Standard Operating Procedure:

Laboratory Gravimetric Analysis of $PM_{2.5}$ Air Quality Filter Samples

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Table of Contents

Standard Operating Procedure for Particulate Matter ($PM_{2.5}$) Analysis

1.0 Procedural

1.1 Scope

This document describes the methodology used by the Monitoring & Quality Assurance Program's Air Quality Laboratory staff to analyze the mass of fine particulate matter ($PM_{2.5}$) samples collected on Teflon® filters. The particular handling and sampling criteria are taken from the EPA's Code of Federal Regulation (CFR), specifically 40 CFR Part 50, Appendix L and Quality Assurance Guidance Document 2.12

1.2 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 the filters during the sample duration of 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 for a minimum of 5 years, of which the first 12 months are required to be a temperatures below 4 °C.

Major steps in the handling and processing of filters are:

- Obtaining filters from the EPA
- Conditioning and pre-weighing filters
- Packaging and shipping filters into and from the field
- Receiving, conditioning, and post-weighing filters
- Laboratory and weighing room environmental control
- Archiving filters

1.3 Definitions

Gravimetric Analysis—Determination of particulate concentration based on weight difference.
$PM_{2.5}$—Particulate Matter with an aerodynamic diameter less than or equal to 2.5 microns.
PM_{10}—Particulate Matter with an aerodynamic diameter less than or equal to 10 microns.

Filter Lot—Units of filters sent from the manufacturer, manufactured at essentially the same time and conditions.

Filter Batch—Units of un-sampled filters that are inspected and equilibrated at essentially the same time and conditions.

Weighing Session—Duration in which filter batches are weighed by a single

laboratory analyst, on a single balance, on a single date, interrupted by only brief breaks of no more than 15 minutes duration.

2.0 Interferences

The weights of filters are sensitive to certain interfering factors and conditions. The following are several common precautions to prevent unnecessary interference.

- During the transport of filters, there is a potential for weight loss through thermal or chemical decomposition and/or evaporation. This can be minimized by keeping the filters cool during transport.
- The potential effect of body moisture or oils contacting the filters is minimized by using non-sterile lab-grade gloves. This measure also moderates interference due to static electricity.
- Teflon® filters accumulate a surface electrical charge that may affect filter weight. Static electricity is controlled by holding filters near a Polonium-210 (^{210}Po) antistatic strip for approximately 20-30 seconds prior to weighing.
- 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 30-40% (±5% SD over 24 hrs.) and air temperature at a mean of 20-23 degrees Celsius (±2˚C SD over 24 hrs.).
- Airborne particulates can adversely affect an accurate mass measurement of the filter. Equilibrating filters shall 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 regularly, installing sticky floor mats at the entrance to the balance room, and wearing clean lab coats over regular clothing.

3.0 Apparatus and Materials

- Mettler XPR6UD5 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.
- Calibration weights, utilized as working standards(non-corrosive), are 300 mg and 500 mg and are certified as traceable to National Institute of Standards and Testing (NIST) mass standards. The working weights used during weighing procedures should be Class 1 with a tolerance of 0.01 mg.
- Calibration weights, utilized as primary standards (non-corrosive), range in weight from 300 mg to 500 mg and are certified as traceable to National Institute of Standards and Testing (NIST) mass standards. The Primary mass standards should be Class 0 with a tolerance of 0.005 mg.
- Radioactive (alpha-particle) Polonium-210 (^{210}Po) antistatic strips and/or a powered ionizer for static charge neutralization.
- Anti-static mat.

- Non-metallic, non-serrated forceps.
- Filter: Teflon® membrane, 46.2 mm in diameter with a polypropylene support ring.
- Filter support cassettes.
- Filter equilibration racks.
- Vaisala automated relative humidity/temperature recording system.
- Psychrometer (NIST certified) for calibration of relative humidity readings.
- Precision thermometer (NIST traceable) for calibration of temperature readings.
- Light box.
- Antistatic, powder-free vinyl gloves.
- Plastic Petri-slide filter containers.
- Disposable laboratory wipes.
- Filter equilibration cabinets.

