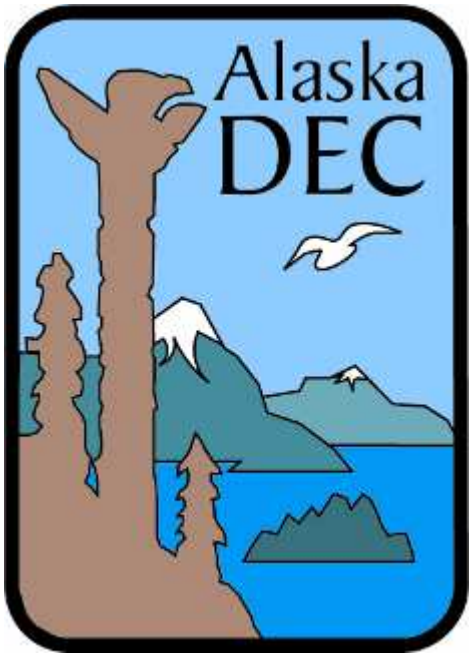
Standard Operating Procedure
For
Laboratory Gravimetric Analysis of
Fine Particulate Matter ($PM_{2.5}$)
Air Quality Filter Samples



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$PM_{2.5}$ FILTER PROCESSING STANDARD OPERATING PROCEDURES

1.0 SCOPE

This document describes the methodology used by the Monitoring & Quality Assurance Program's Juneau Air Quality Laboratory staff to analyze the mass of fine particulate matter ($PM_{2.5}$) samples collected on Teflon™ filters. Activities addressed by this method include:

- Instrument calibration and verification;
- Receipt of filters from EPA;
- Filter integrity check;
- Pre-sampling filter conditioning and weighing;
- Shipment of filters into and from the field;
- Post-sampling filter conditioning and weighing;
- Laboratory and weighing room environmental control; and
- Filter handling and storage.

2.0 METHOD SUMMARY

Individual Teflon™ filters (46.2 mm in diameter) are weighed on an electronic micro-balance before and after field sampling. Particulate matter less than 2.5 micrometers in diameter is collected from ambient air on one of these filters. The sampling duration is 24-hours. The net difference between pre- and post sampling filter weights is used to calculate the ambient air mass concentration. After post weighing, filters are stored.

3.0 INTERFERENCES

3.1 The potential effect of body moisture or oils contacting the filters is minimized by using non-serrated forceps to handle the filters at all times. This measure also moderates interference due to static electricity.

3.2 Teflon™ filters accumulate a surface electrical charge that may affect filter weight. Static electricity is controlled by treating filters on ^{210}Po antistatic strips prior to weighing. Filters are held near a ^{210}Po antistatic strip for 60 seconds before any filter can be weighed.

3.3 Moisture content can affect filter weight. Filters must be equilibrated for a minimum of 24 hours in a controlled environment prior to pre- and post weighing. During the equilibration period, relative humidity must be maintained at a mean value of 35-40% and air temperature at a mean of 21-23 degrees Celsius.

3.4 Airborne particulates can adversely affect an accurate mass measurement of the filter. Equilibrating filters should not be placed within airflow paths created by air conditioning ductwork, near computer printers or turbulence created by opening and closing doors. Dust contamination can be further minimized by

cleaning the lab work surfaces and weighing areas daily, installing Asticky≅ floor mats at the entrance to the balance room, and wearing clean lab coats over regular clothing.

4.0 APPARATUS

4.1 Mettler MT5 electronic microbalance with a minimum resolution of 0.001 mg (i.e., 1 microgram) and a precision of $\pm$ 0.001 mg, supplied with a balance pan. The microbalance is positioned upon a vibration-damping balance support table and is interfaced with a Laboratory Information Management System (LIMS) database system.

4.2 Calibration weights, utilized as Mass Reference Standards, non-corroding, range in weight from 100 mg to 200 mg and be certified as traceable to National Institute of Standards and Testing (NIST) mass standards. Two sets: one set as a working standard and one set as the primary standard. The mass standards should be Class 1 category with a tolerance of 0.01mg.

