



CALIFORNIA

AIR RESOURCES BOARD

Standard Operating Procedure for Determination of PM_{2.5} Mass and PM Coarse Mass by Gravimetric Analysis

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Northern Laboratory Branch
Monitoring and Laboratory Division

Approval Signatures	Approval Date
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Table of Contents

1.	Scope and Application.....	1
2.	Summary of Method	1
3.	Acronyms and Definitions.....	2
4.	Interferences.....	3
5.	Personnel Qualifications and Training	4
6.	Safety Requirements and Hazardous Waste.....	5
7.	Equipment and Supplies.....	5
8.	Procedures	7
9.	Quality Control.....	14
10.	Sample and Data Management	23
11.	Calculations.....	24
12.	Revision History.....	26
13.	References	27

Appendix A

1.	Procedures to Calculate Daily Balance Room Conditions.....	28
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Appendix B

1.	Procedures to Calculate Quarterly Verifications of Working Control Standards..	31
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Appendix C

1.	Example of Quarterly Verification of Working Control Standards Using Double Substitution Method.	32
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Standard Operating Procedure

Determination of PM_{2.5} Mass and PM₁₀ 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 (PM_{2.5}) 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 PM_{2.5}, 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, PM_{2.5} SASS™ (Speciation Air Sampling System) mass Teflon filters are analyzed and processed following PM_{2.5} methods described within this standard operating procedure (SOP).

2. Summary of Method

PM_{2.5} and PM Coarse are collected on 46.2 mm in diameter polytetrafluoroethylene (PTFE or Teflon) filters over a 24-hour period. The sampling of PM_{2.5} 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.

PM_{2.5} Mass and PM Coarse Mass is determined by gravimetric analysis in an environmentally controlled room with guidance from the EPA Quality Assurance Guidance Document 2.12, January 2016. Before field sampling, individual filters are pre-weighed on an electronic micro-balance that is interfaced with the Laboratory Information Management System (LIMS). 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 PM_{2.5}, and the other sampler collects PM₁₀. PM Coarse Mass concentrations are calculated by the arithmetic difference of the PM₁₀ concentration and the PM_{2.5} 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 and control standards are all NIST certified and traceable. PM_{2.5} and PM Coarse filters are weighed in accordance with quality control protocols, identified in the NLB QC Manual, and EPA Guidance Document 2.12. PM_{2.5} 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 PM_{2.5} and PM Coarse filter sample is entered in the Northern Laboratory Branch's (NLB) 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.

PM_{2.5} 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 PM_{2.5} analyst, a second level peer reviewer, and finally reviewed and approved by NLB's management. PM_{2.5} 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
°C	Degrees Celsius
Cw	Mass Correction
mg	Milligram
mm Hg	Millimeters mercury
µm	Micrometer
µg	Microgram
µg/m ³	Microgram per cubic meter
AQS	Air Quality System
AQSB	Air Quality Surveillance Branch
ASTM	American Society for Testing and Materials
CARB	California Air Resources Board
CFR	Code of Federal Regulations
FRM	Federal Reference Method
IDOC	Initial demonstration of capability

ILS	Inorganics Laboratory Section
LCD	Liquid crystal display
LIMS	Laboratory Information Management System: Database containing sample metadata, raw and reported concentration results, and quality control samples and results.
LIMS Link	Software allowing review of raw sample data and quality control results, and transfer of data electronically retrieved from the analytical balance to LIMS.
MFC	Mass flow controller
MLD	Monitoring and Laboratory Division
MRS	Mass reference standard
NAAQS	National Ambient Air Quality Standard
NIST	National Institute of Standards and Technology
NLB	Northern Laboratory Branch
PM _{2.5}	Particulate matter with an aerodynamic diameter less than or equal to a nominal 2.5 micrometers
PM ₁₀	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
PTFE	Polytetrafluoroethylene (Teflon)
QA	Quality assurance
QC	Quality control
RH	Relative humidity
SASS™	Speciation Air Sampling System
SOP	Standard operating procedure
TSA	Technical systems audit
U.S. EPA	United States Environmental Protection Agency
VFC	Volumetric flow controller
Mass Reference Standards	1. Working Control Standards: Certified weights used for routine filter weighing and balance QC checks. 2. Primary Control Standards: Certified weights used for quarterly verification checks of the working control standards.

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 strips 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 PM_{2.5} and PM Coarse programs. This includes the use of the temperature and humidity sensors, interpretation of protocol, process of pre- and post-inspection of filters, use of the analytical balance, filter weighing methodology, and operation of LIMS. Training is maintained through

repetition of pre- and post- mock 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 in the laboratory notebook.

Documentation of training will be included in the program's laboratory notebook, and maintained by the laboratory supervisor. Each training date must be initialed by the trainer, trainee, and laboratory management.

Proof of competence must be demonstrated and approved by management before independent analyses may occur.

