TITLE: Determination of Particulate Matter (PM) Gravimetric Mass for the Chemical Speciation Network

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1.0 Purpose

The purpose of this Standard Operating Procedure (SOP) is to provide consistent guidance to Wood Environment & Infrastructure Solutions, Inc. (Wood) laboratory personnel for the determination of particulate matter gravimetric mass for the Chemical Speciation Network (CSN).

2.0 Scope

This method provides for the measurement of the mass concentration of particulate matter (PM) samples collected on Teflon filters with an aerodynamic diameter less than or equal to 2.5 micrometers ($PM_{2.5}$). This measurement process is nondestructive, and $PM_{2.5}$ samples can be subject to subsequent physical and chemical analyses.

Note: Final data validation, including the application of flags or codes, is performed external to the laboratory by CSN Program Management personnel.

2.1 Detection Limit

Detection limits and sensitivity will vary with the measured airflow through the filter and the balance's precision. A nominal instrument detection limit of 3 micrograms (μg) per filter is established as three times the instrument's specified precision of 1 μg and verified by replicate measurements of mass reference standards. A program to determine the mass method detection limit (MDL) using measurements from approximately 10 sites using monthly field blanks has been initiated for the CSN program. Calculations of results for that program will be performed external to the laboratory by the CSN Program Management personnel.

2.2 Holding Time

Holding time from pre-weighing of the filter to sampling is 30 days. The holding time from the completion of sampling to final weighing is determined based on the temperature measured upon arrival at the CSN Filter Shipping and Handling Unit (FiSH) and the average temperature during sampling. The criteria is slightly different for CSN sites than for tribal or other mass only sites. These criteria (and the corresponding AQS flags (which are applied external to the laboratory) are provided below.

For CSN sites that require gravimetric mass determination (e.g., have no Federal Reference Method (FRM) sampler), the following criteria exist:

- Sample arrives below 4 degrees C – no AQS flag, 30 days to weigh
- Sample arrives above 4 degrees C but below average temperature during sampling – TT AQS validity flag applied and 30 days to weigh
- Sample arrives above 4 degrees C and above average temperature during sampling – TT AQS validity flag applied and 10 days to weigh

For these 3 scenarios, if the window for weighing is missed and/or the sample arrives above 25 degrees C, then the TS (holding time or transport temperature is out of specs) AQS null code should be applied (invalidating the sample).

For tribal and other gravimetric mass only sites:

- Average ambient temperature during sampling was below 4 degrees C and the sample arrives below 4 degrees C – 30 days to weigh
- Average ambient temperature during sampling was below 4 degrees C and the sample arrives above 4 degrees C – 10 days to weigh
- Average ambient temperature during sampling was above 4 degrees C and the sample arrives below the average ambient temperature during sampling – 30 days to weigh
- Average ambient temperature during sampling was above 4 degrees C and the sample arrives above the average ambient temperature during sampling – 10 days to weigh

For these 4 scenarios, if you miss the window for weighing and/or the sample arrives above 25 degrees C, then the TS (holding time or transport temperature is out of specs) AQS null code should be applied (invalidating the sample). The gravimetric laboratory does not have access to the ambient temperature data and these codes are applied external to the laboratory.

3.0 Summary of Method

After moisture equilibration in a controlled atmosphere, each filter is weighed before and after exposure to determine the net weight (mass) increase on an exposed $PM_{2.5}$ filter.

Data are captured into an Access© database (AutoWeight v36). Sample data are held according to analysis date and employee number. Data are exported into spreadsheet files and uploaded into the Element™ Laboratory Information Management System (LIMS) where batches are created (see GLO-3180-035). The report files serve as log pages.

This method uses an electronic microbalance to make precision measurements in the microgram range in a controlled environment. These balances are by nature delicate and precise.

3.1 Definitions

- Gravimetric Analysis – Determination of particulate concentration based on weight difference.
- $PM_{2.5}$ – PM with an aerodynamic diameter less than or equal to 2.5 microns.

- Filter Lot – Units of filters from a single type, grade, class, size and composition, manufactured under essentially the same conditions and time by the same manufacturer.
- Filter Batch – Units of unsampled filters inspected, equilibrated under essentially the same conditions and time and weighed in the Gravimetric Laboratory for delivery to FiSH.
- Weighing Session – Period of time in which filters for one client are weighed by one Laboratory Analyst on one balance on one date, interrupted only by brief breaks of no more than 15 minutes.

3.2 Method Interferences

Several types of effects, if present, may contribute to interferences in the determination of $PM_{2.5}$ mass. They can be summarized as follows:

- Loss of mass due to chemical decomposition or vaporization of collected compounds. Semi-volatile organic compounds on the filters can be lost during shipment or storage. Collected compounds such as ammonium nitrate can break down and the products evolve as gases. The conditioning processes as outlined in Section 6.5 of this SOP are designed to minimize these losses.
- Positive errors due to the retention of gaseous species such as sulfur dioxide and nitric acid. The increase in mass resulting from the formation of sulfate particles from chemical reactions of sulfur dioxide gas at the filter surface can be minimized by using an essentially neutral Teflon® filter for the collection surface. Errors of this nature are expected to be minimal for most sampling locations.
- Adsorption or desorption of water vapor on the filter surface or on the collected particulate matter can result in either positive or negative errors. The conditioning process outlined in the Section 6.5 of this SOP is designed to minimize biasing moisture effects.
- Electrostatic interferences - polymer membrane filters, being poor electrical conductors, are the most prone to this problem. The buildup of electrostatic charges on the filters during manufacture or the sampling process can interfere with the microbalance and cause significant errors. A&D Company, Limited produces an anti-static device that eliminates static electricity effectively. The additional use of Polonium-210 (^{210}Po) anti-static strips before weighing further reduces static buildup.
- Air- or surface-borne particulate matter in the form of dust or other debris can positively bias either the initial or final mass measurement. It is imperative that the filter weighing and storage areas are clean.
- To minimize contamination from the external environment, laboratory personnel will always wear clean laboratory coats, gloves and shoe covers.
- Filters will only be handled with clean forcepts.

