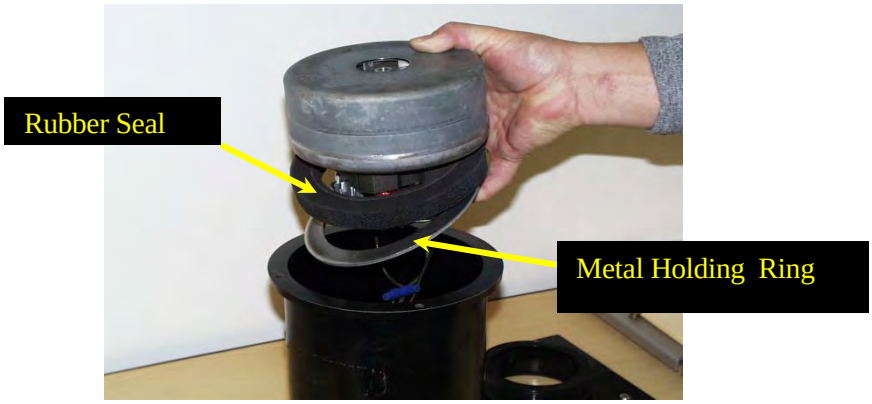


Figure 5-9 Blower Motor Maintenance

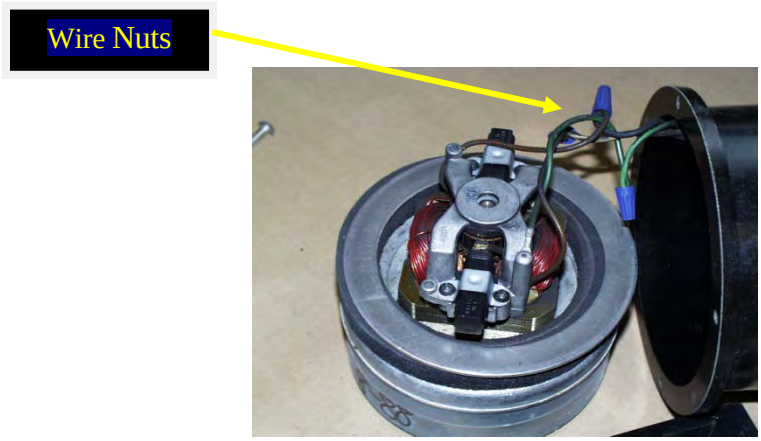


Figure 5-10 Blower Motor Maintenance

Figure 5-11 Blower Motor Maintenance

The carbon brushes must be replaced after 600 hours of run time. They should be replaced either after a quarterly audit or before calibration of the air sampler for the next quarter.

To replace the brushes, stand the motor up to expose the brushes, motor rotor and motor windings. Using a small flat screw driver gently pry the brush electrical connection out of the brushes. If the brushes have been loosened from their mounting plates this may be a difficult task to accomplish. Once this is done remove the 2 Philips head retaining screws from the brush retainer holders and remove the carbon brushes. (Figures 5-12 & 5-13)

