CALIFORNIA
AIR RESOURCES BOARD

Standard Operating Procedure for Determination of PM2.5 Mass and PM Coarse Mass by Gravimetric Analysis

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Northern Laboratory Branch
Monitoring and Laboratory Division

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Standard Operating Procedure Determination of PM2.5 Mass and PM Coarse Mass by Gravimetric Analysis

1. Scope and Application

This document describes the methodology used by the Monitoring and Laboratory Division (MLD) Inorganics Laboratory Section (ILS) staff to analyze the mass of particulate matter with an aerodynamic diameter less than or equal to 2.5 μm (PM2.5) and 2.5 μm-10 μm (PM Coarse) from samples collected on Teflon filters. In accordance with 40 Code of Federal Regulations (CFR), Part 50, Appendix L for PM2.5 (PM2.5 FRM), and Appendix O for PM Coarse, concentrations are used for designation of attainment status and maintenance of the National Ambient Air Quality Standard (NAAQS). Additionally, PM2.5 SASS™ (Speciation Air Sampling System) mass Teflon filters are analyzed and processed following PM2.5 methods described within this standard operating procedure (SOP).

2. Summary of Method

PM2.5 and PM Coarse are collected on 46.2 mm in diameter polytetrafluoroethylene (PTFE or Teflon) filters over a 24-hour period. The sampling of PM2.5 and PM Coarse follows the United States Environmental Protection Agency's (U.S. EPA) monitoring schedule. This monitoring schedule is referenced in CFR, Title 40, Chapter 1, Part 58, Subpart B.

PM2.5 Mass and PM Coarse Mass are determined by gravimetric analysis in an environmentally controlled room with guidance from the EPA Quality Assurance Guidance Document 2.12, January 2016 (EPA Guidance Document 2.12). Before field sampling, individual filters are pre-weighed on an electronic micro-balance that is interfaced with the Laboratory Information Management System (LIMS) via LIMSLink. The filters are sampled on federally-approved air samplers for a 24-hour period. Filters are returned to the lab and post-weighed. The difference between the two weights and the total sample volume are used to determine the final mass concentration.

PM Coarse Mass is determined using two separate, but concurrent, collocated, federally-approved air samplers. One sampler collects PM2.5, and the other sampler collects PM10. PM Coarse Mass concentrations are calculated by the arithmetic difference of the PM10 concentration and the PM2.5 concentration.

Individual Teflon filters are weighed on an electronic micro-balance before and after field sampling in an environmentally controlled room (i.e., balance room). National Institute of Standards and Technology (NIST) traceable environmental sensors are used to continuously monitor the temperature and relative humidity (RH) in the balance room.

The balance must be calibrated prior to any pre- or post-sampled weighing session. The balance calibration and control standards are all NIST certified and traceable. PM2.5 and PM Coarse filters are weighed in accordance with quality control protocols seen in this SOP the Northern Laboratory Branch's (NLB) Laboratory Quality Control Manual, and EPA Guidance Document 2.12. PM2.5 filters are sampled on different types of samplers: either a federally approved single channel or sequential sampler, or a Speciation Air Sampling System (SASS™) low-volume sampler. The net difference between pre- and post-sampling filter weights and the total sample volume are used to calculate the ambient air mass concentration. All data associated with the PM2.5 and PM Coarse filter sample is entered in NLB's LIMS. After post-weighing, all filters are archived; however, samples from the SASS™ sampler are stored for subsequent analysis. All filters are archived for five years, plus the current year, in compliance with U.S. EPA's guidelines.

PM2.5 and PM Coarse Mass data, all quality control data, and documentation detailing pertinent information are compiled into a monthly data package. The data package is reviewed by the PM2.5 analyst, a second level peer reviewer, and finally reviewed and approved by NLB management. PM2.5 and PM Coarse data are then submitted to U.S. EPA's Air Quality System (AQS) database.

3. Acronyms and Definitions

Acronym or Term	Definition
24-Hr Report form	A report sent out with each filter includes the following sections: chain of custody, field information and laboratory notes. Also known as CARB 24 Hour- Field Sample Report.
°C	Degrees Celsius
Cw	Mass Correction
mg	Milligram
μm	Micrometer
μg	Microgram
$\mu g/m^3$	Microgram per cubic meter
AQS	Air Quality System
ASTM	American Society for Testing and Materials

Acronym or Term	Definition
Calibration	Weighing NIST traceable standards to certify the balance is measuring within the acceptable uncertainties. Performed annually by an approved source.
Calibration verification	Weighing NIST traceable standards (i.e., working control standards) to check the balance is performing within accepted specifications. Performed on a daily basis.
CARB	California Air Resources Board
CFR	Code of Federal Regulations
Duplicate	Two filters collected in separate samplers during the same period and under the same conditions. The duplicate samples are then analyzed separately for the purpose of checking sampler precision. Duplicates are also known as collocated samples.
Field blanks	Unexposed filters conditioned in the laboratory and shipped with a batch of filters. The field blanks are loaded into the sampler without airflow, then removed, and sent back to the lab with the same batch of filters or a subset of the batch. The field blanks are used to determine if contamination occurs during sampling.
FRM	Federal Reference Method
ID	Identification
IDOC	Initial demonstration of capability
ILS	Inorganics Laboratory Section
Lab blanks	Unexposed filters conditioned and weighed with a pre-weight session. The lab blanks remain in the laboratory conditioning cabinet for a minimum of 30 days. After 30 days the lab blanks are weighed to determine whether contamination occurs in the laboratory.
LCD	Liquid crystal display
LIMS	Laboratory Information Management System: Database containing sample metadata, raw and reported concentration results, and quality control samples and results.
LIMSLink	Software allowing review of raw sample data and quality control results, and transfer of data electronically retrieved from the analytical balance to LIMS.
Lot Blanks	Unexposed filters from a new lot used to determine the length of time it takes to stabilize in the balance room environment.
Mass Reference Standards	ASTM- or NIST- traceable weighing standards, generally in the range of masses expected for the filters.
MLD	Monitoring and Laboratory Division
NAAQS	National Ambient Air Quality Standard
NIST	National Institute of Standards and Technology
NLB	Northern Laboratory Branch

Acronym or Term	Definition
Precision	Degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions.
PM2.5	Particulate matter with an aerodynamic diameter less than or equal to a nominal 2.5 micrometers
PM10	Particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers
PM Coarse	Particulate matter with an aerodynamic diameter between a nominal 2.5 and 10 micrometers
Primary Control Standards	NIST-traceable weights used for quarterly calibration verification of the working control standards.
PTFE	Polytetrafluoroethylene (Teflon)
QA	Quality assurance
QC	Quality control
Replicate	An additional analysis of the same sample. The sample used for replicate analyses must be chosen at random. Replicate analyses results are used to evaluate analytical precision, but not the precision of sampling, preservation, or storage internal to the laboratory.
RH	Relative humidity
SASS™	Speciation Air Sampling System
SD	Standard deviation
Working Control Standards	Weights used for routine filter weighing and balance QC checks. The weights are verified using the certified primary control standards on a quarterly basis.
SOP	Standard operating procedure
Stability blanks	Lot blanks with an assigned acceptable weight range. The stability blanks are stored in the conditioning area and are weighed as part of the external calibration verification. The stability blanks are used to determine whether contamination occurs in the conditioning area.
Teflon ID	A unique combination of alphanumeric numbers and letters lasered on the filter's support ring during the manufacturing process.
Temperature at Receipt	Equal to the maximum temperature during travel to the laboratory.
Trip blanks	Unexposed filters conditioned in the laboratory and shipped with a batch of filters. The filters are sent back with the same batch or a subset of the batch. The trip blanks are used to determine if contamination occurred during transportation.
U.S. EPA	United States Environmental Protection Agency

4. Interferences

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

4.2. Teflon, or Polytetrafluoroethylene (PTFE), filters accumulate a surface electrical charge, which may affect filter weight. Static electricity is controlled by treating filters with a static charge neutralizer, such as “Static Master”, prior to weighing. Static charge neutralizers must also be taped inside each balance chamber to aid in static mitigation.

4.3. Moisture content can affect filter weight. Filters must be equilibrated for a minimum of 24 hours in a controlled environment (balance room) prior to pre- and post-weighing. See QC Section 9.3 for limits.

4.4. Airborne particulates can adversely affect accurate mass measurement of the filter. Cleaning laboratory bench-tops and weighing areas prior to weighing, installing “sticky” floor mats at doorway entrances to the balance room, and wearing clean lab coats over regular clothing can further minimize dust contamination.

