

STATEWIDE STANDARD OPERATING PROCEDURE

FOR THE
DETERMINATION OF PARTICULATE MATTER (PM_{2.5}) IN
AMBIENT AIR BY GRAVIMETRIC ANALYSIS



STATE OF FLORIDA
DEPARTMENT OF ENVIRONMENTAL PROTECTION
OFFICE OF AIR MONITORING

Prepared by: OAM Gravimetric Lab

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Approved by: _____
Designated Approving Manager

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Document History

Revision No.	Date	Responsible Person	Description of Change
-	-	DEAR	The following revisions were made to SOP revision PM-001-3.15 from the prior version PM-001-3.14: • Incremented the SOP number. • Made all updates as requested in EPA SEDS's August 3, 2015 review of the lab SOP. • Updated holding time determination per DARM's Statewide Data Handling and Data Validation SOP (DEP 18-17), Section 12.2.2. The lab does not have sufficient information from submitting field agencies to evaluate filter holding times. The holding time evaluation will need to be performed by each field agency following receipt of the lab data.
-	June 2016	DEAR	June 3, 2016 - Made updates as requested in EPA SEDS's February 12, 2016 review of the lab SOP.
0	March 2018	Anna Lomasney	Initial release from DARM; adapted from DEAR PM-001-3.16; organizational and procedural updates corresponding to lab transition from DEAR to DARM. Document assigned a DEP tracking number (DEP 16-23) and updated to DARM format.
1	November 2021	Cadedra Hodge	The following updates were made: • Updated DEP logo to most recent version. • Removed Section 18.0 and incorporated it into this table (Document History). • Moved Appendix 19.13 Filter Holding Time Decision Flowchart to Section 12.0. • Reformatted Section 19.0.

Revision No.	Date	Responsible Person	Description of Change
2	September 2022	Chris Armour	Updated signature requirements regarding the Cooler Receiving Log; clarified cooler tape usage; replaced Greyhound procedures and references to FedEx; updated Cooler Receiving Log form in Appendix.

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1.0 SCOPE AND APPLICATION

The principle objective of this air quality monitoring project is to measure the concentration ($\mu\text{g}/\text{m}^3$) of particles $\leq 2.5 \mu\text{m}$ in diameter ($\text{PM}_{2.5}$) that have been collected on 47-mm Teflon filters in accordance with the reference method set forth in the U.S. Environmental Protection Agency's (EPA's) Code of Federal Regulations (CFR), specifically 40 CFR Part 50, Appendix L.

This standard operating procedure (SOP) details the operations of the $\text{PM}_{2.5}$ gravimetric laboratory ("lab"). These operations include:

- Maintenance and operation of the weighing lab environment, equipment, and supplies;
- Filter handling and storage;
- Conditioning and weighing the filters;
- Shipping and receiving filters; and
- Reporting the gravimetric results.

The lab is operated and managed within the Division of Air Resource Management (DARM) Office of Air Monitoring (OAM). Prior to 2017, the lab was operated and managed within the Division of Environmental Assessment and Restoration (DEAR) Chemistry section.

The procedures described within this SOP meet the criteria established in EPA 40 CFR Part 50, Appendix L for $\text{PM}_{2.5}$ measurements. Additional guidance is provided in the EPA Quality Assurance Guidance Document 2.12 (January 2016), also referred to as Method 2.12. Low-volume PM_{10} samples may also be analyzed according to the same requirements and guidance.

2.0 SUMMARY OF THE METHOD

The PM_{2.5} lab was established for the gravimetric analysis of the quantity of atmospheric particulate matter ≤ 2.5 μm in diameter (without regard to chemical speciation) collected using reference methods set forth in 40 CFR Part 50, Appendix L. Teflon filters are conditioned in a clean room environment with carefully regulated temperature and relative humidity to ensure stable, reproducible masses. These filters are then pre-weighed on a microbalance, packaged, and shipped to various sampling sites for collection of particulates. Upon return of the filters from the sampling sites, they are again conditioned and post-weighed. The lab reports the pre- and post-weights and the resulting delta, calculated as the difference between the pre- and post-weights, along with any associated qualifiers and comments. The particulate concentration, in $\mu\text{g}/\text{m}^3$, is subsequently calculated within the DARM Particulate Matter Tracking System (PMTS) database using the pre- and post-weights reported by the lab and the measured volume of air drawn through the filter during sampling.

3.0 EQUIPMENT AND SUPPLIES

Equipment and supplies necessary to perform PM_{2.5} gravimetric analysis in accordance with Method 2.12 are listed below:

- Mettler-Toledo Microbalances (2 units)
- Marble or Granite Balance Tables Equipped with Anti-vibration Pads
- Rupprecht & Patashnick (R&P) 47-mm Filter Beveled Cassettes with Screens
- Rupprecht & Patashnick (R&P) Magazines with Caps
- MTL (Measurement Technologies Laboratories) 47-mm Teflon Filters or Equivalent
- Millipore Plastic Petri Slides or Equivalent
- Anti-Static Electronic U-Electrode (2 units)
- Staticmaster Polonium-210 strips
- Nylon Forceps
- Powder-Free Vinyl Gloves
- Certified 300 mg and 500 mg Working/Primary Mass Standards (Class 0)
- Vaisala National Institute of Standards and Technology (NIST) Traceable HMT333 Digital Hygrometer/Thermometer (3 units)
- Vaisala Veriteq ViewLinc Software
- Large Hard-sided Coolers
- Small Soft-sided Coolers
- Ice-Substitute (“Blue Ice”) Gel Packs, Large and Small
- Re-sealable Plastic Bags
- Lab Refrigerators
- Fisher Brand Tacky Mop or Equivalent
- Tyvek Lab Coats, Hair Covers, and Shoe Covers
- Lint-free Cloth “Texwipes”
- “Balance Command” WinWedge Version 3.4.3 Standard Edition Software, 2014, by TAL Technologies
- Solid Metal Shelves, Heavy-duty
- Siemens Insight, Version 3.9.1 Electronic AC Controller Unit

- Neptronic SK302M Humidifier
- Munters Model HC-300 Dehumidifier
- Infrared Thermometer NIST Traceable
- Light Box and Magnifying Glass
- Staticide Antistatic Solution, ACL Inc.
- Alcohol (i.e. Methanol, Absolute Ethanol, or Isopropyl Alcohol)

4.0 EQUIPMENT AND ENVIRONMENT MAINTENANCE AND SPECIFICATIONS

4.1. Weighing Equipment and Supplies

4.1.1. Analytical Microbalances

- Located in the conditioned environment (see Section 4.2.1.).
- Readability of 1 µg and repeatability of at least 1 µg.
- Internally calibrated prior to weighing (see manual for instructions on auto-calibration).
- Electrically grounded and operated with the leveling bubble at the level position.
- Serviced by a Mettler–Toledo technician (or other qualified service provider) once a year. Additional service technician visits may be required if a balance is not operating within the acceptance limits of the method or is otherwise malfunctioning. Documentation of instrument service is kept on file by the lab.

4.1.2. Mass Reference Standards

- The primary mass reference standards (“primary standards”) are certified annually by a NIST-traceable weights and measures lab, and should be ASTM Class 0, Class 1, or Class 2. The primary standards and certifications may be acquired through Troemner, Rice Lake Weighing Systems, or equivalent vendor.
- The working mass reference standards (“working standards”) are verified quarterly against the primary standards by the PM_{2.5} lab staff. Working standards are used for routine filter weighing; however, the primary standards may be used for filter weighing if necessary. The working standards are generally older but undamaged primary standards that are no longer in current certification.
- Documentation of the primary standard certifications and working standard verifications are stored electronically or in hardcopy.
- Standards must be handled and stored properly to avoid damage. The standards are stored in containers labeled with a serial number, handled only with labeled nylon forceps, and placed only on clean, dry surfaces. Working standards are assigned to a specific balance – take care not to mix them. Document and

verify the correct standard serial number on each lab weigh sheet (see Section 8.3).

- Standards that have been scratched or bent, show corrosion, or no longer meet the weight specifications must not be used.

4.1.3. Computers and Associated Software

The lab uses WinWedge Balance Command, Microsoft Excel, and a customized Laboratory Information Management System (LIMS) for data acquisition and reporting.

- The software programs Balance Command and Excel are used for automated data collection from the microbalances.
- The Scientific Services and Support (SSS) Program within DEAR is responsible for maintenance of LIMS. Contact that program administrator or designee for LIMS issues or updates including lab automated email distribution list changes, filter status change requests, lab analyst permissions changes, and the annual LIMS calculation and rules review.
- Computer hardware or other software related problems are reported through the DEP Office of Technology and Information Services (OTIS) Help Desk.

4.1.4. Balance Weighing Tables

Microbalances are located on sturdy granite or marble tables to ensure balance stability. Isolation spacers are used to reduce vibration. The table placement in a controlled environment (room within a room) minimizes air drafts and thermal instabilities. Table surfaces are kept free of clutter, paper, chemicals, oils, electrostatic charge, and particulates. The weighing table surface should be cleaned weekly with Staticide.

4.1.5. Conditioning Shelves

Industrial, heavy-duty steel shelves are used for conditioning the filters. Shelves should be smooth, level, and stable, and must be cleaned with Staticide before filters are placed for conditioning. Shelves are grounded to prevent static.

4.1.6. Anti-Static Devices

A variety of anti-static devices, supplies, and procedures are employed in the lab, including U-electrodes, Polonium strips, and grounding wires or cables, and Staticide cleaning solution. U-electrodes should be

cleaned periodically to prevent dust accumulation. Polonium strips should be replaced every 6 months. The newer strips should be used inside the balance chamber, while older strips can be rotated out for balance tabletop use.

4.1.7. 47-mm Teflon Filters

The 47-mm polytetrafluorethylene (PTFE or Teflon) filters are acquired from the EPA through the Office of Air Quality Planning and Standards. EPA will usually contact the lab for the filter order in the fall of each year. Around the same time, EPA publishes the annual sampling schedule.

Filter shipments include a memo detailing the lot characteristics and acceptance testing results, which should be kept in lab records.

Each new lot of filters is subject to a lab filter lot stability test to establish the lot conditioning period (see Section 7.3). Filter lots can be used for longer than one year, but this is generally not necessary.

Filters are stored in the anteroom prior to use, in the weigh room during processing, and in the lab refrigerators or storage areas when archived. Archived filters must be stored for five years total (see Section 13).

The lab may keep old or unused filter lots for demonstration, testing, or training. These demo filters should be stored separately and marked clearly.

4.2. Maintenance and Monitoring of the Air Conditioning Equipment

4.2.1. Conditioned Environment

The microbalance and filter conditioning environment temperature and humidity data are recorded at ≤ 5 -minute intervals and evaluated based on 24-hour averages and standard deviation of the 24-hour averages.

NOTE: *Twenty-four (24) hours is the minimum required conditioning period and may be longer depending on the filter lot stability test. See Section 7.3.*

- The 24-hr average relative humidity (RH) must be 30.0-40.0% RH, with a variability not to exceed 5.0% RH standard deviation.
- The 24-hr average temperature must be 20.0-23.0 °C, with variability not to exceed 2.0 °C standard deviation.
- The difference in 24-hr average RH for the pre- and post-weights must be less than or equal to $\pm 5.0\%$ RH.

Filters are weighed only when environmental conditions are within these acceptance limits. Weight variations due to absorption or desorption of water vapor on the filter surface or on the particulate matter collected will be minimized by conditioning the pre- and post-sampled filters within these temperature and humidity ranges. The weighing room conditions are easier to maintain if foot traffic and the number of personnel in the weighing environment is minimized.

4.2.2. Air Conditioning Maintenance

The DEAR Laboratory Engineering Specialist is responsible for the following:

- The air-conditioning pre-filter should be changed every 3 months or as needed to minimize the pressure drop across the filtration system. Fan speed may be adjusted seasonally or as needed to maintain controlled lab conditions. Increasing the fan speed tends to increase humidity in the weigh room; decreasing fan speed tends to decrease humidity in the weigh room. See Section 18-3 for details.
- The HEPA filter should be changed about every four years. The condition of the HEPA system is monitored by a pressure gauge measuring the pressure drop across the filter media. The media should be replaced when the drop exceeds 2.0 inch water gauge (w.g.) (498 Pa).
- The Neptronic SK302M humidifier and the Munters HC-300 dehumidifier should be inspected/monitored regularly as part of the general air condition service. The dehumidifier desiccant cartridge requires replacement about once a year, or as needed. The humidifier requires cleaning of the evaporation chamber about once a year, or as needed. Refer to the operator manuals for details.
- Verify AC fan belt tension and condition. Fan belt should not slip during operation and should not be frayed or cracked.
- The lab will document HVAC, Neptronic, and Munters maintenance, service, and adjustments in the lab notebook.

4.2.3. Vaisala Veriteq Software

The PM_{2.5} lab uses Vaisala Veriteq software to monitor and record the room temperature and humidity. The Vaisala Veriteq data is maintained

on a DEP server, manually backed up weekly using the Veriteq software report functions and archived in the PM_{2.5} network folders. DARM OAM Quality Assurance/Audit staff also access the data for monthly and annual reviews and verifications.

4.2.4. Siemens Insight Software

The lab conditions are also monitored using Siemens Insight software, maintained by DEAR. When possible, the lab will archive this data.

4.3. Forceps for Handling Filters and Standard Weights

Filters and standards are handled only with forceps specifically denoted for the type of handling to be performed. Nylon forceps used to handle filters and standards are labelled on the handles with “Pre,” “Post,” or “Weights” and must be used only for those purposes. Metal or plastic-coated forceps must not be used. Discard defective, worn forceps.

4.4. Laboratory Attire

Tyvek lab attire, including coats, caps, and shoe covers, must be worn by anyone entering the weighing environment. Powder-free gloves should be worn when handling filters or standards. The Tyvek lab apparel is donned in the anteroom prior to entering the weighing environment. The protective clothing is disposable but can normally be used for about 1 week before it needs to be discarded. The primary purpose for the attire is to minimize particles being introduced into the weighing environment. For this reason, clothing that generates dust or has loose fibers should not be worn into the weighing area.

4.5. Refrigeration for Filter Storage

Refrigerators for archival of sampled filters must maintain a temperature range of 1-4 °C. The temperature is recorded using either a log on the exterior door or an electronic data recorder where the temperature of each refrigerator is recorded at least daily. All archived filters are stored in the refrigerator for a minimum of one calendar year. These refrigerators may also be used to store post-sampled filters if received early or when conditioning or weighing is delayed. Thermometers associated with the electronic data loggers should be verified at least annually, and the logged data should be archived at least quarterly. Currently, these electronic data loggers and the Checkpoint software used to access the data are provided by DEAR.