6. Safety Requirements and Hazardous Waste

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

Expired radioactive (alpha particle) Polonium-210 antistatic strips used for static charge neutralization need to be disposed of properly. Upon receipt of new strips, return expired 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. 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).
- 7.2. Laboratory Information Management System (LIMS), and LIMSLink.
- 7.3. 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 a vibration-damping balance support table with interface capability to an instrument controller.
- 7.4. 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 (i.e. 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.

- 7.5. Laboratory ionizer stand (positioner) for use with the antistatic strips. This stand positions the antistatic strips for optimal static charge neutralization.
- 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.
- 7.7. Metal non-serrated forceps for handling filters.
- 7.8. Non-metal, non-serrated forceps for handling weights.
- 7.9. Digital timer/stopwatch.
- 7.10. 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.
- 7.11. Clean filter support cassettes and covers. The support cassettes must be compatible with the air samplers.
- 7.12. Filter cassette opener.
- 7.13. Filter equilibration racks and/or trays.
- 7.14. Two relative humidity/temperature recorders and probes must be placed in the balance room. One recorder is designated as the "primary" and the additional recorder is used as a "secondary" recorder. All recorders 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.
- 7.15. 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 (i.e., "Sticky") floor mats.
- 7.20. Metal filter-shipping canisters.

- 7.21. Light source for inspecting filters.
- 7.22. Rubber air dust blower.
- 7.23. Anti-vibration balance support table.
- 7.24. Self-adhesive labels (i.e. Avery 5293, 1 2/3 inch diameter).
- 7.25. PM2.5, PM2.5 SASS™, and PM Coarse chain-of-custody forms.
- 7.26. Isopropanol for cleaning.
- 7.27. Fine brush.
- 7.28. Archive refrigerators.

8. Procedures

8.1. Filter Pre-Inspection and Stability

- 8.1.1. 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:
 - 8.1.1.1. Pinhole—A small hole appearing as a distinct and obvious bright point of light when examined over, and up to, a light source.
 - 8.1.1.2. Separation of ring—Any separation or lack of seal between the filter and the filter support ring.
 - 8.1.1.3. Chaff or flashing—Any extra material on the reinforcing ring or on the heat-seal area that would prevent an airtight seal during sampling.
 - 8.1.1.4. Loose material—Any extra loose material or dirt particles on the filter.
 - 8.1.1.5. Discoloration—Any obvious discoloration that might be evidence of contamination.

- 8.1.1.6. Other—A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.
- 8.1.2. 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.
- 8.1.3. 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.
- 8.2. Pre-Weighing of Unsampled Filters
 - 8.2.1. 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 9.3.6 for limits. Procedures for calculating the temperature and relative humidity are kept in the lab procedures notebook which is located in the balance room. Also, make certain the selected filters have been conditioned in the balance room for at least 24 hours.
 - 8.2.2. 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.
 - 8.2.3. Perform an internal and external calibration of the micro-balance as described in Section 9.1.1.3 prior to beginning the weighing session. Transfer the external calibration values into LIMS.
 - 8.2.4. Weigh the four stability blanks. See section 8.4.6.
 - 8.2.4.1. 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.

- 8.2.4.2. Leave the stability blank on the balance for 30 seconds. Transfer the mass value into LIMS. Using metal non-serrated forceps, remove the stability blank and return it to the petri slide.
- 8.2.5. 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.
- 8.2.6. 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.
- 8.2.7. Create a work list in LIMS using the barcode numbers from the 24-Hr Report forms. All pre-weight work lists include duplicates, a lab blank, and upper and lower working control standards.
- 8.2.8. There must be a duplicate mass rate of $\geq 10\%$. Duplicates are automatically inserted by LIMS after every nine filters using the first filter of each set. A set can be up to nine filters.
- 8.2.9. Working control standards are automatically inserted by LIMS at the beginning and at the end of the work list. After each duplicate sample, the upper and lower working control standards are alternately inserted automatically by LIMS.
- 8.2.10. 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 LIMS after 30 seconds. Repeat these steps with the upper working control standard.
- 8.2.11. 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 LIMS. Record on the 24-Hr Report form the following:
 - 8.2.11.1. Cassette identification (I.D.) number (each support cassette rim is marked with an I.D. number).