4.5. Air conditioning ductwork, printers, and frequently opened doorways may create undue air flow. This airflow can adversely affect mass measurements. Appropriate placement of the balance, and use of a double door entry, can minimize airflow. Filters undergoing conditioning should not be placed within an airflow path created by air conditioning ductwork, computer printers, or frequently opened doorways.

4.6. Vibrations can cause micro-balance instability and potentially biased results. The micro-balance must be stationary and level on a sturdy, vibration-free weighing table.

5. Personnel Qualifications and Training

Prior to performing this method, new personnel must be trained by staff with expert knowledge of this method. Personnel must be trained to understand the program’s requirements per any applicable State and federal regulations and guidance, and this SOP. Personnel will also be trained on how to safely and properly operate the equipment needed to perform the method, the quality assurance components, and LIMS functionality pertaining to the program. In addition, an initial demonstration of capability (IDOC) should be met and documented to show proper training in this method, prior to

performing this method on real-world samples (i.e., data for record). A thorough understanding of the method ensures the quality and integrity of data produced by the analyst.

Training provides a general understanding of the PM2.5 and PM Coarse programs. This includes the use of the temperature and humidity sensors, review of quality control criteria, process of pre- and post-inspection of filters, use of the analytical balance, filter weighing methodology, and operation of LIMSLink and LIMS. Training is maintained through repetition of pre- and post- filter inspections and weighing sessions with oversight by the trainer. If accurately completed, these actions will be signed off by both the trainer and laboratory management on the Employee Training Checklist.

Proof of competence must be demonstrated and approved by management before independent analyses may occur. Documentation will be maintained by the laboratory supervisor.

6. Safety Requirements and Hazardous Waste

All personnel must follow the general health and safety requirements found in NLB's Chemical Hygiene Plan.

Used radioactive (alpha particle) Polonium-210 antistatic strips used for static charge neutralization need to be disposed of properly. Upon receipt of new strips, return used strips to the manufacturer for proper disposal. While it is imperative the Polonium-210 antistatic strips be disposed of properly, Polonium-210 emits alpha radiation, which cannot penetrate paper or skin. According to the manufacturer, external exposure does not pose a health risk.

No other hazardous waste is generated by this method.

7. Equipment and Supplies

7.1. Environmentally controlled laboratory (i.e., balance room). Specifications are described in Section 9.1.3.1.

7.2. Two relative humidity/temperature data loggers and probes must be placed in the balance room. One logger is designated as the "primary" and the additional logger is used as a "secondary" logger. All probes must be calibrated and certified annually as NIST traceable, by an outside source. Minimal performance specifications: 20-50% RH, 18-25°C, readable to the nearest 0.5% RH and 0.1 °C, accurate to within 2% RH and 2°C, and at least a five minute logging interval.

7.3. Laboratory Information Management System (LIMS), and LIMSLink.

7.4. Electronic micro-balance with a minimum resolution of 0.001 mg and a precision of ± 0.001 mg, supplied with a balance pan. The micro-balance must be positioned on an anti-vibration balance support table with interface capability to an instrument controller.

7.5. Calibration weights, utilized as Mass Reference Standards (control standards), should be non-corroding, and be certified as traceable to NIST mass standards. Lower and upper control standards that “bracket” the expected filter masses are needed (e.g., 100 mg and 300 mg, or 100 mg and 500 mg). Two sets are needed, one set as a working control standard and one set as the primary control standard. The weights should be American Society for Testing and Materials (ASTM) and at least a Class 2 category.

7.6. Radioactive (alpha particle) Polonium-210 antistatic strips for static charge neutralization. At least eleven (11) strips are needed per balance: six (6) 1”x1” strips for inside the balance chamber, two (2) 3”x1” strips for the positioner, and three (3) 3”x1” strips for use on the workbench. Replace strips every six months.

7.7. Laboratory ionizer stand (positioner) for use with the antistatic strips. This stand positions the antistatic strips for optimal static charge neutralization.

7.8. Metal non-serrated forceps for handling filters.

7.9. Non-metal, non-serrated forceps for handling weights.

7.10. Digital timer/stopwatch.

7.11. Filter: PTFE (Teflon) membrane, 46.2 mm diameter with a polypropylene support ring. Teflon filters must meet the U.S. EPA’s requirements defined in Appendix L of 40 CFR, Part 50.

7.12. Clean filter support cassettes, screens, and covers. The support cassettes must be compatible with the air samplers.

7.13. Filter cassette opener.

7.14. Filter equilibration racks and/or trays.

7.15. Antistatic, nitrate-free, phosphate-free, sulfate-free, and powder free nitrile gloves.

7.16. Plastic petri slide filter containers.

7.17. Disposable laboratory wipes.

7.18. Filter equilibration cabinets.

7.19. Clean room floor mats (e.g., “sticky mats”).

7.20. Light source for inspecting filters.

7.21. Manual rubber air dust blower.

7.22. Self-adhesive labels (e.g., Avery 5293, 1 2/3 inch diameter).

7.23. PM2.5, PM2.5 SASS™, and PM Coarse CARB 24-Hr Field Sample Report forms.

7.24. Isopropanol for cleaning.

7.25. Fine brush.

7.26. Refrigerators.

8. Procedures

Procedures apply to filters for PM2.5 FRM, PM2.5 SASS, and PM Coarse programs, unless stated otherwise.

8.1. The following portrays a recommended analytical sequence procedure for both pre- and post- weigh sessions. Sections 8.1.1 through 8.1.3, must be performed daily prior to any weigh sessions.

8.1.1. Calculate 24-hour Average for Balance Room Environmental conditions (i.e., temperature and RH)

8.1.2. Internal Adjustment Check (of balance)

8.1.3. External Calibration Verification

a. Lower and upper Working Control Standards

b. Stability Blanks- Four

c. Lower and upper Working Control Standards

8.1.4. Recommend Pre-weight and Post-weight Sequence

a. Lower and upper Working Control Standards

b. Set of samples (up to 9)

c. Replicate

d. Alternate lower and upper Working Control Standards

e. Repeat steps b-d

f. Last Set of samples (up to 9)

g. Replicate

h. Lab Blank

i. Lower and Upper Working Control Standards

8.2. Filter Pre-Inspection and Equilibration

8.2.1. Transfer filters from their sealed manufacturer's packaging to a plastic petri slide within the environmentally controlled laboratory. Handle filters with metal non-serrated forceps. Lab personnel must wear nitrile gloves that are free of contaminant ions, powder-free, and anti-static when preparing filters for conditioning and weighing. Before any filter is placed in a conditioning container, it must be inspected for defects. Examine each filter by holding it under, and up to, a light source. A filter must be discarded if any of the following defects are identified:

8.2.1.1. Pinhole—A small hole appearing as a distinct and obvious bright point of light when examined over, and up to, a light source.

8.2.1.2. Separation of ring—Any separation or lack of seal between the filter and the filter support ring.

8.2.1.3. Chaff or flashing—Any extra material on the reinforcing ring or on the heat-seal area that would prevent an airtight seal during sampling.

8.2.1.4. Loose material—Any extra loose material or dirt particles on the filter.

8.2.1.5. Discoloration—Any obvious discoloration that might be evidence of contamination.

8.2.1.6. Other—A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

8.2.2. Maintain a logbook to track filter equilibration. Track balance room location, tray identification, filter number range, date and time of pre-condition start, and analyst initials. This logbook is kept in the balance room.

8.2.3. Maintain an adequate supply of equilibrating filters so the minimum equilibration period is always met before the filters are pre-weighed. The equilibration time is determined by conducting a stability study each time a new lot of filters is opened. See section 9.2.1.

8.3. Pre-Weighing of Unsampled Filters

8.3.1. Ensure that the temperature and the relative humidity of the balance room have remained, and are currently, within the allowable limits throughout the preceding 24 hours prior to the start of the weigh session. See Section 9.3.7 for limits. See Section 11.2 for calculating the average and standard deviation for temperature and relative humidity. Also, make certain the selected filters have been conditioned in the balance room for at least 24 hours.

8.3.2. Clean the micro-balance weighing chamber with a fine brush. Clean the surfaces near the micro-balance with disposable lab wipes moistened with isopropanol. Prior to each weighing session, clean the forceps used for handling the mass reference standards and the forceps used for handling the filters with isopropanol and wipe dry with disposable lab wipes.

8.3.3. Perform an internal adjustment check and external calibration verification of the micro-balance as described in Section 9.1.1.2 prior to beginning the weighing session. Transfer the external calibration verification values from the LIMSLink worksheet into LIMS.