4.6. Digital Hygrometer & Temperature Control Probes and Software

The digital sensors perform several functions in the lab environment, including interface with the air condition (AC) system and recording and transmitting temperature/humidity data in the conditioning/weighing lab. The Siemens

sensor is used to interface with the building HVAC system to help regulate the temperature in the weighing room. The Vaisala sensors interface to the Vaisala Veriteq software which is used to monitor, record, and report the lab temperature and humidity data. The lab is equipped with multiple Vaisala sensors for backup and verification.

- NIST certification is maintained by sending units to Vaisala for recalibration annually. The Vaisala sensor calibration criteria for accuracy is $\pm 0.5\text{ }^{\circ}\text{C}/\pm 2\% \text{ RH}$ and for resolution is $\pm 0.1\text{ }^{\circ}\text{C}/\pm 0.5\% \text{ RH}$.
- Verification of the Vaisala sensors is performed on a quarterly basis with criteria of $\pm 2\text{ }^{\circ}\text{C}/\pm 2\% \text{ RH}$ by comparing the working and back-up sensor readings to the primary sensor readings. The primary sensor unit is labeled and designated in the lab notebook. The verifications are documented in the Quarterly Temperature-Humidity Verification Form (Figure 18-9.).
- When possible, the Siemens sensor functionality is verified on a quarterly basis by comparing the 5-minute data archived in the DEAR monitoring system against the Vaisala primary unit. The Siemens sensor is not used to validate lab data and is not subject to annual calibration.

4.7. Filter Shipping Supplies

4.7.1. Rupprecht & Patashnick (R & P) Magazines

Each magazine is assigned a unique magazine number (“mag ID”). A mag ID sticker is attached to the side of the magazine and is assigned in the LIMS system for custody tracking. Magazine port O-rings or caps should be replaced if worn. Repair or discard magazines with faulty pistons, ports, or damage.

4.7.2. R & P Filter Cassettes and Screens

Use only R&P cassettes with beveled tops. The older 90-degree angle tops are not suitable for the PM_{2.5} samplers since they result in equipment jams. Special attention should be given to the “fit” of the top and bottom components. Maintain cassette components in pairs; however, any screen can be used with any cassette. Discard worn or damaged cassettes and screens.

4.7.3. Insulated Shipping Containers (Soft-Sided and Hard-Sided Coolers)

All shipping containers must be inspected after every use for cleanliness and damage. Replacement coolers are purchased by the lab or the local program agencies, as needed.

4.7.4. Ice-substitute Gel Packs (“Blue Ice”)

These are used to maintain the proper preservation temperature during filter transport. They are packed tightly in the coolers to minimize shifting during transport. The blue ice packs are kept in sealed plastic bags but are discarded if leaking. Replacement blue ice is purchased by the lab or the local programs as needed.

4.8. Infrared Thermometer Gun

An infrared thermometer gun (“IR gun”) is used to measure the temperature of the incoming magazines upon receipt into the lab documented on the Cooler Receiving Log, and for any other routine magazine temperature checks. The IR guns are purchased with a NIST-traceable certificate of calibration that is usually certified for a 2-year period over a calibration range suitable for ambient temperatures. No quarterly or annual verifications are required for the IR guns, which are valid through the certified dates.

5.0 GENERAL PM_{2.5} LABORATORY CLEANING PROCEDURES AND SCHEDULE

5.1. Laboratory and Equipment Cleaning

- Workbenches and filter shelves must be wiped down at least weekly using Texwipes and Staticide.
- Clean forceps with Texwipes and alcohol or Staticide weekly or as needed.
- The floor of the weighing environment and of the anteroom must be rolled using the Tacky Mop at least weekly. Use a Tacky Mop sheet to clean the floor behind the weighing tables as needed.
- All trash receptacles are placed outside the lab entry door for custodian disposal.
- Minimize foot traffic in weighing environment to reduce contamination.
- A sticky floor mat at the entrance into the weigh room is used to reduce the amount of particulates brought into the weighing environment. Replace as necessary.
- Use a clean soft brush to clean the balance weighing pan and surrounding area around the pan as needed. Perform the cleaning with great care to minimally disturb the sensitive balance weighing pan.
- Upon receipt, wipe out hard and soft coolers to dry, or use bleach cleaning solution as needed, and leave open to air dry. Dry the bags with blue ice. Handles, latches, and hinges on the coolers must be functional. When possible, replace broken components using spare parts.

5.2. R&P Cassette Cleaning Procedure

The filter cassettes must be cleaned after every use with Texwipes and alcohol:

- The cassette top, bottom, and screen are separated and placed on the workbench.
- Wipe each component of the cassette.
- Inspect and reassemble the cassette. Cassettes with hairline cracks, roughened areas, or poor fit should be discarded. Screens with rust or serious dents should be discarded.
- Place the clean, assembled cassettes in a labeled plastic bag.

5.3. R&P Magazine Cleaning Procedure

The magazines must be cleaned after every use with Texwipes and alcohol:

- Remove the top orange cap on the magazine.

- Wipe the underside of the orange cap.
- Wipe the inside of the magazine column and the top of the magazine piston.
- Use the hand pump to push the piston to the top of the magazine.
- Remove any fibers left by the cleaning process.
- Place the orange cap on the magazine.
- Check the pneumatic port O-rings and the fit of the caps. Discard loose or damaged caps.

Table 5-1. PM_{2.5} Laboratory Maintenance, Calibration/Verification, and Archive Schedule

Item	Responsibility	Frequency
Trash	Lab Staff	Daily
Cleaning Weigh Tables, Workbenches, Conditioning Shelves, Floors	Lab Staff	Weekly
Cleaning Cassettes, Magazines, Coolers	Lab Staff	Weekly
Replace Sticky Floor Mat	Lab Staff	As Needed
HVAC and HEPA Filter Maintenance	Lab Engineering Specialist	See Section 4.2.2.
Humidifier and Dehumidifier	Lab Engineering Specialist	See equipment manuals
Microbalance Calibration Service	Authorized Service Provider	Annually
Vaisala Temperature/Humidity Sensor Verification	Lab Staff	Quarterly
Vaisala Temperature/Humidity Sensor Calibration	Lab Staff	Annually
Vaisala Data Archive	Lab Staff	Weekly
Vaisala Software Calculations Verification	OAM QA/Audit Staff	Annually
LIMS Rules and Calculations Verification	Lab Supervisor/DEAR Scientific Services Support (SSS) Program Staff	Annually
Primary Mass Standard Replacement or Recertification	Lab Staff	Annually
Working Mass Standard Verification	Lab Staff	Quarterly
IR Gun Replacement	Lab Staff	Per Certificate Dates
Refrigerator Temperature Sensor Verification	Lab Staff/Standards Lab	Annually
Refrigerator Temperature Data Archive	Lab Staff	Quarterly
Filter Orders	Lab Supervisor/EPA Designee	Annually
Filter Archive	Lab Staff	Annually

6.0 FILTER HANDLING PROCEDURES AND INTERFERENCES

Careful handling of the filters during conditioning, weighing, and packing is necessary to avoid measurement errors, filter contamination, interferences, and damage.

6.1. Handling Filters in the Conditioning Environment

All filters, regardless of status, are handled only in the conditioning environment with appropriately labeled forceps and while wearing powder-free gloves. The filters must not be removed from the lab environment unless they are protected inside a magazine or Millipore Petri slide.

6.2. Handling Filters with Forceps

All filters are to be handled using appropriately labeled forceps by the plastic filter support ring. Forceps should never touch the Teflon filter media. The filter media is delicate, and contact may damage the filter surface.

6.3. Millipore Petri-slide Storage of Filters

Each filter is stored in a Millipore Petri-slide, always covered except when being conditioned, weighed, and processed for shipping or receiving. The Millipore Petri-slide has a cutaway section to make it easier to pick-up the filters with forceps.

6.4. Laboratory Interferences

Most analytical interferences can be mitigated by proper lab practices, training, and instrument/work environment maintenance.

6.4.1. Particulates

PM_{2.5} filters may become contaminated during conditioning or weighing from particulates in the air, on the microbalance or working surfaces, and on personnel who enter the lab. To reduce these sources of contamination, personnel must wear Tyvek coats, hair covers, and shoe covers when in the weighing room; the air in the room is purified with a HEPA filtration system, and all work surfaces must be cleaned regularly.

6.4.2. Static Charge

Static electrical charge is a common interference in performing microbalance measurements. To reduce this source of interference, the microbalances are electrically grounded, the working surfaces are wiped with Staticide, and the weights and filters are discharged using the U-electrode and polonium strips.

6.4.3. Environment Conditions

Excessive air movement or large changes in the room temperature or humidity may interfere with balance performance. The room temperature and humidity must be closely regulated by the air handling system and air movement minimized with proper baffling and work habits.

6.4.4. Vibrations

The lab environment must be free of vibrations. Minimize foot traffic. The mass of the balance bench and use of vibration absorbers can also minimize vibration interferences.

6.4.5. Balance Drift and Taring

The balance should not drift or require excessive taring during a typical weigh session if the instrument is grounded and lab conditions are properly maintained. Drift may be observed if the balance has been powered-on recently; if so, allow the instrument to stabilize for 20 minutes prior to use. Excessive balance drift may be indicative of more serious equipment or environment problems and should be reported to the lab supervisor.

7.0 **FILTER CONDITIONING PROCEDURES AND CRITERIA**

The PM_{2.5} filters must be inventoried, inspected, and conditioned prior to weighing. The procedures described in this section include verifying new filter lots, conditioning pre-sampled filters, and conditioning post-sampled filters. The weighing procedures, described briefly in this section, are given in detail in the next section.

7.1. **Recording Environment Conditions**

The conditioning environment for the filters must be within the acceptance criteria given in Section 4.2.1. These environmental conditions must be documented in the weigh sheets, for the previous 24-hour period checked no more than 30 minutes from the beginning of the weigh session. In addition, a plot of the 24-hour temperature and humidity data from the Vaisala Veriteq software, including temperature and humidity minimum/maximum/average and standard deviation (SD), must be recorded, verified, and added to lab records. All filters processed during a given weigh session must be completed within 10 hours of the assigned filter conditioning end time. A final 24-hour temperature and humidity data set (“end-of-weigh” data) is collected, verified, and recorded at the end of the weigh session to verify that the weighing room was in control during the weigh session.

NOTE: *Twenty-four (24) hours is the minimum required conditioning time. Longer times may be required depending on the filter lot stability test performed for each new lot of filters provided by EPA – see Section 7.3. For simplicity, the conditioning period is generally described as 24 hours in this SOP; however, if the lot stability test determines that a longer conditioning period is required for a given lot of filters, each conditioning period or procedure described in this SOP would be based on that determined time.*

- Open the Vaisala Veriteq software program. Software updates may change details of the following steps.
- Enter Name (“admin”) and Password (“admin”).
- Select “Options” and “Locations” and select the “Trend” tab at the top of the dashboard.
- Select the working Vaisala unit. Display the Relative Humidity and Temperature selections in the plot interface.
- Make sure the “Start Date and Time” and “Duration” represents the past 24 hours.

- Verify that the data captured includes plot axis labels, time and date stamps, sensor ID, max, min, and average values, standard deviations, and number of samples (datapoints).
- Create an Adobe Portable Document Format (PDF) file of the plots and statistics, save in the lab network drive folders, and print a copy for the weekly folder.

NOTE: One analyst will create and initial the Vaisala plot data file using a Word template. Then a second analyst independently verifies the data, initialing and dating the Word file. The verified Word document is then saved as a PDF. The final record is the PDF document.

- Verify that the humidity average was maintained at 30.0-40.0% ($\leq 5\%$ SD) for the past 24-hours.
- Verify that the temperature average was 20.0-23.0 °C (± 2 °C SD) for the past 24-hours.
- On the weigh sheet, record the temperature/humidity minimum, maximum, and 24-hour averages to four significant figures, exactly as presented by the Veriteq software program.
- On the weigh sheet, record the temperature/humidity 24-hour standard deviations to two decimal places, exactly as given by the Veriteq software program.
- The Excel formatting in the weigh sheet may drop the trailing zeroes for these data entries.
- Filters are weighed only when environmental conditions are within the temperature and humidity acceptance limits.
- The impact of relatively brief temperature or humidity excursions (“transients”) outside of the limits described in Section 4.2.1 must be considered when weighing filters. A transient is defined as a brief humidity and/or temperature excursion outside acceptable limits that has an insignificant impact on the overall environmental conditions and measurements. The length of delay time required between the transient and the beginning of a weigh session depends on the duration of the transient. The table below (Table 7-1) summarizes acceptable delay times for weighing filters after a transient.
 - The temperature and humidity values during the transient must be included in calculation of the 24-hour criteria.

- The 24-hour criteria for humidity and temperature must still be achieved after a transient has passed the minimum delay times given in Table 7-1.
- Temperature/humidity excursions lasting longer than 45 minutes are not considered transient.
- Temperature/humidity excursions large enough to create standard deviation results outside acceptable limits are not considered transient.

Table 7-1. Delay Times for Weighing Filters After a Humidity/Temperature Transient

Temp/Humidity Transient Duration	Minimum Delay for Weighing from Transient
< 10 minutes	30 minutes
10-30 minutes	60 minutes
30-45 minutes	180 minutes

- Transients during a weigh session should be evaluated based on extent and duration and include assessment of weigh session quality control (QC) checks and analyst observations of balance behavior. The end-of-weigh data must meet acceptance criteria for the weigh session to be valid. When acceptance criteria for end-of-weigh data are not met and the lab conditions cannot be brought back into control within the 10-hour weigh session limit, filters may be re-refrigerated, re-conditioned, and re-weighed later, if the total conditioning time does not exceed 72 hours for post-sampled filters and filter holding times are still met. Under certain circumstances, it may be necessary to qualify the filter data for failing lab temperature or humidity conditions (see Section 12.1 and Table 12-1).

7.2. Filter Inventory

All filters must be inventoried before use. Filters are normally received in allotments sufficient for at least one year. Each new shipment of PM_{2.5} filters are removed from the original shipping box and externally inspected to ensure the plastic boxes of filters are not damaged or unsealed. The Lot #, Box # and Filter ID ranges are organized in sequential order on a designated storage shelf in the anteroom. Packs of filters are removed from storage as needed, using the filters in sequence.