- Relative humidity (rH) and temperature can also impact the ability to achieve accurate measurements. The procedures outlined in Section 6.1 of this SOP address corrective measures whenever environmental controls are out of specification.

3.3 Personnel Qualifications

Personnel employed to perform weighing operations must have a minimum of a high school diploma with at least 6 months experience in laboratory sample handling and record-keeping practices. Training is conducted by a lead analyst with a minimum of a bachelor's degree in a science related field and at least 6 months of experience in the gravimetric laboratory (a minimum of 10 years gravimetric laboratory experience may be substituted for the degree requirement).

3.4 Deviations from the Method

A general overview of the steps described in this SOP is depicted in Figure 1. Deviations from the analytical method described in this SOP are not permitted.

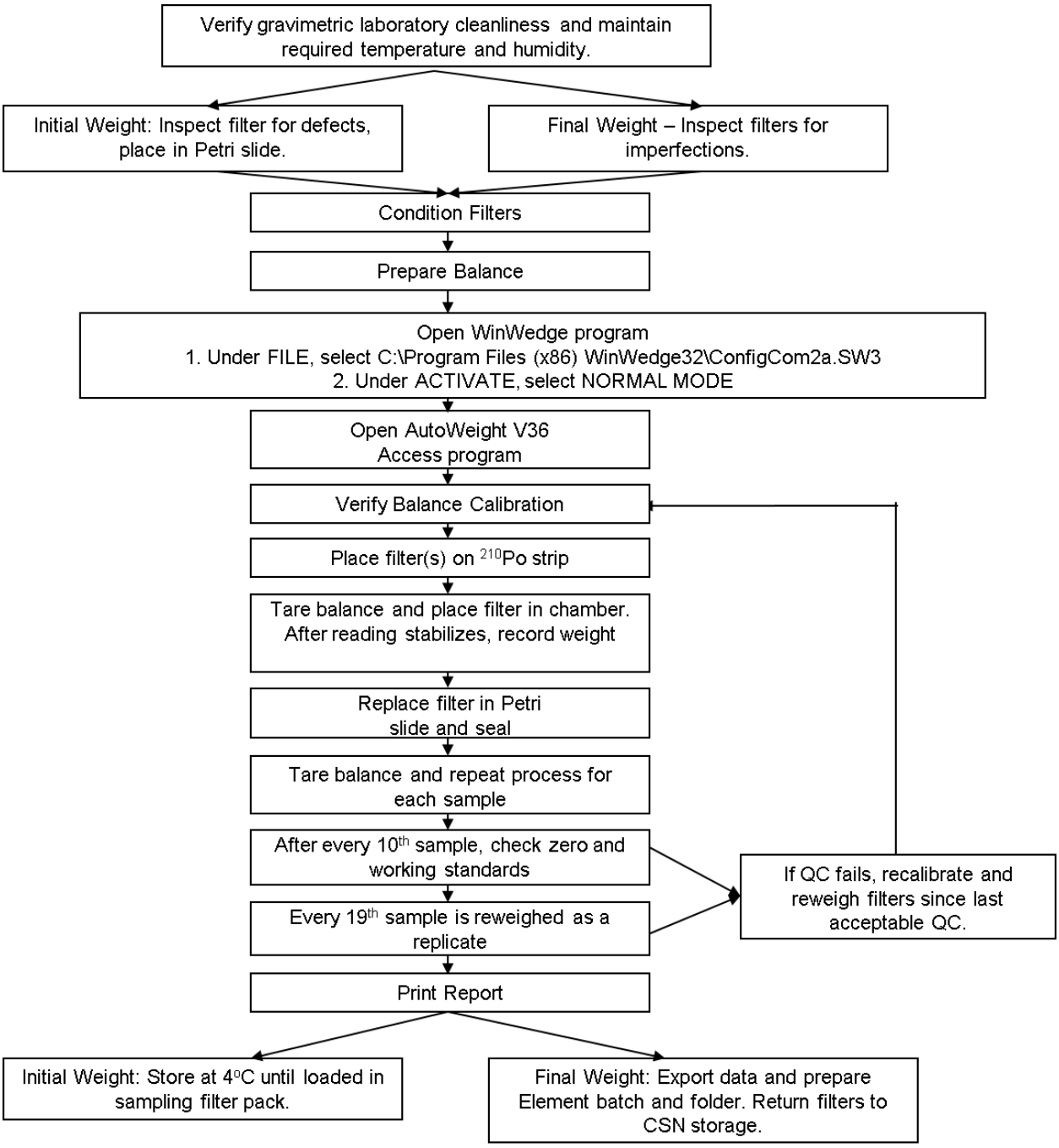

Figure 1. Overview – Determination of Particulate Matter

4.0 Materials

- Sartorius ME-5 Microbalance (or equivalent, see Figure 2) with a display of 0.001 milligram (mg) or 1 μg with a reproducibility of ± 1 μg. The balance must be identified by a unique balance identification sequence (or serial number) and calibration verified prior to each weighing session according to the manufacturer's instructions.
- Controlled weighing environment. Sufficient size for weighing apparatus, with consistent and adjustable temperature and humidity. Must be free from air currents.
- AD-1683 DC Anti-static device, A&D Company Limited
- ^{210}Po anti-static strips
- High-efficiency particulate air (HEPA) filtration system
- Commercial dehumidifier, adjustable
- Dickson Data Logger, National Institute of Standards and Technology (NIST)-traceable annual calibration, for recording temperature and humidity.
- Adhesive disposable floor mats for removal of particulates from shoes
- Light system to inspect filters for defects
- Anti-static mat (optional).
- Commercial anti-static spray (optional).
- Alcohol wipes, individually wrapped.
- Powder-free gloves.
- Disposable protective booties.
- Kimwipes
- Smooth plastic forceps for use with standards. These forceps should be clearly labeled for use with mass reference standards only.
- Two sets of mass reference standards (one working set and one primary set), each consisting of one each American Society for Testing and Materials (ASTM) Class 1 100, 200, 300 and 500 mg weights. Each weight is NIST certified with an individual tolerance less than or equal to 0.010 mg. Each standard weight will be re-certified yearly at an NIST or NVLAP (National Voluntary Laboratory Accreditation Program) accredited calibration laboratory. Copies of the relevant certificates must be included in every data batch. Class designation should be verified by ASTM E617-13.
- Clean, smooth (non-serrated) stainless steel (conductive) forceps labeled for filter use only.
- Plastic forceps, for handling calibration check weights only.
- 2-μm pore size PTFE (polytetrafluoroethylene) 47-mm membrane filters for $PM_{2.5}$ (pre-numbered).