4.0 Instrument Calibration and Verification

4.1 Analytical Micro-balance

The micro-balance will be calibrated/certified annually and maintained according to the manufacturer's recommendations. The calibration will use NIST-traceable mass reference standards. If at any time the micro-balance is found to be out of calibration, it will be recalibrated and recertified 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., 300 mg and 500 mg) will be routinely weighed each weighing session.

If verified and measured values of a working standard differ by more than 3 µg (i.e., three times the micro-balance's precision), the analyst will reweigh both working standards. If a discrepancy 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.

4.2 Mass Reference Standards

Mass reference standards should be 300 mg and 500 mg, which bracket the typical weight of a 46.2 mm Teflon® filter (325-425 mg). These standards must be NIST-traceable certified. Primary mass standards are Class 0 with a tolerance of 0.005 mg. Working mass standards are Class 1 with a tolerance of 0.01 mg.

Mass reference standards should be recertified annually at a National Voluntary Laboratory Accreditation Program (NVLAP) accredited 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 standards. Working standards are recommended for routine filter weighing and should be kept next to the balance in a protective container. Primary standards

should be handled carefully and kept in a secured compartment. Working standards should be verified against the lab's primary standards once a quarter. Always use smooth, non-metallic forceps for handling mass reference standards. These forceps should be used 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 QC logbook.

4.3 Recording Thermometer

The Vaisala Temperature meter will be certified annually. Additionally, periodic checks (a minimum of once every quarter) will be conducted with certified temperature standards to verify the accuracy of the Vaisala meter.

If the recording thermometer's value differs by more than 0.5˚C from the calibration thermometer, the analyst will investigate the problem and take appropriate corrective action which may include comparing the Vaisala thermometer to a different temperature standard or returning the recording thermometer to the manufacturer for service, calibration and certification.

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

4.4 Recording Hygrometer

The Vaisala Relative Humidity meter will be certified annually. Additionally, periodic checks (a minimum of once every quarter) of the recording hygrometer will be verified against the laboratory reference standard (a NIST traceable hygrometer). The verification will be performed at a minimum of one %RH level, which may be the current level in the conditioning environment.

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 corrective action which may include comparing the Vaisala thermometer to a different temperature standard or recertifying the lab's reference probe against NIST-traceable standard.

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

5.0 Sample Preparation and Analysis

5.1 Filter Receipt and Inspection

Shipment of 46.2 mm filters are sent annually 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.

After inspection, filters are placed in individual filter carriers, scanned into the MTL LIMS program, and loaded into the automated filter weighing chamber. Each slide is given a unique barcode label corresponding to the 7-digit numeric identification on each filter. The digits are preceded with a letter that identifies the filter media (T=Teflon®, N=Nylon, etc.)

5.2 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. The analyst will wear antistatic powder-free gloves. 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 also reduce the potential contamination of filters with body moisture/oils.

In the laboratory, each filter will remain in its sealed manufacturer's packaging until it is ready to be loaded into a filter carrier, scanned into the LIMS, and loaded into the automated filter weighing chamber. After the filter is weighed, it will be loaded into a filter cassette. Each filter will have a unique identification number. Filters will be processed sequentially to the extent possible.

5.3 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 manner that allows retrieval when necessary.

After sample post-weighing, filters will be archived to cold (≤4˚C) storage for a minimum of one year, followed by 4 years in storage at room temperature. Filters will be stored in sealed, marked containers to facilitate easy retrieval.

5.4 Blanks

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

5.4.1 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). Filters can be 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.

Clean unexposed filters will be randomly selected and designated as lot blanks. 9 random filters (3 random filters from 3 random boxes) from a single lot should be selected for the lot blank procedure test. Lot blanks are removed from their sealed manufacturer's packaging, scanned into the LIMS system, and loaded into the automated filter weighing chamber. The batch of lot blanks will be weighed, and then set to weigh 24 hours after the end of the first weighing procedure. The weight difference between the 2 filter weights, (initial weight and post-conditioning weight) must be less than 15 µg (i.e., 3 times the precision for weighing unexposed filters). If the weight difference is greater than 15 µg, then filters should be weighed periodically until the weight difference is less than 15µg.