7.3. Filter Lot Stability Test

A filter lot stability test must be performed before a new lot of filters is used for routine sampling. A separate stability test must be performed for each new lot of filters received and is valid for the entire lot of filters. If a shipment includes multiple filter lots, then a stability test must still be performed for each lot in the shipment. For this reason, it is best to insist that filter shipments be filled from one lot number if possible. The procedure for this lot test is outlined below:

7.3.1. Obtain a PM_{2.5} Filter Lot Stability Test Form (See Figure 18-1).

7.3.2. Select 3 boxes of filters at random from the lot.

7.3.3. Record the lot #, filter box #'s, and lab analyst's initials.

7.3.4. Record the room temperature and humidity conditions on the form.

7.3.5. Select three filters from each of the three boxes (nine filters total for each lot) and inspect them for damage. Filters with defects should not be used for the lot stability test and should be set aside and later cancelled in the LIMS PM_{2.5} Manager (See Section 7.5.6). If the quantity of defective filters is greater than 15% per box, the lab supervisor should be notified.

- Filter rings must be intact on both sides and not doubled.
- Filter must not have holes as viewed against a good light source.
- Filter must not have discoloration or water marks.
- Filters must not have scratches or dents in the Teflon surface.
- Filter number must be present, legible, and unique.

7.3.6. Place the filters in individual Millipore Petri-slides. Record the filter ID number for each of the filters on the form. Weigh the filters as described in Section 8 and record the weights for Day 1 on the form.

7.3.7. Place the Petri-slides containing the filters on the conditioning shelves. Place the Petri-slides covers such that the lids cover $\frac{3}{4}$ of the filter surface. Allow the filters to condition in the controlled environment, then reweigh the filters every 24 hours until the weight difference between successive 24-hour measurements is less than 15 µg. The date for each measurement set is recorded at the top of the data set for Day 1, Day 2, etc. The weight difference between successive 24-hour measurements is calculated and recorded on the Lot Stability Test Form.

7.3.8. Calculate the lot conditioning period. The times required for each of the 9 filters to meet the weight difference criteria are calculated and

averaged. This average time, expressed in hours, is used as the minimum pre-conditioning time for all subsequent filters conditioned from the same lot. Twenty-four (24) hours is the minimum required conditioning period for pre-sampled filters. See Section 10.5 in the Method 2.12 guidance document for more information on lot stability tests.

7.4. Pre-Conditioning Filter Inspection

Each filter must be inspected for damage as it is initially removed from its box and placed into a Petri slide on the shelf for conditioning. It is also important to take notice of the filters every time they are handled in case some defect has been missed during the initial inspection or the filter has suffered subsequent damage.

7.4.1. A defective or damaged filter may include holes, creases, scratches in the surface, separation of the ring, faulty ID numbers (i.e. missing, duplicate, or obscured, etc.), foreign material, discoloration, non-uniformity, or any other defect that may render the filter unsuitable for sampling.

7.4.2. Label the slides with damaged filters as “TBC”, indicating “To Be Cancelled”. The defective filters are cancelled in the PM_{2.5} Manager, accompanied by a comment and recorded on the Pre-Sampling Filter Layout sheet (see Figure 18-4). Filters with minor defects may instead be used as lab blanks. The damaged filters should be archived after they are cancelled in LIMS (see Section 7.5.6.).

7.4.3. If the quantity of defective filters is greater than 15% per box, the lab supervisor should be notified.

7.4.4. Complete the Pre-Sampling Filter Layout Sheet to document filter lot number, filter ID number range, total number filters laid out, and LIMS exposure # (see Section 7.5.5). Also record the filter IDs of filters cancelled for damage and for unused filters from previous week’s layout.

7.5. Filter Pre-Conditioning

Each filter must be conditioned for the specified amount of time set by the Filter Lot Stability Test. The conditioning procedure for pre-sampled filters is outlined below. Only filters that pass inspection are conditioned.

7.5.1. Obtain Millipore Petri slides from a new container.

7.5.2. Place the Petri slides on the pre-conditioning shelf in rows of 5 filters. For a box of 50 filters there will be 10 rows of five on the shelf. Remove the lids from the Millipore Petri slides.

7.5.3. Filters are laid out in multiples of 50 or 100 for ease in tracking lab blanks from the pre-conditioning and weighing process through the post-conditioning and weighing process (see Section 8.5).

7.5.4. Using forceps labeled “Pre”, remove the filters one at a time from the original filter box and place the filters on the shelf in numerical order, 1 per Petri slide. Any damaged filters are initially placed into Petri slides on the pre-conditioning shelf but covered and marked “TBC” (indicating “To Be Cancelled”) as a place holder. Each filter position is maintained in sequence to ensure that filters with duplicate or ambiguous ID numbers will be identified, and to simplify layout.

7.5.5. The filters that pass inspection are then $\frac{3}{4}$ covered with the Petri slide lids and stay in this position until the end of the conditioning period, which is determined by the lot stability test.

- The filters laid out for conditioning must be entered into the LIMS PM_{2.5} Manager. Create “New Filters” by following the instructions below. Open the PM_{2.5} Manager in LIMS.
- Under Filters, select “New Filters”.
- In the “Start Serial Number”, type the ID of the first filter of the box that is to be added.
- In “Number to Add” type in the total number of filters laid out.
- Type in the Lot # and Exposure #. The Lot # is assigned in the Filter Lot Stability Test, while the Exposure # is assigned sequentially for each pre-conditioning period.
- Click “Add”.
- Click “OK” when finished.

7.5.6. At this time, any damaged filters marked “TBC” should be cancelled in the LIMS PM_{2.5} Manager with the comment “Damage observed during filter layout.” Filters leftover from the previous week’s layout should be cancelled with the comment “Filter unused.” Analyst initials and date should also be added to the comments in LIMS.

7.5.7. Start Pre-Conditioning in LIMS:

- Open the PM_{2.5} Manager in LIMS.
- Under Filters, select “Pre”.
- Select “Start Conditioning”.

- A window will appear listing filters available for pre-conditioning.
- Ensure the filter ID numbers correspond to the filter layout. Select “Add All”.
- Enter Analyst Name, Date, and Time (the time when filter layout was completed).
- Click “OK” when finished.

7.5.8. End Pre-Conditioning in LIMS: To end pre-conditioning, follow the instructions in Section 7.5.7 above, except select “End Conditioning” instead of “Start Conditioning”.

7.6. Filter Post-Conditioning

Once filters are received from the field, they must condition for a minimum of 24 hours, but should be weighed within 72 hours from start of conditioning. Under certain circumstances, post-filters set out for conditioning can be returned to cold storage (i.e., 1-4°C). Consult with lab supervisor. The reasons for returning filters to cold storage should be documented. Prior to weighing, filters must be re-conditioned for a minimum of 24 hours, but weighing should be completed after no more than 72 hours of total conditioning time, including the initial conditioning time before filters were returned to cold storage. The conditioning time starts when the layout is completed. The date and time are recorded on the Post-Sampling Filter Layout Sheet (Figure 18-5) and entered into LIMS (see Section 7.5.8). The instructions to start and end post-conditioning are identical to the pre-conditioning procedures provided in Sections 7.5.7. and 7.5.8. above, except there are separate tabs in the PM_{2.5} LIMS module for the pre- and post- workflow. Also, the filter layout shelf configuration for post-conditioning is in rows of 7 filters. Use forceps labelled “Post”.

NOTE: This is different from the layout configuration for pre-conditioning, which is in rows of five filters. The reason for this difference is the pre-filters are in sequential order and placing them in rows of 5 filters assists in filter management. However, the post-filters are not received sequentially and are laid out in the order returned. In the case of the post-filters, the conditioning layout is optimized for space.

8.0 **FILTER WEIGHING PROCEDURES AND CRITERIA**

8.1. Inspect the Weigh Table and Balance

The weigh table should be wiped with a dry Texwipe, if necessary. Avoid cleaning the balance table with Staticide or alcohol immediately prior to a weigh session, because evaporation will affect the balance performance. Check the balance level bubble position and the ground wire connection.

8.2. Perform Microbalance Automatic Calibration

Auto-calibrate the balance and wait for the balance reading to return to zero. See balance manual for auto-calibration instructions.

8.3. Manual Filter Weighing Procedure

NOTE: A batch of filters is defined as the filters weighed on one weigh sheet. Filters are weighed in sets of up to 100 filters per weigh sheet, including 1 or more lab blanks, 1 batch duplicate, and 300 mg and 500 mg mass reference standards check weights after every 10 filters. Excel worksheets are stored as Excel files and text tab delimited files on the PM_{2.5} network drive. The text tab delimited files are used to upload data into the LIMS.

8.3.1. Creating a Weigh Sheet

- Open the WinWedge Balance Command software program **first**. Opening the WinWedge program **after** the weigh sheet is created will not enable data transfer correctly.
- Open the Excel template for data collection (see Figure 18-7) and save as a new weigh sheet in the appropriate network folder with the filename format MMDDYYYY-Balance ID-X, where balance ID is either AXP6 or BMT5, and X is “PRE” or “POST”. The filename may also include the letter A, B, etc. indicating the 1st, 2nd, etc. sheet of the day, or other notation indicating a corrected weigh sheet or a weight confirmation.
- Enable the Excel worksheet macro functions by clicking the “Enable Content” button in the tool bar, to allow correct data transfer and timestamps from the balance. Do not “Enable Content” when opening a completed weigh sheet for validation or review.
- Complete the weigh sheet information - only cells highlighted in yellow can be modified.
 - PRE- or POST - The pre- and post- filters are not weighed together in a given weigh session.
 - Analyst Name

- Vaisala Unit Serial Number (SN)
- Conditioning Start and End Date/Time (see Section 7.0).
- 24-Hour Filter Conditioning Values taken from the Vaisala data (see Section 7.1):
 - Humidity Average, Minimum, Maximum, and Standard Deviation.
 - Temperature Average, Minimum, Maximum, and Standard Deviation.
- Balance ID - after entering ID, the balance SN and Working Weights values and SNs will automatically update. Verify the standards in use are documented correctly. Document the balance auto-calibration on the weigh sheet (see Section 8.2.).
- Enter the Filter ID numbers. It may be convenient to add filter IDs to the weigh sheet in sets of ten as the weigh session progresses. Never ‘cut-and-paste’ or ‘click-and-drag’ to add or to auto-fill the filter IDs.
- Be sure to enable macros (click “Enable Content” button near the top of the weigh sheet before starting the weigh session) so that the mass data transferred from the balance is time-stamped correctly. Filter and QC data must be time-stamped in chronological sequence. Reweighs or confirmations may be documented on a separate weigh sheet. If a filter or working standard must be reweighed for correction on the original sheet, any filter or QC check time-stamped after the correction must be reweighed as well, to maintain the chronological sequence of weight data. Weigh sheets will have, at minimum, starting and ending QC checks, a QC check bracketing each set of up to ten sample filters, up to ten lab blanks, and a batch duplicate filter (see Sections 8.4 and 8.5).
- Currently the DEAR Scientific Services Support (SSS) staff have password access to the Excel weigh sheet template. Contact that program administrator or designee for assistance with any functional issues with the weigh sheet macros, QC rules, formatting, or file upload into the LIMS.

8.3.2. QC Check Weights

- Expose the 300 mg working standard between the U-shaped electrode or between a pair of Polonium-210 strips installed in a gooseneck assembly for a minimum of 30 seconds to discharge any static on the standard. Open the draft shield, place the standard on the balance pan, and close the draft shield. A stable reading has been obtained once the indicating circle in the top left corner of the balance disappears – see balance manual for details. Press the Print key once a stable reading is achieved. The WinWedge Balance Command software will automatically import the mass data and time-stamp the weigh sheet. Verify that the mass value is recorded in the correct cell of the Excel weigh sheet and that the time-stamp is correct. Remove the standard and close the draft shield on the microbalance.
- Repeat the process above for the 500 mg working standard.
- The weight of each of the working standards must not exceed ± 3 μg of the certified (i.e., conventional mass) value. The weigh sheet formatting will indicate pass (“Accept” in green) or fail (“Reject” in red) based on this limit. If failing, working standards may be re-weighed at analyst’s discretion until a passing weight is obtained. Troubleshooting or corrective action may be required; document appropriately. If 3-5 attempts are required to achieve a passing weight check, it may be more efficient to close the weigh sheet, obtain an alternate mass standard, and start a new weigh sheet. Be sure the alternate standard chosen has a current certificate or verification and document its SN and conventional mass value on the new weigh sheet. Consult with the lab supervisor as needed.
- Open the first Millipore Petri slide and remove the filter using the appropriate forceps. Discharge the static on the filter as described for the working standards, above.
- While the filter is being discharged, observe that the balance returns to zero. Manually tare the balance to zero if necessary; however, if the balance consistently fails to return to zero or takes too long to stabilize, troubleshoot to determine and correct the cause of the drift.
- Once the balance indicates that a stable zero reading has been obtained, open the draft shield, place the filter on the microbalance pan, and close the draft shield.

- Wait until the balance indicates that a stable reading has been obtained.
- Press the Print key. Observe that the filter weight and timestamp has been recorded in the appropriate cells on the weigh sheet.
- Open the draft shield, remove the filter with forceps, and place it back into the Millipore Petri slide with the lid covering it completely.

8.4. Batch Duplicate Filter Sample (QC Check Parameter)

The first sample filter on each weigh sheet is designated as the batch duplicate filter. This filter is the first and last sample filter weighed in each batch. The first and second weights of the batch duplicate filter must be within $\pm 15\mu\text{g}$. If the criteria are not met, then the entire batch must be reweighed.

8.5. Lab Blank Filter Sample (QC Check Parameter)

8.5.1. Each box of 50 filters laid out for conditioning must have at least five filters assigned as lab blanks (at least 10% of total pre-sampled filters weighed) to provide enough lab blanks to be pre-weighed with a specific batch of filters, and then post-weighed with the same batch or subset of that batch of filters. The lab blank filters are normally the first five filters in each box (filters ending in “01”, “02”, “03”, “04”, “05”, or “51”, “52”, “53”, “54”, “55”). If one or more of these filters is too damaged to be used as a lab blank, then the next acceptable filter is assigned as a lab blank for that filter box.

8.5.2. The lab blanks are treated as routine filters. The pre- and post-weight for the lab blanks are documented on the weighing sheets along with the sample filters. The acceptance criterion for lab blanks is $\pm 15\mu\text{g}$ between weights. If the lab blank weight change exceeds $\pm 15\mu\text{g}$, implement appropriate troubleshooting and corrective actions. Consult with the lab supervisor as needed.

8.5.3. Once the weight of the Lab Blank is determined in the pre-weigh session, the filter is placed in a Millipore Petri slide and covered. The top of the Millipore Petri slide is labeled with the “LB” (to denote that it is a lab blank), the filter ID, and the weight of the filter. Then the filter is placed on the lab blank shelf until conditioned for use in a subsequent post-weigh session. Lab blanks are documented on the post-filter layout sheet and later archived in sequence with sampled filters.