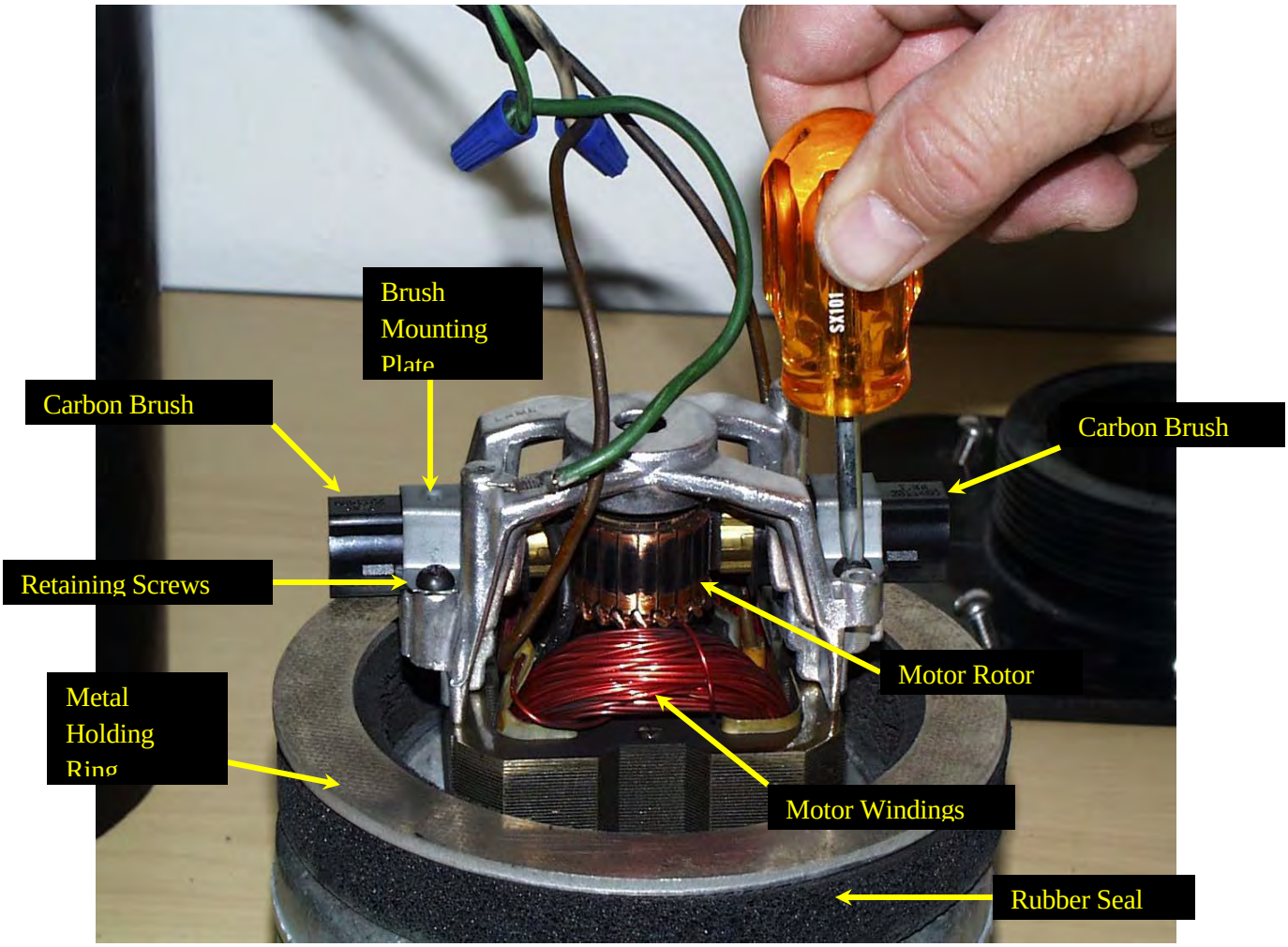


Figure 12 Blower Motor Maintenance

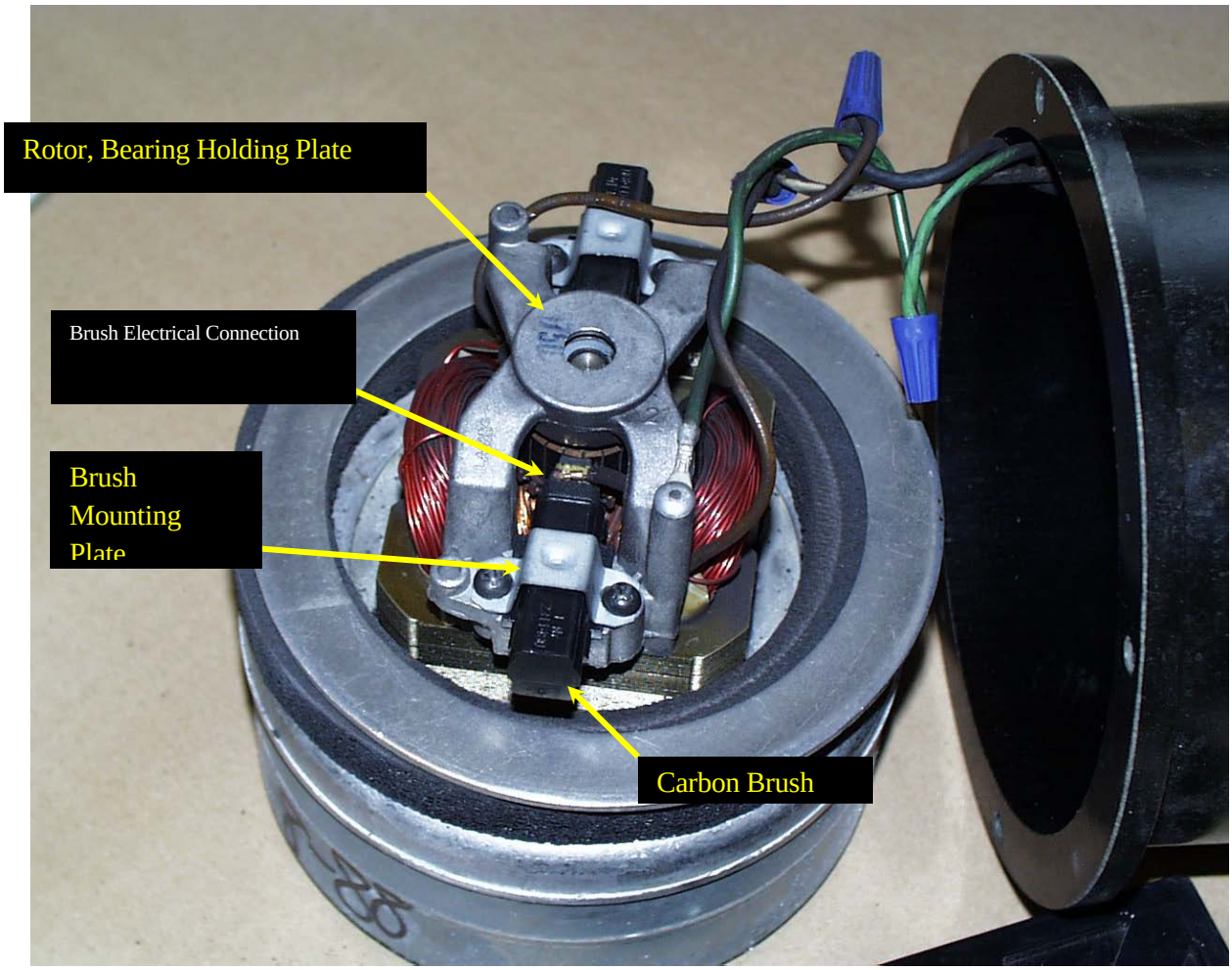


Figure 13 Blower Motor Maintenance

Install new brushes in reverse order of disassembly. Discard old brushes.

Reassemble motor in reverse order. For proper motor performance and maximum brush life expectancy it is necessary to seat or "Break-in" the brushes. Using a variable power supply apply approximately 50 % or half voltage to the motor for at least thirty minutes. A brush miser can also be ordered from the factory to accomplish this.

Caution: Direct application of full voltage after changing brushes will cause arcing, pitting, surging and reduce overall life of the motor.

A leak test is recommended after brush changes or any blower motor maintenance.