- 8.2.11.2. Pre-weight mass of the filter.
- 8.2.11.3. Date of pre-weight measurement.
- 8.2.11.4. Analyst's initials.
- 8.2.11.5. Filter expiration date, which is 30 days from the pre-weight date.
- 8.2.12. Place the weighed filter into the appropriate filter support cassette or petri slide for speciation pre-weigh.
- 8.2.13. 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 speciation sample label. If the filter is for a SASS™ sampler and is a blank, a "field blank" 24-Hr Report form is used.
- 8.2.14. Repeat above steps (8.2.11 through 8.2.13) for subsequent filters and duplicate weighings.
- 8.2.15. Duplicate weighings—See Section 9.3.2 for criteria.
- 8.2.16. Working control standards are both weighed (100 mg and 500 mg) at the beginning of a weigh sessions, alternated (100 mg, 500 mg, 100 mg, etc.) after every set of filters plus a duplicate, and both weighed again at the end of a weigh session—See Section 9.3.1 for criteria.
- 8.2.17. Repeat above steps (8.2.11 through 8.2.16) for subsequent sets of filters.
- 8.2.18. At the end of the weighing session, weigh the lab blank(s) following pre-weight procedures and enter the filter number. Weigh the upper and lower working control standards. After the last control standard has been transferred to the work list, examine the data for errors, and document and correct, if necessary.
- 8.2.19. Transfer data to LIMS, print a pre-weight summary report. See Section 10.2.
- 8.2.20. After pre-weighing is complete, deliver the filters along with the corresponding 24-Hr Report form to the designated space in the sample handling room.

- 8.2.21. When the pre-weighing for SASS™ samplers is done, deliver the filters contained in labeled petri slides along with the corresponding 24-Hr Report form to the designated space in the speciation sample processing lab.

8.3. Post-Sample Handling

- 8.3.1. 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 sample may remain valid in LIMS until after post-weigh.
- 8.3.2. Filters received with “Not Used” notation are discarded, and the 24-Hour Report filed in the designated Quarterly archive “Not Used” folder.
- 8.3.3. PM2.5, PM Coarse, and SASS™ samples shipped and stored at a constant 4°C or lower before equilibration must be weighed within 30 days of the sampling date. Samples shipped and stored at a constant range between 4°C and 25°C before equilibration must be weighed within 10 days of the sampling date, unless the average ambient temperature during sampling was above 25°C, as reported on the 24-Hr Report form by the site operator. In that case, the sample must be weighed within 30 days of the sampling date. Samples received at temperatures above 25°C must be weighed within 10 days of the sampling date. Any samples exceeding the time limit between sampling and post-weighing must be noted on the matching 24-Hr Report. LIMS will automatically invalidate these samples.

Temperature at Receipt	Days Allowed Between Sampling and Weighing
4°C or lower	30
Between 4°C and 25°C Average ambient temperature >25°C	30
Between 4°C and 25°C Average ambient temperature <25°C	10
25°C or higher	10

8.4. Post-Weighing of Field Samples

- 8.4.1. 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 9.3.6 for limits. Procedures for calculating the temperature and relative humidity are kept in the lab procedures notebook located in the balance room. Also, make certain the selected filters have been conditioned in the Balance Room for at least 24 hours.
 - 8.4.1.1. Once the LIMS post-weigh worksheet is created, LIMS will automatically upload the calculated temperature and humidity values for each sample from their pre-weigh session. LIMS identifies the post-weight sample by its barcode, and retrieves the calculated temperature and humidity from that filter's pre-weight. These values were entered into LIMS during the pre-weight calibration session. LIMS will automatically compare the pre- and post- weight calculated values for each sample. If the pre- and post-sampling RH conditions are not within ± 5 % RH, LIMS will flag the sample. See Section 9.3.9 for limits. In the event a sample exceeds the control requirement, the filter should be 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 invalid.
- 8.4.2. 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.
- 8.4.3. Working control standards are automatically inserted by LIMS at the beginning and at the end of the work list. After each duplicate sample, the upper and lower working control standards are alternately inserted automatically by LIMS. There must be a duplicate mass rate of $\geq 10\%$. Duplicates are automatically inserted

by LIMS after every nine filters using the first filter of each set. A set can be up to nine filters.

- 8.4.4. After the samples have equilibrated for at least 24 hours, arrange the 24-Hr Reports of the samples that are to be post-weighed in the same order as the LIMS work list. Generate matching barcode labels on an equal number of petri slides. Remove the sampled filters from the conditioning cabinet. Match up the filter cassette I.D. numbers with the correct 24-Hr Reports 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 LIMS work list.
- 8.4.5. Perform an internal and external calibration of the micro-balance as described in Section 9.1.1.3 prior to beginning the weighing session. Transfer the external calibration values into LIMS.
- 8.4.6. Weigh the four stability blanks following procedures in Section 9.1.1.3. See section 9.2.2 for limits.
- 8.4.7. 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 work list. 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 LIMS worksheet. Record on the 24-Hr Report form the following:
 - 8.4.7.1. Post-weight mass of the filter.
 - 8.4.7.2. Date of post-weight measurement.
 - 8.4.7.3. Analyst's initials.
- 8.4.8. Place the weighed filter into the labeled petri slide.
- 8.4.9. Repeat steps 8.4.7 and 8.4.8 for subsequent filters and duplicate weighings. See Section 9.3.2 for criteria.
- 8.4.10. 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 work list, examine the data for errors, and document and correct, if necessary.