8.3.4. Confirm an appropriate number of CARB 24-hour Field Sample Report (24-Hr Report) forms with site name(s) and barcodes. Confirm appropriate number of field and trip blanks. See Section 9.2.3.1 for blank frequency. For filters designated for SASS™ samplers, use the SASS sample and field blank forms. For filters designated as PM Coarse, use the appropriate sample and field blank forms.

8.3.5. Gather the appropriate number of clean filter support cassettes and metal covers. For filters being sent to monitoring sites using SASS™ samplers, use petri slides.

8.3.6. Create a worksheet in LIMSLink using the LIMS ID from the 24-Hr Report forms. All pre-weight worksheets include replicates, a lab blank, and upper and lower working control standards.

8.3.7. There must be a replicate mass rate of ≥10%. Replicates are automatically inserted by LIMSLink after every nine or less filters using the first filter of each set. A set can be up to nine filters.

8.3.8. Working control standards are automatically inserted by LIMSLink at the beginning and at the end of the worksheet. After each replicate sample, the upper and lower working control standards are alternately inserted automatically by LIMSLink.

8.3.9. Place the lower working control standard on the balance pan using a pair of non-metal non-serrated forceps and close the chamber. Transfer the mass value into the LIMSLink worksheet after 30 seconds. Repeat these steps with the upper working control standard.

8.3.10. Using metal non-serrated forceps, grip a filter only by the outer polypropylene support ring and place the filter onto a static neutralizer strip for a minimum of 30 seconds. Then, using forceps, pass the filter through the static strip ionizer positioner 3 times before placing on the balance. Using forceps, place the filter on the balance pan and close the chamber. At the end of 30 seconds, transfer the mass data into LIMSLink. Record on the 24-Hr Report form the following:

8.3.10.1. Cassette identification (ID) number (each support cassette rim is marked with an ID number).

8.3.10.2. Pre-weight mass of the filter.

8.3.10.3. Date of pre-weight measurement.

8.3.10.4. Analyst’s initials.

8.3.10.5. Filter expiration date, which is 30 days from the pre-weight date.

8.3.11. Place the weighed filter into the appropriate filter support cassette or petri slide for PM2.5 SASS pre-weigh.

8.3.12. After each set of nine (9) filters has been weighed, snap on the top of the plastic filter support cassette, then place the protective metal covers on the bottom and top of the cassette. If the filters are for a SASS™ sampler, place the filter into a petri slide and apply a PM2.5 SASS sample label. If the filter is for a SASS™ sampler and is a blank, a “field blank” 24-Hr Report form is used.

8.3.13. Repeat above steps (8.3.9 through 8.3.12) for subsequent filters and replicates.

8.3.14. Replicates— See Section 9.3.2 for criteria.

8.3.15. Working control standards are both weighed (e.g., 100 mg and 300 mg or 100 mg and 500 mg) at the beginning of a weigh session, alternated (e.g., 100 mg, 300 mg, 100 mg, etc.) after every set of filters plus a replicate, and both weighed again at the end of a weigh session—See Section 9.3.1 for criteria.

8.3.16. Repeat above steps (8.3.14 through 8.3.15) for subsequent sets of filters.

8.3.17. At the end of the weighing session, weigh the lab blank(s) following pre-weight procedures and type the Teflon ID into the designated row in the worksheet. After weighing, place the filter in a petri slide and label. The label must include the following: “PM2.5 Lab Blank” or “PM2.5 LB”, pre-weight time, pre-weight date and the pre-weight mass. Store the filters in the designated area until the filter is ready for post-weight.

8.3.18. Weigh the upper and lower working control standards. After the last control standard has been transferred to the worksheet, examine the data for errors, and document and correct, if necessary.

8.3.19. Transfer data on LIMSLink worksheet to LIMS, print a pre-weight summary report. See Section 10.2.

8.3.20. After pre-weighing is complete, prepare the pre-weight peer review package, which contains the Peer Review Checklist, calibration printout, pre-weight summary report, and 24-Hr Report forms. When

the peer review is complete, the samples and respective 24-Hr Report forms are ready to be processed for shipment to sites.

8.4. Post-Sample Handling

8.4.1. If there is evidence of contamination and/or damage to the filter (e.g., dark spots, cuts, etc.), make note of it in the “lab comments” section of the 24-Hr Report form and write “INVALID” at the top of the report form. The analyst will update the sample validity status in LIMS after post-weight.

8.4.2. Filters received with “Not Used” notation are discarded without weighing, and the 24-Hour Report filed in the designated Quarterly archive “Not Used” folder.

8.4.3. Samples shipped and stored at a constant 4°C or lower before equilibration must be weighed within 30 days of the sampling date. A sample shipped and stored at a constant range between 4°C and 25°C before equilibration must be weighed within 30 days of the sampling date, provided that its temperature at receipt is less than its average ambient temperature during sampling, as reported on the 24-Hr Report form by the site operator. In the event that sample receipt temperature is greater than or equal to the average ambient temperature during sampling, the sample must be weighed within 10 days of the sampling date.

Field blanks and trip blanks do not have a recorded average ambient temperature. A field blank received between 4°C and 25°C must be weighed within 10 days of the sampling date.

Samples, field blanks, and trip blanks exceeding 25 ˚C at receipt are invalid.

Any samples exceeding the time limit between sampling and post-weighing must be noted on the matching 24-Hr Report form. LIMS will automatically invalidate these samples.

Filters	Temperature at Receipt	Days Allowed Between Sampling and Weighing [a)]
Samples	4°C or lower	30
	4-25°C & Average ambient temperature > Receipt temperature	30
	4-25°C & Average ambient temperature ≤ Receipt temperature	10
	Greater than 25°C	Invalid
Field Blanks and Trip Blanks	4°C or lower	30
	4-25°C	10
	Greater than 25°C	Invalid
All	Unknown	Invalid

a) For Trip Blanks, Days allowed between receipt and weighing.

8.5. Post-Weighing of Field Samples

8.5.1. Ensure that the temperature and the relative humidity of the balance room have remained, and are currently, within the allowable limits throughout the preceding 24 hours prior to the start of the weigh session. See section 9.3.7 for limits. See section 11.2 for calculating the average and standard deviation for temperature and relative humidity. Also, make certain the selected filters have been conditioned in the Balance Room for at least 24 hours.

8.5.1.1. Once the LIMSLink post-weigh worksheet is created, LIMSLink will automatically upload the calculated relative humidity values for each sample from their pre-weigh session. LIMSLink identifies the post-weight sample by its LIMS ID, and retrieves the calculated relative humidity from that filter's pre-weight. These values were entered into LIMS during the pre-weight calibration session. LIMSLink will automatically compare the pre- and post-weight calculated relative humidity values for each sample. LIMSLink will notify on the worksheet if the pre- and post-sampling RH conditions are not within ±5 % RH. See Section 9.3.10 for limits. In the event a sample exceeds the control requirement, the filter is removed from the worksheet and weighed another day. If subsequent daily calculations still don't

meet the control requirements for a filter, the filter must be weighed by the weigh-by date. If the filter does not meet the relative humidity control requirements, it should be weighed, but will be invalidated by the analyst.

8.5.2. Clean the micro-balance weighing chamber with a fine brush. Clean the surfaces near the micro-balance with disposable lab wipes moistened with isopropanol. Prior to each weighing session, clean the forceps used for handling the mass reference standards and the forceps used for handling the filters with isopropanol and wipe dry with disposable lab wipes.

8.5.3. Working control standards are automatically inserted by LIMSLink at the beginning and at the end of the worksheet. After each replicate sample, the upper and lower working control standards are alternately inserted automatically by LIMSLink. There must be a replicate mass rate of ≥10%. Replicates are automatically inserted by LIMSLink after every nine filters using the first filter of each set. A set can be up to nine filters.

8.5.4. After the samples have equilibrated for at least 24 hours, arrange the 24-Hr Report forms of the samples that are to be post-weighed in the same order as the LIMSLink worksheet. Generate matching barcode labels on an equal number of petri slides. Remove the sampled filters from the conditioning cabinet. Match up the filter cassette ID numbers with the correct 24-Hr Report forms and petri slides matched by barcode. For samples received from SASS™ samplers, arrange the 24-Hr Report forms and petri slides in same order as the LIMSLink worksheet.

8.5.5. Perform an internal adjustment check and external calibration verification of the micro-balance as described in Section 9.1.1.2 prior to beginning the weighing session. Transfer the external calibration values into LIMS.