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 lab QC notebook or an equivalent database.

5.4.2 Laboratory Blanks

Laboratory blanks are conditioned, clean, unexposed filters that are used to determine any weight change between pre- and post-sampling weighing due to contamination in the microbalance environment. One laboratory blank should be weighed with each batch (up to 9 filters) of a weighing procedure. Laboratory blanks should be stored in the filter weighing chamber to remain conditioned and protected from contamination. Upon post-sampling weighing of field filters, the laboratory blank weighed with that batch prior to sampling should be weighed with that batch. The pre- and post-sampling weights are recorded in the LIMS database. If the weight change exceeds 15 µg, contamination in the conditioning chamber may be occurring. Notify the QA Officer and Manager of Operations, and take appropriate troubleshooting and corrective actions. The pre- and post-sampling weights are recorded in the LIMS database.

5.4.3 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 in their protective containers,

momentarily installed in the sampler, removed and placed back in their protective containers during transport to the lab.

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.

The pre- and post-field blank weights are recorded in the LIMS database. If the weight change between pre- and post-field blank weighing exceeds 30 µg, contamination during transportation or at the sampling site may be occurring. Notify the QA officer and Manager of Operations, and take appropriate troubleshooting and corrective actions.

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.

5.5 Filter Conditioning and Weighing

5.5.1 Micro-balance Environment

The micro-balance is located in the environmentally-controlled automated filter weighing chamber. Dust contamination will be minimized by cleaning the weighing area frequently. Other measures for dust control will be implemented if necessary.

5.5.2 Initial Conditioning of Exposed Filters

Filters will be conditioned for a minimum of 24 hours to allow their weights to stabilize before being weighed.

Maintain consistent environmental conditions during pre- and post-sampling weighing. Mean %RH will be between 30 and 40 %RH ± 5% SD over 24 hours. Automatic room conditioning %RH and temperature will be measured continuously and recorded by the LIMS.

Within the filter conditioning environment, filters are placed in the AH500 weighing chamber. The weighing chamber provides a HEPA-filtered environment to prevent contamination of the filter. During conditioning, care should be taken to avoid cross-contamination of the filters by other materials that release particulates inside the chamber, such as quartz and glass media. Lab blanks should be used to check for potential cross-contamination from airborne particulates. If contamination occurs, take appropriate corrective action(s).

Filters are conditioned in the filter carriers in the automated filter weighing chamber. Filters should be scanned into the MTL LIMS software so the true conditioning time can be recorded and tracked.

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 using the lot blank procedure.

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. Additionally, filters will pass through a powered U-shaped ionizer prior to being placed on the micro-balance. Low %RH, heavy filter loading, particle materials, etc. can affect the time needed to neutralize electrostatic charges. Balance performance will ultimately determine the required measures to reduce or minimize electrostatic buildup.

Polonium antistatic strips reduce electrostatic buildup through charge neutralization. Electrostatic charges will be neutralized on items brought within an inch of the strips. These antistatic strips should be changed every 6 months. Properly dispose of used strips according to the manufacturer's recommendations.

5.6 Pre-sampling Gravimetric Analysis of Unexposed Filters

Pre-sample filters must be weighed in the same room that the filters were conditioned in. Filters must be weighed in a controlled environment.

5.6.1 Filter Lab Environmental Conditions

The weighing lab %RH and temperature will be recorded in the LIMS database. Analyst will verify that mean temperature and %RH for the last 24 hours has remained between 20 – 23˚C (± 2˚C SD over 24 hrs.) and 30 – 40%RH (±5 % SD over 24 hrs.). **If these conditions are not met, filters will not be weighed.** Appropriate troubleshooting and corrective actions will then be taken, and lab conditions brought into required specifications before any filters are weighed.