6 Performance Audits

Performance audits are designed to evaluate the accuracy of the sampler in measuring the key parameters involved in collecting a valid TSP lead sample. Performance audits should be conducted in accordance with the frequency and personnel requirements specified in the QAPP. In general, however, performance audits are to be conducted by personnel not directly involved in the routine operation, data processing or reporting of the TSP-lead monitoring network. Furthermore, performance audits are to be conducted using transfer standards different from those used in the routine calibration and operation of the sampler. It is acceptable for the audit transfer standards to be certified against the same local primary standard as the routine transfer standards.

6.1 Audit Criteria Table

Parameter	Tolerance	Frequency	Comments
TSP Hi-Volume Sampler design flow rate audit	Percent Difference ≤ 7 % within 1.1 to 1.7 m^3/min	Once per quarter	
Flow rate audit	Percent Difference ≤ 10 %	Semi-annually (2/year)	

6.2 Performance Audit Reporting

Upon completion of the performance audit, immediately inform the site operator of the audit results so that corrective action may be taken, if necessary. An audit report should be prepared for submittal to the ADEC-AMQA within 30 days of the completion of the audit.

7 Laboratory Analysis for Lead

The DEC Environmental Health Laboratory will analyze the TSP samples for lead content using inductively coupled plasma-mass spectrometry (ICP-MS). All procedures applicable to the sample preparation and quantitative determination of lead in the TSP filter samples will be conducted in accordance with the Standard Operating Procedure titled *Standard Operating Procedure – Determination of Lead ion TSP by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Heated Ultrasonic Nitric and Hydrochloric Acid Filter Extraction.*

The DEC EH laboratory will analyze the TSP lead samples on a quarterly basis. The resulting data package will reviewed in accordance with QA/QC procedures as specified in the above SOP. The data package provide by the EH lab to DEC Air Quality will consist of a data table generated by the Laboratory Information Management System (LIMS) and a QA/QC report.

8 Data Calculations and Validation

The following subsections describe the routine procedures used to calculate 24-hour TSP lead concentrations and to validate individual samples.

8.1 Calibration Calculations

To calculate an estimated set point use following equations:

1. Estimated set point, in inches = $\frac{[(1.3 \times m_{act}) + b_{act}]^2 \times P_{amb}}{T_{amb}}$

Where: m_{act} = slope (from calibration sheet in calibration kit)
B_{act} = intercept (from calibration data sheet in calibration kit)
P_{amb} = ambient pressure (mm hg)
T_{amb} = ambient temperature (°K)

2. Seasonal Temperature Set point = $(m_{Qstd} \times 1.3 \times 760 / T_S \times 298/760) + b_{Qst}$

Where: m_{Qstd} = slope (calculated based on 5 calibration points

8.2 Data Calculations

To calculate sampler flow rates, sample volumes, and sample concentrations use the following equations:

Where:
Sample flow rate in cubic meter/minute corrected to 760 mm Hg and 298° K
Avg. Indicated Flow = average of pre and post indicated flow

Intercept Qstd. = intercept from the calibration spreadsheet
Slope Qstd. = slope from the calibration spreadsheet

Where:
Sample flow rate in cubic meter/minute at local ambient site conditions in mm Hg and ° K
Avg. Ambient Temp = average temperature for the sample run date in ° K
Avg. Ambient Press = average pressure for the sample run date in mm Hg

Where
Sample Volume Std. = cubic meters corrected to 760 mm Hg and 298 ° K
Sample time = minutes

Where:
Sample Volume Act. = cubic meters at local ambient site conditions in mm Hg and ° K
Sample time = minutes

Where:
Pb Conc. Std. = total lead in micrograms per cubic meters corrected to 760 mm Hg and 298 ° K
Total Pb/strip (as determined by the laboratory analysis) = the total Pb derived from one ¾ by 8-inch strip as cut from the 8 by 10-inch glass-fiber sample filter.
A = area correction to equate the area of the filter strip analyzed to the total area of the exposed sample filter. The exposed area of the sample filter is 7 by 9 inches or 63square inches. The area of the analyzed ¾ by 8 inch filter strip is 5.25 square inches. (A = 63 in^2/5.25 in^2 = 12)

Where:

Pb Conc. Act. = total lead in micrograms per cubic meters at local ambient site conditions in mm Hg and ° K

Total Pb/strip (as determined by the laboratory analysis) = the total Pb derived from one ¾ by 8-inch strip as cut from the 8 by 10-inch glass-fiber sample filter.

A = area correction to equate the area of the filter strip analyzed to the total area of the exposed sample filter. The exposed area of the sample filter is 7 by 9 inches or 63square inches. The area of the analyzed ¾ by 8 inch filter strip is 5.25 square inches. (A = 63 in^2/5.25 in^2 = 12)

An MS Excel data calculation spreadsheet will be used for all the above calculations.

8.3 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.

1. Verify that the run data from the hi-vol are within the following limits:
   Average volumetric flow rate = 1.1 to 1.7 m^3/min.
   Total sample duration = 24 hours (1440 minute) ± 60 minutes
2. Verify that the sample dates are correct and that all QC procedures for calibration and monthly flow rate checks were completed and validated.
3. Verify that the site technician did not flag the sample as "questionable" on the Field Data Log.
4. Verify that free-form notes on the Field Data Log do not indicate an invalid sample.
5. Verify that the sample was not damaged during shipping and was transferred to the DEC EH Lab with appropriate chain of custody.
6. 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 AMQA air quality specialist assigned as project coordinator. The data will then receive a second-level review and validation by a senior air quality specialist.

8.4 Quarterly Reports

A lead TSP quarterly report is a report that puts all of the operational information recorded from each calendar quarter into one file for review, processing, and data submission. The lead quarterly reports must include copies of all of the data used to calculate $\mu g/m^3$.