4.3 Radioactive (alpha-particle) Polonium-210 (^{210}Po) antistatic strips for charge neutralization. At least 3 strips are needed per balance.

4.4 Non-metallic, non-serrated forceps.

4.5 Digital timer/stopwatch.

4.6 Filter: Teflon™ membrane, 46.2 mm in diameter with a polypropylene support ring.

4.7 Filter support cassettes.

4.8 Filter equilibration racks.

4.9 Automated relative humidity/temperature recording system.

4.10 Psychrometer (NIST certified) for calibration of relative humidity readings.

4.11 Precision thermometer (NIST traceable) for calibration of temperature readings.

4.12 Light box.

4.13 Antistatic, powder-free vinyl gloves.

4.14 Plastic Petri-slide filter containers.

4.15 Zip-lock plastic bags, 5"x8".

4.16 Disposable laboratory wipes.

4.17 Filter equilibration cabinets.

5.0 INSTRUMENT CALIBRATION AND VERIFICATION

5.1 Analytical Micro-balance

The micro-balance will be calibrated/certified annually and maintained according to the manufacturer's recommendations. The calibration will be traceable to NIST-traceable mass reference standards. If at any time the micro-balance is found to be out of calibration, it will be recalibrated according to the manufacturer's directions.

Calibration of the micro-balance will be verified during each weighing session. Working standards bracketing expected unexposed and exposed filter masses (e.g., 100 and 200 mg) will be routinely weighed each weighing session.

If verified and measured values of a working standard differ by more than 3 ug (i.e., 3X micro-balance's repeatability), the analyst will reweigh both working standards. If disagreement persists, the analyst will investigate the problem and take appropriate corrective action. Corrective action may include: recertifying the working standards against the lab primary standards, and/or repairing the microbalance.

5.2 Mass Reference Standards

Mass reference standards should be in range of 100 to 200 mg. Since a typical 46.2 mm Teflon™ filter weighs between 110 – 160 mg. These standards must be NIST traceably certified. Additionally, they should have a tolerance of 0.01mg.

Mass reference standards should be recertified annually at a NVLAP accredited calibration laboratory or at a State Weights and Measures Laboratory holding a NIST Certificate of Traceability.

Two sets of mass reference standards are recommended: primary and working. Working standards are recommended for routine filter weighing and should be kept next to the balance in a protective container. Laboratory primary standards should be handled carefully and kept in a secured compartment. Working standards should be verified against the lab's primary standards every 3 to 6 months. Always use smooth, nonmetallic forceps for handling mass reference standards. These forceps should be identified only for this purpose and should be cleaned with alcohol and lint-free wipes and allowed to air dry before handling standards.

Working standards verifications to the primary standards will be documented in

the lab's QC logbook.

5.3 Recording Thermometer

Calibration of the recording thermometer will be verified quarterly using the calibration thermometer. Temperature verification will be done at three different temperatures over the expected range of the conditioning environment.

If the recording thermometer's value differs by more than 0.5^{o}C from the calibration thermometer, the analyst will investigate the problem and take appropriate action. Corrective actions may include: (1) adjusting the recording thermometer to agree with the calibration thermometer or (2) returning the recording thermometer to manufacturer for service, calibration and certification.

All verifications/calibrations will be documented in the lab's QC logbook.

5.4 Recording Hygrometer

Calibration of the recording hygrometer will be verified quarterly against the laboratory's reference standard. The verification will be performed at a minimum of one %RH level, which may be the current level in the conditioning environment. The laboratory's reference standard will be verified annually against ADEC's primary %RH standard (a NIST traceable Assmann type psychrometer).

If the recording hygrometer's value differs by more than 2% RH from the lab's reference standard, the analyst will investigate the problem and take appropriate action. Corrective actions may include: (1) adjusting the recording hygrometer to agree with the reference probe and/or (2) recertifying the lab's reference probe against NIST-traceable standard.