8.5.4. Lab blanks are intended as single-use filters; however, they may need to be weighed more than once under certain circumstances, such as

insufficient number of lab blanks available for all post-filter batch subsets, lab blank failure corrective actions, or sample weight confirmations. Consult with the lab supervisor as needed.

8.6. Upload Filter Weight Data into LIMS

Follow the outline below to upload filter weights into the LIMS:

- Weigh sheets must be cross-verified and initialed by an independent lab analyst (an analyst that did not weigh the filters on that sheet). Cross-verification includes careful review of all weigh sheet header information, including weigh session ID, Balance ID and auto-calibration confirmation, Vaisala unit S/N, Conditioning start and end date/time, lab temperature and humidity data, as well as filter weigh data and chronological time sequence. Filter weigh data and timestamps must pass all QC checks built into the weigh sheet and must also be reasonable (filter IDs and weights within reasonable range; timestamps at least 30 seconds apart). If any corrections are needed, save the weigh sheet as “{file name} Corrected” and indicate what correction was made in the batch sheet comments section on the corrected sheet. Save the final verified and initialed Excel file as a text tab delimited file with the same filename in the appropriate network folder.

NOTE: *Analysts must exercise caution when creating, cross-verifying, and saving weigh sheets. Inadvertent keystrokes can affect timestamps or automated QC functions within the weigh sheets. Back-up copies of the weigh sheets are created and saved automatically when the sheet macros are enabled and may be used to check questionable data in the original files.*

- Open the PM_{2.5} Manager in LIMS.
- Under Filters, select “Pre” or “Post” and then select “Weigh”.
- Under File, select “Open”.
- From the file manager, select the text tab delimited file that contains the data to be uploaded.
- In LIMS, highlight filters and the associated Lab Blanks and Batch Duplicate.
- Under File, select “Assign Batch ID” then select “New” to assign the next available number; in certain circumstances, a previously assigned batch ID number may need to be entered. Then select “Upload”. Verify correct upload in the LIMS Review/Authorize screen.

9.0 FILTER SHIPPING PROCEDURES AND DOCUMENTATION

The filters must be packaged in such a way to maintain chain-of-custody, protect them from damage or exposure, and control post-sampling shipment temperature. To achieve this, the filters are placed in cassettes, sequentially stacked in magazines, and packaged in soft-side coolers within the large hard-sided coolers, along with sufficient blue ice (room temperature for pre-filter shipments, frozen for post-filter shipments).

9.1. Filter Scheduling in LIMS

- Requests for filters are received from clients and are scheduled in LIMS. Typically, each client will submit an annual filter request in advance.
- Filter shipments are packed according to the requests received, generally during the week prior to cooler shipping.

9.2. LIMS Program Instructions for Filter Packing

- Open the PM_{2.5} Manager in LIMS.
- Under Filter, select “Send”.
- Select the Schedule for a Customer.
- Under Magazines, highlight one row and double-click the appropriate magazine. That magazine ID will appear in the Magazine box. The number to the right will be the number of filters to be packaged in that magazine.
- With the Magazine ID highlighted, go to the list of available filters and select one (or the control key may be used to select multiple filters). This procedure is done until all the filters for that magazine have been selected. If another magazine is needed to fill the request, repeat magazine and filter selection steps until the request is complete.
- Once a request is complete, select the Analyst name at the top of the screen and select the Finished button.
- If the request is not filled in properly, the Finish button will be gray and not permit the user to complete the task.

9.3. Cassette Loading Instructions

A filter cassette is composed of a cassette bottom, cassette metal screen, and a cassette top.

- Place the cassette bottom on the workbench.
- Place the cassette screen, with the screen ID down, in the cassette bottom.

- Remove the filter from a Millipore Petri-Slide with the forceps labeled “Pre”.
- Center the filter on the cassette screen.
- Finally, place the cassette top on the filter. Press firmly to ensure the cassette top and bottom make a tight seal.

9.4. Magazine Loading Instructions

- Cassettes are packed in numbered magazines according to the LIMS filter request completed in Section 9.2. above.
- Obtain a manual hand pump and attach it to the pneumatic port located at the base of the magazine. Pump up the magazine so that the piston is level with the top of the magazine.
- Insert the cassettes in the magazine with the filter media exposed upward and the cassette screen facing down. Filters are given a final inspection and loaded into the magazine one at a time, starting with the highest filter ID # loaded first. To load the cassettes into the magazine, place a cassette on the magazine piston and push gently down until the top of the cassette is flush with the top of the magazine. Place the next cassette on top of the first and repeat the process until the magazine is loaded. This technique will ensure that filter cassettes are not placed into the magazine at an angle where damage to filters above or below may occur. The filters will end up stacked in the magazine in ascending filter ID# order, with the lowest filter ID# at the top and the highest filter ID# at the bottom of the magazine.

NOTE: *The filter order in the magazines must agree with the order on the Chain-of-Custody Log (see Figure 18-6) and each of the magazine and filter ID numbers must be recorded accurately.*

- Place the orange cap on the magazine after all the cassettes are loaded in the magazine.
- Place a protective cap on the magazine pneumatic port.
- Pack all magazines required for the client request in a labelled, clean, dry soft-side cooler. Store the soft-side coolers in the anteroom until ready for shipment.

9.5. Shipping Documentation

- FedEx labels will be generated by lab staff and placed on coolers. Copies of the shipping labels will be saved in PDF format will be archived electronically as well as provided to agencies receiving the shipments.

- In the PM_{2.5} Manager → Filters → Print → Custody Sheet, generate a PDF Chain-of-Custody Log (see Figure 18-6) for each customer, time-stamped according to the date and time the coolers are placed on receiving dock for pickup by FedEx.
- The lab analyst must electronically sign the Chain-of-Custody Log, send to the customer via email, and save a copy to the appropriate file folder on the network. Customers will return a signed copy of the custody log via email, which is also saved in this folder.

9.6. Cooler Packing for Shipment

- Move the packed soft-side coolers from the anteroom to the hallway.
- Obtain a large hard-sided cooler labelled with the name of the district or local agency customer receiving the shipment. The cooler should be clean and dry.
- Place large and/or small blue ice packs (room temperature for outgoing pre-filter shipments) in the soft-side cooler to secure the magazines.
- Place the soft-side cooler in the hard-sided cooler. Center the soft-side cooler in the larger hard-sided cooler, then place blue ice packs on both sides to secure the soft-side cooler. (These blue ice packs are provided to the customers to freeze at their facility for adequate cooling of the return shipment of sampled filters.). Verify the soft-sided cooler label matches the hard-sided cooler label.
- Close the hard-sided cooler and secure the lid all the way around the cooler and cover latch or latches if present with tape to protect in shipping. Fiber reinforced packing tape is preferred, but clear packing tape can be used.
- Attach the FedEx shipping label to the cooler and tape securely with clear packing tape. Verify the shipping label matches the cooler label.

9.7. Cooler Shipping

Coolers are routinely shipped by commercial carrier, currently FedEx. Procedures for using the FedEx carrier shipping service are provided in the DARM SOP DEP 03-14.

10.0 FILTER RECEIVING PROCEDURES AND NON-CONFORMANCE REPORTING

10.1. Cooler Inspection

10.1.1. As soon as possible after a cooler is received from the field, it must be inspected to make sure it is sealed properly. Then, the cooler should be opened to inspect the contents and measure the magazine temperature. Make note of any damage or irregularities on the Cooler Receiving Log (Figure 18-2).

10.1.2. Measure the received temperature on the surface of a magazine inside the soft-side cooler using an IR gun and record the temperature on the Cooler Receiving Log. The IR Gun serial number and certificate date are also documented on this log.

10.1.3. If a cooler is received with magazine temperatures above 4.0 °C, the qualifier “ISP” (Inadequate Sample Preservation) and a comment including the filter magazine receipt temperature will be added to the associated filter IDs in the LIMS system, and in the final report to the customer.

10.1.4. Received temperatures greater than 25 °C will result in data qualification with the qualifier “FSP” (Failing Sample Preservation) and a comment including the filter magazine receipt temperature in the LIMS system, and in the final report to the customer.

10.1.5. PM_{2.5} Filter Holding Time Determination - Per 40 CFR Part 50, Appendix L, Section 8.3.6, a 10-day holding time (i.e., time between end of sampling and final weighing) is applicable for samples collected at average ambient temperatures > 4 °C; however, a 30-day holding time is allowable if samples are maintained and received at < 4 °C. Since the lab does not have access to the average ambient sampling temperature data, DARM or local agency designees will determine the allowed holding time for all filters based on the lab’s reported received temperature and weigh date, and will apply appropriate null or qualifier codes to the data. Filter data may be qualified or void if the average ambient sampling temperature is lower than the received temperature depending on the holding time requirement, or if the filters are received above 25 °C, as specified in 40 CFR Part 50, Appendix L, Section 8.3.6. Refer to DARM’s Statewide Data Handling and Data Validation SOP (DEP 18-27) for additional details. The lab will always weigh the filters as soon as practical after receipt and conditioning. See Figure 12-1 for a flowchart that can be used to help determine filter holding times.

10.1.6. The signed Cooler Receiving Log, with cooler receipt temperatures and any irregularities noted, is added to the weekly folder with the printed Field Filter Logs (see Figure 18-3).

10.1.7. Remove and inspect the magazines from the cooler. Pre-printed dividers are used to identify the magazines from the different agencies.

10.1.8. Move the sorted magazines from each cooler on a cart into the anteroom. This anteroom is conditioned the same as the weighing room and allows the magazines to come to equilibrium in a controlled environment before unpacking the filters. Typically, the coolers are received in the morning and the filters are unpacked from the magazines that afternoon.

10.2. Non-Conformance Reporting

- Irregularities or damage are recorded on the Cooler Receiving Log, the Filter Layout Sheet, and in the LIMS Receiving module, as appropriate. It is important to note abnormalities associated with shipments (i.e. broken or damaged magazines, missing items, improper packing or paperwork).
- These records will be used to add appropriate qualifiers and comments when reporting the data in LIMS and to the customers.

10.3. Post Sampling Filter Layout

- Move the magazines into the conditioned weigh room.
- Select the first set of customer magazines to receive into the lab and remove the orange cap from one of the magazines.
- Use the manual hand pump to pressurize the magazine piston and remove filters one-by-one. Place the filters on the receiving desk left to right in the same sequence/order they are removed.
- Gently open the cassette to expose the filter. Visually inspect the filter for damage. Note any filter damage on the Post-Filter Layout Sheet (see Figure 18-5). Recommended LIMS codes and comments for damage, voided filters, or holding times are provided in Table 10-1. These codes and comments will be added to the filter record in LIMS and will qualify the data in the final reports (see Section 14.2.). Very minor damage such as small dents or a very small number of visible particles that will not affect the weight of the filter may be noted on the layout sheet with the comment “NC” (“no code”). This description of minor damage may be added to the filter record in LIMS but will not qualify the data in the final reports.

Table 10-1. Recommended Data Qualifiers and Comments for LIMS

Data Qualifier	Code	Recommended Comments
Sample Integrity Suspect	SIS	Filter damage observed upon receipt (ex: scuff, pinhole). Foreign object on filter surface (ex: particle, spot).
Sample Integrity Compromised	SIC	Destructive filter damage observed upon receipt (ex: hole, tear).
Lab Accident	LAC	Laboratory accident or damage.
Holding Time Exceedance	HTE	Post-weighed more than 30 days after sampling. Holding time in field exceeded 177 hours. Sampled more than 30 days after pre-weight.
Voided Filter	VOD	Filter voided by customer.
Lab Temperature criteria exceeded	FLT	Lab Temperature criteria exceeded.
Lab Humidity criteria exceeded	FLH	Lab Humidity criteria exceeded.
Improper Sample Preservation	ISP	Filter Magazine received above 4 °C.
Failing Sample Preservation	FSP	Filter Magazine received above 25 °C.

- Using the post filter layout sheet, list the customer name, the sequence/order # of the filter, the filter ID, the magazine ID, and the date of shipment receipt.
- Remove the filter with the appropriate forceps taking extra precautions not to disturb the filter media and the particulate matter on the filter.
- Place the filter, sampled side facing up, in a Millipore Petri-slide.
- The filter ID should be visible through the top of the Millipore Petri-slide.
- Place the filter on the post-conditioning shelf with the slide cover $\frac{3}{4}$ over the filter.

NOTE: *The order of the filters on the layout sheet documents the received order of the filters and the order of the filters on the shelves.*

- Repeat the above steps until all filters are removed from the magazine.
- After all filters have been removed from all magazines received and the layout is complete for the week, record the date and time as start of conditioning (see Section 7.6.)

10.4. Receive Shipments into the LIMS

- Open the PM_{2.5} Manager in LIMS.
- Under Filter, select “Receive”.
- Select a Customer and then choose “Next”.
- Select “Load”.
- Select the appropriate customer’s filter log text file. (Filter logs are emailed to the lab in excel format from the customer and saved in text/tab delimited format on the server). See Figure 18-3 for an example Field Filter Log.
- Select the date on which the filters arrived into the lab.
- Select “Upload” and the filter log data will be transferred to LIMS.
- Check the list to ensure the correct filter ID’s and magazines were selected. Review comments from the filter log that automatically upload with the filter data. Filters with zero run time should be identified as field blanks. Confirm any the (Holding Time Exceedance) qualifiers added to the data; ask the customer to confirm sample start, stop, or pick-up times and dates as needed. Add comments from the cooler receiving log or layout sheet.

NOTE: Customers may indicate that a filter will be void or cancelled on the Filter Log. See Section 18.2 for additional information on void and cancel filter status and how to handle these filters in the lab workflow.

- Select “Analyst ID”.
- Add the cooler temperature as recorded on the Cooler Receiving Log.
- Select “Finish” to upload data to LIMS.

NOTE: If customers cannot provide a complete and correct filter log in a timely manner, the lab may refrigerate their received filter magazines, and postpone layout, conditioning, and post-weighing until the next weekly post-weigh session. Customers will be notified in advance and filter storage will be documented.

11.0 MASS STANDARD AND BALANCE CERTIFICATIONS/VERIFICATIONS

11.1. Annual NIST Traceable Certifications

NIST traceable certifications are required for the primary mass standards and the temperature/humidity sensor units.

- Primary Standard Annual Certifications – purchased new each year or recertified by Troemner, Rice Lake Weighing Systems, or an equivalent vendor.
- Vaisala Humidity and Temperature Sensor Units – certified annually by Vaisala Inc.
- Certification documents are scanned and archived electronically on the network server and in hard-copy in the PM_{2.5} Lab.