5.6.2 Balance Maintenance

Prior to weighing, inspect the balance weighing chamber for dust and debris. Clean the balance weighing chamber with alcohol-moistened disposable laboratory wipes as needed. Routine cleaning of the balance weighing chamber should be performed once a month. Clean the standard forceps with a lint-free cloth and the filter forceps with moistened wipes. Allow the forceps to air-dry. A more detailed laboratory cleaning schedule is described in section 5.11.

5.6.3 Balance Stabilization

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

For **manual filter weighing**, follow the below steps in section 5.6.4

5.6.4 Manual Filter Weighing

If manual weighing must be performed, ensure that it is being done in a climate-controlled environment.

In MTL's Filter Weighing System, go to "Manual Weighing">"Manual Filter Weighing" and select the "Operator". For a typical weighing session, the "Weighing Repetition" setting should be set at "1" and the "Method" should be set to "Direct Read." Verify that the current "Environment" setting is "In Tolerance." If the LIMS software has recently been restarted, it will take a few minutes (~3) for the software to register environmental conditions.

5.6.4.1 Select “Calibrate Balance”, wait for procedure to complete.

5.6.4.2 Select “Weigh Calibration Weight” and then click on the links for one of the two working mass reference standards (300 mg and 500 mg). The balance will tare automatically and the door will open. Use the standard forceps to handle the mass standards and follow the prompts on the screen to determine when to gently place and remove weights from the balance. (Warning: Always handle calibration weights with forceps rather than hands.) If there is more than a 3 µg discrepancy between the verified and measured values of the mass reference standard, reweigh the working standard. If the discrepancy remains, troubleshoot and take appropriate corrective action. Repeat the procedure with the remaining Calibration Weight.

5.6.4.3 Place filters to be weighed (about 6 at a time) on an anti-static mat. Use a ^{210}Po anti-static fan on open filter petri-slides or pass filters through the U-shaped ionizer before placing filter on the balance pan. Fan runs for 10 seconds at a time. Before starting the weighing procedure, run the fan about 4-6 times (40-60 seconds). After this, when a filter is weighed, add a new filter to the line-up and run fan 1-2 times.

5.6.4.4 One *Laboratory Blank* must be weighed every weighing event. A weighing event contains a maximum of 8 pre/post sampled filters and the laboratory blank. To weigh blanks, insert cursor in the “Filter ID:” box and then scan barcode with barcode reader. The LIMS system will automatically recognize that the filter is a blank. Follow instructions on the screen to weigh filter.

5.6.4.5 After weighing both the calibration weights and the laboratory blank, weigh the pre/post sampled filters. Eight filters (including one replicate weight) can be weighed sequentially. Scan the filter barcode of each filter for each weighing, taking care to make sure LIMS registered the correct filter id. The balance should automatically re-zero. 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. Immediately transfer the filter to the microbalance pan and close the weighing chamber door. Ensure that the filter ID, filter lot number and tare-weight (pre-sampling mass) have been recorded in the LIMS database. After a maximum of eight filters (and the replicate) are weighed, repeat steps 5.6.4.2 and 5.6.4.3 before more filters are weighed.

5.6.4.6 For the replicate filter, if the two measurements differ by more than 15 µg, reweigh the filter. If the measurements still disagree, troubleshoot and take 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.

5.6.4.7 At the end of the weighing session, reweigh a laboratory blank and both working standards. If either of the working standard measurements disagree from the verified value by more than 3µg, reweigh that standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action.

For **automated filter weighing**, follow the below steps in section

5.6.5 Automated Filter Weighing

In MTL's Filter Weighing System, go to "Automated Filter Weighing" and select the "Operator".

5.6.5.1 Click the dropdown list next to "Procedure" and select "Standard Procedure".

5.6.5.2 Select "Configure Procedure" and edit the "Standard Procedure" to modify the supply and storage silos for the procedure. The supply silo is where the arm will take filters from, the storage silo is where the arm will place the filters after weighing them. Save changes and return to the "Automated Filter Weighing" screen.