- 60 x 15 mm and/or 50 x 9 mm plastic Petri slides.
- Conditioning racks.

5.0 Safety

Although no chemical reagents are used in this method, proper laboratory procedures should be followed at all times to prevent accidents and to minimize sample contamination. No food or drinks are allowed in the filter room. Powder free gloves, disposable booties, and lab coats will be worn at all times.

Personnel must exercise caution when using antistatic devices containing radioactive polonium sources. The devices must be disposed of according to the manufacturers' specifications, Wood health and safety guidelines and state and local regulations.

Figure 2. Sartorius ME-5 Microbalance

6.0 Procedure

Micro-analytical weighing is a technique-dependent process. Every attempt should be made to establish and follow routines that will ensure the accuracy and precision of the measurements made.

6.1 Environmental Conditions

1. Micro balance environmental conditions are pivotal in generating high quality data. The microbalance is located in a clean, climate controlled, draft-free laboratory space equipped with a HEPA filtered air supply system and is dedicated to the storage, conditioning, and weighing of filters only.
2. The laboratory and the analytical balance should be cleaned routinely and an adhesive floor mat placed in the room entrance to control dust contamination. Traffic is minimized in the controlled weighing environment. The cleaning performed is recorded in the Cleaning Schedule Logbook. See Attachment C for a room cleaning checklist and schedule (includes changing of adhesive floor mats).
3. The anti-static precautions are important. The AD-1863 generates bipolar ions that are continuously directed in an air stream that is sufficiently balanced in ion polarity to eliminate static charges on objects regardless of the polarity of the charge. Additional precautions may include the use of an anti-static pad placed under the balance to minimize electrostatic buildup. An anti-static solution may be applied to the nonmetallic surfaces around the balance as needed.
4. The balance is set up on a marble slab table to maximize stability and minimize vibration. The microbalance should remain powered on at all times to maximize stability.
5. A Dickson Data Logger (see Figure 3) is used for recording temperature and humidity measurements at five minute intervals. The data logger sensors are returned to Dickson annually to be recertified with NIST-traceable calibration standards.
6. Appropriate humidity controls ensure measurements that are unbiased by water mass. Use a dehumidifier, as necessary, to maintain a mean relative humidity between 30 – 40%, with a standard deviation of not more than $\pm$ 5.1 % RH over 24 hours. Mean temperature should be held from 20 – 23 °C (68 – 73.4 °F) with a variability of not more than $\pm$ 2.1 °C ($\pm$ 3.8 °F) over a 24 hour period. Filters will not be weighed if these conditions are not met and the HVAC contractor will be notified to service the unit. Filters will not be weighed until the room conditions meet the 24 hour requirement.
7. The readings from the data logger will be downloaded daily to a USB jump drive. The results will be transferred to an Excel spreadsheet for calculation of maximum, minimum, average and standard deviation for the 24 hour period. A chart of the results will be printed and stored in a notebook in the gravimetric lab (see Attachment H).

8. The weight room maintenance logbook is used to record any abnormalities in room conditions.

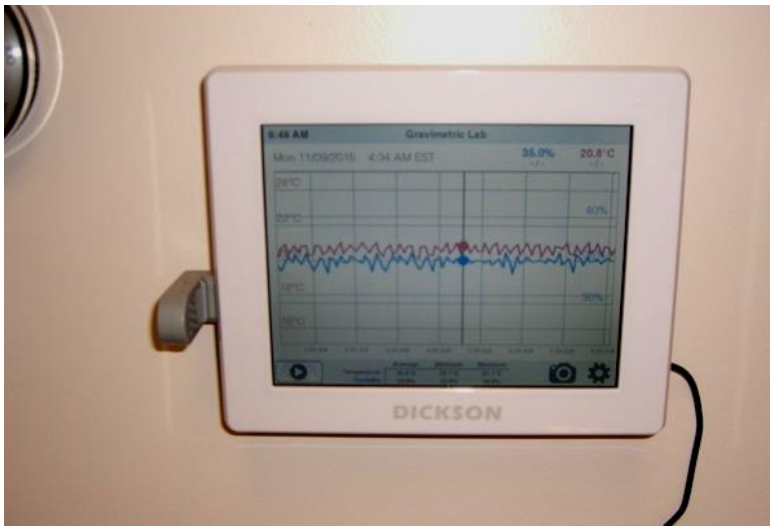

Figure 3. Dickson Data Logger

6.2 Filter Handling

1. Extreme caution in handling the filters is necessary to avoid damaging the filter or dislodging any of the collected particulate matter.
2. Powder-free gloves must be worn whenever filters are handled. Please note that gloves that are packed in a box can carry an electrostatic charge. Touching a ^{210}Po strip should effectively discharge each glove.
3. Touch only the polyolefin reinforcing ring on the outside edge of the filter with the forceps when handling filters. Use only the forceps designated for filter handling.

6.3 Filter Integrity Check

Perform filter inspection before obtaining initial weights. Using a light table, visually inspect all filters for defects prior to initial weighing. Discard any found to have defects and record the filter number in the Rejected Filter Logbook. Examples of defects are as follows:

- Pinholes – small holes visible as bright points of light when viewed over a light table.
- Chaff or flashing – extra material found on the polyolefin reinforcing ring or on the heat-seal area that inhibits an air-tight seal.
- Filter discoloration – obvious discoloration may be a sign of filter contamination.
- Loose material – any extra material or dirt particles on the filter surface.
- Filter non-uniformity – any visible indication of gradation in porosity or density across the filter surface.