11.2. Quarterly Verification of Working Standards

Working standards must be verified against the primary standards on a quarterly basis using the following procedures. See Section 9.7.2 in the Method 2.12 guidance for more information and an explanation of the double substitution calculation.

11.2.1. Use the Quarterly Working Standard Verification Form (see Figure 18-8).

11.2.2. Inspect each working standard for cleanliness and wear. The working standards may be cleaned by rinsing with alcohol and air dried on a Texwipe, but it may take at least an hour to obtain a stable weight after this cleaning.

11.2.3. Fill out the form with the Balance ID, Analyst Name, Date, and Time. Record the Vaisala unit SN and 24-hour Humidity and Temperature data and save the Vaisala data as described in Section 7.1. Look up the previous quarterly verification form (or certificate if the working standard has not been verified previously) to obtain the Initial Mass Correction (C_w) for each working standard. Note the certificate or Test ID from this form.

11.2.4. Weigh the 300 mg standards following the order listed on the form (working, primary, primary, working). Then repeat with the 500 mg standards. The WinWedge Balance Command software can transfer the mass data from the balance into the selected cells on the form.

11.2.5. Calculate the Working Standard Mass Correction (C_w) values for each working standard.

11.2.6. The acceptance criteria for the quarterly verification of the working standard C_w is $\pm 2 \mu\text{g}$ compared to the previously determined (initial) C_w . If the verification fails, a second trial the following day is performed. Cleaning or troubleshooting must be documented. If the acceptance criteria are not met in two consecutive trials, then the working standard must be removed from service and labeled “Do not use” or discarded. Notify the lab supervisor of verification failures.

NOTE: This calculated mass correction is not used to generate a “new mass” for the working standard; it is intended only as a verification (check) of the standards. The conventional mass value given by the standard’s original certification is always used as the reference value for routine QC checks. See Section 9.7.1 in the Method 2.12 guidance for more information on conventional mass values.

11.2.7. Submit the working standard verification to the lab supervisor. Maintain records in the PM_{2.5} certificates folder on the network.

11.2.8. Upon acceptable verification, the working standards may be used for routine weigh sessions.

11.3. Annual Microbalance Maintenance Service and Calibration

- A Mettler-Toledo technician will complete this service annually or more often if required.
- Certification documents and service records are stored on the network server and in hard copy in the PM_{2.5} Lab.

12.0 QUALITY CONTROL

The quality control procedures discussed below are described in detail within the text of this document but are summarized here. Table 12-1 provides qualifiers and comments for failed quality control. A summary of all automated weigh sheet and LIMS checks is provided in Table 18-1.

12.1. Conditioning Environment

The average, minimum, maximum, and standard deviation of the temperature and relative humidity values are recorded with each weigh session. The conditioning room 24-hour average temperature must be 20.0-23.0 °C and the Relative Humidity (RH) in the range of 30.0-40.0%. The following temperature and humidity checks are also performed by the LIMS. Permanent digital records of the temperature and humidity are maintained.

- Verify and record the preceding 24-hour average temperature (20.0-23.0 °C) with the standard deviation not to exceed 2.0 °C.
- Verify and record the preceding 24-hour average Relative Humidity (30.0-40.0% RH) with the standard deviation not to exceed 5.0% RH.
- Verify that the difference in the pre- and post- relative humidity does not exceed $\pm 5.0\%$.
- Refer to Table 12-1 for automated LIMS flagging of temperature/humidity control failures.

12.2. Filter Holding Times

- Filters must be sampled within 30 days of pre-weighing.
- Filters must be collected from the sampler within 177 hours of sampling.
- Filters received into the lab at temperatures below the average ambient sampling temperature must be post-weighed within 30 days of sampling. Agencies must compare relevant temperatures and dates and verify the holding time requirement.
- Filters received into the lab at temperatures above the average ambient sampling temperature must be post-weighed within 10 days of sampling. Filters received at temperatures higher than 4 °C will be reported to the agency with an “ISP” (Inadequate Sample Preservation) qualifier. Agencies must compare relevant temperatures and dates and verify the holding time requirement.

- Filters received into the lab at temperatures above 25 °C do not meet method criteria and are invalid; however, they will be weighed and reported to the agency with an “FSP” (Failing Sample Preservation) qualifier.
- Refer to Table 12-1 for automated LIMS flagging of holding time exceedances.
- Refer to Figure 12-1 for filter sample holding time decision flowchart. This figure is taken directly from the Method 2.12 version identified in the reference section of this SOP. This flowchart may be used to assist the laboratory analyst in determining post-weighing time frames. In most cases, all filters arriving at the laboratory should be post-weighed. While the laboratory analyst may have determined using the flow chart or visual inspection that a filter will be invalid, the monitoring organization’s QA Coordinator (or decision maker) should make the final determination of validity. However, it is the laboratory analyst’s responsibility to clearly flag the filter so that the QA Coordinator can make an accurate assessment of validity.

Figure 12-1. Filter Sample Holding Time Decision Flowchart

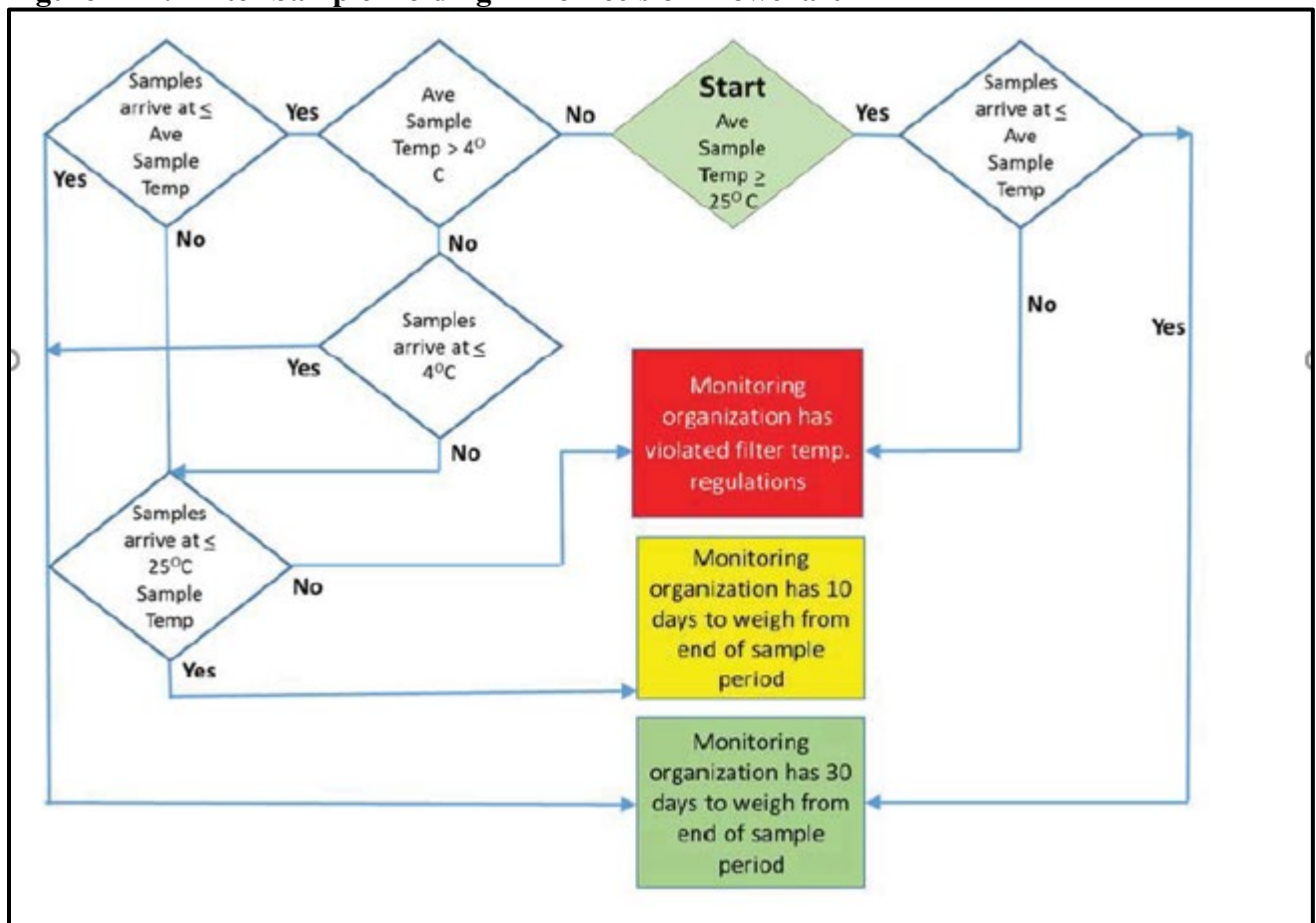


Table 12-1. Automated LIMS Flags and Comments for Holding Time and Temperature/Humidity Control

Specification	Qualifier	Failure Comment
Filter holding time from pre-weight ≤ 30 days	HTE	Sampled more than 30 days after pre-weighing.
Filter holding time from sampling ≤ 30 days	HTE	Weighed more than 30 days after sampling.
Filter collection ≤ 177 hours after sampling	HTE	Filter collected more than 177 hours after date sampled.
Lab conditioning average temperature 20.0-23.0 °C	FLT	Lab average temperature criterion exceeded.
Lab temperature control 24-hour standard deviation ≤ 2.0 °C	FLT	Lab temperature 24-hour standard deviation criterion exceeded.
Lab conditioning humidity average 30.0-40.0% RH	FLH	Lab average humidity criterion exceeded.
Lab conditioning humidity 24-hour standard deviation $\leq 5.0\%$	FLH	Lab humidity 24-hour standard deviation criterion exceeded.
Pre-/post- sample conditioning humidity difference does not exceed $\pm 5.0\%$ RH	FLH	Pre-/post- sample humidity average difference of $\pm 5.0\%$ RH exceeded.
Received temperature 4.1 - 25 °C	ISP	Received at [X] degrees C. Verify average ambient sampling temperature and holding time.
Received temperature ≥ 25.1 °C	FSP	Filter magazine received above 25 °C.

12.3. Working Standard QC checks

Mass reference standards of 300 mg and 500 mg are weighed at the beginning and end of each weigh sheet and after every 10th filter on a weigh sheet and must not exceed ± 3 μ g compared to their conventional mass values.

12.4. Batch Duplicate Filter Weighing

A duplicate filter is weighed with each batch of filters. The first filter on any weigh sheet is denoted the Batch Duplicate (BD). The difference of the duplicate filter weights must not exceed $\pm 15\mu\text{g}$.

12.5. Lab Filter Blanks

Lab blanks are weighed with every batch of filters and must meet the delta criteria of $\pm 15\mu\text{g}$, compared to the original mass measurement made when the blank filter was first weighed.

NOTE: *The lab does not track or validate field blanks (FB). However, FB results are verified when the delta exceeds $30\mu\text{g}$ if filters are identified as field blanks on filter logs. If the information is available on the filter logs, customers are notified for FB deltas exceeding the $30\mu\text{g}$ specification.*

13.0 FILTER STORAGE AND RECORDS MAINTENANCE/ARCHIVAL

13.1. Filter Storage and Archival

- Upon completion of filter sampling, post-weighing and reporting, the filters are stored in Millipore Petri-slides.
- Filters are stored in numerical order in Petri-slide trays.
- When all the filters have been post-weighed from a box of 50 filters, the associated lab blanks must be placed in the Petri-slide tray, along with any filters in the sequence that were cancelled. Label the series of filters contained in each tray, seal the tray in a clear plastic bag, and store in the lab refrigerator at 1-4 °C.
- Archived filters will be stored for five years. The filters are stored in refrigeration for the first year and then at room temperature for four additional years, free from excessive heat, humidity, and contamination.
- Archived filters may be made available upon request to federal, state, or research agencies for supplemental analysis.

13.2. Data Records Maintenance and Archival

- Store all paper and electronic records for a minimum of ten years.
- The PM_{2.5} electronic documentation is stored in MS Excel, MS Word, PDF, and/or text tab delimited files, organized by year, month, and/or agency.
- The Excel spreadsheets containing the pre- and post-weight data are saved as text tab delimited files. The text tab delimited file is used to upload the data into LIMS. Excel spreadsheets for both pre- and post- weights are electronically stored in the PM_{2.5} directory on a network server.
- The paper records are archived for the most recent years in the PM_{2.5} filing cabinets. Older documents are archived according to the lab's data archival procedures.

13.3. Logbook and Other Supporting Document Storage

- Purchasing and Service Records, Equipment Manuals – When hardcopies of invoices, maintenance reports, and manuals are provided, they will be kept in the PM_{2.5} lab filing cabinet.
- Shipping & Receiving Documentation – Shipping and Receiving documents are electronically stored in the PM_{2.5} directory on a network server.

- Lab Logbook – The PM_{2.5} lab maintains electronic files and/or a hardcopy notebook for recording issues regarding the lab equipment maintenance, lab condition controls and maintenance, cleaning activities, and other PM_{2.5} lab activities.

14.0 DATA AUTHORIZATION AND REPORTING

The procedures provided in this section are an overview of the data review process, responsible parties, and final report authorization necessary for reporting results from the PM_{2.5} lab.

14.1. Data Review and Authorization

The primary responsible parties for reviewing the PM_{2.5} lab data are the Lab Analysts and the Lab Supervisor. They have the responsibility to review and verify all data and documents relevant to PM_{2.5} filter preparation, shipping/receiving, weighing, and reporting.

14.1.1. Open the PM_{2.5} Manager module in the LIMS.

14.1.2. Under Filters, select “Review/Authorize”.

14.1.3. Select “09-Weight-2-Complete”. Filters in this category are ready for data review. In this portion of the PM_{2.5} LIMS, one can view data associated with any filter by searching for a filter ID, selecting a specific filter status in LIMS, and sorting by the assigned sampling agency. Corrections or comments to data may be added in this LIMS module.

14.1.4. The following documents must be included in the PM_{2.5} data review:

- Weigh Sheets and Vaisala data - Must be cross-verified by two lab analysts. Corrections and weight confirmations must be documented.
- Filter Logs - Review for accuracy, completeness, consistency, and comments. Corrections must be documented.
- Cooler Receiving Log and Filter Layout Sheets - Must be consistent with the Filter Log and LIMS records. Cooler Receiving Log must be signed by lab analyst(s) receiving and unpacking coolers in the appropriate fields.

Layout sheets must be cross verified by two lab analysts.

- Custody Logs - Must be signed by both lab and field staff.

14.1.5. LIMS records general filter information such as status, report SN, schedule request ID, filter lot #, exposure #, calculated delta, shipping data, receiving data, conditioning and weigh session data and results, filter comments and data qualifiers, batch IDs, and weigh sheet QC results.