The quarterly report is a MS Excel® workbook with individual worksheets as labels tabs. The quarterly report will consist of the following individual worksheets:

- Comments tab with explanation of data invalidation or qualifiers
- Quarterly Data tab with all parameters and equations used to calculate TSP Lead in $\mu g/m^3$.
- Flow Orifice Calibration spreadsheet and certification of traceability
- TSP calibration spreadsheet
- Monthly Flow Rate Check spreadsheet
- Copies of TSP Field Log Sheets and Dickson Charts
- QA Performance Audit spreadsheet
- Calculation of Network Precision

9 References Cited

Code of Federal Regulation, Title 40, Parts 50, 53, and 58

Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, EPA-454/B-08-003, December, 2008

Appendix B – Standard Operating Procedures (SOP) for Lead Analyses

	Alaska Department of Environmental Conservation Environmental Health Laboratory 5251 Dr. MLK Jr. Ave., Anchorage, AK 99507 Standard Operating Procedure
SOP Title:	**Determination of Lead in TSP by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Heated Ultrasonic Nitric and Hydrochloric Acid Filter Extraction**
Version/Revision Date:	11-2010
Supersedes:	02-2010
Reference Method:	EPA 3050B, EPA 6020A, EQL-0510-191, QP-07, QP-10, QP-13, QP-20, WI-CHEM-020

STANDARD OPERATING PROCEDURE

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Periodic Review:

Signature	**Title**	**Date**

If printed on purple paper or present on the EHL Intranet, this is a controlled document with the most recent updated revision. Uncontrolled copies should be viewed as obsolete and verified upon each use. This SOP should be reviewed on an annual basis. If the SOP is found adequate, the SOP cover page is signed and dated for documenting the review. If major revisions are needed, a new revision will be released with a new signature page. The above signatures reflect periodic review of the Standard Operating Procedure.

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1.0 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) is for the Determination of Lead in TSP by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Heated Ultrasonic Nitric and Hydrochloric Acid Filter Extraction.

NOTE: This SOP is based on USEPA's Office of Air Quality Planning and Standards (OAQPS), EPA Contract No. EP-D-08-047 specifically Method EQL-0510-191, and Office of Solid Waste (SW-846) Method 6020A – Inductively Coupled Plasma Mass Spectrometry.[3] Wording in certain sections of this SOP are paraphrased or taken directly from Method 6020A.

1.1 Inductively coupled plasma mass spectrometry (ICP-MS) is applicable for the sub-µg/mL determination of lead in a wide variety of matrices. This procedure describes a method for the acid extraction of lead in particulate matter collected on high-volume, glass fiber, TSP filters and measurement of the extracted lead using ICP-MS.

1.2 Due to variations in the isotopic abundance of lead, the value for total lead must be based on the sum of the signal intensities for isotopic masses, 206, 207, and 208. Most instrument software packages are able to sum the primary isotope signal intensities automatically.

1.3 ICP-MS requires the use of an internal standard. ^{115}Ge, ^{165}In, and ^{209}Tb are used as internal standards for the determination of lead in this procedure.

1.4 Use of this method is restricted to use by, or under supervision of, properly trained and experienced personnel. Requirements include training and experience in inorganic sample preparation including acid extraction and also knowledge in the recognition and in the correction of spectral, chemical and physical interference in ICP-MS. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 This method describes the acid extraction of lead in particulate matter collected on 8 x 10 inch, glass fiber, ambient air filters using a high-volume TSP sampling device as described in 40 CFR Part 50, Appendix B[5] with subsequent measurement of the dissolved lead by ICP-MS.

2.2 This SOP includes one extraction method. In this method, a solution of nitric and hydrochloric acids are added to the filter samples in plastic tubes and the tubes are placed in a heated ultrasonic bath for one hour to facilitate the extraction of lead. Following ultrasonication, the samples are brought to a final volume of 40mL, vortex mixed, and centrifuged prior to aliquots being taken for ICP-MS analysis.

2.3 Calibration standards and check standards are prepared to matrix match the acid composition of the samples. ICP-MS analysis is then performed. With this method, the samples are first aspirated and the aerosol thus created is transported by a flow of argon gas into the plasma torch. The ions produced (e.g., Pb^{+1}) in the plasma are extracted via a differentially-pumped vacuum interface and are separated on the basis of their mass-to-charge ratio. The ions are quantified by a channel electron multiplier and the signal collected is processed by the instrument's software. Interferences must be assessed and corrected for, if present.

3.0 DEFINITIONS

CCB – Continuing Calibration Blank
CCV – Continuing Calibration Verification
CRM – Certified Reference Material
ICB – Initial Calibration Blank
ICP-MS - Inductively Coupled Plasma Mass Spectrometer
ICV - Initial Calibration Verification
IDC – Initial Demonstration of Capability
LCS –Laboratory Control Sample (Blank Spike)
LLCCV – Lower Level Continuing Calibration Verification, equal to the low level of the calibration range, serves as LLICV and LLCV
LLCV – Lower Level Calibration Verification, equal to the low level of the calibration range, serves as LLICV and LLCCV
LLICV – Lower Level Initial Calibration Verification, equal to the low level of the calibration range, serves as LLCV and LLCCV
MB - Method Blank
MDL – Method Detection Limit
MS/MSD – Matrix Spike/ Matrix Spike Duplicate
MSDS – Material Safety Data Sheet
NIST – National Institute of Standards and Technology
Pb – Elemental or ionic lead
RL – Reporting Limit
RPD – Relative Percent Difference

RSD – Relative Standard Deviation
SOP – Standard Operating Procedure
SRM – NIST Standard Reference Material
USEPA – U.S. Environmental Protection Agency
v/v – volume to volume ratio

4.0 INTERFERENCES

4.1 Reagents, glassware, plasticware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All containers and reagents specified in this SOP have been checked for contamination. A certificate of analysis is supplied by the vendors for each container and/or reagent received.