All verifications/calibrations will be documented in the lab's QC logbook.

6.0 FILTER SHIPPING, RECEIVING AND TRACKING

A filter inventory sheet will accompany filter shipments to and from the field. Site operators will determine from the inventory sheet the appropriate filters for each sampling event. Filters will be used within 30 days of the pre-sampling weighing date.

Filters will be assigned unique alpha-numeric ID numbers as follows:

The first 2 alpha values identify the type of filter as being either a routine filter (RF), a field blank (FB), a lab blank (LB), or a stagnation blank (SB). The third alpha value identifies the filter media (T = Teflon™, N = nylon, etc.). The next 7 numeric values are those stamped on the filter support ring by the manufacturer.

6.1 Filter Shipping Requirements

The $PM_{2.5}$ network has rigid requirements for preventing sample contamination. Deliberate care must be exercised while handling filter cassettes. Once filter cassettes are taken outside the weigh room, they should never be opened as damage may result to the Teflon™ filter. Once samples have been collected and recovered, they are to be stored with the particulate side up. Samples should also be stored in static-resistant zip lock bags.

Temperature requirements of the $PM_{2.5}$ network are explicitly detailed in 40 CFR Part 50, Appendix L. There are no specific requirements for temperature control during transport from the weigh room to the sample location; however, excessive heat must be avoided. Pre-weighed filters must also be used for sampling within 30 days of the pre-sampling weigh date, which will be identified on the filter inventory sheet delivered to the operator with the filters.

96 hours is the maximum allowable time for recovery of a sample filter from completion of a sample run. During this time the temperature within the sampler's filter housing is not to exceed the ambient temperature by more than $5^{o}C$.

Ideally filter transport will not exceed 24 hours. If filters are stored at less than $4^{o}C$ from the end of the sample run to the date of laboratory conditioning for post weighing, the 30 day holding time is allowed. If filters are maintained at less than $25^{o}C$ from the end of sample run to the date of laboratory conditioning for post-weighing, a 10 day holding time is allowed.

Note: Permissible $PM_{2.5}$ sample holding times are detailed in both 40 CFR Part 50 Appendix L and Section 2.12 of the U.S. EPA QA Handbook.

Field operators will be supplied with insulated shipping containers and ice substitute gel pouches that can maintain the filters' temperature at $< 25^{o}C$ during shipment back to the weighing lab. Samples should be stored in antistatic zip lock bags with particulate side facing up. Bubble wrap will be placed around the samples. The probe for the min/max thermometer will be placed in the samples proximity. Ice substitutes will be packed around the samples. A closed cell foam insert will be placed securely over the ice substitutes, with the wire from the temperature probe running up the side4 so the temperature can sit on top of the foam. Samples will be shipped by US priority mail.

6.2 Filter Receiving and Tracking

Upon receipt of the samples, filter will be logged into the sample receipt log. Sample information will be entered into the log book should include: filter ID, shipper, min/max temperature, data/time/analyst receiving shipment, any other

significant information recorded on the filter inventory sheet and apparent integrity of the sample. Samples are to be removed from their container and immediately placed in the lab refrigerator.

7.0 FILTER RECEIPT, INSPECTION, HANDLING AND STORAGE

7.1 Filter Receipt and Inspection

Shipment of 46.2mm filters will be periodically sent from EPA to the ADEC. Cases will be labeled with the date of their receipt and used in the order they were received. They will be opened one at a time and all filters in the case will be used before the next case is opened.