14.1.6. Review the uploaded results in LIMS and reweigh filters with delta values less than negative 10 μg or more than 500 μg , for confirmation. Also, reweigh any identified field blank filters where the delta exceeds 30 μg . If the filters can be reweighed within the allowed 10-hour window for the weigh session and temperature and humidity criteria are acceptable, a new weigh sheet and additional QC checks are not required; the weight confirmation can be documented on a Post Weight Confirmation form. If upon re-weighing, the first weight differs more than $\pm 15 \mu\text{g}$ compared to the second weight, a new weigh sheet must be created with all required weigh session information and QC data. The filter should be weighed twice on this confirmation weigh sheet, once as the first filter on the sheet and once as the batch duplicate. If confirmed in the duplicate weighing on this sheet, this data should be uploaded to LIMS with a unique batch ID (if not confirmed, alert the lab supervisor). Add a comment to the LIMS data indicating that the results were confirmed.

14.1.7. LIMS is designed to automatically add some qualifiers and comments (see Table 12-1), others must be entered manually (see Table 10-1). All qualifiers and comments should be verified during data review.

14.1.8. When the data has been thoroughly reviewed, highlight the filters to be authorized.

14.1.9. Under Action, select Authorize. A window will appear listing all the filters selected.

14.1.10. Highlight the filters and select Authorize Selected.

14.2. PM_{2.5} Analysis Reports

The primary responsible party for generating the final PM_{2.5} reports is the Supervisor (or designee) using the following procedures.

- Open the PM_{2.5} Manager module in the LIMS.
- Under “Utilities”, select “Build Final Reports”.
- Select “New Reports”.
- Choose a “Customer”. All filters ready for reporting will appear in the Available Filters column.
- Choose the filters that are to be reported by highlighting each one.

- Select “Add”.
- At the top are options to “Preview” the report or to “Certify Filter Data”. Filter data, qualifiers, and comments can be reviewed in the Preview option. Choose the appropriate option. Once a filter has been reported on a PM_{2.5} Analysis Report, it is assigned a Report Number. The filter will be assigned to that Report Number unless it is unauthorized and reported again.
- The PM_{2.5} Laboratory Analysis Report (Figure 18-10) includes lab and recipient contact information, data qualifier codes and definitions, received temperature of the filters, magazine ID, filter ID, pre- and post-weight results and dates, calculated delta, and any qualifiers and comments applied to the data.
- The final PM_{2.5} report will automatically print at the lab’s designated printer. The paper report is filed with the weekly lab folder. The LIMS system also automatically generates an email to the customer with a link to the File Transfer Protocol (FTP) site where the custody records and lab reports are accessible.
- The lab has a paper-free policy for reporting data to our clients. Upon authorization, an email with a link to the electronic copy of the final report is automatically sent to each client.
- Reporting the filter data also makes some of the lab data available in the DARM database PMTS for calculation of PM_{2.5} concentration and QA review. See the DARM SOP DEP 18-27 for more details.

14.3. Turn-Around Time

The lab’s expected turn-around time (time from receipt of sampled filters to reporting) for PM_{2.5} data is 7 to 12 days. Recipients will be notified of delays.

15.0 HAZARDOUS WASTE & SAFETY PROCEDURES

To prevent personal injury or damage to lab instruments, employees must heed all warnings associated with operation of equipment and handling of chemicals.

- All chemicals and hazardous materials used in the lab must be accompanied by material safety data sheets (MSDS). This documentation must be readily available to all lab personnel.
- Use care when using solvents such as alcohol. Use gloves when recommended and wash hands thoroughly after use.
- Use safe lifting techniques when moving coolers and supplies.

16.0 METHOD VALIDATION AND PERSONNEL QUALIFICATIONS

The lab must perform a formal validation of the PM_{2.5} gravimetric method prior to generating reportable data. Also, all lab personnel using the method must be trained on the lab procedures and successfully demonstrate their ability to perform the analysis in an Initial Demonstration of Capability (IDOC) and Annual Evaluations thereafter. Shadowing, hands-on practice, and/or supervised demonstration of lab activities is recommended for any personnel responsible for PM_{2.5} lab duties or data review and reporting activities.

16.1. Analytical Method Validation

A method validation exercise must initially be performed on every instrument used to collect data and must be repeated any time there is a major change to the analytical method or instrumentation. The validation procedure is the repeated measurement of standard weights, demonstrating that the instrumentation and personnel can meet all the method criteria. Method validation documentation is archived in the network folder.

16.2. Personnel Qualifications

All personnel who generate reportable data must first successfully perform an IDOC and then an Annual Proficiency Evaluation thereafter. It is imperative that the staff be part of a continuing education and training program to ensure that their skills and knowledge stay up-to-date. As indicated, some of the training items listed below are required, while some are recommended. Training documentation is archived in the network folder.

16.2.1. Method IDOC (required for analysts)

Demonstrate the ability to operate the microbalances and anti-static devices. This involves taking multiple measurements of standard weights and filters. The acceptance criteria for the measurements are established by the method (see Quality Control requirements in Section 12). The IDOC also includes demonstration of the ability to handle sample filters within the lab workflow, to perform calibration and verification procedures, to assess and document lab activities and environmental conditions, and to maintain a clean working environment.

The Annual Proficiency Evaluation is a demonstration of an analyst's ongoing capability. If an analyst has participated in regular lab activities including weigh sessions at least once per quarter, they are considered proficient; otherwise, the analyst should complete another IDOC before returning to regular lab activities.

16.2.2. SOP Training

- PM_{2.5} Lab SOP (required)
- DEP 03-14, Cooler Shipping (required)
- DEP 03-8 (2025) and DEP 03-15 (2025i) Field Sampler SOPs (recommended)
- DEP 18-27 Data Handling and Data Validation SOP (recommended)

16.2.3. Lab Training (required)

- Quality Assurance Guidance Document 2.12 Monitoring PM_{2.5} in Ambient Air Using Designated Reference or Class I Equivalent Methods, January 2016

16.2.4. Regulatory Training

- 40 CFR Part 50 Appendix L (required)
- 40 CFR Parts 53 & 58 (recommended)
- Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Quality Monitoring Program, EPA-454/B-17-001, January 2017 (required)

16.2.5. Air Division Training (recommended)

- Quality Management Plan for the Florida Department of Environmental Protection's Division of Air Resource Management
- Quality Assurance Project Plan for the State of Florida Ambient Air Particulate Matter (PM_{2.5} & PM₁₀) Monitoring Program
- APTI SI-434 Introduction to Air Monitoring
- APTI SI-470 Quality Assurance for Air Pollution Measurements

17.0 **REFERENCES**

Quality Assurance Guidance Document 2.12. Monitoring PM_{2.5} in Ambient Air Using Designated Reference or Class I Equivalent Methods. U.S. EPA, National Exposure Research Laboratory. Research Triangle Park, NC, January 2016.

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

Quality Assurance Handbook for Air Pollution Measurement Systems Volume II, U.S. EPA, Ambient Air Quality Monitoring Program, Research Triangle Park, NC, January 2017.

EPA Additional Guidance on PM_{2.5} Cassette Handling and Transportation, January 2000.

Draft Guidance for Acceptable Procedures for Archiving PM_{2.5} Samples, February 2000.

Memorandum that Transmits Information on a National User Modification Allowing an Extension of the PM_{2.5} Mass Filter Retrieval Period from 96 Hours to 177 Hours, February 5, 2002.

Clarification on Use of PM_{2.5} Field and Laboratory Requirements for Low Volume PM₁₀ Monitoring to Support PM₁₀ NAAQS, March 3, 2016.

Guidance on the Interpretation of the PM Filter Archive Requirements Described in 40 CFR Part 58.16, July 30, 2012.

18.0 APPENDICES

The Appendices contain examples of the referenced forms or documents and additional guidance. The current version of each form is maintained in the Lab's network folders.

18.1. Forms and Documents

Figure 18-1. Filter Lot Stability Test

Filter Lot Stability Test												PM-LOT-STABILITY-1.0 Effective Date: 07/21/2017 Revision Date: 07/21/2017					
Lot Number:		020		Filter Box Numbers:		44300-44399		Analyst Initials:		CAA 7/25/17							
Balance ID:		A-XP6				Boxes Pulled: 44310, 44360, 44390		Reviewer Initials:		JRJ 7/26/2017							
QC Check Weight Values:		QC1: 300.002		QC2: 500.002		QC1 SER#: 1000122776		QC2 SER#: 1000123031									
	Filter ID	Date 7/25/17	Time 7:12	Date 7/26/17	Time 7:12	Date	Time	Date	Time	Date	Time	Total Days	Average days				
QC1 300mg		300.002		300.001		N/A		N/A		N/A							
QC2 500mg		500.000		500.002		N/A		N/A		N/A							
1	T7715501	381.330	0.000	381.330		N/A		N/A		N/A		1					
2	T7715508	373.804	0.003	373.801		N/A		N/A		N/A		1					
3	T7715536	372.649	0.003	372.646		N/A		N/A		N/A		1					
4	T7718018	377.022	0.007	377.015		N/A		N/A		N/A		1					
5	T7718042	375.663	0.004	375.659		N/A		N/A		N/A		1					
6	T7718050	377.338	0.001	377.337		N/A		N/A		N/A		1					
7	T7719509	372.739	0.001	372.738		N/A		N/A		N/A		1					
8	T7719520	368.538	0.001	368.537		N/A		N/A		N/A		1					
9	T7719542	373.603	0.001	373.602		N/A		N/A		N/A		1					
QC1 300mg		300.001		300.002		N/A		N/A		N/A							
QC2 500mg		500.001		500.001		N/A		N/A		N/A			1				
Vaisala Unit S/N:		L0930143															
DAY 1 7/25/17 7:12		Temperature		Humidity		DAY 4		Temperature		Humidity		DAY 5		Temperature		Humidity	
		Average	20.97	33.46	Average			N/A	N/A	Average	N/A			N/A			
		Minimum	20.75	32.11	Minimum			N/A	N/A	Minimum	N/A			N/A			
		Maximum	21.18	34.69	Maximum			N/A	N/A	Maximum	N/A			N/A			
		Std. Dev.	0.06	0.56	Std. Dev.			N/A	N/A	Std. Dev.	N/A			N/A			
DAY 2 7/26/17 7:12		Temperature		Humidity		DAY 3		Temperature		Humidity				Temperature		Humidity	
		Average	20.94	33.39	Average			N/A	N/A	Average	N/A			N/A			
		Minimum	20.60	32.52	Minimum			N/A	N/A	Minimum	N/A			N/A			
		Maximum	21.11	35.28	Maximum			N/A	N/A	Maximum	N/A			N/A			
		Std. Dev.	0.08	0.72	Std. Dev.			N/A	N/A	Std. Dev.	N/A			N/A			
DAY 3		Temperature		Humidity				Temperature		Humidity				Temperature		Humidity	
		Average	N/A	N/A	Average			N/A	N/A	Average	N/A			N/A			
		Minimum	N/A	N/A	Minimum			N/A	N/A	Minimum	N/A			N/A			
		Maximum	N/A	N/A	Maximum			N/A	N/A	Maximum	N/A			N/A			
		Std. Dev.	N/A	N/A	Std. Dev.			N/A	N/A	Std. Dev.	N/A			N/A			

Figure 18-2. Cooler Receiving Log

Effective Date: 08/22/2022					PM-RECEIVINGLOG-2.0			
Revision Date: 08/18/2022								
Revision Author(s): C. Armour								

DEP PM2.5 Cooler Receiving Log								
Infrared Thermometer SN:					Certification Exp:			
Agency	Incoming Y/N	Received Y/N	Date/Time Received	Received By	Shipment Integrity OK?	Received Temp °C	Unpacked By Initials	Comments
Broward								
Central								
Miami-Dade								
Duval								
Hillsborough								
Tallahassee								

Shipment Integrity: Tape intact on hard cooler and lid closed. Softside inner cooler closed. No damage to magazines.

Coolers Unpacked By Signature:

Lab Staff Signature #1: _____ Date/Time: _____

Lab Staff Signature #2: _____ Date/Time: _____

Equilibrium Time ≥ 2.5Hrs: _____ or Equalized Temperature in Degrees Celsius: _____

Additional Comments:

PM-RECEIVINGLOG-2.0.xlsx

Figure 18-3. Field Filter Log

DEP 03-8 REV. 3: 08-2015
 Effective Date: April 20, 2016

INCOMING				FILTER RUN DATA				FILTER STORAGE DATA				OUTGOING		
REC. DATE	MAG #	FILT. #	OPER IN	START		STOP		SAMPLER REMOVAL		Frozen Blue Ice Yes/No	OPER IN	SHIP DATE	MAG #	LAB FILTER COMMENTS
				DATE	TIME	DATE	TIME	DATE	TIME					
04/26/16	M0236	T5562025	AH	05/10/16	00:00	05/11/16	00:00	05/17/16	10:55	Yes	AH	05/17/16	M0236	VOID
05/03/16	M0058	T5562184	AH	05/11/16	00:00	05/11/16	00:00	05/17/16	10:55	Yes	AH	05/17/16	M0236	BLANK
05/03/16	M0058	T5562185	AH	05/11/16	00:00	05/12/16	00:00	05/17/16	10:55	Yes	AH	05/17/16	M0236	
05/03/16	M0058	T5562186	AH	05/12/16	00:00	05/13/16	00:00	05/17/16	10:55	Yes	AH	05/17/16	M0236	
05/03/16	M0058	T5562187	AH	05/13/16	00:00	05/14/16	00:00	05/17/16	10:55	Yes	AH	05/17/16	M0236	
05/03/16	M0058	T5562188	AH	05/14/16	00:00	05/15/16	00:00	05/17/16	10:55	Yes	AH	05/17/16	M0236	
05/03/16	M0058	T5562189	AH	05/15/16	00:00	05/16/16	00:00	05/17/16	10:55	Yes	AH	05/17/16	M0236	
05/03/16	M0058	T5562190	AH	05/16/16	00:00	05/17/16	00:00	05/17/16	10:55	Yes	AH	05/17/16	M0236	
04/26/16	M0284	T5562033	AH	05/10/16	00:00	05/11/16	00:00	05/17/16	12:50	Yes	AH	05/17/16	M0284	
05/03/16	M0287	T5562193	AH	05/11/16	00:00	05/11/16	00:00	05/17/16	12:50	Yes	AH	05/17/16	M0284	BLANK
05/03/16	M0287	T5562194	AH	05/11/16	00:00	05/12/16	00:00	05/17/16	12:50	Yes	AH	05/17/16	M0284	
05/03/16	M0287	T5562195	AH	05/12/16	00:00	05/13/16	00:00	05/17/16	12:50	Yes	AH	05/17/16	M0284	
05/03/16	M0287	T5562196	AH	05/13/16	00:00	05/14/16	00:00	05/17/16	12:50	Yes	AH	05/17/16	M0284	
05/03/16	M0287	T5562197	AH	05/14/16	00:00	05/15/16	00:00	05/17/16	12:50	Yes	AH	05/17/16	M0284	
05/03/16	M0287	T5562198	AH	05/15/16	00:00	05/16/16	00:00	05/17/16	12:50	Yes	AH	05/17/16	M0284	
05/03/16	M0287	T5562199	AH	05/16/16	00:00	05/17/16	00:00	05/17/16	12:50	Yes	AH	05/17/16	M0284	
04/26/16	M0425	T5562041	AH	05/10/16	00:00	05/11/16	00:00	05/17/16	9:40	Yes	AH	05/17/16	M0425	
05/03/16	M0314	T5562202	AH	05/11/16	00:00	05/11/16	00:00	05/17/16	9:40	Yes	AH	05/17/16	M0425	BLANK
05/03/16	M0314	T5562203	AH	05/11/16	00:00	05/12/16	00:00	05/17/16	9:40	Yes	AH	05/17/16	M0425	
05/03/16	M0314	T5562204	AH	05/12/16	00:00	05/13/16	00:00	05/17/16	9:40	Yes	AH	05/17/16	M0425	
05/03/16	M0314	T5562205	AH	05/13/16	00:00	05/14/16	00:00	05/17/16	9:40	Yes	AH	05/17/16	M0425	
05/03/16	M0314	T5562206	AH	05/14/16	00:00	05/15/16	00:00	05/17/16	9:40	Yes	AH	05/17/16	M0425	
05/03/16	M0314	T5562207	AH	05/15/16	00:00	05/16/16	00:00	05/17/16	9:40	Yes	AH	05/17/16	M0425	
05/03/16	M0314	T5562208	AH	05/16/16	00:00	05/17/16	00:00	05/17/16	9:40	Yes	AH	05/17/16	M0425	
04/26/16	M0457	T5562042	AH	05/12/16	00:00	05/13/16	00:00	05/17/16	12:40	Yes	AH	05/17/16	M0438	
ENTER FIELD FILTER COMMENTS BELOW														
T5562025	Filter exchange failed during sampler audit and leak check. Void sample.													
T5562184	Designated Blank Filter													
T5562193	Designated Blank Filter													
T5562202	Designated Blank Filter													

Figure 18-4. Pre-Sampling Filter Layout Sheet

Effective date: 09/28/2016			PM-PRELAYOUTLOG-1.0	
Revision Date: 09/27/2016				
Revision Author(s): P. Kaus				
Pre-Sampling Filter Layout Analyst/Date/Time:				
Lot Number	Filter ID Number Ranges		Number Accepted	LIMS Exposure #
	From	To		

Cancelled Filters (Damaged):

Cancelled Filters (Unused):

Figure 18-6. Chain-of-Custody Log

DEP 03-8-7 Printed: 7/31/2017 9:03:25 AM		PM FILTER CUSTODY LOG		Page 1 of 1
Miami - Dade County Dept. of Env. Resources 701 North West 1st Court 5th Floor Miami, FL 33136 (305) 372-6515 Attention: Georgina Loo		Florida Department of Environmental Protection Division of Air Resource Management Office of Air Monitoring 2600 Blair Stone Road MS 5500 Tallahassee, Florida 32399-2400 Request Number: PM2.5-RQ-2017-07-31-08		
Magazine # M0116				
<u>Filter #</u>	<u>Date Weighed</u>	<u>Filter Order</u>	<u>Expiration Date</u>	
T8529695	24-JUL-2017	1	23-AUG-2017	
T8529696	24-JUL-2017	2	23-AUG-2017	
T8529697	24-JUL-2017	3	23-AUG-2017	
T8529698	24-JUL-2017	4	23-AUG-2017	
T8529699	24-JUL-2017	5	23-AUG-2017	
T8529700	24-JUL-2017	6	23-AUG-2017	
T8529746	24-JUL-2017	7	23-AUG-2017	
T8529748	24-JUL-2017	8	23-AUG-2017	
Magazine # M0304				
<u>Filter #</u>	<u>Date Weighed</u>	<u>Filter Order</u>	<u>Expiration Date</u>	
T8529749	24-JUL-2017	1	23-AUG-2017	
T8529750	24-JUL-2017	2	23-AUG-2017	
T8529756	24-JUL-2017	3	23-AUG-2017	
T8529757	24-JUL-2017	4	23-AUG-2017	
T8529758	24-JUL-2017	5	23-AUG-2017	
T8529759	24-JUL-2017	6	23-AUG-2017	
T8529760	24-JUL-2017	7	23-AUG-2017	
T8529761	24-JUL-2017	8	23-AUG-2017	
Magazine # M0379				
<u>Filter #</u>	<u>Date Weighed</u>	<u>Filter Order</u>	<u>Expiration Date</u>	
T8529762	24-JUL-2017	1	23-AUG-2017	
T8529763	24-JUL-2017	2	23-AUG-2017	
T8529764	24-JUL-2017	3	23-AUG-2017	
Magazine # M0578				
<u>Filter #</u>	<u>Date Weighed</u>	<u>Filter Order</u>	<u>Expiration Date</u>	
T8529765	24-JUL-2017	1	23-AUG-2017	
Relinquished by: <u>Ashley Stroman</u> Ashley Stroman Date/Time: <u>07/31/2017 08:10 AM</u> Received by: <u>Cristina Presmanes</u> Digitally signed by Cristina Presmanes Date: 2017.08.01 14:18:25 -04'00' Date/Time: <u>08/01/2017 01:40 PM</u>				

Figure 18-7. PM_{2.5} Data Acquisition Weigh Template

	A	B	C	D	E	F	G	H	I	J	K
1	PRE/POST:	POST	Vaisala Unit SN:		L0930143		24 hr Average Environmental Conditions Control				Document Control
2			Humidity:		Temperature:		Conditioning Start Date/Time:	07/13/2017 08:00			Form: PM-002-1.6
3	Analyst:	JONES_JR	24 h Avg:	32.61	24 h Avg:	21.2	Conditioning End Date/Time:	07/14/2017 08:00			DEP SOP: PM-001-3.16
4			Minimum:	31.21	Minimum:	20.83					
5	Verified By:	ARMOUR_C	Maximum:	35.37	Maximum:	21.54					
6			Std. Dev.:	0.82	Std. Dev.:	0.16					
7	File Name:	07142017-BMT5-POST.xlsm									
8	Balance ID:	B-MT5									
9	Balance S/N:	1117513806									
10	Auto-Cal:	YES									
11											
12	Filter ID	Filter Type	Weight	Date/Time	Difference	Acc/Rej	Balance ID		Lab Blank		Comments
13	300 mg	MR	300.001	07/14/2017 08:12:36	0	Accept	B-MT5		Pre Weight		
14	500 mg	MR	499.999	07/14/2017 08:13:39	-0.002	Accept	B-MT5				
15	T6528765	SF	387.191	07/14/2017 08:16:38		Accept	B-MT5				
16	T6528981	SF	388.635	07/14/2017 08:17:42		Accept	B-MT5				
17	T6528982	SF	381.659	07/14/2017 08:18:40		Accept	B-MT5				
18	T6528791	SF	385.294	07/14/2017 08:20:02		Accept	B-MT5				
19	T6528794	SF	382.434	07/14/2017 08:20:58		Accept	B-MT5				
20	T6528428	SF	389.154	07/14/2017 08:21:39		Accept	B-MT5				
21	T6528427	SF	375.514	07/14/2017 08:22:15		Accept	B-MT5				
22	T6528426	SF	380.36	07/14/2017 08:24:28		Accept	B-MT5				
23	T6528425	SF	386.75	07/14/2017 08:25:20		Accept	B-MT5				
24	T6528424	SF	374.934	07/14/2017 08:26:16		Accept	B-MT5				
25	300 mg	MR	300	07/14/2017 08:27:07	-0.001	Accept	B-MT5				
26	500 mg	MR	499.999	07/14/2017 08:28:04	-0.002	Accept	B-MT5				
27	T6528423	SF	382.662	07/14/2017 08:29:06		Accept	B-MT5				
28	T6528422	SF	382.28	07/14/2017 08:30:20		Accept	B-MT5				
29	T6528247	SF	384.204	07/14/2017 08:31:18		Accept	B-MT5				
30	T6528438	SF	378.368	07/14/2017 08:32:20		Accept	B-MT5				
31	T6528437	SF	384.999	07/14/2017 08:35:07		Accept	B-MT5				
120		SF					B-MT5				
121	300 mg	MR	300.002	07/14/2017 09:52:25	0.001	Accept	B-MT5				
122	500 mg	MR	500.002	07/14/2017 09:53:08	0.001	Accept	B-MT5				
123	T6528205	LB	379.298	07/14/2017 09:53:59	-0.006	Accept	B-MT5		379.304		
124	T6528353	LB	387.786	07/14/2017 09:55:02	0	Accept	B-MT5		387.786		
125	T6528404	LB	380.699	07/14/2017 09:55:54	-0.001	Accept	B-MT5		380.7		
126	T6528501	LB	379.401	07/14/2017 09:56:44	-0.001	Accept	B-MT5		379.402		
127	T6528602	LB	373.869	07/14/2017 09:57:52	-0.001	Accept	B-MT5		373.87		
128	T6528703	LB	379.06	07/14/2017 09:58:45	-0.003	Accept	B-MT5		379.063		
129	T6528902	LB	385.078	07/14/2017 09:59:40	-0.002	Accept	B-MT5		385.08		
130		LB					B-MT5				
131		LB					B-MT5				
132		LB					B-MT5				
133	T6528765	BD	387.192	07/14/2017 10:04:03	0.001	Accept	B-MT5				
134	300 mg	MR	300.003	07/14/2017 10:05:58	0.002	Accept	B-MT5				
135	500 mg	MR	500.003	07/14/2017 10:07:02	0.002	Accept	B-MT5				
136											
137											
138	Batch Sheet Comments:										
139											
140											
141											

Figure 18-8. Quarterly Working Standard Verification Form

FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION PM2.5 Quarterly Working Masses and Environmental Verifications						Revision No. 1 August 2017 Eff. Date: 08/11/2017
Balance ID: _____		Date: _____		Test No.: _____		
Balance S/N: _____		Time: _____				
Analyst: _____						
Auto-Cal (Y/N) _____						
Cert. / Test ID: _____		ID Date: _____				
Initial mass correction is taken from previous verification result						
Working Standards:						
Standard ID	Serial Number	Working Nominal Mass (N _w) (mg)	Initial mass Correction, C _w (mg)	Working Mass Correction, C _w (mg)	Within Tolerance (Y/N)?	
300 mg (W ₃₀₀)				0	Yes	
500 mg (W ₅₀₀)				0	Yes	
Criteria for working masses: C _w must be within ±3 µg of initial C _w value.						
Primary Standards:						
Standard ID	Date of Calibration	Certificate Number	Serial Number	Primary Nominal Mass (N _p) (mg)	Primary Mass Correction (C _p) (mg)	
300 mg (P ₃₀₀)						
500 mg (P ₅₀₀)						
300 mg Test Results:			500 mg Test Results:			
Measurement	Weight ID	Value (mg)	Measurement	Weight ID	Value (mg)	
O ₁	W ₃₀₀		O ₅	W ₅₀₀		
O ₂	P ₃₀₀		O ₆	P ₅₀₀		
O ₃	P ₃₀₀		O ₇	P ₅₀₀		
O ₄	W ₃₀₀		O ₈	W ₅₀₀		
Environmental Conditions:						
Vaisala Unit S/N: _____		Date: _____		Time: _____		
		Time: _____				
Measurement	Minimum	Maximum	24-hour Average	Standard Deviation		
Temperature (°C)						
Humidity (%RH)						
Conventional Mass Calculations:						
Formula: $C_w = C_p + [(O_1 - O_2 - O_3 + O_4)/2] + N_p - N_w$						
Where C _p = Primary standard mass correction; C _w = Working standard mass correction; O _x = Observed weight; N _p = Primary nominal mass; N _w = Working nominal mass.						
Comments:						
Completed by: _____			Reviewed by: _____			

Figure 18-9. Quarterly Temperature-Humidity Verification Form

Vaisala Temperature and Humidity Quarterly Verification											
Quarter:											
Date:											
Analyst:											
Primary Unit S/N:											
Calibration Due Date:											
Working Unit S/N:											
Calibration Due Date:											
Reviewed By:											
Review Date:											

The temperature and RH data logger of the environmental control system for the PM2.5 weighing room is an integral part of ensuring 40 CFR Part 50 Appendix L weighing criteria are met and should be audited on a routine frequency. Dataloggers may fail in different ways. A comparison of all numbers, logged and real-time, is essential to ensure that the loggers are both functioning properly. An independent audit standard or logger (Primary Unit) should be used to compare against the laboratory temperature and RH unit (Working Unit) every 90 days; however, more frequently is recommended. The standard used for the audit must be at least as accurate as the weighing room's logger. The acceptance criteria for the temperature and RH audit is $\pm 2^\circ\text{C}$ for temperature and $\pm 2\%$ for RH.

Observation	Time	Primary Unit (real-time)		Working Unit				Nominal Difference (= Primary - Working)			
		Temp $^\circ\text{C}$	%RH	Logged Data		Real-Time Data		Logged Data		Real-Time Data	
				Temp $^\circ\text{C}$	%RH	Temp $^\circ\text{C}$	%RH	$^\circ\text{C}$	%RH	$^\circ\text{C}$	%RH
1								0	0	0	0
2								0	0	0	0
3								0	0	0	0
4								0	0	0	0
5								0	0	0	0
6								0	0	0	0
Means:		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!	0.00	0.00	0.00	0.00

Acceptance Criteria: (Average Difference) Temp: $\pm 2^\circ\text{C}$ %RH: $\pm 2\%$

Pass	Pass	Pass	Pass
------	------	------	------

Instructions: The audit standard (Primary Unit) should be placed in the weighing room and allowed to equilibrate for at least 30 minutes prior to conducting the audit. After the audit standard has equilibrated to room conditions, record the audit standard temperature and RH readings and the temperature and RH from the laboratory logging system (Working Unit) every five minutes for 30 minutes. Calculate the differences for both temperature and RH for all points. Calculate and compare the means of the audit device and the logger. If the comparison is outside of the acceptance criteria, troubleshoot and repeat the process. If the problem persists, cease weighing, replace the laboratory logger, and investigate the impact on the filter weighing data.