5.6.5.3 Select "Begin Procedure" and set the start date to be at least 24 hours from the current date and time. This is the conditioning period for those filters.

5.6.5.4 When the procedure runs, it will begin with a start bracket QC protocol which includes a 5-step internal balance calibration and weighing of the 300 mg and 500 mg Class 1 working weights. Following this QC protocol, it will weigh the 500 mg Class 1 working weight to bracket the first batch of filters. 9 filters are weighed per batch, with the first filter being reweighed at the end (replicate sample) for a total of 10 filter weights per batch. Between each batch is the QC protocol of the 5-step internal balance calibration and weighing of the 500 mg Class 1 working weight. Once all filters have been weighed, the system will perform an end bracket QC protocol similar to the start bracket QC protocol where a 5-step internal balance calibration and weighing of the 300mg and 500mg class 1 working weights are performed.

5.6.5.5 Any unused filter whose weight is outside the normal range of 325-425 mg should be investigated. If there is a consistent negative replicate mass reading (>15 µg) for reweighed filters, it is usually a sign that the filters have not equilibrated long enough. In this case, troubleshoot and take corrective action.

5.6.5.6 Following pre-sample weighing, filters should be placed in a filter cassette and protective tin, labeled with the unique filter ID, batch number, and weigh date.

5.7 Pre-Sampled Transport of Filters

5.7.1 The lab analyst will place pre-sampled weighed filters into clean filter

cassettes and stored in individual tins, clearly marked with the filter's pre-sampling weighing date.

5.7.2 When it is time for filters to be used at sites, the site technician will place cassette assembly into protective containers for transport to site. Prepare and transport several extra filters to the site in case a filter must be invalidated during the installation process.

5.7.3 If filters are to be shipped out of town, be sure the site operator has bubble wrap, insulation, gel packs, min/max thermometer, address label, and a cooler to ensure that exposed filters are protected and chilled to ≤ 4˚C during shipment back to the analytical laboratory. Filter slides are individually marked with initial weighing date for operator's reference, and operators will mark filter tins with sampling date for the lab analyst's reference. Un-sampled filters are sent to operators at room temperature and are to be sampled within 30 days of the initial weighing date.

5.8 Post Sampling Conditioning of Exposed Filters

Filters will be kept at a temperature of < 25˚ C, and preferably < 4˚ C, from retrieval from the sampler until the start of post-sampling conditioning in the weighing lab. All exposed filters will be weighed within 10 days from the sampling end date if kept and shipped at ≤ 25˚ C, or within 30 days from the sampling end date if kept and shipped at ≤ 4˚ C. Upon receipt of the filters from the field, the laboratory analyst will follow these steps:

5.8.1 Lab analyst who receives and logs in sample shipment will verify that the temperature of the interior of the insulated shipping container was maintained at ≤25˚C, or at ≤4˚C, depending upon interim storage conditions. The maximum temperature registered by the shipping thermometer will be recorded in the LIMS database or an appropriate laboratory notebook. Filters will be removed from the cooler and checked to see that lids of the filter tins are secure. If any lids opened during shipment, or loose particulate matter or debris is found, the analyst will record on the appropriate shipping form and in the LIMS database that the sample has been flagged for possible voiding. After sample receipt and filter inspection, the analyst will store filters/filter containers, exposed side up, in the lab refrigerator (≤4˚C and ≥ 0°C) until ready for filter post sampling equilibration and weighing.

5.8.2 The lab analyst will receive data from all site samplers directly from the site operator. Instrument data download files will be sent along with sample filter paperwork.

5.8.3 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.

5.8.4 Prior to post-sampling filter conditioning, remove the exposed filters from the lab refrigerator and transfer to the automated filter weighing chamber. 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 annotated in the LIMS database. 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, scan each filter barcode and place in the filter weighing chamber to begin the equilibration process. Filters must be equilibrated within required lab %RH and temperature specifications for a minimum of 24 hours and a maximum of 48 hours prior to weighing.