All filters should be visually inspected for defects before the initial weighing. A filter should be discarded if any defects are found. Any lot of filters containing a high number of defects should be returned to the supplier. Specific filter defects are:

- Pinhole – A small hole appearing as a distinct and obvious bright point of light when examined over a light table.
- Separation of ring – Any separation or lack of seal between the filter and the filter border reinforcing the ring.
- Chaff or flashing – Any extra material on the reinforcing, polyolefin ring or on the heat seal area that would prevent an airtight seal during sampling.
- Loose material – Any extra loose material or dirt particles on the filter.
- Discoloration – Any obvious discoloration that might be evidence of contamination.
- Filter nonconformity – Any obvious visible nonconformity in the appearance of the filter when viewed over a light table that might indicate gradations in density or porosity across the face of the filter.
- Other – A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

7.2 Filter Handling

Filters will be handled carefully during filter conditioning and weighing to avoid measurement errors due to damaged filters or a gain or loss of collected particles on the filters. If necessary, the analyst will wear antistatic-powder-free gloves. Gloves should be touched to ground to remove any static charge. Filters will be handled with smooth, non-serrated forceps that will be used only for that purpose. These forceps will be marked to distinguish them from those used to handle mass standards. Forceps will be cleaned with alcohol and lint-free wipes and allowed to air-dry before handling filters. Filters will be handled carefully by the support ring rather than the filter material. If the forceps touch the filter material of an exposed filter, the forceps will be cleaned with antistatic disposable laboratory wipes to avoid cross-contamination. These precautions will reduce the potential

contamination of body moisture/oils on filters.

In the laboratory, each filter will be transferred from its sealed manufacturer's packaging to a clean plastic Petri dish, to reduce the risk of contamination. The filter will remain in this container, except for weighing, until it is loaded into a filter cassette prior to sampling. Each filter will have a unique identification number. Filters will be processed sequentially.

7.3 Filter Storage

After sample pre-weighing, filters will be returned to the filter-handling containers and stored in the filter equilibration/weighing room. Containers will be identified by filter numbers and stored in an orderly way that allows retrieval when necessary.

After sample post-weighing, filters will be archived to cold ($4^{o}C$) storage for at least one year or until EPA specifically requests specific samples be held for future analysis. Filters will be returned to the filter handling containers and stored in an orderly way to facilitate easy retrieval.

8.0 LOT BLANKS, LABORATORY BLANKS, AND FIELD BLANKS

Three types of blank filters will be used in the $PM_{2.5}$ filter processing procedure.

8.1 Lot Blanks

Lot blanks are clean unexposed filters, that are used to determine filter weight stability over long periods of time (e.g., 4 – 6 weeks), affected by the volatilization of material from the filter or to the absorption of gaseous materials into the filter from the atmosphere. A filter lot is defined as a single shipment of filters from a filter manufacturer. A filter exposure lot is defined as a subsample of filters from the filter lot to be conditioned within a specified period of time.

Three clean unexposed filters will be randomly selected and designated as lot blanks. Lot blanks are conditioned for an initial 24 hours prior to the initial pre-weight determination. These exposure lot blanks are reweighed periodically (e.g., daily, weekly) and stored in the conditioning chamber (with other filters) between weighings. These weighings should be continued until the weekly weight change is less than 15 μg (i.e., 3 times the precision for weighing unexposed filters).

This filter weight stability experiment will determine the period that the entire filter lot should be conditioned before it can be used for routine sampling. These measurements will be recorded in the QC notebook or an equivalent database.

8.2 Laboratory Blanks

Laboratory blanks are conditioned, clean unexposed filters that are used to determine any weight change between pre- and post sampling weighings due to contamination in the microbalance environment. Laboratory blanks should be kept inside the conditioning chamber except during weighing sessions. Weigh enough laboratory blanks during a pre-sampling weighing session to provide at least one single-use laboratory blanks during each subsequent post sampling weighing session. If the weight change exceeds 15 µg, contamination in the conditioning chamber may be occurring. Take appropriate troubleshooting and corrective actions. The pre- and post sampling weights are to be recorded in the QC notebook or an equivalent database.

8.3 Field Blanks

Field blanks are conditioned, clean unexposed filters that are used to determine whether contamination occurs during sample transport, setup and recovery. Field blanks should be transported to the sampling site, momentarily installed in the sampler, removed and stored in their protective containers inside the sampler's case at the sampling site until the exposed filters are retrieved for post sampling weighing. Site operators are encouraged to occasionally install field blanks in a non-active sampler's filter holder over a 24 hour period during which the sampler is not scheduled to be sampling (stagnation blank).