- 8.4.11. After the data transfer into LIMS is complete, run the PM2.5-Post Weight Summary Report. Confirm that the percentage of duplicates to filters is not less than 10 percent and that there are no duplicates out of range. File the report in the designated binder located in the balance room.
- 8.4.12. Make sure field and/or trip blanks are within control limits. See Section 9.3.4 and 9.3.5 for criteria. If not, reweigh the field and/or trip blanks and indicate that the post-weight was verified in the comments section of the 24-Hr report form.
- 8.4.13. Update LIMS with any invalid filters determined during the weighing session.
- 8.4.14. Exceedances of the National Ambient Air Quality Standard for PM2.5 should be reported to the lab supervisor. Abnormally high exceedances (i.e. above 100 $\mu\text{g}/\text{m}^3$) should be reported to the site operator and lab supervisor as soon as possible. If the mass variation between the pre-weight and post-weight of any blank filter is greater than ± 0.030 mg, notify the site operator and lab supervisor.
- 8.4.15. Place the weighed filter into a labeled petri slide, close tightly, and store cold 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.

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. Quarterly Calibration - Each quarter, 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 $\pm 2 \mu\text{g}$ 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 $\pm 2 \mu\text{g}$ of the initial calculated mass correction, see Section 9.3.7.
- 9.1.1.2.1. Quarterly Calibration Worksheets are then printed, peer reviewed, approved, and scanned as a PDF to the designated PM2.5 Calibrations file on the ILS Drive. See Appendix C for example.
- 9.1.1.3. Daily Calibration - The micro-balance must be calibrated daily, prior to any weighing session. This will be done by internal and external micro-balance calibrations and checked by alternating mass reference standards.
 - 9.1.1.3.1. 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.
 - 9.1.1.3.2. Internal Calibration: Press the "Internal Calibration" key on the balance liquid crystal display (LCD). The LCD should display "OK" when the balance has finished the internal calibration. Press the "OK" button and the balance is now ready for external calibration.
 - 9.1.1.3.3. External Calibration: Create a Calibration worksheet in LIMS. Insert the current set of Stability Blanks and insert the lower and upper working mass reference standards (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 LIMS. 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 LIMS. Repeat these steps for the Stability Blanks and final QC controls. Also, record and send the date, 24-hour calculated temperature and relative humidity of the balance room to LIMS.

External calibration 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 (i.e. 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 working control standards and 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.

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 duplicate weighing.

9.1.2.4. Each quarter, the masses of the working control standards are compared to the primary control standards. See Section 9.1.1.2.

9.1.3. Temperature and Percent Relative Humidity Recorder (Data Loggers)

9.1.3.1. The balance room’s relative humidity must be maintained at a mean value range of 30-40% and standard deviation $\leq \pm 5\%$ over the preceding 24 hours. Additionally, its air temperature must be maintained at a mean value range of 20.0-23.0 °C and standard

deviation $\leq \pm 2^{\circ}\text{C}$ 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. The data loggers should be set to record a temperature and relative humidity data point every two (2) minutes.
- 9.1.3.3. Each quarter, the primary temperature and humidity recorder should be checked against a NIST traceable temperature and humidity instrument standard. The standard must be recertified annually by an outside source. Record the temperature and humidity readings every two minutes from both the primary recorder and the standard (secondary recorder) at least ten times. Determine the averages of the temperature and humidity for both the primary recorder and the standard. Subtract the averages for the standard from the averages for the primary recorder. The differences should be within $\pm 2^{\circ}\text{C}$ for temperature and $\pm 2\%$ for relative humidity. If the differences are not within ± 2 , no weighing shall occur. Notify the lab supervisor and schedule a repair visit.
- 9.1.3.4. Quarterly Temp and Humidity Calibration Worksheets are then printed, peer reviewed, approved, 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 January 2016 EPA QA 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. Weigh lot blanks every 24 hours. In an Excel spreadsheet, record the lot number, filter number, mass, and dates of the lot blanks. Send the information to LIMS staff to update the Lot Blank Determination worksheet and criteria in LIMS. Once the mass difference between weighing is less than 0.015 mg for all 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, and trip 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. Document the lot blank study and pertinent information in the laboratory notebook.