8.5.6. Weigh the upper and lower working control standards. Post-weigh the samples, field and trip blanks, and lab blanks in the same order as the worksheet. Using metal non-serrated forceps, grip a filter only by the outer polypropylene support ring and place the filter onto a static neutralizer strip for a minimum of 30 seconds. Then, using forceps, pass the filter through the static strip ionizer positioner 3

times before placing on the balance. Using forceps place a filter on the balance pan and close the chamber. After at least 30 seconds, transfer the mass data into the LIMSLink worksheet. Record on the 24-Hr Report form the following:

8.5.6.1. Post-weight mass of the filter.

8.5.6.2. Date of post-weight measurement.

8.5.6.3. Analyst's initials.

8.5.7. Place the weighed filter into the labeled petri slide.

8.5.8. Repeat steps 8.5.6 and 8.5.7 for subsequent filters and replicate weighings. See Section 9.3.2 for criteria.

8.5.9. Make sure field and/or trip blanks are within control limits. See Section 9.3.4 for criteria. If not, tare the balance and reanalyze the field and/or trip blanks. If blanks are still outside of the criteria limits, comment the exceedance on the 24-Hr report form.

8.5.10. At the end of the weighing session, weigh appropriate lab blanks (See Section 9.2.5), and the lower and upper working control standards. After the last control standard has been transferred to the worksheet, examine data for errors and QC, and document and correct, if necessary.

8.5.11. After the data transfer to LIMS is complete, run the MLD055 Post Transfer Report. Confirm replicates and control meet QC criteria then sign and date the report.

8.5.12. Organize the post weight report to include a Peer Review Checklist, daily calibration check printout, Environmental Conditions during Weigh Session printout, and MLD055 Post Transfer Report from 8.5.11. See Appendix A for example. Submit the report for peer review. Confirm the peer reviewer signed the reports. File the report in the designated binder located in the balance room.

8.5.13. Update LIMS with any invalid filters determined during the weighing session.

8.5.14. Exceedances of the National Ambient Air Quality Standard for PM2.5 should be reported to the lab supervisor. Abnormally high exceedances (e.g., above 100 μg/m3) should be reported to the site

operator and lab supervisor as soon as possible. Notify the site operator and lab supervisor if the mass difference between the pre-weight and post-weight of any trip and field blank filters exceed the criteria outlined in section 9.3.4.

8.5.15. Place the weighed filter into a labeled petri slide, and organize in petri trays by site name, sample date, and filter type. A full tray of 50 samples is secured in a Ziploc bag. The filters are stored in refrigerators for at least one year after sampling. After one year, the samples can be stored at room temperature. Samples must be kept for five years, plus the current year. For additional information see section 10.3.

8.5.16. All PM2.5 FRM and PM Coarse filters are stored following the procedure from 8.5.15.

8.5.17. PM2.5 SASS samples are placed in a designated refrigerator for further analysis.

9. Quality Control

9.1. Instrument Calibration and Verification

9.1.1. Analytical Micro-Balance

9.1.1.1. Annual Calibration - The micro-balance must be calibrated and certified annually as NIST traceable by an outside vendor. If the micro-balance is found to be out of calibration, no weighing can occur and the balance will need to be recalibrated according to the manufacturer’s directions.

9.1.1.2. Daily Calibration verification - The micro-balance must be verified daily, prior to any weighing session. This will be done by internal and external micro-balance calibration verifications and checked by alternating mass reference standards.

9.1.1.2.1. Prior to any daily filter weighing session, the micro-balance must be calibrated. First, check the micro-balance base level and adjust as needed. To ensure maximum stability, the micro-balance must remain “ON” at all times.

9.1.1.2.2. Internal Adjustment check: Press the “Internal Adjustment” key on the balance liquid crystal display (LCD). The LCD

should display “OK” when the balance has finished the internal adjustment check. Press the “OK” button and the balance is now ready for external calibration verification.

9.1.1.2.3. External Calibration verification: Create a Calibration worksheet in LIMSLink. Insert the current set of Stability Blanks and insert the lower and upper working control standards to bracket the stability blanks. For example, an external calibration sequence would be: Lower working control standard, upper working control standard, Stability Blank 1, Stability Blank 2, Stability Blank 3, Stability Blank 4, lower working control standard, upper working control standard. Place the lower working control standard onto the micro-balance pan with non-metal, non-serrated forceps and close the chamber. After 30 seconds, send the mass value to the LIMSLink worksheet. Remove the lower working control standard and place the upper working control standard on the balance pan and close the chamber. After 30 seconds, send the mass value to the LIMSLink worksheet. Repeat these steps for the Stability Blanks and final QC controls. Also, record and send the weight data 24-hour calculated temperature and relative humidity of the balance room to LIMS. External calibration verification must be performed each day that filters are pre-weighed and/or post-weighed.

9.1.2. Mass Reference Standards

9.1.2.1. Mass reference standards (control standards) should be non-corroding and be certified as traceable to NIST. Lower and upper control standards that “bracket” the expected filter masses (e.g., 100 mg and 300 mg or 100 mg and 500 mg) are needed for each balance. Two sets are needed, one set as working control standards and one set as primary control standards. The standards should be at least ASTM Class 2 category.

9.1.2.2. The primary control standards must be recertified by an outside source annually. The outside source must provide a NIST certificate for each mass reference standard. The working control standards are verified against the primary control standards on a quarterly basis. See Section 9.1.2.4.

9.1.2.3. The working control standards will be used during each weighing session as controls. The two standards that are selected to “bracket” the expected mass of the samples will be weighed at the beginning and the end of each session. The standards will also alternately be weighed after each replicate weighing.

9.1.2.4. Quarterly Calibration Verification - Each quarter before any weighing sessions occur, the primary and the working standards are weighed on the balance following the double substitution method described in Section 9.7.2 of the EPA Quality Assurance Guidance Document 2.12, January 2016. If the calculated mass readings are not within ±2 µg of the initial calculated mass correction (Cw value), the primary and the working standards must be weighed again. If the difference is still greater than ±2 µg of the initial calculated mass correction, see Section 9.3.8.

9.1.2.4.1. Quarterly Calibration Worksheets are then printed, peer reviewed, approved by the lab supervisor, and scanned as a PDF to the designated PM2.5 Calibrations file on the ILS Drive. See Appendix B for example.

9.1.3. Temperature and Percent Relative Humidity Data Loggers

9.1.3.1. The balance room’s relative humidity must be maintained at a mean value range of 30.0-40.0% and standard deviation ≤ ± 5.0% SD over the preceding 24 hours. Additionally, its temperature must be maintained at a mean value range of 20.0-23.0 °C and standard deviation ≤ ± 2 °C SD over the preceding 24 hours. If the balance room is out of range for temperature or humidity, no weighing can occur until the room is back in range for at least 24 hours. If the balance room does not meet these specifications, notify the lab supervisor and schedule a repair visit. Procedures for calculating the temperature and relative humidity are kept in the lab procedures notebook located in the balance room.

9.1.3.2. Two sets of data loggers and probes are kept in each balance room. One set is designated as a primary logger and another as the secondary logger. The primary logger is used to calculate the 24-hr balance room conditions. The secondary logger is used to verify the primary logger’s temperature and humidity on a

quarterly basis. The probes are purchased annually or existing probes are certified annually by an outside source.

9.1.3.3. The data loggers should be set to record a temperature and relative humidity data point every two (2) minutes.

9.1.3.4. Each quarter before any weighing sessions occur, the primary temperature and humidity data logger should be checked against a NIST traceable temperature and humidity instrument standard. Record the temperature and humidity readings every two minutes from both the primary logger and the standard (secondary logger) at least ten times. Determine the averages of the temperature and humidity for both the primary logger and the secondary logger. Subtract the averages for the secondary from the averages for the primary logger. The differences must be within ± 2°C for temperature and ± 2% for relative humidity. If the differences are not within ± 2, no weighing shall occur. Refer to section 9.3.9 for additional QC information. Notify the lab supervisor, and repair or replace logger or sensor as needed.

9.1.3.5. Quarterly Temp and Humidity Calibration Worksheets are then printed, peer reviewed, approved by lab supervisor, and scanned as a PDF to the designated PM2.5 Calibrations file on the ILS Drive.

9.2. Blanks

Five types of blanks are used in the method: Lot Blanks, Stability Blanks, Field Blanks, Trip Blanks, and Lab Blanks. The limits, corrective action, and troubleshooting criteria are described in section 9.3.