Field blanks should occur at a frequency of 10 to 15% of a sampler's routine operating frequency. Field blanks should be scheduled to ensure that a post sampling weighing session contains 10% blanks, or at least one field blank.

If the weight change between pre- and post filed blank weighings exceeds 30 µg, contamination during transportation or at the sampling site may be occurring. Take appropriate troubleshooting and corrective actions. The pre- and post field blank weights are to be recorded in the QC notebook or an equivalent database.

Note: A difference between pre- and post field blank weights may be attributable to moisture mass loss or gain due to difference in %RH/Temperature of the conditioning chamber compared to %RH/Temperature that the field blank was collected under.

9.0 FILTER CONDITIONING AND WEIGHING

9.1 Micro-balance Environment

The micro-balance will be located in the same environmentally controlled room that conditions sample filters. The micro-balance rests on a balance table which itself rests on a 3 foot by 4 foot isolated cement pad. Dust contamination will be minimized by cleaning the weighing area frequently. Other measures for dust

control will be implemented if necessary.

9.2 Initial Conditioning of Exposed Filters

9.2.1 New, clean unexposed filters should be placed in the conditioning environment immediately upon arrival and should be stored there until the pre-sampling weighings. Filters will be conditioned for a minimum of 24 hours to allow their weights to stabilize before being weighed.

9.2.2 Condition filters at the same conditions before the pre- and post sampling weighings. Mean %RH will be between 30 and 40 %RH ± 5%RH over 24 hours. However, where it can be shown that the mean %RH during sampling is less than 30%RH, conditioning can occur at a mean ambient %RH ± 5%RH, but not less than 20%RH. Automatic room conditioning %RH and temperature will be measured continuously and recorded by the LIMS.

9.2.3 Within the filter conditioning environment, filters should be placed in a cabinet that will allow air circulation over the filters while reducing the chance for airborne materials to settle on the filters. Care should be taken to avoid contaminating the $PM_{2.5}$ filters inside the conditioning environment with particulates released by other media (e.g., quartz and glass) that are also being conditioned in the chamber. Lab blanks should be used to check for potential cross-contamination from airborne particulates. If contamination occurs, take appropriate corrective action(s).

9.2.4 Condition filters in their filter-handling containers (e.g., slide Petri dishes). Label both the lid and slide portions of the Petri dish with the sample filter's unique ID. During filter conditioning, place the lid so it partially covers the open container.

9.2.5 For new filter lots, remove filters from their sealed packages and place in their own filter containers. Allow sufficient time for filter weights to stabilize before use. Required sample conditioning time for filters within a lot is determined upon the receipt from the manufacturer.

9.2.6 Electrostatic charge within the balance will be minimized by placing a radioactive antistatic strip (i.e. 500 picocuries of ^{210}Po) in the balance's weighing chamber. In addition, filters should be held for 60 seconds near an antistatic strip (but not touching) before weighing. Charge neutralization times may need to be longer than 60 seconds. Low %RH, heavy filter loading, particle materials, etc. can affect the necessary time to neutralize electrostatic charges. Balance performance will ultimately determine the required measures to reduce or minimize electrostatic

buildup.

^{210}Po antistatic strips are used to reduce electrostatic buildup by charge neutralization. They will neutralize electrostatic charges on items brought within an inch of them. Since ^{210}Po has a half-life of 138 days, these antistatic strips need to be changed every 6 months. Dispose of old strips according to the manufacturer's recommendations.

9.3 Pre-sampling Gravimetric Analysis of Unexposed Filters

Pre-sample filters must be weighed in the same room as the filters were conditioned in. Filters must be weighed without intermediate or transient exposure to other conditions or environments.