- Any other imperfection such as irregular filter surface that would indicate poor quality control.
- Place each filter to a Petri slide. Store the filters in these containers except when weighing.

6.4 Initial Lot Stability Check

When filters are received from a manufacturer or lot not previously used in the laboratory, an Initial Lot Stability Check must be performed to determine the minimum amount of time required to condition the filters. The results of the test will be recorded in the Lot Stability Logbook.

1. All new boxes of filters will be stored in the gravimetric laboratory for a minimum of 60 hours before initial weights are taken for the lot stability test or field sampling.
2. Randomly select two filters from each of six boxes of the lot and inspect as described in Section 6.3. Weigh each of the filters as outlined in Sections 6.7-6.9 and place each in separate Petri slides.
3. Allow the filters to equilibrate for 24 hours and reweigh.
4. Continue the 24 hour equilibration and weighing for 5-7 days. The filters are considered equilibrated when the weight change for each filter is less than 15 µg in a 24 hour period. This will be the minimum time that all filters from this lot must equilibrate prior to performing a batch stability test as outlined in Section 6.6.
5. The Laboratory Operations Manager (LOM) will review the data for trends. Even if the weights are within $\pm$ 15 µg, continuously decreasing weights could indicate outgassing and the test should be continued until the downward trend stops. If the trend is an increase in weight, it may indicate laboratory contamination which must be identified, rectified and the test repeated.

6.5 Filter Conditioning

1. Filters must be conditioned (equilibrated with the atmosphere of the laboratory) before both the initial and final sample weighing. The filters are considered equilibrated when the weigh change for each filter is less than 15 µg in the period determined by the Initial Lot Stability Test (see Section 6.4). Condition new filters in an open petri slide for a minimum of 24 hours on an elevated stand. Allow returning filters to equilibrate in the laboratory for a minimum of 24 hours (additional equilibration may be necessary for very damp filters).
2. Ensure that the temperature and humidity are within the control limits specified in Section 6.1.6 of this SOP. If the relative humidity and/or temperature are not within control limits, do not weigh filters. Take the steps necessary to bring the laboratory within parameters and maintain them for a 24-hour period prior to weighing filters.
3. Initial and post-sampling conditioning periods must be within +/- 2 ^{o}C and +/- 5 % rH before starting a weighing session for final weights.

4. If the exposed filters cannot be weighed promptly, they must be stored at 4°C until the post-sampling conditioning can occur.

6.6 Batch Stability Test

1. Randomly select three filters from a batch of filters that were conditioned for the minimum required time as outlined in Section 6.5.
2. Weigh each of the three filters (see Sections 6.7-6.9) and record their weights in the laboratory $PM_{2.5}$ QC logbook.
3. Allow the filters to equilibrate overnight and reweigh.
4. If the average weight loss for the 3 filters is less than 5 µg, the remaining conditioned filters re ready to weigh.
5. If the average weight loss exceeds 5 µg, repeat the 24-hour equilibration and weighing until the average weight loss for the 3 filters is less than 5 µg.

6.7 Balance Preparation

1. The microbalance will be serviced by a factory authorized technician from an accredited vendor annually, after the balance is moved, or if the balance fails daily verification checks. During the service call, the balance is cleaned internally and externally, the internal weight is checked and cleaned (if applicable), and the balance is calibrated.
2. Plug in the antistatic device. Make sure that a ^{210}Po strip is placed within the "X" marking the range of the device (see Figure 4).
3. Clean the microbalance. Turn the balance off; remove the cover, and dust the platform and tray thoroughly with a brush. Clean with an alcohol wipe, if necessary, and air-dry. Do not use pressurized gas; damaging debris and oils may be blown into the microbalance mechanism. Dust the interior before replacing the cover, making sure the grooves are aligned properly. Check level indicator on top of the balance and make sure the bubble is centered before proceeding. If the balance is moved and/or the bubble is not centered, the balance will require recalibration. Turn the balance back on once the cleaning procedure is completed.
4. Clean the surfaces near the balance with an anti-static solution or alcohol-moistened wipes. Wipe both sets of forceps with a lint-free laboratory wipe. If necessary, clean with an alcohol-moistened wipe. Be sure to allow the forceps to dry thoroughly before using; a small amount of moisture can cause a significant measurement bias.

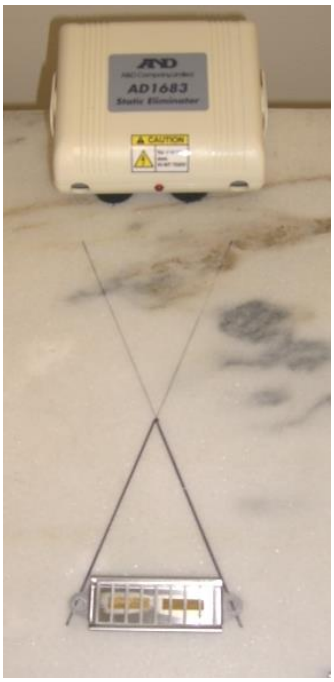

Figure 4. Antistatic Device and ^{210}Po strip

6.8 Balance Calibration Verification

The balance calibration must be verified each day prior to weighing filters. The $PM_{2.5}$ QC Logbook is used for recording the QC associated with each weighing session (see Attachment E).

1. AutoWeight Set-Up
   - Open WinWedge by selecting the WinWedge shortcut icon.
   - Under “FILE”, select C:\Program Files (x86) WinWedge32\ConfigCom2a.SW3
   - Under “ACTIVATE”, select NORMAL MODE
   - Log into the AutoWeight database.
   - Open the AutoWeight database by selecting the *AutoWeight v36* shortcut icon.
   - Fill in the **Options** form with Analyst, Balance, and Mode (i.e. Initial Weights or Final Weights).
   - Select the Start Weighing Operations button.
2. Place the references on the ^{210}Po strip within the antistatic device range.
3. Press either *Tare* button on the balance keypad to zero the balance (tare the balance), or, alternately, by clicking on the *Tare Balance* button on the Weighing Operations screen. Verify the zero by pressing the button located directly beneath the Cal option on the balance display twice. Record the zero weight in the $PM_{2.5}$ QC Logbook. The zero must read 0.000 ± 2 μg.