5.9 Post Sampling Gravimetric Analysis of Exposed Filters

5.9.1 Pre- and post-sampling filter weighing are to be performed on the same micro-balance. Different analysts can perform the pre- and post-sampling filter weighing. 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 ≤ 4˚C during the entire time between sample filter retrieval and the start of filter equilibration, in which case weighing must occur within 30 days. If the filters cannot be weighed within 10 days, then the temperature cannot exceed 4˚C, or else the filter becomes invalidated. Ensure that filters never get so cold that they freeze, as this may affect the structural integrity of the filter.

5.9.2 For manual weighing, repeat steps 5.6.4.1 through 5.6.4.7 in the pre-sampling weighing procedure. For automated filter weighing, repeat steps 5.6.5.1 through 5.6.5.4 in the pre-sampling weighing procedure.

5.9.3 If the pre- and post-sampling weights for the lab blanks disagree by more than 15 µg, 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 as instructed by the QA officer or Manager. Observations and corrective actions should be noted in the lab notebook and the filter lab data review checklist. 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.

5.9.4 Return filters to the filter handling containers and store in the lab freezer until transfer to filter archiving.

5.10 Shipping Overview

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 that were pre-weighed on the earliest date should be the first to be deployed in samplers so that all filters will be used within 30 days of the pre-sampling weighing date. Filters are printed with a unique 7-digit ID number by the manufacturer. The digits may be preceded by a letter which identifies the filter media (T = Teflon®, N = nylon, etc.).

5.10.1 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 weighing 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.

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 for pre-sampled filters (from the weighing room to the sample location); however, excessive heat should be avoided. Pre-weighed filters must 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.

For post-sampled filters, 177 hours is the maximum allowable time for recovery of a sample filter from the monitor following completion of a sample run. Aside from being a *critical criteria*, according to 40 CFR part 50 App. L, a lengthy recovery time may impact the ability of lab analysts to perform final weights within 10 days. During this time prior to sample recovery, the temperature within the sampler's filter housing (T_f) is not to exceed the ambient temperature (T_a) by more than 5˚C.

Ideally, filter transport will not exceed 24 hours. Filters stored at ≤4˚C, from the end of the sample run to the date of laboratory-conditioning for post-weighing, have a 30-day holding time. Filters maintained at ≤25˚C, from the end of sample run to the date of laboratory-conditioning for post-weighing, have a 10-day holding time.

Note: Permissible $PM_{2.5}$ sample holding times are detailed in 40 CFR Part 50 Appendix L, Section 8.1 of the U.S. EPA QA Handbook for Air Pollution Measurement Systems, Vol II., EPA Technical Note—Holding Time Requirements for PM2.5 Filter Samples October 23, 2015, and Quality Assurance Guidance Document 2.12 "Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods"

Field operators will be supplied with insulated shipping containers and ice substitute gel pouches that can maintain the filters' temperature at ≤4˚C during shipment back to the weighing lab. Samples should be stored in canisters 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 sample's proximity. Ice substitutes will be packed around the samples. Samples will be shipped using an express delivery service that will minimize shipping time, such as Alaska Airlines Goldstreak or FedEx.

5.10.2 Filter Receiving and Tracking

Upon receipt of the samples, filters will be stored in a refrigerator at a temperature between 0˚C and 4˚C. Filters are logged (scanned) into the LIMS database and moved into the filter-weighing lab for lab-conditioning at least 24-48 hours prior to weighing. Sample shipping information will also be entered into the lab logbook or equivalent. This should include location of filter samples, shipper, min/max temperature, date/time/analyst receiving shipment, any other significant information, and the apparent integrity of samples.

5.11 Laboratory Cleaning Schedule

5.11.1 Prior to Weighing

Prior to weighing, the balance should be dusted with an anti-static brush. Surfaces near the balance and laboratory forceps should be wiped down with lint-free wipes (such as Kimwipes) moistened with isopropyl alcohol or water and allowed to air-dry prior to use.