9.3.1 Record the conditioning room's %RH and temperature in lab notebook or an equivalent database. Analyst will verify that mean temperature and %RH for last 24 hours has remained between 20 – 23^{o}C (with instantaneous readings within ± 2^{o}C) and 30 – 40%RH (with instantaneous readings within ±5 %RH. **If these conditions are not met, filters will not be weighed.** Subsequent appropriate troubleshooting and corrective actions will then be taken.

9.3.2 Clean the balance's weighing chamber with a fine brush, if necessary. Clean the surfaces near the micro-balance with alcohol-moistened disposable laboratory wipes. Clean the standard forceps with a lint-free cloth and the filter forceps with moistened wipes. Allow the forceps to air-dry.

9.3.3 To ensure maximum stability, leave the micro-balance on at all times.

9.3.4 Zero (i.e., tare) and calibrate the micro-balance according to the manufacturer's directions.

9.3.5 Using the standard forceps, weigh two working mass reference standards (e.g., 100 mg and 200 mg) as a QC check. Record certified and measured values in the lab's QC notebook or an equivalent database. If verified and measured values disagree by more than 3 µg, reweigh the working standard. If the values still disagree, troubleshoot and take appropriate corrective action.

Weigh enough laboratory blanks during a pre-sampling weighing session to provide at least 10% or one single-use laboratory blank during each subsequent post sampling weighing session. Likewise, weigh enough field blanks to provide at least 10% or one single-use field blank during each subsequent post sampling weighing session

9.3.6 Weigh the filters. Operate the balance according to the manufacturer's directions. Between each filter weighing, re-zero the balance. Remove the filter from the Petri dish by gently slipping the filter forceps under the outer polyolefin support ring. Hold the filter only by the ring. Pass the filter, support ring side up, near a ^{210}Po antistatic strip for 30 - 60 seconds immediately before weighing. Immediately transfer the filter to the microbalance's pan and close the weighing chamber door. Record the filter ID, filter lot number and tare-weight (pre-sampling mass) in the LIMS or an equivalent database.

9.3.7 After every 10^{th} filter, reweigh on of the working standards and record the measurement data. If this measurement disagrees from the verified value by more than 3μg, reweigh the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action.

9.3.8 Reweigh one of the previously weighed sample filters. Record the replicate measurement in the LIMS or an equivalent database. If the replicate measurement disagrees from the original by more than 15 μg, reweigh the filter. If the measurements still disagree, troubleshoot and take corrective action.

9.3.9 At the end of the weighing session, reweigh both working standards. Record the measurement in the LIMS or an equivalent database.

9.3.10 Any unused filter whose weight is outside the normal range of 110 – 160 mg should be investigated. If there is a consistent negative replication (>15 μg) for laboratory blank filters, it is usually a sign that the filters have not equilibrated long enough. In this case, troubleshoot and take corrective action.

9.3.11 Return filters to the filter handling container, replace the lid, and return it to the conditioning environment to protect it from contamination prior to sampling.

9.3.12 Check filter cassettes and the backing screens for fractures, cracks, evidence of wear, or contamination. Clean or replace as necessary. Cassettes can be washed and thoroughly rinsed with deionized water.

9.3.13 When it is time for filters to be used at sites, install filters in filter cassettes, and place filter/cassette assembly into protective containers for transport to site. Attach a label with filter ID to the outside of the protective container. Prepare several extra filters in case a filter is invalidated during the installation process.

9.3.14 If filters are to be mailed, the operator should be supplied with bubble wrap, etc., in addition to the protective container, to protect exposed filters

during their shipment back to the analytical laboratory. A filter inventory sheet with filter ID and pre-sampling weighing dates shall also be supplied to the operator.