- 9.2.1.1. Lot blanks should be replaced at least every 6 months, using the same procedure above.
- 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 ± 0.015 mg of its determined weight from the lot blank study. If the stability blanks are not within 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, consult with the lab supervisor.
 - 9.2.2.1. Stability blanks are replaced at least 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 sampling. 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
- 9.2.3.2. If the weight change between pre- and post- field blank weighings exceeds ± 0.030 mg, contamination may have occurred. Verify the mass value by reweighing the filter. If the mass difference still occurs, notify the site operator and lab supervisor. See section 9.3.
- 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 Field Blanks for suggested scheduling).
 - 9.2.4.2. If the weight change between pre- and post- trip blank weighings exceeds ± 0.015 mg, contamination may have occurred. Verify the mass value by reweighing the filter. If the mass difference still occurs, notify the site operator and lab supervisor. See section 9.2.3.2 and 9.3.
- 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. If the weight change between pre- and post- lab blank weighings exceeds ± 0.015 mg, 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	Solution
9.3.1	Mass Reference Standards (Working) Reference: U.S. EPA QA Handbook, Volume II, Appendix D	$< \pm 0.003$ mg from certified weight	Investigate the discrepancy further. Tare the balance, reweigh the weights and confirm mass values. If in a weigh session, confirm mass values of filters in the preceding set of 9. If still not within required ± 0.003 mg of certified weight, verify the balance is calibrated properly and check another set of QC weights. Verify the room is within the allowable limits. If the problem persists, weighing cannot proceed. Weights (primary and working) will either need to be replaced or recertified at the corrected or adjusted weight by an approved source. Or, if it's a balance issue, the balance may need recalibration or adjustment. Notify and consult with the lab supervisor. Document in the balance room laboratory notebook.
9.3.2	Duplicate Weighings Reference: U.S. EPA QA Handbook,	$< \pm 0.015$ mg from initial weight Duplicate mass rate of $\geq 10\%$	Investigate the discrepancy further. Tare the balance, reweigh the filter and confirm mass value. Confirm mass values of filters in the preceding set of 9. If duplicate is still not within required ± 0.015 mg of initial weight, verify the balance is calibrated properly and check QC weights. Verify the room is within the allowable limits. If the problem persists, notify

	QC	Acceptable Criteria/Range	Solution
	Volume II, Appendix D		the lab supervisor prior to proceeding with any weighing. Document in the balance room laboratory notebook.
9.3.3	Mass Reference Standards (Primary) Reference: U.S. EPA QA Handbook, Volume II, Appendix D	< $\pm$ 0.003 mg from certified weight	Investigate the discrepancy further. Tare the balance, reweigh the weights and confirm mass values. Verify the balance is calibrated properly and check another set of QC weights. Verify the room is within the allowable limits. If the problem persists, weights will either need to be replaced, or recertified at the corrected or adjusted weight by an approved source. Or, if it's a balance issue, the balance may need recalibration or adjustment. Notify and consult with the lab supervisor. Document in the balance room laboratory notebook.
9.3.4	Field Blanks Reference: U.S. EPA QA Handbook, Volume II, Appendix D	< $\pm$ 0.030 mg from initial weight	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 supervisor and site operator that the blank was > $\pm$ 0.030 mg. Make appropriate notations on the chain-of-custody form. Document in the balance room laboratory notebook.
9.3.5	Stability Blanks, Lot Blanks, Trip Blanks and Lab Blanks Reference: U.S. EPA QA Handbook,	< $\pm$ 0.015 mg from initial weight	Tare the balance, re-weigh the stability blanks to verify the weights. Check QC, mass reference standards, and verify the balance internal calibration has been performed. Check room conditions are within acceptable limits. If the discrepancy still remains, contact the ILS supervisor for direction. Document in the balance room laboratory notebook.

	QC	Acceptable Criteria/Range	Solution
	Volume II, Appendix D		
9.3.6	24-Hour mean Balance Room Temp. and RH Reference: U.S. EPA QA Handbook, Volume II, Appendix D	20.0 – 23.0 °C <i>and</i> Control of $\leq \pm 2$ °C 30 – 40 % and Control of $\leq \pm 5$ %	Determine if the equipment or HVAC system needs an adjustment and schedule if needed. No weighing can occur until the room is within limits for 24 hours. 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.
9.3.7	Quarterly Calibration Cw Reference: U.S. EPA QA Guidance Document 2.12, Section 9.7.2, January 2016	$\leq \pm 2$ µg from initial Cw value	Tare the balance, re-weigh the reference standards. Investigate the discrepancy further. Reweigh the weights and confirm mass values. Verify the balance is calibrated properly and check another set of QC weights. Verify the room is within the allowable limits. If the problem persists, weights (primary and working) will either need to be replaced, or recertified at the corrected or adjusted weight by an approved source. If the discrepancy still remains, contact the ILS supervisor for direction. Document in the balance room laboratory notebook.
9.3.8	Quarterly Calibration Temp. and RH Reference: U.S. EPA QA Handbook,	For differences between the averages for the standard from the averages for the primary recorder: $\leq \pm 2$ °C	Repeat the calibration process. If it's still out, notify the lab supervisor and schedule a repair visit. Document in the balance room laboratory notebook.