4.2 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z) as the species of interest. There are no species found in ambient air that will result in isobaric interference with the three lead isotopes, 206, 207, and 208, being measured. Polyatomic interferences occur when two or more elements combine to form an ion with the same mass-to-charge ratio as the isotope being measured. Lead is not subject to interference from common polyatomic ions and no correction is required.

4.3 The distribution of lead isotopes is not constant. The analysis of total lead is based on the summation of signal intensities for the isotopic masses 206, 207, and 208. The instrument software will perform this summation.

5.0 HEALTH AND SAFETY CAUTIONS

5.1 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to the skin. Use these reagents in a hood, and if eye and skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection when working with these reagents. The component of this procedure requiring the greatest care is nitric acid (HNO_3). Nitric acid is a strong, corrosive, oxidizing agent that requires protection of the eyes, skin, and clothing. Items to be worn during use of this reagent include:

- Laboratory Coat
- Safety goggles (or safety glasses with side shields)
- Acid resistant rubber gloves

If acid is spilled onto the skin, wash immediately with large amounts of water. Medical attention is not required unless the burn appears to be significant. Even after washing and drying, the nitric acid may leave the skin slightly brown in color.

5.2 Lead (Pb) salts and lead solutions are toxic. Great care must be taken to ensure that samples and standards are handled properly; wash hands thoroughly after handling.

5.3 Care must be taken when using the ultrasonic bath as it is capable of causing mild burns.

5.4 Consult the Chemical Hygiene Plan (QP-07) for EHL safety policies and waste procedures.

6.0 EQUIPMENT AND SUPPLIES

6.1 Apparatus

6.1.1 Perkin Elmer ELAN DRC II Inductively Coupled Plasma Mass Spectrometer (ICP-MS), or equivalent.

6.1.2 Heated ultrasonic bath capable of maintaining a temperature of 80°C; Crest Ultrasonics, Model CP1200, or equivalent.

6.1.3 Laboratory centrifuge, Thermo Jouan G4i, or equivalent.

6.1.4 Vortex mixer, Fisher miniRoto S56, or equivalent.

6.2 Materials and Supplies

6.2.1 Argon Gas, high purity, 99.99%, or better, Air Liquide, or equivalent.

6.2.2 Water, Milli-Q, Type I, or equivalent.

6.2.3 50mL Falcon centrifuge tube, or equivalent.

6.2.4 Pipettes, Eppendorf Reference or equivalent, various dispensing volumes.

6.2.5 Tweezers, plastic, or equivalent.

6.2.6 Scissors, ceramic, or equivalent.

6.2.7 Cylinders, graduated, various volumes.

6.2.8 Flasks, volumetric, class A, various volumes.

6.2.9 High purity glass fiber filters, or equivalent.

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7.0 REAGENTS AND STANDARDS

7.1 Nitric acid, concentrated, Ultrex II Ultrapure Reagent HNO_3, JT Baker or equivalent.

7.2 1% (v/v) nitric acid solution

Add 20mL concentrated HNO_3 solution to approximately 1500mL Milli-Q (reagent) water. Mix well and dilute to 2000mL with Milli-Q (reagent) water.

7.3 Hydrochloric acid, concentrated, Ultrex II Ultrapure Reagent HCl, J.T. Baker or equivalent.

7.4 Reagent water – ASTM Type I, Milli-Q water or equivalent.

7.5 Extraction solution (1.03M HNO_3 + 2.23M HCl).

Prepare by adding 500mL of Milli-Q water to a 1000mL flask, adding 64.4mL of concentrated HNO_3 and 182mL of concentrated HCl, shaking to mix, allowing solution to cool, diluting to volume with reagent water, and inverting several times to mix. This is used for preparing the ICV, CCV, LCS, and calibration standards.

7.6 Standard stock solutions may be commercially purchased for each element or as a multi-element mix. Internal standards may be purchased as a mixed multi-element solution. The manufacturer's expiration date and storage conditions must be adhered to.

7.6.1 At a minimum the curve must contain a blank and five (5) lead containing calibration standards. The calibration standards shall be stored at ambient laboratory temperature. Calibration standards must be prepared weekly and verified against a freshly prepared ICV using a NIST-traceable source different from the calibration standards.

7.6.2 Prepare calibration standards at five levels by diluting commercially obtained Lead stock solutions using the extraction solution from 7.5 to cover the working range of the instrument, typically 0.1µg/L through 100µg/L.

7.7 Internal standards are added by on-line addition. Prepare an internal standard working solution at a concentration of 0.25µg/mL by diluting a commercially obtained Tb standard using the Extraction solution from 7.5.

7.8 Three laboratory blank solutions are required for analysis: (1) the calibration blank is used in the construction of the calibration curve and as a periodic check of system cleanliness (ICB and CCB); (2) the Method Blank (MB) is carried through the extraction process to assess possible contamination; and (3) the rinse blank is run between samples to clean the sample introduction system. If calibration blanks or Method Blanks yield results above the detection limit, the source of contamination must be identified.

7.8.1 The calibration blank is prepared in the same acid matrix as the calibration standards (0.38M HNO_3 + 0.84M HCl) and samples and contains all internal standards used in the analysis.

7.8.2 The method blank (MB) contains all reagents used in the extraction and is carried through the extraction procedure at the same time as the samples.