9.4 Post Sampling Conditioning of Exposed Filters

Filters will be kept at a minimum of less than 25^{o} C (preferably 4^{o} C) from retrieval from the sampler until the start of post sampling conditioning. All exposed filters will be weighed within 10 to 30 days from sample filter retrieval depending upon the maximum exposure temperature. Upon receipt of the filters from the field, the laboratory analyst will follow these steps:

9.4.1 Lab analyst who receives and logs in sample shipment will verify that the temperature of the insulated shipping container's interior was maintained at <25^{o}C, or at <4^{o}C for shipment received via air priority mail. The maximum temperature registered by the shipping thermometer will be recorded in LIMS or an appropriate laboratory database. Filters will be removed from the zip lock bags and the bags inspected for loose particulate matter or debris. If loose particulate is found, the analyst will record that the sample has been flagged for possible voiding in LIMS and noted on the appropriate shipping form. The filter will be set aside for subsequent inspection by the laboratory supervisor. After sample receipt and filter inspection, the analyst will store filters/filter containers, exposed side up, in the lab refrigerator (<4^{o}C) until ready for filter post sampling equilibration and weighing.

9.4.2 The lab analyst will access the data recorded from all site samplers by contacting each site via phone/modem and directly downloading the electronic data files. For sites not connected to a phone system, data will be downloaded to disk on site and sent to the lab with the appropriate sample filter paperwork.

9.4.3 The analyst will match the filter identification number with the correct LIMS data on which the filter number, pre-sampling filter weight and other information were recorded.

9.4.4 Prior to post sampling filter conditioning, remove the exposed filters from the lab refrigerator and transfer to the equilibration cabinet. The analyst will allow each filter/filter container to warm to room temperature before opening the filter container to preclude water condensation on a cold filter. After filters have warmed to room temperature, open the filter container and begin the filter equilibration process. During filter conditioning, place the lid so it partially covers the open container. The analyst will inspect each filter for any damage that may have occurred during sampling. If any damage is found, the analyst will note that the sample has been flagged and the reason recorded in LIMS or in an equivalent database. These

filters will be set aside for subsequent inspection by the laboratory supervisor. Allow filters to equilibrate for a minimum of 24 hours.

9.5 **Post Sampling Gravimetric Analysis of Exposed Filters**

Pre- and post sampling filter weighings are to be performed on the same micro-balance. Different analysts can perform the pre- and post sampling filter weighings as long as the appropriate SOPs have been followed and as long as the working standard and replicate measurements are within specifications. Use an effective technique to neutralize static charges on the filter. Post sampling conditioning and weighing should be completed within 240 hours (10 days) after the end of the sampling period, unless the filter is maintained at $\leq 4^{o}$ C during the entire time between sample filter retrieval and the start of filter equilibration. If an exposed filter is maintained at $\leq 4^{o}$ C, the maximum time between sample filter retrieval and post sample filter conditioning can not exceed 30 days.

9.5.1 Repeat steps 9.3.1 through 9.3.11 in the pre-sampling weighing procedure.

9.5.2 Reweigh at least one lab blank and field blank (or 10% of the weighed filters, if larger). If the pre- and post sampling weights for the lab blanks disagree by more than 15μg, repeat the measurements. If pre- and post sampling weights for the field blanks disagree by more than 30 μg, repeat the measurements. If the two measurements still disagree, troubleshoot and take appropriate corrective action. Sample filter measurements should not be corrected to account for blanks measurements. High blank values should not cause automatic invalidation of sample filters that were measured during the same weighing session. Instead, a high blank value should trigger troubleshooting and corrective action to reduce blank values to acceptable levels.

9.5.3 Reweigh one routine filter at the end of the weighing session. Record the replicate measurement in LIMS or in an equivalent database. If the replicate measurement disagrees from the original by more than 15μg, reweigh the filter. If the measurements still disagree, troubleshoot and take appropriate corrective action. Corrective action may include: (1) reweighing all or some of the previously weighed filters, (2) reweighing the working standards, or (3) servicing the micro-balance.

9.5.4 Return filters to the filter handling containers for transfer to filter archiving.

10.0 DATA ACQUISITION, CALCULATIONS, VALIDATION AND REPORTING

10.1 Data Acquisition

The analytical microbalance is linked to a computer and acquires filter weights, continuous weighing room temperature and %RH electronically through Measurement Technologies Laboratory's (MTL) laboratory information management system (LIMS). LIMs will access site sample run data through phone or modem. For sites not connected to a phone system, data will be downloaded on site, copied to disk, and sent to the lab with the appropriate sample filter paperwork via email or floppy disk. Laboratory analysts subsequently upload the electronic data to LIMS.