5.11.2 Weekly Cleaning Schedule

At least a day prior to weighing, laboratory surfaces should be dusted (including on top of cabinets and air conditioner). Lab floors should be mopped, and lab surfaces (including the interior of the filter equilibration chamber) should be wiped down with pre-moistened wipes.

5.11.3 Filter Cassette Cleaning Schedule

Filter cassettes, tins, and screens are cleaned after being returned to the lab after sampling and final weighing. Isopropyl alcohol pre-moistened wipes and/or Kimwipes are used to remove any residue. Cassettes, tins, and screens are left to air dry before further use.

6.0 Data Acquisition, Calculations, Validation, and Reporting

6.1 Data Acquisition

The analytical microbalance is linked to a computer which records filter weights, continuous weighing room temperature and %RH electronically through Measurement Technologies Laboratory's (MTL) laboratory information management system (LIMS). Filter run data will be downloaded from particulate monitors on-site and sent to the lab with the appropriate sample filter paperwork. The laboratory analyst will subsequently upload the electronic data to LIMS.

6.2 Calculations

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

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).

Using the post-sample and pre-sample filter weights, 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)

Calculate the $PM_{2.5}$ concentration as:

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

Calculate the PM_{10} concentration as:

$$PM_{10,actual} = M_{10}/V_a \qquad \text{Equation (4)}$$

Calculate the standard PM_{10} concentration as:

$$PM_{10,standard} = (PM_{10,actual})(T_{ambient}/T_{standard})(P_{standard}/P_{ambient}) \qquad \text{Equation (5)}$$

Where: $PM_{10,\ standard}$ = standard PM_{10} concentration (µg/m^3)

$PM_{10,actual}$ = actual PM_{10} concentration

$T_{ambient}$ = average ambient temperature of sample (Kelvin)

$T_{standard}$ = 298 (Kelvin)

$P_{standard}$ = 760 (Torr)

$P_{ambient}$ = average ambient pressure of sample (Torr)

6.3 Data Validation

Data is exported directly from the LIMS system and imported into the Air Vision database for further review. Data validation is the responsibility of the local air monitoring network including site operators. The MTL LIMS software calculates sample $PM_{2.5}$ concentrations based upon pre- and post-sample filter net mass and sample data. Monthly, final sample concentrations with attendant raw data spreadsheets are imported into the Air Vision database.

6.3.1 Data imports into Air Vision

Following the import of sampler data into the LIMS program, where sampler data and filter weight data are combined to determine PM concentration and data validity, the lab operator exports the combined data from LIMS and imports the files into the Air Vision database. Sample and field blank files are created for each sampler and imported into the Air Vision database for operators and data reviewers to verify and validate the data. If any scheduled sample days were missed in the time period of the export, empty rows containing the scheduled date and sampler ID

should be included, as missed scheduled sample days must be included and nullified.

6.4 Data Reporting

The method detection limit is 2 µg/m^3, however, data below this limit is still reported to AQS.

7.0 Quality Control and Quality Assurance

A laboratory QC notebook, LIMS electronic database, and monthly lab data review checklists 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 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. Internal QA audits will be performed annually, and an external EPA Technical Systems Audit will be performed every 3 years.

Semi-annually, a batch of pre-sample and post-sample filters will be conditioned and weighed by one gravimetric lab and sent to the other to be conditioned and weighed for QC audit purposes. Filters will be conditioned and weighed mimicking normal filter weighing equipment, operations, and procedures. Weight difference between the two labs should be less than 15 µg.

8.0 References

EPA 2006. 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 2016. 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.

EPA. Filter Conditioning and Weighing Facilities and Procedures for PM2.5 Reference and Class I Equivalent Methods.

EPA. Technical Note—Holding Time Requirements for PM2.5 Filter Samples. October 23, 2015.

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

RTI International. Standard Operating Procedure for Particulate Matter (PM) Gravimetric Analysis. Environmental and Industrial Sciences Division RTI International. Research Triangle Park, North Carolina.