Figure 18-10. PM_{2.5} Laboratory Analysis Report

PM_{2.5} Analysis Report

Florida Department of Environmental Protection
 Division of Air Resource Management
 Office of Air Monitoring
 2600 Blair Stone Road, MS 5500
 Tallahassee, Florida 32399-2400

DEP South West District Office
13051 North Telecom Parkway
Temple Terrace, FL 33637-0926
(813) 632-7600
Attention: Patrick Washinton

For Additional Information Contact:
Anna Lomasney, Environmental Administrator
Phone (850) 717-9053
Saphique Thomas, Quality Assurance Manager
Phone (850) 717-9015

Printed: 8/7/2017 4:29:41 PM

Abbreviations and Data Remark Codes

<u>Code</u>	<u>Definition</u>	<u>Code</u>	<u>Definition</u>	<u>Code</u>	<u>Definition</u>
ALT	Alternate Measurement	FLB	Failed Laboratory Blank	LTC	Less Than Criteria of Detection
AVG	Average Value	FLD	Failed Laboratory Duplicate	NAR	No Analysis Result
BDL	Below Detectable Limits	FLH	Failed Laboratory Humidity	PSD	Possible Shipping Damage
BLQ	Below Limit of Quantitation	FLT	Failed Laboratory Temperature	REJ	Rejected
CAN	Canceled	FQC	Failed Quality Control	REQ	Requeue for Re-Analysis
CBC	Cannot be Calculated	FRW	Failed Replicate Weight	RET	Return(ed) for Re-Analysis
EER	Entry Error	FSP	Failing Sample Preservation	RIN	Re-Analyzed
FBK	Found in Blank	HTE	Holding Time Exceeded	SIC	Sample Integrity Compromised
FCS	Failed Collocated Sample	ISP	Improper Sample Preservation	SIS	Sample Integrity Suspect
FFB	Failed Field Blank	LAC	Laboratory Accident	STD	Internal Standard
FIS	Failed Internal Standard	LLS	Less Than Lower Standard	UND	Analyzed But Undetected
				VOD	Void Sample

Certified By:

Anna Lomasney

Date: 07-AUG-2017 16:28

Received Temperature: -1.8 degC

Filters Received in Magazine: M0443

<u>T6528626</u>	<u>Weight (mg)/Delta (ug)</u>	<u>Date</u>	<u>Qualifier</u>	<u>Comment</u>
PRE	380.454	19-JUN-2017 09:33		
POST	380.573	28-JUL-2017 09:14		
DELTA	119	NA		

<u>T6528629</u>	<u>Weight (mg)/Delta (ug)</u>	<u>Date</u>	<u>Qualifier</u>	<u>Comment</u>
PRE	378.811	19-JUN-2017 09:33		
POST	378.916	28-JUL-2017 09:13		
DELTA	105	NA		

<u>T6528630</u>	<u>Weight (mg)/Delta (ug)</u>	<u>Date</u>	<u>Qualifier</u>	<u>Comment</u>
PRE	373.994	19-JUN-2017 09:34		
POST	374.134	28-JUL-2017 09:12		
DELTA	140	NA		

--- End of Report ---

Serial # 14251

Page 1 of 1

Table 18-1. PM_{2.5} Laboratory Automated Weigh Sheet and LIMS QA Verification Checks

Check	Purpose	Action on Failure	Qual.	Comment
Weigh sheet: Analytical low mass $\pm 3 \mu\text{g}$ at start of batch	Verify balance calibration	Flag Accept/Reject in sheet		
Weigh sheet: Analytical high mass $\pm 3 \mu\text{g}$ at start of batch	Verify balance calibration	Flag Accept/Reject in sheet		
Weigh sheet: Analytical low mass $\pm 3 \mu\text{g}$ at end of batch	Verify balance calibration	Flag Accept/Reject in sheet		
Weigh sheet: Analytical high mass $\pm 3 \mu\text{g}$ at end of batch	Verify balance calibration	Flag Accept/Reject in sheet		
Weigh sheet: Filters in batch bracketed by passing analytical masses	Verify balance calibration bracketing	Flag Accept/Reject in sheet		
Weigh sheet: Lab blank(s) within $\pm 15 \mu\text{g}$ of original mass	Verify blank mass stability	Flag Accept/Reject in sheet		
Weigh sheet: Filter duplicate within $\pm 15 \mu\text{g}$ of original mass	Verify filter duplicate mass stability	Flag Accept/Reject in sheet		
Weigh sheet upload: Filter in correct status	Verify filter is in the correct status to process	Prevent data upload		
Weigh sheet upload: Filter ID exists	Verify the filter ID exists in the database	Prevent data upload		
Weigh sheet upload: Analyst name exists	Verify that the analyst name is a valid user	Prevent data upload		
Weigh sheet upload: Balance ID is not blank	Verify that the weigh sheet includes a balance ID	Prevent data upload		
Weigh sheet upload: Weight is a valid number	Verify that weight is a valid non-negative number	Prevent data upload		
Weigh sheet upload: Dates are valid values	Verify that dates are formatted correctly	Prevent data upload		
Weigh sheet upload: Pre-condition start date < Pre-condition end date	Verify date order	Prevent data upload		
Weigh sheet upload: Pre-condition end date $\leq$ Pre-weigh date	Verify date order	Prevent data upload		
Weigh sheet upload: Pre-weigh date < Sample date	Verify date order	Prevent data upload		
Weigh sheet upload: Sample date < Post-condition start date	Verify date order	Prevent data upload		
Weigh sheet upload: Post-condition start date < Post-condition end date	Verify date order	Prevent data upload		
Weigh sheet upload: Post-condition end date $\leq$ Post weigh date	Verify date order	Prevent data upload		
Weigh sheet upload: Pre-condition end date – Pre-condition start date ≥ 24 hours	Verify minimum conditioning period	Prevent data upload		
Weigh sheet upload: Post-condition end date – Post-condition start date ≥ 24 hours	Verify minimum conditioning period	Prevent data upload		
Weigh sheet upload: Pre-weigh date – Pre-condition end date ≤ 10 hours	Verify weighed within allowed time since conditioning	Prevent data upload		
Weigh sheet upload: Post-weigh date – Post-condition end date ≤ 10 hours	Verify weighed within allowed time since conditioning	Prevent data upload		

Check	Purpose	Action on Failure	Qual.	Comment
Receive: Magazine ID exists	Verify the magazine ID exists in the database	Error message in interface		
Receive: Filter ID exists	Verify the filter ID exists in the database	Error message in interface		
Receive: Collected date - Sample date ≤ 177 hours	Verify holding time after field sampling	Flag filter in interface		
Review: Sample date - Pre weigh date ≤ 30 days	Verify holding time before field sampling	Qualify/comment record	HTE	Sampled more than 30 days after Pre Weighing.
Review: Collected date - Sample date ≤ 177 hours	Verify holding time after field sampling	Qualify/comment record	HTE	Filter collected more than 177 hours after Date Sampled.
Review: Received temp ≤ 4 °C → Post weigh date - Sample date ≤ 30 days	Verify 30 day hold time	Qualify/comment record	HTE	Weighed more than 30 days after Sampling.
Review: Received temp 4.1 - 25 °C	Verify received temp	Qualify/comment record	ISP	Received at [X] degrees C. Verify average ambient sampling temperature and holding time.
Review: Received temp ≥ 25.1 °C	Verify received temp	Qualify/comment record	FSP	Filter magazine received above 25 degrees C.
Review: Pre and Post lab blank weights within 15 µg	Verify blank mass stability	Qualify/comment record	FLB	Post Weight Lab Blank out of range.
Review: Pre-conditioning Temperature Average 20 – 23 °C	Verify conditioning period temperature criteria	Qualify/comment record	FLT	Lab temperature average criterion exceeded.
Review: Pre-conditioning Temperature Standard Deviation ≤ 2 °C	Verify conditioning period temperature criteria	Qualify/comment record	FLT	Lab temperature 24-hour standard deviation criterion exceeded.
Review: Pre-conditioning Humidity Average 30 – 40 % RH	Verify conditioning period humidity criteria	Qualify/comment record	FLH	Lab humidity average criterion exceeded.
Review: Pre-conditioning Humidity Standard Deviation ≤ 5 % RH	Verify conditioning period humidity criteria	Qualify/comment record	FLH	Lab humidity 24-hour standard deviation criterion exceeded.
Review: Post-conditioning Temperature Average 20 – 23 °C	Verify conditioning period temperature criteria	Qualify/comment record	FLT	Lab temperature average criterion exceeded.
Review: Post-conditioning Temperature Standard Deviation ≤ 2 °C	Verify conditioning period temperature criteria	Qualify/comment record	FLT	Lab temperature 24-hour standard deviation criterion exceeded.
Review: Post-conditioning Humidity Average 30 to 40 % RH	Verify conditioning period humidity criteria	Qualify/comment record	FLH	Lab humidity average criterion exceeded.
Review: Post-conditioning Humidity Standard Deviation ≤ 5 % RH	Verify conditioning period humidity criteria	Qualify/comment record	FLH	Lab humidity 24-hour standard deviation criterion exceeded.
Review: Pre- and Post-conditioning Humidity Averages within 5% RH	Verify conditioning period humidity criteria	Qualify/comment record	FLH	Pre-/Post-Sample Humidity Average Difference of 5 % RH exceeded.

18.2. PM_{2.5} Lab Guidance for Void and Cancel Status

Table 18-2. PM_{2.5} Lab Guidance for Void and Cancel Status

	Cancel	Void
Installed in a sampler?	No	Yes
Weighed upon receipt in lab?	No	Yes
Will appear on the customer's report?	No	Yes

The PM_{2.5} lab has developed this document to clarify the difference between Void and Cancel for filter status on filter logs and subsequent reports. See Table 18-2 for a brief summary of the differences. See detailed explanations below.

- Filter logs should not have any cells merged or any text/letter characters in the time- or date-formatted cells, for example in the columns for Filter Run Data Start and Stop Date and Time.
 - Text in these fields or merged cells anywhere in the data table section of the field sheet prevents upload of filter data into our LIMS system and delays the filter receiving process.
 - Text comments can be entered in the Lab Filter Comments column of the data table or in the Field Filter Comments section below the data table.
 - Information in the Lab Filter Comments column will be uploaded to LIMS and may appear on the customer's report.
 - Information in the Field Filter Comments section is for lab, field, and QA reference only, and will not be uploaded into LIMS.

Two types of filters often create problems on the filter log and confusion for lab staff and customers.

- Filters that were **never installed** in a sampler, and, therefore have no run or sampler removal time/date associated.
 - These filters should be **cancelled**.
 - Customers should remove filter/magazine IDs that they wanted to be cancelled from the data table and list them in the Field Filter Comments section beneath with the comment "Cancel".
 - These filters will not be weighed upon receipt into the lab.
 - These filters will be cancelled in the LIMS system and will not appear on the customer's report.

- Filters that were installed in a sampler, but, for some reason did not run properly or at all. In other words, these filters have install/removal and at least intended run date/time data, but the agency knows the reason the filter data will be invalid.
 - These filters should be **voided**.
 - Customers should enter Filter Run Start/Stop dates and times as well as the Sampler Removal dates and times in the data table of the field sheet. These dates and times can be any reasonable data. For example, customers often use the date and time the magazine was installed in the sampler for both the Start and Stop Run Data.
 - The comment “Void” should be entered in the Lab Filter Comment column for these filters.
 - Other comments or information about the reason for the void can be entered in the Field Filter Comments section.
 - These filters will be weighed upon receipt.
 - Void filters will appear on the report with the data qualifier “VOD” and the comment “Voided by customer”.

18.3. Lab Humidity Control Guidance

Table 18-3. Lab Humidity Control Guidance

Outside temp and RH	When to Adjust	Solution	Result	Possible Consequence
Cold and Dry	When Humidity drops in a constant manner (6 or more hours of Vaisala data) below 32.	Turn off dehumidifier and increase fan speed to 46,000 HZ. Make sure humidifier is on. Limit ingress and egress to lab.	Humidity will be maintained at near minimum for most of time. Dropouts be low 30 are highly likely.	Received filters may have to be refrigerated. May need to lay out and weigh on another day. Pre-filters may have to be weighed on alternate day.
Wet and warm	Only if adjusted prior to ambient conditions changing or if RH values are changing rapidly (2 hour timeframe more than 3 RH units not during a significant weather event or weigh session).	Normal fan speed of 38,000 to 43,000 HZ with dehumidifier on.	Humidity should remain in range. May be on high side of range.	Possible delayed weigh sessions based on when and how long conditions are out of range. See SOP for details (transient event).
Wet and cold	If humidity levels are dropping in wet ambient conditions, follow the cold and dry procedure.	Normal fan speed of 38,000 to 43,000 HZ with dehumidifier on. Humidifier can be left on.	Humidity should remain in range. May still be low.	Possible delayed weigh sessions based on when and how long conditions are out of range. See SOP for details (transient event). Also see Cold and dry.
Warm and dry	When Humidity levels decrease in a constant manner (6 or more hours of Vaisala data) below.	Turn off dehumidifier and increase fan speed to 46,000 HZ. Humidifier should be left on.	Humidity should remain in range. May still be low.	Possible delayed weigh sessions based on when and how long conditions are out of range. See SOP for details (transient event).
Note: Lower fan speeds with dehumidifier on will decrease humidity levels during warm weather. Higher fan speeds with dehumidifier off and humidifier on will increase (to a point) humidity levels during cold dry weather. Other than cold dry weather, Humidity levels can usually be kept within limits with simple adjustments of fan speed. Most times outside of winter, the same setting can be used for the majority of the time. This is just a broad guide for making adjustments to room conditions. Temperature was not mentioned as it seems largely unaffected by these ambient conditions and resulting adjustments. It does not usually require adjustment. Adjustments should be avoided during weigh sessions as an over adjustment may compromise the session. Every effort should be made to make the adjustments 2 days prior to a weigh session and monitor for anomalies.				
All adjustments should be noted in the laboratory notebook.				

The humidifier is always left on, because it automatically shuts off when humidity rises. The following pages show equipment to be adjusted, where to adjust and how.

The fan speed can be adjusted on this unit (see Figure 18-11). Only use arrow keys to adjust; nothing else is touched (see Figure 18-12).

Figure 18-11. Fan Speed



Figure 18-12. Fan Speed (Close-Up)



The dehumidifier is pictured below (see Figure 18-13). Only use “OFF” and “AUTO” buttons to adjust (see Figure 18-12).

Figure 18-13. Dehumidifier



Figure 18-14. Dehumidifier (Close-Up)