7.8.3 The rinse blank is a solution of 1% nitric acid (v/v) in reagent grade water. A sufficient volume should be prepared to flush the system between all standards and samples analyzed.

7.8.4 EPA procures filters and distributes them to the state monitoring agencies collecting Pb in support of the National Ambient Air Quality Standard (NAAQS). If filter lot blanks are provided to the laboratory for analysis, consult 40CFR, Appendix G to Part 50, Section 6.1.1 for guidance on testing.

7.9 The Initial Calibration Verification (ICV), Lower Level Calibration Verification (LLCV), and Continuing Calibration Verification (CCV) solutions are prepared from a different lead source than the calibration curve standards and at a concentration that is either at or below the midpoint on the calibration curve, but within the calibration range. Both are prepared in the same acid matrix as the calibration standards. Note that the same solution may be used for both the ICV and CCV. The ICV/CCV and LLCV solutions must be prepared fresh daily.

7.10 Tuning Solution (6020TS – Inorganic Ventures): 1ppb ELAN 6100DR setup solution. Prepare a tuning solution according to the instrument manufacturer's recommendations. This solution will be used to verify the mass calibration and resolution of the instrument.

7.11 Daily Performance Check Solution (made by diluting Inorganic Ventures standards of Mg, In, Ce, and Ba): 1ppb ELAN 6100 sensitivity detection Limit solution.

8.0 QUALITY CONTROL

8.1 IDC/MDL Studies: An IDC must be conducted by each new analyst. An MDL study must be conducted annually. If appropriate, the IDC and MDL studies may be combined.

8.1.1 Four passing LCS's must be provided to validate an analyst for this method.

8.1.2 An MDL study should be conducted when significant changes in instrument occur, or when a new instrument is purchased for the analysis.

8.1.3 MDLs must be calculated by analyzing seven replicates of a known low-level spike added to a filter strip and carried through the entire extraction procedure. The test solutions shall be spiked at 2-5 times the estimated MDL. The MDL is defined as 3.143 times the standard deviation of the seven replicates in accordance with 40 CFR Part 136, appendix B[6]. The reporting limit, RL, is defined as the lowest calibration standard included in the curve or the LLCV concentration. Results reported below this limit must be qualified as estimated results

8.2 Standard quality control practices shall be employed to assess the validity of the data generated. Included are: Method Detection Limit (MDL), Reporting Limit (RL), Method Blank (MB), duplicate samples, spiked samples, serial dilutions, ICV, CCV, LLCV, ICB, and CCB.

8.3 For each batch of samples, one method blank (MB) and one Laboratory Control Sample (LCS) spiked at the same level as the Matrix sample spike (MS) must be prepared and carried throughout the entire process. The results of the MB must be below the RL. The recovery for the LCS must be within ± 20% of the expected value, or alternatively control limits may be established when sufficient data points exist for statistical analysis (control limits = ±3sigma). If the MB yields a result above the RL, the source of contamination must be identified and the extraction and analysis repeated. Reagents and labware must be suspected as sources of contamination.

8.4 Any samples that exceed the highest calibration standard must be diluted and rerun so that the concentration falls within the curve. The minimum dilution will be 1 to 5 with a 0.38M HNO_3 and 0.84M HCl solution.

8.5 The internal standard response must be monitored during the analysis. If the internal standard response falls below 70% the sample must be diluted and reanalyzed. The minimum dilution will be 1 to 5 with a 0.38M HNO_3 and 0.84M HCl solution. If the first dilution does not correct the problem, additional dilutions must be run until the internal standard falls within the specified range.

8.6 For every batch of samples prepared, there must be one duplicate (DUP) and one Matrix Spike (MS) sample prepared. The spike added is to be at a level that falls within the calibration curve, nominally the midpoint of the curve. The initial plus duplicate sample must yield an RPD of $\leq$ 20%. The spike must be within ± 20% of the expected value or alternatively control limits may be established when sufficient data points exist for statistical analysis (control limits = ±3sigma).

8.7 For each batch of samples, one extract must be diluted five-fold and analyzed. The corrected dilution result must be within ±10% of the undiluted result. The sample chosen for the serial dilution should have a concentration at or above 10X the lowest standard in the curve to ensure the diluted value falls within the curve. If the serial dilution fails, chemical or physical interference should be suspected.

8.7.1 This requirement is only applicable if there is a sample in the batch at a concentration at or above 10x the lowest standards in the curve.

8.8 The quality control requirements are described in Table 1.0. If any of these QC samples fails to meet specifications, the source of the unacceptable performance must be determined, the problem corrected, and any samples not bracketed by passing QC samples must reanalyzed.

8.9 For each run, a LLCV must be analyzed. The LLCV shall be prepared at a concentration not more than three times the lowest calibration standard and at a concentration not used in the calibration curve. The LLCV is used to assess performance at the low end of the curve. If the LLCV fails the run must be terminated, the problem corrected, the instrument recalibrated, and the analysis repeated.

8.10 Pipettes used for volumetric transfer must have the calibration checked at least annually and pass ± 1% accuracy.

9.0 CALIBRATION

9.1 Power up the ICP/MS and workstation. Turn on the argon supply at the tank and adjust the regulator to 50 psi.

9.2 Check that the sample tubing and drain tubing leading from the spray chamber are properly set up. Click on the instrument icon and then on the front panel button to open the ELAN control panel. The status indicator should indicated "ready" if all system hardware is operating properly and the plasma can be ignited. If the indicator shows "not ready" refer to the operator manual to determine which system component is causing the fault. Tune to meet the instrument manufacturer's specifications. After tuning, place the sample aspiration probe into a 2% nitric acid rinse solution for at least 5 minutes to flush the system.