9.2.1. Lot Blanks: Once a new lot of filters is opened, randomly select one set of nine filters, from three different boxes, as lot blanks. Per the EPA Guidance Document 2.12, a lot is “defined as a group of filters from the same manufacturer, manufactured using the same materials and process, and having the same physical and chemical characteristics.” Place the filters in individual petri slides. Equilibrate the exposed filters in a filter equilibration cabinet in the balance room that allows air circulation, but still reduces extraneous airborne particles from settling on filters. Send the Teflon ID numbers and Lot Numbers to the LIMS administrator to update LIMS. Generate a worksheet in LIMSLink with the new lot blanks and controls (i.e.,

stability study), weigh lot blanks, and transfer the data to LIMS. Lot blanks are weighed every 24 hours. Once the mass difference between weighing is less than or equal to ±0.015 mg for each of the nine lot blanks, the filters have stabilized. Note the time taken from initial exposure of the filters to attainment of mass stability. This information is designated as the minimum equilibration period required before filters from the same lot can be pre-weighed and used for routine sampling. Once this minimum equilibration period is determined, designate eight of the nine lot blanks to become stability blanks (four for each balance room) which are set aside for long term exposure in the same equilibration cabinet where routine samples, field blanks, trip blanks, and lab blanks are equilibrated prior to pre- or post-weighing. Set aside the remaining lot blank as a backup stability blank, in case a working stability blank is damaged. Print the LIMS report “LoVol Mass Stability Study Summary and Limit Determination.” Place the report in the ILS bin for peer review, and lab supervisor review and approval. Email the LIMS administrator the approved report along with which lot blanks are now a stability blank and the assigned balance room. LIMS will be updated with this information. Document the lot blank study in the laboratory notebook, include start date and lot number. Attach the original signed and approved “LoVol Mass Calibration/Stability Blank Summary” Report to the laboratory notebook.

9.2.2. Stability Blanks: Weigh the four stability blanks prior to each weighing session day. The stability blanks are used to determine if there is any contamination in the conditioning area. Acceptable range of the expected stability blank weight is less than or equal to ± 0.015 mg of its determined weight from the lot blank study. If the stability blanks are not within the required ± 0.015 mg, reweigh the stability blanks. Investigate the issue further to determine if contamination occurred, verify QC, room conditions, and that the balance was calibrated properly. If the stability blanks continue to be out of the acceptable range no weighing is performed. Consult with the lab supervisor.

9.2.2.1. Stability blanks are replaced as needed or every 6 months and recorded in the laboratory notebook.

9.2.3. Field Blanks: Field blanks are conditioned, unexposed filters that are used to determine whether contamination occurs during both laboratory and field activities. Field blanks are included in weighing

sessions and handled in the same manner as a filter destined for sampling. Field blanks are loaded on a sampler, without airflow.

9.2.3.1. Ensure that field and trip blanks are included in at least 10 percent of the sampler's operating frequency. Suggested scheduling:

For daily schedule—10 field and 10 trip blanks per quarter

For 1 in 3 schedule—4 field and 4 trip blanks per quarter

For 1 in 6 schedule—3 field and 3 trip blanks per quarter

For 1 in 12 schedule—1 field and 1 trip blank per quarter

For PM2.5 SASS schedule—1 field blank per quarter.

9.2.3.2. The difference between pre- and post- field blank weighings must be less than or equal to ± 0.030 mg. If the mass difference exceeds the criteria, contamination may have occurred. Verify the mass value by reweighing the filter. If the mass difference still exceeds the criteria, notify the site operator and lab supervisor. See section 9.3.4.

9.2.4. Trip Blanks: Trip blanks are conditioned, unexposed filters that are used to determine whether contamination occurs during the transportation of the filters. Trip blanks are included in weighing sessions and handled in the same manner as a filter destined for sampling. The trip blanks travel with the filters, but are not loaded into the sampler.

9.2.4.1. Ensure that trip blanks are included in at least 10 percent of the sampler's operating frequency (see Section 9.2.3.1).

9.2.4.2. The weight difference between pre- and post- trip blank weighings must be less than or equal to ± 0.015 mg. If the mass difference exceeds the criteria, contamination may have occurred. Verify the mass value by reweighing the filter. If the mass difference still exceeds the criteria, notify the site operator and lab supervisor. See section 9.3.4.

9.2.5. Lab Blanks: Lab Blanks are conditioned, unexposed filters that are used to determine whether contamination occurs in the laboratory. One Lab Blank is included in each pre-weigh session and remain

covered in the laboratory for at least thirty (30) days. After the 30th day, the lab blanks are exposed to the environmental chamber conditions for at least 24 hours, and post-weighed with the next consecutive post-weigh session. Lab blanks are then archived following the same process as all filter samples.

9.2.5.1. The difference between pre- and post- lab blank weighings must be less than or equal to ± 0.015 mg. If the mass difference exceeds the criteria, contamination may have occurred. Reweigh the filter and verify the mass value. Investigate the issue further to determine if contamination occurred, and verify the balance was calibrated properly. If the lab blank continues to be out, consult with the lab supervisor.

9.3. QC Summary Values, Ranges, and Corrective Action

QC criteria must be met for all weigh sessions and balance room operations. The table below describes the QC, acceptable range for the criteria, potential issues, and solutions.

	QC	Acceptable Criteria/Range	Corrective Action
9.3.1	**Working Control Standards**	≤ ± 0.003 mg from certified weight Working control standards must be both weighed at the beginning of a weigh session, alternated (e.g., 100 mg, 300 mg, 100 mg, etc.) after every set of filters plus a replicate, and both weighed again at the end	If working control standard is outside of the criteria limit, then stop the weighing session and close the worksheet. All samples must be bracketed by valid working control standards to be valid. Verify the room is within the allowable limits. Tare the balance, reweigh the controls and check mass values. If still not within required ± 0.003 mg of certified weight, then check the primary control standards. If the primary control standards are within acceptable values, then perform a quarterly calibration verification as outlined in section 9.1.2.4. If the working control standards are not within the acceptable certified value, then replace or recertify. Perform a Quarterly Calibration and re-weigh all samples by the hold time. Any samples weighed after the hold times are invalidated. Notify and consult with

	QC	Acceptable Criteria/Range	Corrective Action
		of a weigh session.	the lab supervisor. Document in the balance room laboratory notebook.
9.3.2	**Replicates**	≤ ± 0.015 mg from initial weight Replicate mass rate of ≥10%	If replicate result is outside of the criteria limit, samples in the associated batch (i.e., the preceding set of 9 samples) must be reanalyzed to be validated. Verify the room is within the allowable limits. If replicate is still not within required ± 0.015 mg of initial weight, verify the balance is calibrated properly and check working control standards. Reweigh the previous set of filters. If the problem persists, notify the lab supervisor prior to proceeding with any weighing. Document in the balance room laboratory notebook.
9.3.3	**Primary Control Standards**	≤ ± 0.003 mg from certified weight Criteria is verified after annual certification and quarterly calibration	If primary control standard is outside of the criteria limit, no weighing is performed. Verify the room is within the allowable limits. Tare the balance, reweigh the weights. Verify the balance is calibrated properly and check another set of certified QC weights. If the problem persists, then check primary, working and QC weights on another balance. If all weights are within acceptable criteria, then the balance needs to be adjusted and re-calibrated by an approved source. If the weights are out of range then replace, or recertify by an approved source. Notify and consult with the lab supervisor. Document in the balance room laboratory notebook.
9.3.4	**Field Blanks and Trip Blanks**	≤ ± 0.030 mg from initial weight for Field Blanks	The blank is still considered valid, but corrective actions must take place to determine if contamination is a possibility. Tare the balance, re-weigh the sample to verify the weight. Check QC, balance, and room conditions. If the blank is still out, notify the lab

	QC	Acceptable Criteria/Range	Corrective Action
		≤ ± 0.015 mg from initial weight for Trip Blanks at least 10 percent of the sampler's operating frequency	supervisor and site operator that the blank exceeded the acceptance criterion. Make appropriate notations on the 24-Hr Report form. Document in the balance room laboratory notebook.
9.3.5	**Lot Blanks and Stability Blanks**	≤ ± 0.015 mg from initial weight Every 6 months	If the blank is outside of the criteria limit, no weighing is performed. Check room conditions are within acceptable limits. Tare the balance, re-weigh the stability blanks to verify the weights. Check QC, mass reference standards, and verify the balance internal adjustment check has been performed. If the discrepancy still remains, stop weighing and close the weighing session. Prepare a new set of lot and stability blanks. Notify and consult with the lab supervisor. Document in the balance room laboratory notebook.
9.3.6	**Lab Blanks**	≤ ± 0.015 mg from initial weight One per pre-weigh session. Post-weigh after 30th day	Check room conditions are within acceptable limits. Tare the balance, re-weigh the lab blanks to verify the weights. Check QC, mass reference standards, and verify the balance internal adjustment check has been performed. If the discrepancy still remains, stop weighing and close the weighing session. Notify and consult with the ILS lab supervisor for corrective action direction. Document in the balance room laboratory notebook.
9.3.7	**24-Hour mean Balance**	20.0 – 23.0 °C and	No weighing can occur until the room is within limits for 24 hours. Determine if the equipment or HVAC system needs an adjustment and