4. Weigh the two working calibration standards, or references that bracket the filter weights. Make sure each weight is centered on the platform. Do not touch the weights, use plastic forceps; even fingerprints will change the mass considerably.
5. Select RF (reference) as the sample type; the default value is DA (data point).
6. The Filter ID field is completed using the following format: RYY-0000#, where "YY" is the two-digit year and "#" is 1 for the initial references. "#" increases incrementally with each additional reference.
7. Fill in the Description field with STDxxx.
8. Place the mass on the balance using smooth angled plastic forceps. The balance door can be opened and closed from either side using the large arrows located on either end of the balance keypad area. On-screen buttons are also available within the AutoWeight database forms for these functions, i.e. *Open Balance Left*, *Open Balance Right*, and *Close Balance.*
9. Select *Capture Balance Weight* to bring up the previous reference's mass. Click the same button again to capture the current data.
10. Select *Save Sample Data* to save captured data and advance to the screen for the next reference
    - Remove mass, close the door, and tare balance for the next reference
    - Repeat for the remaining reference(s).
11. Record the certified and measured values for each reference in the $PM_{2.5}$ QC Logbook with any comments necessary. See Attachment A for acceptance criteria and corrective actions.

6.9 Method Blanks

Method Blanks (MB), or laboratory blanks, are conditioned as outlined in Section 6.5 and the Batch Stability Test is performed as described in Section 6.6. After the balance calibration verification (Section 6.8), the initial weights for the MBs are determined as follows:

1. A minimum of three MBs will be prepared. If more than 30 sample filters are in the batch, an additional MB will be prepared for each additional 10 sample filters.
2. Lay them out on the ^{210}Po strips before weighing them. Begin weighing with the blank filter on the strip in front of the antistatic device (see Figure 5). Carefully wipe the strip between each sample with a clean Kimwipe to prevent cross contamination.
3. Select MB for sample type. Use the form MYY-0000# for the Filter ID field. In Initial Weight Mode, use the drop-down options to select "MB1," "MB2," etc. to fill the Description field. Using smooth metal forceps place the first blank filter on the balance platform and close the door.
4. Allow the reading to stabilize (steady reading for at least thirty seconds) before capturing data. While the balance is stabilizing, move one of the other filters already on the ^{210}Po strips to the strip in front of the anti-static device. Shift the

other filter to the most recently vacated ^{210}Po strip and lay out another filter on the last strip. Repeat this shifting and cleaning process as each filter is being weighed (see Figure 5).

5. Remove filter, tare, and repeat for remaining blank filters.
6. Record the weight of each blank in the $PM_{2.5}$ QC Logbook.

Figure 5. Sample Set-Up

6.10 Sample Analysis – Initial Weights

1. Determine the number of sample filters required by the CSN FiSH group.
2. The sample filters are conditioned (see Section 6.5), the room conditions verified (see Section 6.1.6), the balance calibration verified (see Section 6.8), and the MBs are prepared (see Section 6.9).
3. Lay out ten Petri dishes at a time (see Figure 5).
4. Place two filters out at a time on the ^{210}Po strips, numbered side up. Begin weighing with the filter on the strip in front of the antistatic device.
5. Open AutoWeight, select Initial Weights and enter the Filter ID. Verify that the filter number of the first filter matches the sequence before proceeding.
6. Using the smooth metal forceps, place the first filter on the balance platform, touching only the reinforcing ring and making sure the filter is centered on the

platform. Close the balance door. Allow the filter to remain on the balance until a stable reading is reached (thirty seconds without the reading changing) before capturing the data. The Initial Weight is also recorded in the CSN Initial Weight Logbook (see Attachment F).

7. Remove the filter using the forceps and replace in the Petri slide. Close the door and tare balance for the next sample.
8. On the nineteenth sample, place the filter back on the ^{210}Po strip to be used as a replicate and capture the data (If less than 19 samples are being prepared, reweigh the last filter). Acceptance criteria for the duplicate are listed in Attachment A.
9. After every tenth sample and at the end of the run, check the zero and the working standards (see Section 6.8). Acceptance criteria for the duplicate are listed in Attachment A.
10. Be sure that the sequence numbers increment from the last reference samples. Record the values for the references in the appropriate logbook or form.
11. Unplug the antistatic device at the end of the analysis.
12. Initial Weights Summary Report: After finishing weighing for the day, return to the **Options** screen.
    - Select Review and Edit Data.
    - Choose Initial weights and select the filter type.
    - Click on the *Edit Report* button.
    - Highlight the data in the Date field and click the *Sort Ascending* icon on the toolbar to sort the data by date and time. Make any necessary changes and close the report form.
13. Click on the *Print Report* button and then click the *Print* icon on the toolbar to print to the local printer. Check over the Initial Weights Summary Report. Staple the pages together and forward to the LOM along with the pre-weighed filters.
14. The LOM will review the Summary Report and, if acceptable, give the filters to the CSN FiSH group.