9.3 Click on the plasma start button to ignite the plasma. The ELAN initiates the ignition process which requires a little over a minute to complete. The process of ignition sequence is shown in the ignition sequence status bar. After the plasma ignites allow the instrument to warm up for approximately 30 minutes.

9.4 Open the EPA 6020A tuning method. Aspirate the tuning solution, and click the analyze sample button. When analysis of the tuning solution is complete check the report to ensure the mass values are ±0.1 amu of the actual mass values and the resolution is less than 0.9 ± 0.1amu.

9.5 If you have not yet reached the target resolution, open the optimization method for the optimization procedure. If you pass the tuning, click the EPA 6020A method, aspirate the daily performance check solution. Click the analyze sample button to run the daily check. When the measurement is complete, compare the result with the table below. If any value is outside the range, repeat the procedure.

Daily Performance Check Criteria

Parameter	Requirement
Mg	>8,000
In	>40,000
U	>30,000
Precision	<3%
Ba^{2+}/Ba	<0.03
CeO/Ce	<0.03
Bg level	<2

9.6 In the EPA 6020A workspace, open the "EPA 6020A quantitative analysis method". Enter a blank and six calibration standards on the calibration page. Select the appropriate (µg/L) unit and linear calibration type.

9.7 Press the sample icon; enter the autosampler locations for the blanks and standards. Enter the appropriate sample flush delay (typically 35s) and rinse (typically 45s) parameters. Type a new data name, description, and save the new data file.

9.8 Place the calibration standard tubes into the positions indicated in EPA 6020A quantitative page. Return to the main EPA 6020A quantitative page; Press the "Analyze Batch" icon to begin the calibration. Once finished running all the calibration points, click the "CalibView" icon. Select linear calibration algorithm with $R \geq 0.995$.

9.9 If there is difficulty meeting the linear calibration algorithm with $R \geq 0.995$, the instrument will require recalibration and/or maintenance

9.10 Immediately after the calibration curve is completed, analyze an ICV and an ICB. The ICV must be prepared from a different source of lead than the calibration standards. The ICV must recover 90-110 % of the expected value for the run to continue. The ICB must be less than the RL. If either the ICV or the ICB fails, the run must be terminated, the problem identified and corrected, and the analysis re-started.

9.11 A LLCV, CCV and a CCB must be run after the ICV and ICB. A CCV and CCB must be run at a frequency of not less than every 10 extracted samples. The CCV solution is prepared from a different source than the calibration standards and may be the same as the ICV solution. The LLCV must be within ± 30% of expected value. The CCV value must be within ± 10% of expected for the run to continue. The CCB must be less than the RL. If either the CCV, LLCV, or CCB fails, the run must be terminated, the problem identified and corrected, and the analysis re-started from the last passing CCV/LLCV/CCB set.

9.12 A LLCV, CCV, and CCB set must be run at the end of the analysis. The LLCV must be within ± 30% of expected value. If either the CCV, LLCV, or CCB fails, the run must be terminated, the problem identified and corrected, and the analysis re-started from the last passing CCV/LLCV/CCB set.

10.0 FILTER STRIP EXTRACTION

10.1 Sample Preparation – Heated Ultrasonic Bath

10.1.1 Using ceramic scissors and non-metal ruler, cut a 3/4 in. x 8 in. strip from the exposed area of the filter by cutting a strip from the edge of the filter where it has been folded along the 10" side at least 1 in. from the right or left side to avoid the un-sampled area covered by the filter holder. The filters must be carefully handled to avoid dislodging deposits.

10.1.2 Using plastic tweezers roll the filter strip up in a coil and place the rolled strip in the bottom of a labeled 50mL extraction tube. In a fume hood, add 15.0 ± 0.15mL of the extraction solution (Section 7.5) using a calibrated mechanical pipette. Ensure that the extraction solution completely covers the filter strip.

10.1.3 Loosely cap the 50mL extraction tube and place it upright in a plastic rack. When all samples have been prepared, place the racks in an uncovered heated ultrasonic water bath that has been preheated to 80 ± 5°C and ensure that the water level in the ultrasonic is above the level of the extraction solution in the tubes but well below the level of the extraction tube caps to avoid contamination. Start the ultrasonic bath and allow the unit to run for 1 hour ± 5 minutes at 80 ± 5°C.

10.1.4 Remove the rack(s) from the ultrasonic bath and allow the racks to cool.

10.1.5 Add 25.0 ± 0.25mL of Milli-Q water with a calibrated mechanical pipette to bring the sample to a final volume of 40.0 ± 0.4mL. Tightly cap the tubes and vortex mix or shake vigorously. Place the extraction tubes in an appropriate holder and centrifuge for 20 minutes at 2500RPM.

10.1.6 Pour an aliquot of the solution into an autosampler vial for ICP-MS analysis to avoid the potential for contamination. Do not pipette an aliquot of solution into the autosampler vial.

10.1.7 Decant the extract to a clean tube, cap tightly, and store the sample extract at ambient laboratory temperature. Extracts may be stored for up to six months from the date of extraction.

11.0 MEASUREMENT PROCEDURE

11.1 Follow the instrument warm up, tuning and instrument calibration procedure described in section 9.0.

11.2 As directed in Section 8 of this SOP, analyze an ICV and ICB immediately after the calibration curve followed by a LLCV, then CCV and CCB. The acceptance requirements for these parameters are presented in Section 8.8.

11.3 Analyze a CCV and a CCB after every 10 extracted samples.

11.4 Analyze a LLCV, CCV and CCB at the end of the analysis.

11.5 Typical run samples will include field samples, field sample duplicates, spiked field sample extracts, serially diluted samples, and the set of QC samples listed in 8.8 above.