	QC	Acceptable Criteria/Range	Corrective Action
	Room Temp. and RH	Control of ≤ ± 2 °C SD over 24 hour 30.0 – 40.0 % and Control of ≤ ± 5.0 % SD over 24 hour	schedule if needed. If the room is still out of acceptable range, relocating to the backup balance room may be required. Notify the lab supervisor. Document in the balance room laboratory notebook.
9.3.8	**Quarterly Calibration Cw**	≤ ± 2 µg from initial Cw value Every quarter before any weighing sessions occur and as needed	If Cw value is greater than 2 µg, no weighing is performed. Verify the room is within the allowable limits. Verify the balance was calibrated within the year. Complete the daily calibration of the micro-balance and the quarterly calibration verification again. To determine if the problem is the weights or the balance continue to section 9.3.3. Document in the balance room laboratory notebook.
9.3.9	**Quarterly Calibration Temp. and RH**	For differences between the averages for the standard from the averages for the primary data logger: ≤ ± 2 °C ≤ ± 2 % RH	Repeat the calibration process. If it's still out, notify the lab supervisor and schedule a repair visit. No weighing will occur. Document in the balance room laboratory notebook.
9.3.10	**Pre/Post Sampling RH 24-Hour mean difference**	≤ ± 5 %	Verify the calculated 24-Hour values for both the pre-weight and post-weight sessions for that filter and confirm the difference is ≤ ± 5% RH. If the pre/post RH 24-Hour mean difference is > ± 5% RH, remove the filter from the weigh session. If subsequent daily calculations still don't meet the control

	QC	Acceptable Criteria/Range	Corrective Action
			requirements for the filter, the filter must be weighed by the weigh-by-date. If the filter does not meet the relative humidity control requirements, weigh the filter then invalidate the sample. Document in the balance room laboratory notebook.
9.3.11	**Pre-sampling**	Pre-weighed filters must be used for sampling ≤ 30 days	If the filter is used after 30 days, invalidate the sample.

10. Sample and Data Management

Data management consists of samples logged into LIMS, documentation of unusual occurrences and their resolutions, creation of data packages (monthly, amendments, and special projects) for peer review and management approval, submittal of data to clients, and archival procedures for sample media and respective 24-Hr Report forms. Program and maintenance notebooks and/or logbooks are to be kept with the instrumentation at all times.

10.1. Data management for this method assumes a familiarity with LIMSLink and LIMS for the transfer and management of mass data.

10.2. After each pre-weigh and post-weigh session, print a summary report from LIMS. Review, initial, and date the report. Prepare a peer review package for each and every weigh session to be checked by a peer reviewer. The peer reviewer will check handwritten values on the 24-Hr Report form and compare to that entered into LIMS. Each report in the peer review package is then signed off, and filed with the completed checklist. Archive the reports for five years, plus the current year.

10.3. After each post-weight session, samples are archived in the designated refrigerators, where they are kept under cold storage for at least one year. After their one-year retention in cold storage, samples are moved to the designated room temperature archive storage, away from light and dust,

for the remainder of the required archive period. Samples are kept for five years, plus the current year.

11. Calculations

11.1 Mass Calculations

The equation to calculate the mass of fine particulate matter collected on a Teflon filter is seen below:

$$M_{2.5}=(M_f-M_i)\times 10^3 \qquad \text{Equation 1}$$

where,
$M_{2.5}$ = total mass of fine particulate collected during sampling period (μg)
M_f = final mass of the conditioned filter after sample collection (mg)
M_i = initial mass of the conditioned filter before sample collection (mg)
10^3 = unit conversion factor for milligrams (mg) to micrograms (μg)

According to 40 CFR Part 50, Appendix L, PM2.5 samplers are required to provide measurements of the total volume of ambient air passing through the sampler (V) in cubic meters at the actual temperatures and pressures measured during sampling. Use the following formula if V is not available directly from the sampler:

$$V=Q_{avg}\times t\times 10^{-3} \qquad \text{Equation 2}$$

where,
V = total sample volume (m^3)
Q_{avg} = average flow rate over the entire duration of the sampling period (L/min)
t = duration of sampling period (min)
10^{-3} = unit conversion factor for liters (L) into cubic meters (m^3)

The equation outlined below is used to determine PM2.5 mass concentration:

$$PM_{2.5}=\frac{M_{2.5}}{V} \qquad \text{Equation 3}$$

where,
$PM_{2.5}$ = mass concentration of PM2.5 particulates (μg/m^3)
$M_{2.5}$ = total mass of fine particulate collected during sampling period (μg)
V = total volume of air sampled taken directly from sampler (m^3)

For PM Coarse, calculations are as follows:

$$M_{10}=(M_f-M_i)\times 10^3 \qquad \text{Equation 4}$$

where,
M_{10} = total mass of PM10 particulate collected during sampling period (μg)
M_f = final mass of the conditioned filter after sample collection (mg)
M_i = initial mass of the conditioned filter before sample collection (mg)
10^3 = unit conversion factor for milligrams (mg) to micrograms (μg)

According to 40 CFR Part 50, Appendix L, PM2.5 samplers are required to provide measurements of the total volume of ambient air passing through the sampler (V) in cubic meters at the actual temperatures and pressures measured during sampling. PM Coarse follows these guidelines as well. Use the following formula if V is not available directly from the sampler:

$$V=Q_{avg}\times t\times 10^{-3} \qquad \text{Equation 5}$$

where,
V = total sample volume (m^3)
Q_{avg} = average flow rate over the entire duration of the sampling period (L/min)
t = duration of sampling period (min)
10^{-3} = unit conversion factor for liters (L) into cubic meters (m^3)

The equation outlined below can be used to determine PM10 mass concentration:

$$PM_{10}=\frac{M_{10}}{V} \qquad \text{Equation 6}$$

where,
PM_{10} = mass concentration of PM10 particulates (μg/m^3)
M_{10} = total mass of PM10 particulate collected during sampling period (μg)
V = total volume of air sampled (m^3)

To determine PM Coarse mass, the final equation is applied:

$$PM\ Coarse=PM_{10}-PM_{2.5} \qquad \text{Equation 7}$$

11.2 Balance Room Environmental Conditions Calculations for Temperature and Relative Humidity.

11.2.1. Average

$$Average=\frac{1}{n}\sum_{i=1}^{n} a_i=\frac{1}{n}(a_1+a_2\ldots a_n) \qquad \text{Equation 8}$$

Where,

n = sample size,
a_i = Individual observed values

11.2.2. Standard Deviation

$$\text{Standard Deviation} = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2} \qquad \text{Equation 9}$$

Where,
n = sample size,
x_i = observed values,
$\bar{x}$ = mean value of observations,

12. Revision History

	Date	Updated Revision	Original Procedure
1	**Description**: Updated SOP from 0.0 to 1.0		
	March 4, 2014	Addition of the Automatic Filter Weighing System, updates to the filter static charge neutralization procedure, new relative humidity/temperature recorders, updates in 40 CFR, Appendix L, and clarification of procedures.	No Automatic Filter Weighing System, anti-static measures not specified in prior SOP.
2	**Description**: Addendum 1, A07 MLD055.1		
	September 1, 2015	Addition of the use of probes to verify sample temperature at receipt from the field.	Only Time/Temp tabs were used to verify sample temperature at receipt.
3	**Description**: Addendum 2, A15 MLD055.2		
	November 22, 2016	Revised process for calculating temperature and relative humidity prior to weigh sessions.	No process defined for calculating 24-hour averages prior to weigh sessions.
4	**Description**: Updated MLD055 SOP from 1.0 to 2.0 and added PM Coarse		
	August 2, 2018	New data loggers, revisions to the quarterly data logger calibration processes, quarterly mass reference standards calibration	Omega data loggers, less elaborate calibration process of weights and temp/humidity data loggers,

	Date	Updated Revision	Original Procedure
		process, and the addition of lab blanks, and clarification of procedures. Added PM Coarse procedures.	and no Lab Blanks in 1.0 version.
5	**Description**: Addendum 1, A29 MLD055		
	January 31, 2019	Specified temperature indicator type used. Updated holding times for samples received > 4°C for all PM2.5 and SASS.	Temperature indicator type not listed previously. PM2.5 SASS™ holding times not previously specified.
6	**Description**: Updated MLD055 SOP from 2.0 to 3.0		
	April 13, 2022	Primary weights require annual NIST traceable calibration. Working weights checked against primary weights every 90 days. Added and removed terms, updated QC criteria and updated general procedures for clarity. Removed specific temperature measurement.	Primary and working weights require NIST traceable annual calibration. Added terms not previously defined. Specified temperature measurement (indicator or manual) for PM2.5 and SASS.