	QC	Acceptable Criteria/Range	Solution
	Volume II, Appendix D	$\leq \pm 2 \% \text{ RH}$	
9.3.9	Pre/Post Sampling RH 24-Hour mean difference Reference: U.S. EPA QA Handbook, Volume II, Appendix D	$\leq \pm 5 \%$	Verify the calculated 24-Hour values for both the pre-weight and post-weight sessions for that filter and confirm the difference is $> \pm 5\% \text{ RH}$. If the pre/post RH 24-Hour mean difference is $> \pm 5\% \text{ RH}$, remove the filter from the weigh session. If subsequent daily calculations still don't meet the control 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, it should be weighed, but will be invalid. Document in the balance room laboratory notebook.

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 LIMS and transferring 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 is then signed off, and the completed checklist filed with the report. 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 cold for at least one year at. 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

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

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

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, PM_{2.5} 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:

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

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 can be used to determine PM_{2.5} mass concentration:

$$\text{PM}_{2.5} = \frac{M_{2.5}}{V} \quad \text{Equation 3}$$

where,

$\text{PM}_{2.5}$	=	mass concentration of PM _{2.5} particulates ($\mu\text{g}/\text{m}^3$)
$M_{2.5}$	=	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:

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

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:

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

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:

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

where,

PM_{10} = mass concentration of PM10 particulates ($\mu\text{g}/\text{m}^3$)
 M_{10} = total mass of coarse particulate collected during sampling period (μg)
V = total volume of air sampled (m^3)

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

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

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 ample 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		
	January 1, 2018	New data loggers, revisions to the quarterly data logger calibration processes, quarterly mass reference standards calibration process, and the addition of lab blanks, and clarification of procedures. Added PM10 Coarse procedures.	Omega data loggers, less elaborate calibration process of weights and temp/humidity data loggers, and no Lab Blanks in 1.0 version.

13. References

- 13.1. Code of Federal Regulations, Title 40, Part 50, Appendix L, Appendix N, and Appendix O.
- 13.2. Chemical Hygiene Plan for 13th and T Facility, Monitoring and Laboratory Division. California Air Resources Board, July 2005.
- 13.3. Quality Assurance Guidance Document 2.12, Monitoring PM_{2.5} in Ambient Air Using Designated Reference or Class I Equivalent Methods. EPA-454/B-16-001. U.S. Environmental Protection Agency, January 2016.
- 13.4. Quality Assurance Project Plan, Chemical Speciation of PM_{2.5} Filter Samples. RTI/0212053/01QA. U.S. Environmental Protection Agency, January 2014.
- 13.5. Quality Assurance Project Plan for the PM_{2.5} Ambient Air Monitoring Program at State and Local Air Monitoring Stations (SLAMS). California Air Resources Board, October 2001.
- 13.6. Quality Assurance Handbook Volume II, Appendix D. U.S. Environmental Protection Agency, January 2017.
- 13.7. Backgrounder on Polonium-210, U.S. Nuclear Regulatory Commission, <https://www.nrc.gov/reading-rm/doc-collections/fact-sheets/polonium.html>, April 2016.

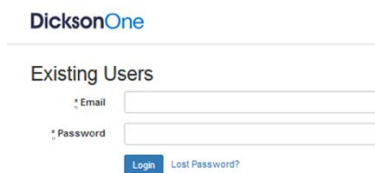
APPENDIX A

1. Procedures to Calculate Daily Balance Room Conditions

- 1) Logon to the Balance Room computer.
- 2) Open the “DicksonOne” website for DicksonOne – 14th/S or 13th/T, depending on location of weigh session.

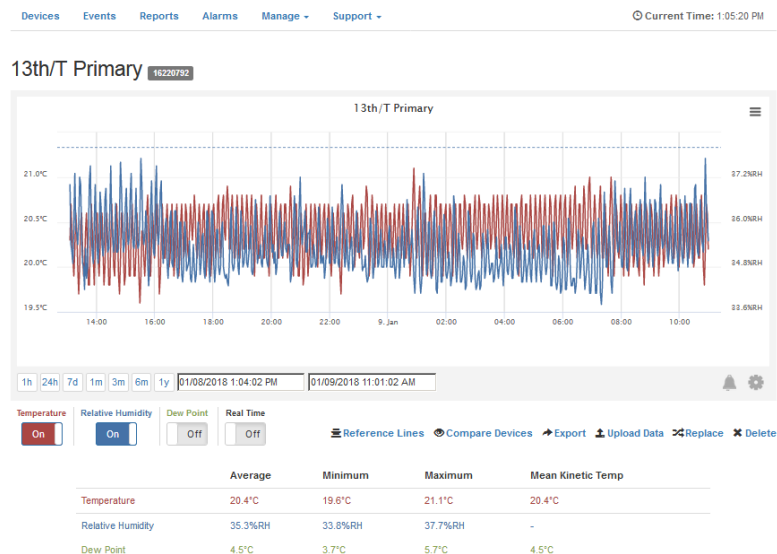
Website: <https://www.dicksonone.com/>

- 3) Login. Note: Analyst must be registered to access DicksonOne.