11.6 Any samples that exceed the highest standard in the calibration curve must be diluted and reanalyzed so that the diluted concentration falls within the calibration curve.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 The filter results must be reported in total µg Pb per strip.

12.1.2 $$\text{Total } \mu g \text{ Pb/strip} = \frac{(\mu g/L) \times (V_t) \times (D)}{\text{Strip}}$$

Where:

µg = micrograms

V_t= total volume of the final extract (L)

D=dilution factor, if needed. If no dilution was made, D=1.

12.2 Data Entry and Review

12.2.1 Select the appropriate batch in Element and create a data entry table.

12.2.2 Open the Data Tool to upload results directly from the ELAN output file stored on the G: drive. Element will calculate final concentrations, adjust reporting limits, calculate % recoveries for LCS/MS/MSD, and RPDs for MS/MSD pairs.

12.2.3 Investigate any results flagged by Element in red. Possible issues could include missed holding time, QC outside acceptance limits, or other issues. In some cases data entry errors during the batching process may have created an erroneous result. Add qualifiers as appropriate.

12.2.4 Print the data entry table. Review for accuracy and completeness.

12.2.5 Assemble a data package consisting (in order);

Final report from the Element LIMS.
Data Review Checklist.
Non Conformance Details report(s) (if applicable).
Sample Preparation Bench Sheet.
ELAN run log.
Place data package in an end tab manila folder, with pre-printed label indicating the method performed and the date.

12.2.6 Update the status of the batch in Element to "Analyzed". Place data package in an end tab manila folder, with pre-printed label indicating the method performed and the date.

12.3 Submit data package to peer or supervisor for second level review. Upon successful review the second level reviewer updates the status of the batch(es) to "Reviewed" in Element

12.4 Submit reviewed package to Chemistry Supervisor for final review. Chemistry supervisor or designee places data package to central files.

12.5 Reporting

12.5.1 The Chemistry supervisor creates final report in hardcopy and .pdf format. Chemistry supervisor or designee forwards the electronic report to the client via email. Hardcopy report is submitted to Administration

Table 1.0: Quality Control Requirements

Description	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	Daily	$r \geq 0.995$	Re-analyze initial calibration
Initial Calibration Verification (ICV)	Immediately following ICAL	%Drift $\leq 10\%$	Recalibrate.
Initial Calibration Blank (ICB)	Immediately following ICV	Less than RL	Investigate source of contamination and correct the problem. Recalibrate after contamination source eliminated.
Lower Level Calibration Verification	Before first sample and after last sample	±30% of the expected value.	Re-analyze LLCV. If second re-analysis of LLCV fails criteria then recalibrate.
Method Blank (MB)	Ever preparation batch, or 20 samples, whichever is less	Less than RL	If samples < RL no action. If samples > RL and level in MB is < 5% of the amount found in samples report data with "B" qualifier. If samples > RL but < 5% of amount found in samples re-extract and re-analyze batch for elements with blank contamination.

Table 1.0: Quality Control Requirements (cont.)

Description	Frequency	Criteria	Corrective Action
Laboratory Control Sample (LCS)	Ever preparation batch, or 20 samples, whichever is less	Limits established by control charts. If no limits established then use advisory limits %R = 75%-125%	If samples ND and LCS %Rec > upper control limit no corrective action. Otherwise re-digest and re-analyze batch for elements with out of control LCS recoveries.
Duplicate	Every preparation batch, or 20 samples, whichever is less.	%RPD ≤ 20% for analyte values greater than 100x the RL.	Qualify data. Possible non-homogeneous sample.
Continuing Calibration Verification (CCV)	Every 10 samples	%Drift ≤ 10%	Re-analyze CCV. If second re-analysis of CCV fails criteria then recalibrate. Samples analyzed prior to failing CCV must be reanalyzed for elements that failed criteria.
Continuing Calibration Blank (CCB)	Follows every CCV	Less than RL	Re-analyze CCB. Investigate source of contamination and correct the problem. Recalibrate after contamination source eliminated. Re-analyze samples or qualify results for elements that failed CCB criteria.

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Table 2.0: Data Review Checklist

Review Item	Analyst	Reviewer	Comments
Calibration			
1. Tune RSD≤5%? Mass calibration≤0.1AMU from true value? Resolution<0.9AMU at 10% height? ELAN daily performance check passed?			
2. Initial Calibration $r^2 \geq 0.995$?			
Batch QC			
1. Initial Calibration Verification (ICV) ±10% of the expected value? ICB ≤ RL?			
2. Lower Level Calibration Verification (LLCV) analyzed before and after every 20 samples? LLCV ±30% of the expected value?			
3. Method Blank (MB) analyzed daily, or every 20 samples? MB ≤ RL?			
4. Laboratory Control Sample (LCS) analyzed daily, or every 20 samples? LCS recovery 75-125%? (advisory)			
5. MS/MSD (Matrix Spike/Matrix Spike Duplicate) analyzed every 20 samples? MS/MSD recovery 80-120%? (advisory)			
6. Sample duplicate every 20 samples? DUP RPD<20%?			
7. Internal Standard (IS) recovery >70%?			
8. Continuing Calibration Verification (CCV) analyzed ever 10 samples? CCV±10% of the expected value?			
9. Continuing Calibration Blank (CCB) after every CCV? CCB ≤ ½ RL?			

SOP Title:	**Determination of Lead in TSP by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Heated Ultrasonic Nitric and Hydrochloric Acid Filter Extraction**
Version/Revision Date:	11-2010
Supersedes:	02-2010
Reference Method:	EPA 3050B, EPA 6020A, EQL-0510-191, QP-07, QP-10, QP-13, QP-20, WI-CHEM-020

Samples			
1. Results within calibration range?			
2. Data entry spot checked at least 10%?			