6.11 Sample Analysis – Final Weights

1. Exposed samples are received from the CSN FiSH group with an internal chain of custody (see Attachment G) which provides the site information including the date collected, date received at Wood and the temperature of the samples when they arrived. The LOM will determine the required weigh by date (see Section 2.2).
2. The sample filters and one MB for each 10 samples from the same initial batch are conditioned (see Section 6.5), the room conditions verified (see Section 6.1.6), and the balance calibration verified (see Section 6.8).
3. In AutoWeight, select Final Weights. The Initial Weights can be retrieved from the $PM_{2.5}$ Filter Initial Weights Logbook or alternately from the relevant Initial Weights Summary Reports for data batch preparation.
4. Fill in the Barcode ID field with the appropriate reference name. The first reference weighed each day should be named “SRM1.” Increment each additional reference through the ninth reference from “SRM2” – “SRM9;” after the ninth reference, increment from “SRMA” through “SRMZ.” Type the same name in the Sample ID field.
5. The first method blanks weighed each day should be named “BLK1” – “BLK3.”
6. Do a basic inspection of the filter. Note any imperfections using the comment codes (See Attachment B) in the Sample Comments field. Include noted imperfections in the batch narrative (See Attachments D).
7. Place two filters out at a time on the ^{210}Po strips, exposed side up. Begin weighing with the filter on the strip in front of the antistatic device.
8. Scan the sample barcode label and enter the Filter ID.
9. Using the smooth metal forceps, place the filter on the balance and allow it to stabilize (at least 30 seconds). This often takes much longer for final weights than for initial weights. Shift the filters on the ^{210}Po strips and cleaning as for initial weights (see Section 6.10). Capture the data.
10. Remove filter from balance and replace in Petri slides. Seal the slide. Close the door and tare balance for the next sample.
11. Fill in the corresponding Final Weight form entry with the correct information.
12. Replace nineteenth sample on the ^{210}Po strip after scanning to be used as a replicate sample. Enter “DUP#” in the Barcode ID field, increasing # incrementally from 1 – 9 through the course of the day’s weighing, Click on the Sample ID field and scan the filter barcode to fill the field with the Laboratory ID. Select RP as the sample type. Fill in the Sample Comment field with the Laboratory ID from the sample label and capture data as before. See Attachment A for acceptance criteria and corrective actions.
13. After every tenth sample and at the end of the run, check the zero and the working standards (see Section 6.8). Be sure that the sequence numbers increment from the last reference samples. Record the values for the references in the appropriate logbook or form. See Attachment A for acceptance criteria and corrective actions.
14. Unplug the antistatic device at the end of the analysis.

15. The petri slides will remain in the gravimetric lab until the LOM has reviewed the data batch.

6.12 Data Analysis

1. Final Weights Summary Report.
   - Repeat process as in Sections 6.10.12, but choose Final weights.
   - Complete a separate report for each filter type weighed that day.
   - The Final Weights Summary Report is placed in the data batch folder.

2. Exporting Data
   - See GLO3180-035 for specific procedures for data exporting and data batch generation.
   - Only export samples after the final weight has been taken and the Final Weights Summary Reports printed and verified. Only QC (SRM and DUP) in Final Weights Mode are uploaded into Element™.

3. Prepare a batch narrative recording any comments or codes for filter imperfections (see Attachment B for comment codes).
   - No field is available in Element for these comments codes; the batch narrative is the only record of any noted defects. Be sure to reference specific samples when using the codes.
   - Include the room temperature and percent humidity data for the day the final weights were taken.
   - See Attachment D for an Element™ Batch Narrative Sample Form.
4. Put together the batch folder. Include copies of the relevant $PM_{2.5}$ QC Logbook pages, the Final Weights Summary Report, the export report, Reference Mass certificates, filter comments codes, and the appropriate batch checklist and submit the batch for review and finalization.
5. After data review, if the results are acceptable, the LOM will transfer the filters and completed internal chain of custody back to the CSN FiSH group and prepare an excel data report for the CSN Program Manager. If the results are not acceptable, the filters will be reconditioned and reweighed.

6.13 Quality Control

1. See Attachment A for a summary of the Quality Control requirements and corrective actions. If an analysis fails criteria and reanalysis cannot be performed, document the problem and consult the LOM.
2. One filter for each 10 pre-weighed filters per batch will be designated as a laboratory blank filter. After initial conditioning, these filters will be stored and re-

weighed in all subsequent weighing sessions involving filters from that batch. These measurements will be recorded in the logbook, and are printed out in the Final Weights Summary Report to be included in the batch folder. The final weight should be within ± 15 μg of the initial weight for each blank sample.

3. Field blanks should be within ± 30 μg of the initial weight.
4. The working standard measurements must agree to within ± 3 μg of the certified weight. If the standards are not within criteria, repeat the calibration verification procedure from Section 6.8 and weigh the standards again. If the standards are still outside of the acceptance criteria, notify the LOM. A service call will be placed for the balance.
5. Duplicate weights must be within ± 15 μg or 10% of the filter's loaded mass. If the duplicate is outside of criteria, reweigh. If the duplicate remains outside of criteria, repeat the calibration verification according to Section 6.8 and reweigh the previous samples and the duplicate sample.
6. The acceptance criteria for the various QC samples are not held in the Element™ programs. Failures must be detected as they occur and the appropriate corrective action taken immediately.
7. If a standard or duplicate does not meet criteria, all samples weighed after the last acceptable standard or duplicate must be reweighed.
8. A second set of standards is used in addition to the working mass reference standards as laboratory primary standards. The primary standard weights must be the same as the working mass standards and should be weighed every three months to check the calibration of the balance. Record the certified and found values for each primary standard in the Laboratory Primary Standard Logbook. The primary standard measurements must agree to within ± 3 μg of the certified weight. If the standards are not within criteria, repeat the calibration verification procedure from Section 6.8 and weigh the standards again. If the standards are still outside of the acceptance criteria, notify the LOM. A service call will be placed for the balance. The primary standards may be used to troubleshoot balance and working reference problems. Keep the laboratory primary standards in a secured location.
9. A manufacturer's representative or a qualified vendor will calibrate the balance and perform any necessary maintenance annually. All certificates and maintenance documents will be filed as permanent records.
10. The data logger sensor will be calibrated for relative humidity and temperature annually using a NIST – traceable source by a manufacturer's representative or a qualified vendor. Calibration certificates will be obtained from each calibration and kept on file.
11. The ^{210}Po strips should be replaced every six months. Dispose of the old strips according to the manufacturer's recommendations.
12. Check total weight results for negative or high positive results. Both are indicators of errors.