The image shows the DicksonOne login interface. At the top is the 'DicksonOne' logo. Below it is the heading 'Existing Users'. There are two input fields: one for 'Email' and one for 'Password'. Below the password field is a blue 'Login' button and a link for 'Lost Password?'.

- 4) Select the “13th/T Primary” or “14th/S Primary” device to retrieve historical READINGS. Wait for the device graph to open.

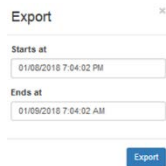


- 5) Click on the “**Export**” link on the lower right side of the device graph.

Devices **Export** Upload

- 6) The conditions range should be the 24-hour period immediately before the time of weighing begins.

Enter the desired conditions range and click .



Export

Starts at
01/09/2018 7:04:02 PM

Ends at
01/09/2018 7:04:02 AM

Export

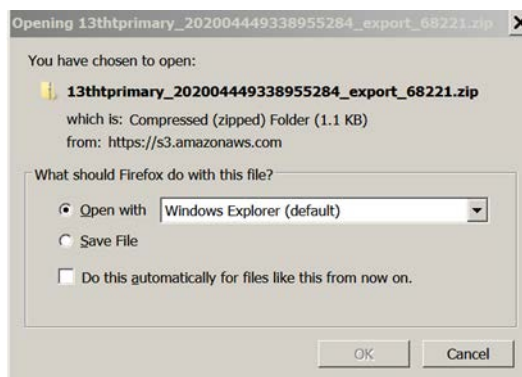
- 5) Open the ARB email account registered to receive exported READINGS from DicksonOne devices. Open the DicksonOne export email, which will appear similar to this:

DicksonOne Export Ready

The export you requested is now ready. You can download the file here:

<https://www.dicksonone.com/exports/68221>

- 6) Click on the link provided to download the file. Windows Explorer automatically opens.



- 7) Double-click on the name of the compressed Comma Separated Values (CSV) file. The EXCEL program automatically opens.

Name ^	Type	Compressed size
 13thprimary_202004449338955284_exp...	Microsoft Excel Comma Separated Values File	9 KB

In the EXCEL program:

- 8) All readings from the conditions range indicated will open on a single spreadsheet page.
- 9) Select the GMT Date and Time columns A and B. Delete these columns.
- 10) For the columns with "Temperature" and "Relative Humidity" header rows, determine the cell array for the 24-hour period of READINGS before weighing begins.
- 11) Use EXCEL formulas to calculate the 24-hour Average and Standard Deviation (SD) separately for Temperature (Temp) and Relative Humidity (RH) using the following cell formulas:
 - Average formula "=Average(cell start:cell stop)"
 - Example: =Average(F2:F722)
 - SD formula "=STDEV(cell start:cell stop)"
 - Example: =STDEV(F2:F722)
- 12) Verify that the calculated results for conditions are valid before starting a weigh session.
 - Criteria Temp: Valid to weigh when calculated Average is 20 to 23 °C and SD is within +/- 2 °C
 - Criteria RH: Valid to weigh when calculated Average is 30 to 40% and SD is within +/- 5%
- 11) The 24-hour conditions file is saved (on the non-weighing Balance Room computer) into "XLSX" format into the "Temp/RH" folder as "Temp RH" and the date of the weigh session(s). Example: Temp RH 01.01.18.xls.

APPENDIX B

1. Procedures to Calculate Quarterly Verifications of Working Control Standards

2.12. 9.0 Gravimetric Lab Design and Set-up

January 2016 Page 12 of 14

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, C_w , 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, C_p , are included. The symbols N_p and N_w refer to the nominal values of p and w , respectively.

$$C_w = C_p + ([O_1 - O_2 + O_4 - O_3]/2) + N_p - N_w$$

8. Subsequent measurements of C_w must be within $\pm 2 \mu\text{g}$ of the initial C_w value.

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

APPENDIX C

1. 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

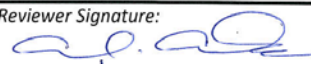
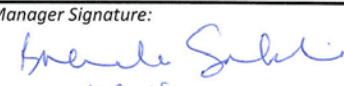
100 mg Control Weights		
Nominal Value (Np, Nw)	100 mg	
Primary 100 mg Mass Correction (Cp)	0.0034 mg	
Primary Serial Number:	1000131217	
Primary Calibration Date:	10/17/2017	
Working 100 mg Mass Correction:	0.0019 mg	
Working 100 mg Conventional Mass:	100.002 mg	
Working Serial Number:	1000131218	
Working 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

To: Brenda Saldana, Manager
Inorganic Laboratory Section

From:  Michael Miguel
Assistant Division Chief
Monitoring and Laboratory Division

Date: January 24, 2019

Subject: STANDARD OPERATING PROCEDURE FOR DETERMINATION OF PM2.5
MASS AND PM COARSE MASS BY GRAVIMETRIC ANALYSIS

Thanks you for the submission of the addendum (see attached) to the Standard Operating Procedure (SOP) for Determination of PM2.5 Mass and PM Coarse Mass by Gravimetric Analysis. The Quality Management Section has reviewed the addendum along with the SOP and determined that it covers all of the required elements. The addendum is approved.