13. References

13.1. Code of Federal Regulations, Title 40, Chapter I, Subchapter C, Part 50, Appendix L, Appendix N, and Appendix O.

13.2. Code of Federal Regulations, Title 40, Chapter I, Subchapter C, Part 58, Subpart B.

13.3. California Air Resources Board, “Chemical Hygiene Plan for Northern Laboratory Branch 1927 13th Street, 1900 14th Street,” November 2021 or current.

13.4. Quality Assurance Guidance Document 2.12, Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods. EPA-454/B-16-001. U.S. Environmental Protection Agency, January 2016.

13.5. Quality Assurance Project Plan, Chemical Speciation of PM2.5 Filter Samples. RTI/0212053/01QA. U.S. Environmental Protection Agency, January 2014.

13.6. Quality Assurance Project Plan for Particulate Matter Pollutant Air Monitoring Program. California Air Resources Board, February 2020.

13.7. Quality Assurance Handbook Volume II, Appendix D. U.S. Environmental Protection Agency, March 2017.

13.8. Backgrounder on Polonium-210, U.S. Nuclear Regulatory Commission, https://www.nrc.gov/reading-rm/doc-collections/fact-sheets/polonium.html, June 2019 (or Current).

APPENDIX A

1. Example of Post Weight Peer Review Checklist

PM2.5 Mass, Coarse, Speciation Weigh Session – Peer Review Checklist

Pre/Post Weigh Session Date: 02/04/2022

- ☐ Mass values (pre and post as applicable) and dates on the chain-of custody sheets match the LIMS Report.
- ☐ All QC values are within acceptable ranges. QC values are:
  - 100 mg and 500 mg at the beginning of all worksheets.
  - 100 mg and 500 mg controls alternated every 9 samples (there may be less than 9 at the end of the worksheet).
  - One duplicate (REP), at the end of 9 sample batch (there may be less than 9 at the end of the worksheet).
  - 100 mg and 500 mg controls at the end of the worksheet.
  - *Acceptable range is +/- 0.003 mg of certified mass values of the controls as follows:*

Target Limits: 100 mg: ~~99.994 - 100.000~~ 99.997 – 100.003 200 mg: ____NA____ ____500 mg: 499.995 - 500.001

Example:

Row #	Pre-Wt Date/Time	Project	Control / Barcode	LIMS Sample_id	Pre-Weight (mg)	Cal/Trip Bl Status
1	07/14/2016 13:49:00	----	CTL100	200483562	99.995	In Limits
2	07/14/2016 13:50:00	----	CTL500	200483563	500.006	In Limits
3	07/14/2016 13:51:00	FRM	PF216725		387.078	
4	07/14/2016 13:52:00	FRM	PF216724	----	353.585	
5	07/14/2016 13:53:00	FRM	PF216725	----	334.683	
6	07/14/2016 13:54:00	FRM	PF216726	----	334.519	
7	07/14/2016 13:55:00	FRM	PF216727	----	351.435	
8	07/14/2016 13:56:00	FRM	PF216728	----	350.159	
9	07/14/2016 13:57:00	FRM	PF216729	----	307.423	
10	07/14/2016 13:58:00	FRM	PF216730	----	386.513	
11	07/14/2016 13:59:00	FRM	PF216731	----	358.710	
12	07/14/2016 14:00:00	FRM	PF216732-REP	200483564	307.079	0.002 - OK
13	07/14/2016 14:01:00	----	CTL100	200483564	99.995	In Limits
14	07/14/2016 14:04:00	----	CTL500	200483565	500.004	In Limits

- ☐ Duplicates are within acceptable ranges. (Range limit is +/- 0.015 mg).
- ☐ Lab Blanks are within acceptable ranges. (Range limit is +/- 0.015 mg).
- ☐ Field & Trip blanks are within acceptable ranges and transferred correctly in LIMS. (Field blank range limit is +/- 0.030 mg, Trip blank range limit is +/- 0.015mg) *N/A for Pre-Weights

Review of Daily calibrations (Attached).

- Verify Stability Blanks within ranges (ranges printed on the Calibration Report attached).
- Temp. and RH within acceptable ranges (20-23 °C and 30-40% RH)
- QC controls within ranges as specified above in "Target Limits."

- ☐ **Return Checklist and Report to Analyst to be filed.**

Peer Reviewer Signature — Date: 02/07/22

Updated: January 11, 2019

2. Example of Daily Calibration Printout

LoVol Mass Calibration/Stability Blank Summary

Weigh Date: 02/04/2022
Balance & Room: Mettler XP6 at 13th and T
QC Batch Name: QCB220204001
QC Batch Status: Initial
Mean Temp (C): 21.5 ✓ Mean RH: 34.7 ✓
Temp/RH Status: DataEntered
Chemist: CTaylor

Row #	QC Name or Filter #	Calib or Non-Calib or Lot #	LIMS ID	Weight Time	Weight (mg)	Range Limit (mg)	In Limits?	LIMS Status
1	qt100	Calib	S220204001	09:18	100.000	99.999 - 100.005	Yes	DataEntered
2	qt500	Calib	S220204002	09:20	500.000	499.995 - 500.001	Yes	DataEntered
3	D9131139	W9320006-E	S220204003	09:21	162.536	162.518 - 162.548	Yes	DataEntered
4	D9131154	W9320006-E	S220204004	09:23	167.340	167.323 - 167.353	Yes	DataEntered
5	D9131165	W9320006-E	S220204005	09:25	162.215	162.200 - 162.230	Yes	DataEntered
6	D9131339	W9320006-E	S220204006	09:27	163.242	163.227 - 163.257	Yes	DataEntered
7	qt100	Non-Calib	S220204007	09:29	99.999	99.999 - 100.005	Yes	DataEntered
8	qt500	Non-Calib	S220204008	09:30	499.999	499.995 - 500.001	Yes	DataEntered

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3. Example of Environmental Conditions during Weigh Session

02-04-22 Temp and Rh

Date	Time	Serial	Pod Serial	Temperature CH:5	Temperature CH:5 Unit	Relative Humidity CH:6	Relative Humidity CH:6 Unit
2/4/2022	10:18:17 AM	16220792	19040958	22	celsius	34.1	%RH
2/4/2022	10:20:17 AM	16220792	19040958	22.1	celsius	34.3	%RH
2/4/2022	10:22:17 AM	16220792	19040958	22.1	celsius	34.1	%RH
2/4/2022	10:24:17 AM	16220792	19040958	22.1	celsius	34.4	%RH
2/4/2022	10:26:17 AM	16220792	19040958	22.1	celsius	34.3	%RH
2/4/2022	10:28:17 AM	16220792	19040958	22.1	celsius	33.8	%RH
2/4/2022	10:30:17 AM	16220792	19040958	22.1	celsius	34	%RH
2/4/2022	10:32:17 AM	16220792	19040958	22.1	celsius	33.9	%RH
2/4/2022	10:34:17 AM	16220792	19040958	22.1	celsius	33.9	%RH
2/4/2022	10:36:17 AM	16220792	19040958	22.1	celsius	34	%RH
2/4/2022	10:38:17 AM	16220792	19040958	22.1	celsius	33.6	%RH
2/4/2022	10:40:17 AM	16220792	19040958	22.1	celsius	33.9	%RH
2/4/2022	10:42:17 AM	16220792	19040958	22.2	celsius	33.9	%RH
2/4/2022	10:44:17 AM	16220792	19040958	22.1	celsius	33.8	%RH
2/4/2022	10:46:17 AM	16220792	19040958	22.1	celsius	34.1	%RH
2/4/2022	10:48:17 AM	16220792	19040958	22.2	celsius	33.9	%RH
2/4/2022	10:50:17 AM	16220792	19040958	22.2	celsius	33.9	%RH
2/4/2022	10:52:17 AM	16220792	19040958	22.2	celsius	34	%RH
2/4/2022	10:54:17 AM	16220792	19040958	22.2	celsius	33.6	%RH
2/4/2022	10:56:17 AM	16220792	19040958	22.2	celsius	34.1	%RH
2/4/2022	10:58:17 AM	16220792	19040958	22.2	celsius	34.1	%RH
2/4/2022	11:00:17 AM	16220792	19040958	22.2	celsius	33.7	%RH
2/4/2022	11:02:17 AM	16220792	19040958	22.3	celsius	34	%RH

√ √ C.P. 02/04/2022

1 of 2 C.P.