13. If the balance does not return to zero after pressing [TARE], leave the balance alone for a few minutes to allow it to reach stability. Tare and reweigh references before moving on to the next filter. Both references must pass the acceptance criteria. If not, follow the entire calibration verification procedure outlined in Section 6.8 and then, if the references meet criteria, reweigh the previous ten samples.

6.13 Calculations

All mass calculations are performed by automated data reduction algorithms that reside in the AutoWeight v36 Access© database. Changes and revisions to all programs are verified prior to use. A separate parameter is included for each sample to hold the calculated mass. The mass calculations are performed in the following manner:

Mass, dry (mg-total) = Final weight (mg) – Initial weight (mg)

7.0 References

Quality Assurance Guidance Document 2.12, *Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods*, U.S. EPA, Research Triangle Park, N.C., January 2016.

EPA/600/B-07/001 – Guidance For The Preparation Of Standard Operating Procedures (SOPs) For Quality-Related Documents, U.S. EPA, Washington, D.C., April 2007.

40 CFR (Code of Federal Regulations), Part 50, Appendix L- Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere.

8.0 Attachments

Attachment A – $PM_{2.5}$ QC Requirements.

Attachment B – Comment Codes for Ambient Filters.

Attachment C – General Filter-room Cleaning Checklist.

Attachment D – Element™ Batch Narrative Sample Form.

Attachment E – $PM_{2.5}$ QC Logbook page.

Attachment F – $PM_{2.5}$ Initial Weights Logbook

Attachment G – Internal Laboratory Chain of Custody

Attachment H – Dickson Daily Temperature and Humidity Graph

Attachment I – Mass Primary Standards Logbook

Attachment A
$PM_{2.5}$ QC Requirements

QC Sample	Acceptance Criteria	Corrective Actions
Working Standards*	±3 µg of the certified range.	Recalibrate, re-zero, and reweigh.
Laboratory (Method) Blanks (10%)	±15 µg of the initial filter weight.	Do not weigh samples. New blanks must be conditioned before any weights can be taken. Also, check room conditions. If conditions are outside of criteria, adjust the air-conditioning and/or dehumidifier. Allow 24 hours to elapse prior to further weighing.
Field Blanks and Trip Blanks		
	±30 µg of the initial filter weight.	See project specific requirements for field blanks.
Sample Replicates** (Min. 1/session)	±15 µg of the previous filter weight.	Recalibrate, re-zero, and reweigh.

Note:

*Note: Weigh each working standard at the beginning and end of each weighing session. Re-weigh all filter samples directly preceding the failed standard. If the results are unacceptable after re-weighing, contact the Laboratory Operations Manager.

**Note: Re-weigh all filter samples since the last acceptable replicate. If the results are still unacceptable after reweighing, contact the Laboratory Operations Manager.

Attachment B
Comment Codes for Ambient Filters

A. Gasket edge smeared.
B. Water spots/wet filter.
C. Insects.
D. Filter torn creating a hole.
E. Poor filter alignment.
F. Wrong side of filter exposed.
G. Pin holes in filter.
H. Scratched filter.
I. Improperly folded.
J. Bird droppings.
K. Loose particulate matter in insert/envelope.
L. No elapsed time.
M. Incomplete pressure data.
N. No filter received.
O. Dirt spots.
P. Audit.
Q. Incorrect sampling time.
R. Wrong filter type.
S. Filter smudges.
T. Excessively dirty filter.
U. Excessively wet filter.
V. Accident in field.
W. Distorted/elongated filter.
X. Marked invalid by site operator.
Y. Lab accident.

Attachment C
General Filter-Room Cleaning Checklist

Before entering the filter-room, always put on protective disposable booties which are located outside the door.

Locount® protective floor mats are to be changed at least once a week or sooner if they become excessively dirty.

Shelving unit must be dusted with a damp cloth or sponge as it is emptied before adding more filters.

Floors must be swept at least once a week, more frequently if they are excessively dirty or there has been excessive traffic in the room.

Filter balance must be taken apart and cleaned each day before use. Remove glass cover, platform, and tray, and dust each piece thoroughly with a brush. Dust around the interior thoroughly before replacing tray and platform. Dust the interior of the cover thoroughly before replacing, making sure to align the grooves in the glass cover and the balance.

Forceps must be cleaned with alcohol swipe before each use.

Dehumidifiers must be emptied every morning and every evening before leaving.

The humidifier is used only when the humidity becomes out of acceptance range (30%-40%).

Shelves and counters should be kept clean of any excess materials to prevent collection of dust. No eating or drinking is allowed in the filter-room.

The HEPA filter pre-filter must be replaced every three months according to the manufacturer’s directions. The primary filter should be replaced annually.

The central air filters are maintained on a regular schedule.

Attachment D
Element™ Batch Narrative Sample Form

Batch: _____ **Date:** _____

Room Temp.: _____ **Date:** _____

Please note the following observations for filters in this batch:

Sample Name (Filter #) Code and description (repeat this information for all samples identified with a code).

Analyst Signature: _____ **Date:** _____

Reviewer Signature: _____ **Date:** _____

Reviewer Signature: _____ **Date:** _____

Reviewer Signature: _____ **Date:** _____

Attachment E
CSN $PM_{2.5}$ PM_{10} QC Logbook

amec foster wheeler

CSN $PM_{2.5}$ PM_{10} QC Logbook

MICROBALANCE #: ME5 ANALYST: _____

FILTER LOT #: _____

STANDARD CERTIFIED VALUES: STD1 _____ mg STD2 _____ mg

SN: _____ SN: _____

ANALYSIS DATE	INITIAL WORKING STD 1 (mg)	FINAL WORKING STD 1 (mg)	INITIAL WORKING STD 2 (mg)	FINAL WORKING STD 2 (mg)	COMMENTS

Lab Blanks (MB)

Filter ID	ANALYSIS DATE	Initial Weight (mg)	ANALYSIS DATE	FINAL WEIGHT (mg)

ANALYST: _____ QC SUPERVISOR: _____

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Attachment F
CSN $PM_{2.5}$ PM_{10} Initial Weights Logbook

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CSN $PM_{2.5}$ / PM_{10} Filter Initial Weights Logbook