Please direct comments or questions to Kyle Vagadori at (916) 445-9391 or by email at kyle.vagadori@arb.ca.gov.

Attachment

cc: Kyle Vagadori
Quality Management Section

QUALITY MANAGEMENT DOCUMENT ADDENDUM

(District completes Sections 1 through 6 -- please type)

Section 1. ARB Document	
☐ Quality Management Plan (QMP)	
☐ Quality Assurance Project Plan (QAPP)	
☒ Standard Operating Procedure (SOP)	

Section 2. District Information		
District Name:	CARB – Monitoring and Laboratory Division – Northern Laboratory Branch	
District Address:	1927 13 th Street, Sacramento CA 95811	
District Contact Name/Phone Number:	Elias Villa	916-327-0886
District Signature/Date:		

Section 3. Document Title (specify exact title, revision #, and date of ARB Document(s) that your District proposes to modify)	Date
MLD055 Revision 2.0 Standard Operating Procedure for Determination of PM2.5 Mass and PM Coarse Mass by Gravimetric Analysis	8/2/18

Section 4. Proposed Deviation(s) (specify exact section(s), page number(s) and language in existing ARB document that your District proposes to modify and then specify proposed modification (including any spreadsheets or forms).)
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Section 7 Equipment and Supplies, Page 7:

Add the following item to the list of equipment and supplies:

7.29. Warm Mark® 5°C and 25°C Temperature Indicators or equivalent.
Indicate if sample temperature exceed 5°C or 25°C during shipment and storage.

Continued on next page...

Section 8.3.3, Page 11:

Samples shipped and stored at a constant range between 4°C and 25°C before equilibration must be weighed within 10 days of the sampling date, unless the average ambient temperature during sampling was above 25°C, as reported on the 24-Hr Report form by the site operator. In that case, the sample must be weighed within 30 days of the sampling date. Samples received at temperatures above 25°C must be weighed within 10 days of the sampling date.

Temperature at Receipt	Days Allowed Between Sampling and Weighing
4°C or lower	30
Between 4°C and 25°C Average ambient temperature >25°C	30
Between 4°C and 25°C Average ambient temperature <25°C	10
25°C or higher	10

Update to:

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 or equal to 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 the average ambient temperature during sampling, the sample must be weighed within 10 days of the sampling date.

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

PM2.5 Mass sample shipping and storage temperature is tracked using temperature indicators that show if a sample has exceeded 5°C or 25°C at any point in the shipping process. Because temperature is not measured directly, samples received with activated 5°C indicators and inactive 25°C indicators are assumed to have been received at 25°C. Samples received with activated 5°C and 25°C indicators are assigned a greater than 25°C receipt temperature.

PM2.5 SASS™ mass shipping temperature is directly measured upon receipt.

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PM2.5 Mass

Temperature at Receipt	Days Allowed Between Sampling and Weighing
4°C or lower	30
4-25°C & Average ambient temperature $\geq$ 25°C	30
4-25°C & Average ambient temperature < 25°C	10
Greater than 25°C	10
Unknown	10
Field Blanks – Greater than 4°C	10

PM2.5 SASS™ Mass

Temperature at Receipt	Days Allowed Between Sampling and Weighing
4°C or lower	30
Average ambient temperature $\geq$ Receipt temperature	30
Average ambient temperature < Receipt temperature	10
Unknown	10
Field Blanks – Greater than 4°C	10

Section 5. Justification for Deviation(s)

(provide explanation of why modification(s) to existing ARB document is necessary)

Proposed modifications more closely aligned with Environmental Protection Agency guidance detailed in the Quality Assurance Guidance Document 2.12, and 40 CFR, Part 50, Appendix L, Sections 8.3.6 & 10.13

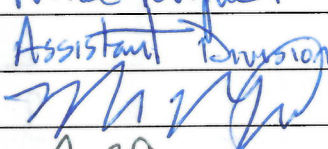
Section 6. Attachment(s) ☒

(specify attachment titles and number of pages, include modified spreadsheets or forms)

of Pages

Section 7. ARB Approval

(completed by ARB)

Name/Phone Number:	Mike Miguel	916-322-0960
Title:	Assistant Division Chief	
Signature/Date:		1/31/19
Addendum Number	A-29	

Completed form must be scanned/emailed or mailed to:

Mr. Patrick Rainey
 1927 13th Street, P.O. Box 2815
 Sacramento, California 95811
prainey@arb.ca.gov