4. Example of MLD055 Post Transfer Report

LoVol Post-Weight Transfer Results

Balance Method MLD055

Post-Weight Date: 02/04/2022

QC Batch ID: QCB22020402

Balance/Room: XP6-13th Chemist: CTaylor

Statistician: CTaylor Date: 02/04/22

Reviewed by: LH Date: 02/04/22

Row #	LIMS ID or QC Name	Project or QC LV ID	Weight Time	Weight Result (mg)	FRM/PM BkRep Diff. Label If Out Of Check	7-RH Diff. Notify If Out	LTP Mass (ug/m3)	Diff Post/ Sampling (Days)	Diff Post Sampling (Days)	Site Name	Start Sample Date-Time	Status	Label If Invalid
1	df500	S22020403	10:17	500.000	In Limits	—	—	--	—	—	--	DataEntered	
2	df500	S22020401	10:18	500.000	In Limits	...	—	--	--	--	--	DataEntered	
3	5778118008	FM2.5 FRM	10:21	170.730		0.8	22.8	10.3	4.55	Quincy	01/24/2022 07:00	DataEntered	
4	S22011D10	FM2.5 FRM	10:22	171.513		0.8	24.1	9.43	5.54	Quincy	01/28/22 05:00	DataEntered	
5	S22011D11	FM2.5 FRM	10:24	171.325		0.8	29.8	8.53	11.54	Quincy	01/26/2022 05:00	DataEntered	
6	S22011D12	FM2.5 FRM	10:26	170.799		0.8	25.6	7.43	7.54	Quincy	01/27/2022 05:00	DataEntered	
7	S22011D13	FM2.5 FRM	10:30	170.410		0.0	16.1	12.44	2.54	Quincy	01/28/2022 05:00	DataEntered	
8	S22011D14	FM2.5 FRM	10:32	168.852	0.003	0.8	—	—	—	Quincy - HS	01/29/2022 05:00	DataEntered	
9	S22011D15	FM2.5 FRM	10:34	167.800	0.002	0.8	—	--	—	Quincy - FS	01/29/2022 05:00	DataEntered	
10	S22011D16	FM2.5 FRM	10:42	166.707	0.032	0.7	—	--	—	Quincy - 7S	01/29/2022 05:00	DataEntered	
11	S2112270D9	FM2.5 CSN	10:44	166.486		0.9	16.4	4.45	25.55	CMAS	01/30/2022 05:00	DataEntered	
12	S2201-9009-Rep	S22020401	10:49	170.731	0.001	—	—	--	—	—	--	DataEntered	
13	df100	S22020101	10:50	99.989	In Limits	—	—	--	—	—	--	DataEntered	
14	9211-227013	FM2.5 CSN	10:53	167.028		0.8	46.7**	17.45	13.55	Portage	01/17/2022 05:00	DataEntered	
15	9211-227014	FM2.5 CSN	10:55	167.191		0.9	21.8	11.45	19.54	Fontana	01/23/2022 05:00	DataEntered	
16	9211-227018	FM2.5 CSN	10:58	166.397		0.9	19.9	5.45	25.54	San-T St	01/29/2022 05:00	DataEntered	
17	S21122701D-Rep	S22020401	10:58	167.828	0.000	—	—	--	—	—	--	DataEntered	
18	D01353-7	S22020401	11:01	168.047	0.001	0.4 / 0.5	—	--	—	—	--	DataEntered	
19	df100	S22020401	11:02	99.980	In Limits	—	—	--	—	—	--	DataEntered	
20	df500	S22020401	11:03	499.880	In Limits	.	.	.	.	.	.	DataEntered	

* = Hold Time Exceeded

** = PM2.5 _TP Meas > Daily Standard

APPENDIX B

1. Procedures to Calculate Quarterly Verifications of Working Control Standards

The following is the recommended procedure. The analyst should weigh the standards at regularly spaced time intervals to average out any effects of instrument drift.

1. Zero and calibrate the microbalance following the microbalance's user guide. Exercise the balance.

2. Open the draft shield. Using cleaned, non-metallic forceps, place the first working standard (for example, a 400 mg weight), *w*, on the weigh pan.

3. Close the draft shield. Wait until the display on the balance has stabilized. Record the weight as Measurement 1.

4. Open the draft shield and remove the weight. Shut the draft shield and allow the microbalance to zero. Tare, if needed.

5. Repeat steps 2-4 for the primary standard of the same weight (for example, 400 mg), *p*, and then the working standard, *w*, again to weigh each standard two times, recording the resulting values as Measurements 2, 3, and 4, respectively. Note that the primary standard is consecutively weighed.

Measurement Number	Weight on Pan	Observation Number
1	*w*	O_1
2	*p*	O_2
3	*p*	O_3
4	*w*	O_4

NOTE: The time intervals between successive trials should not differ from one another by more than 20%. If this difference is exceeded, reject the data and take a new series of measurements.

6. Repeat steps 2-4 for the second mass reference standard utilized (for example, a 300 mg weight).

7. Calculate the mass correction, *Cw*, for the test (working standard weight, *w*) as follows, according to the sequence used. In each case, the apparent mass correction for the primary weight standard, *Cp*, are included. The symbols *Np* and *Nw* refer to the nominal values of p and w, respectively.

$$Cw = Cp + ([O_1\text{-}O_2\text{+}O_4\text{-}O_3]/2) + Np - Nw$$

8. Subsequent measurements of *Cw* must be within ±2 µg of the initial *Cw* value.

The analyst should document the results of this procedure in the laboratory logbook and/or on any required data forms.

2. Example of Quarterly Verifications of Working Control Standards

1st Quarter 2018
MLD055 and MLD071 Mass Standard Calibration Checks: Working and Primary
14th/S Balance Room

100 mg Control Weights		
Nominal Value (Np, Nw)	100	mg
Primary 100 mg Mass Correction (Cp)	0.0034	mg
Primary Serial Number:	1000131217	
Primary Calibration Date:	10/17/2017	
Working 100 mg Mass Correction:	0.0019	mg
Working 100 mg Conventional Mass:	100.002	mg
Working Serial Number:	1000131218	
Working Calibration Date:	10/11/2016	
Measurement Number	*Standard Weight*	*Weight in mg (Observation)*
O1	Working	100.002
O2	Primary	100.003
O3	Primary	100.002
O4	Working	100.003
Observations (O1 - O2 + O4 - O3) =	0	mg
Cw = Cp + (Observations/2) + Np - Nw =	0.003	mg
Initial 100 mg Cw value =	***0.001***	mg
*Difference (Initial Cw - Current Cw) =	***-0.002***	mg

500 mg Control Weights		
Nominal Value (Np, Nw)	500	mg
Primary 500 mg Mass Correction (Cp)	0.0048	mg
Primary Serial Number:	1000131222	
Primary Calibration Date:	10/17/2017	
Working 500 mg Mass Correction:	0.0025	mg
Working 500 mg Conventional Mass:	500.003	
Working Serial Number:	1000131221	
Working Calibration Date:	10/11/2016	
Number	*Standard Weight*	*(Observation)*
O1	Working	500.000
O2	Primary	500.005
O3	Primary	500.004
O4	Working	500.002
Observations (O1 - O2 + O4 - O3) =	-0.007	mg
Cw = Cp + (Observations/2) + Np - Nw =	0.001	mg
Initial 500 mg Cw value =	***0.002***	mg
*Difference (Initial Cw - Current Cw) =	***0.001***	mg

KEY:
Cw = Mass Correction
Cp = Mass correction for Primary Weight Standard (on Certificate)
O1 = Observation weight 1
O2 = Observation weight 2
O3 = Observation weight 3
O4 = Observation weight 4
Np = Primary nominal value
Nw = Working nominal value
Cw = Cp + ([O1 - O2 + O4 - O3]/2) + Np - Nw

Balance Info:
Balance: Mettler Toledo XP6, Serial # B-040074112
Calibration Date: 2/6/2017

*Subsequent measurements of Cw must be within +/- 2 µg of the initial Cw value.
Initial Cw value calculated 4/3/17.

Analyst Signature:
Date: 1/2/18

Peer Reviewer Signature:
Date: 1/4/18

Manager Signature:
Date: 1-9-18

Updated 10/25/16