Microbalance #: _____ Initials: _____ Lot #: _____

Date: _____ Temp (°C): _____ %RH: _____

Filter #	Initial Wt. (mg)	Replicate		Filter #	Initial Wt. (mg)	Replicate

Analyst's Signature _____

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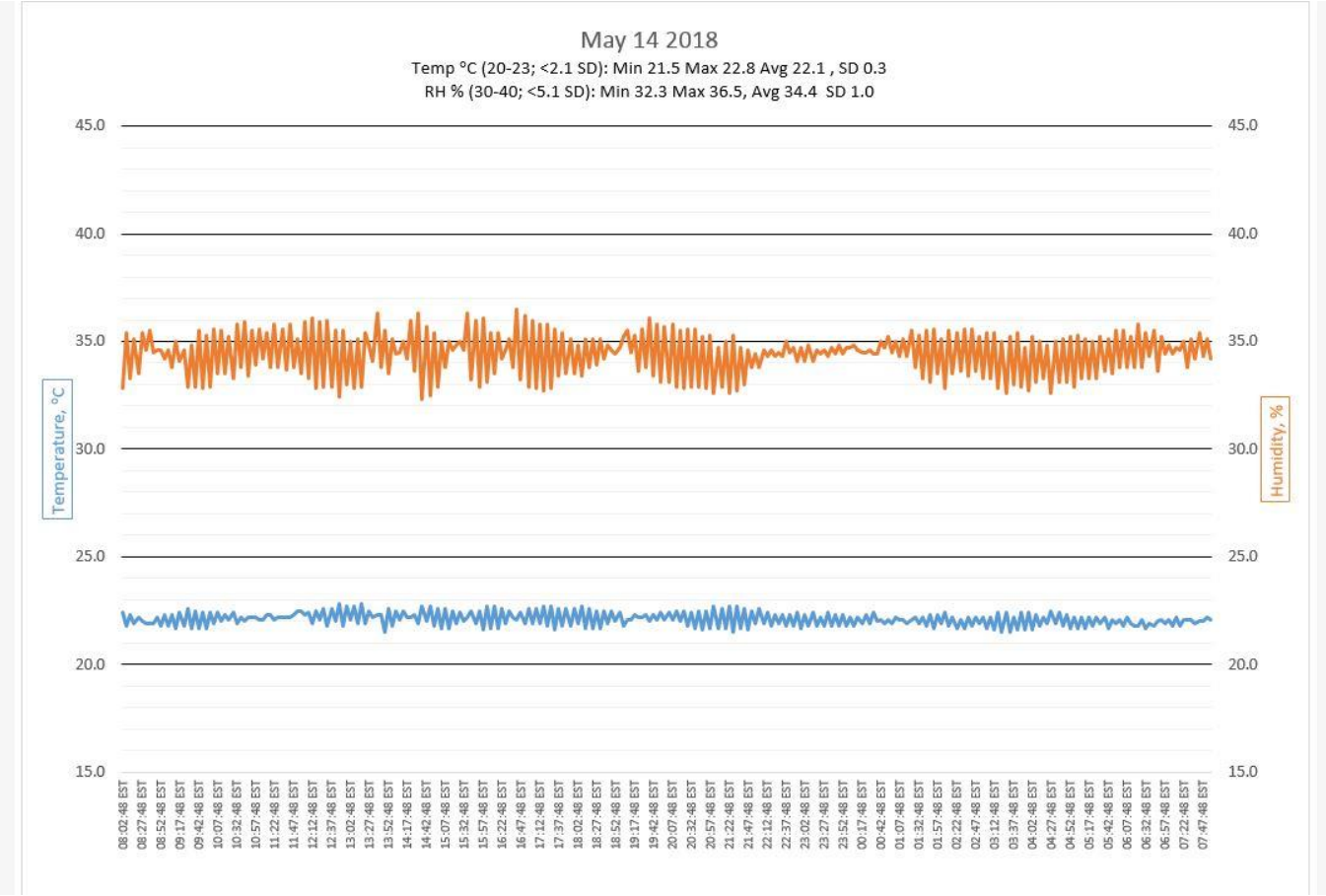
Attachment G
Internal Laboratory Chain of Custody

CSN Filters for Mass
Chain of Custody

Completed by CSN: Initials _____ Date _____					Completed by Lab					
FilterAnalysisID	UnikFilterIDNum	SampleRequestID	Date Rec'd	Temp rec'd oC	Weigh By Date	Date of Initial Weight	Date Rec'd in Lab	Rec'd By	LabID	Date Returned to CSN

GLF3180-014 R0

Attachment H
Dickson Daily Temperature and Humidity Graph



Attachment I
Mass Primary Standards Logbook

Mass Primary Standards Logbook

Microbalance Number: ME5 Date: 4/12/2018

Temperature °C: 20.8 % RH: 35.6

Certified Weights:

Standard	Serial No	Certified Value mg	Uncertainty mg	Range (uncertainty +/- 0.003 mg)
Primary 300 mg	10001168 76	299.9983	0.0025 Mg	+/-0.0051 Mg
Primary 500 mg	1000 116877	499.9988	0.0025 Mg	+/- 0.0051 Mg
Working 300 mg	1000131012	299.9995	0.0025 mg	+/- 0.0051 mg
Working 500 mg	1000131013	500.0056	0.0025 Mg	+/-0.0051 Mg

Measured Weights:

Standard	Acceptable Range	Weight (mg)	Difference from Certified Value (mg)	Pass/Fail
Zero Weight	+/- 0.002 mg	0.000	Ø	(P) / F
Primary 300 mg	+/- 0.003 mg	299.998	-0.0003	(P) / F
Primary 500 mg	+/- 0.003 mg	499.998	-0.0008	(P) / F
Working 300 mg	+/- 0.003 mg	299.999	-0.0005	(P) / F
Working 500 mg	+/- 0.003 mg	500.006	+0.0004	(P) / F

Comments: _____

Analyst: Ruby Wyrsdick Reviewed by: Richard Thomas

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