10.2 Calculations

LIMS automatically calculates 24-hour $PM_{2.5}$ concentrations from information entered into LIMS. Manual recalculations of $PM_{2.5}$ values are determined as follows:

10.2.1 From a LIMS printout, determine the total sample volume (V_a) for the run. If the total sample volume is not available, it can be calculated from the average volumetric flow rate (Q_{avg}) and the total sample time (T) as follows:

$$V_a = (Q_{avg})(T)(10^3) \qquad \text{Equation (1)}$$

where:
- V_a = total sample volume (actual m^3)
- Q_{avg} = average sample flow rate (L/min.)
- T = total sample time (min.)
- 10^3 = units conversion (m^3/L).

10.2.2 Using the post sample filter weight and pre sample filter weight, determine the total filter mass gain ($M_{2.5}$) as:

$$M_{2.5} = (M_{post} - M_{pre}) \qquad \text{Equation (2)}$$

where:
- $M_{2.5}$ = total mass gain (μg)
- M_{post} = post sample filter weight (mg)
- M_{pre} = pre sample filter weight (mg)
- 10^3 = units conversion ((μg/mg)

10.2.3 Calculate the $PM_{2.5}$ concentration as:

$$PM_{2.5} = M_{2.5}/V_a \qquad \text{Equation (3)}$$

10.3 **Data Validation**

Data validation is the responsibility of the local air monitoring network. The Juneau Air Laboratory staff calculates sample $PM_{2.5}$ concentrations based upon pre- and post sample filter net mass and sample data entered into LIMS. Monthly, final sample concentrations with attendant raw data spreadsheets are sent electronically to the local monitoring network. At least 5% of sample field data will be reduced and validated by the local network. The local network will compute the sample concentrations and compare them against Juneau LIMS sample computed results. If sample results agree, the LIMS sample results will be accepted as valid and submitted to the AIRS database manager.

Sample data validity criteria are:

10.3.1 Verify that FRM sample run data are within the following criteria:

Average volumetric flow rate ± 5%	= 16.7 L/min
Flow rate coefficient of variation	≤ ± 4%
Temperature difference (filter – ambient)	≤ ± 5° C
Sample Integration Time sample time ≤ 25 hrs	23 hrs ≤

10.3.2 Verify that the site technician did not flag the sample as "QF1" or "VF1" on the sampler data sheet.

10.3.3 Verify that the free form notes on attached data sheets or in LIMS data records did not indicate an invalid sample.

10.3.4 Verify that the sample was recovered within 96 hours of the completion of the sample run.

10.3.5 Verify that the interior of the sample cooler did not exceed 25° C during shipment.

10.3.6 Verify that sample holding times were not exceeded (30 days if the filter temperature prior to conditioning was maintained at less than 4° C, 10 days otherwise)

10.4 **Data Reporting**

The method detection limit is 2 $\mu g/m^3$.

11.0 QUALITY CONTROL AND QUALITY ASSURANCE

A laboratory QC notebook and/or an equivalent electronic database will be used by the

analyst to record all QC data, including the microbalance calibration and maintenance information, routine internal QC checks of mass reference standards, laboratory field and lot filter blanks and external QA audits. This data will duplicate data that are already recorded in LIMS or other individual notebooks/forms, but will consolidate them such that long-term trends can be identified.

12.0 REFERENCES

EPA 1997. Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere. U.S. Environmental Protection Agency, 40 CFR Part 50, Appendix L.

EPA 4/1998. Quality Assurance Guidance Document 2.12, Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods. . U.S. Environmental Protection Agency.

Draft Idaho State Bureau of Laboratories Standard Operating Procedure for $PM_{2.5}$ FRM Filter Processing for the Idaho Division of Environmental Quality.