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

FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION

DIVISION OF ENVIRONMENTAL ASSESSMENT

AND RESTORATION - CHEMISTRY PROGRAM

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Florida Department of Environmental Protection

Chemistry Program

Tallahassee, Florida

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1. SCOPE AND APPLICATION

The principle objective of the $PM_{2.5}$ ambient air quality monitoring program is to measure the concentration ($\mu g/m^3$) of particles ≤ 2.5 μm in diameter that have been collected on 47-mm Teflon filters. This SOP details the operations of the $PM_{2.5}$ weighing laboratory. These operations include:

- maintenance of the weighing laboratory
- filter handling and storage
- weighing the $PM_{2.5}$ filters
- shipping and receiving filters
- reporting the analytical results.

The quality control requirements for each of these processes are detailed. The procedures described within this standard operating procedure meet the criteria established in EPA 40 CFR Part 50, Appendix L for $PM_{2.5}$ measurements. Additional guidance is provided in the EPA Quality Assurance Guidance Document 2.12 (January 2016).

2. SUMMARY OF THE METHOD

The $PM_{2.5}$ laboratory was established to monitor the quantity of atmospheric particulate matter ≤ 2.5 μm in diameter, generally without regard to chemical speciation. 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 laboratory reports the pre and post weights and the resulting delta, calculated as the difference between the pre and post weights. The particulate concentration, in $\mu g/m^3$, is subsequently calculated within the Division of Air database using the pre- and post-weights and the measured volume of air drawn through the filter during sampling .

3. APPARATUS AND EQUIPMENT

3.1. Mettler-Toledo Microbalances (2 units)

3.2. Marble or Granite Balance Tables equipped with anti-vibration pads

3.3. Rupprecht & Patashnick (R&P) 47-mm Filter Beveled Cassettes with Screens

3.4. Rupprecht & Patashnick (R&P) Magazines with caps

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3.5. MTL (Measurement Technologies Laboratories) 47-mm Teflon Filters or equivalent, 50 filters per box

3.6. Millipore Plastic Petri-slide Dishes or equivalent

3.7. Anti-Static Electronic U-Electrode (2 units)

3.8. 2U500 Staticmaster Polonium-210 strips

3.9. Nylon Forceps

3.10. Static-Free, Powder-Free Vinyl Gloves

3.11. Certified 300 mg and 500 mg Working/Primary Mass Standards (Class 0), Rice Lake or Troemner

3.12. Vaisala NIST Traceable HMT333 Digital Hygrometer/Thermometer (3 units), computer-interfaced

3.13. Vaisala Veriteq ViewLinc Software Version V4.0.0.1212

3.14. Large Igloo Coolers

3.15. Softside Coolers (for placement inside the large coolers)

3.16. Ice-Substitute Gel Packs, Large and Small

3.17. Re-sealable Plastic Bags

3.18. Laboratory Refrigerators

3.19. Fisher Brand Tacky Mop

3.20. Tyvek Lab Coats

3.21. Bonnets and Tyvek Boots

3.22. Lint-free Cloth Texwipes

3.23. WinWedge Version 3.4.3 Standard Edition Software, 2014, by TAL Technologies

3.24. Solid Metal Shelves, Heavy-duty

3.25. Siemens Insight, Version 3.9.1 Electronic AC Controller Unit

3.26. Neptronic SK302M Humidifier

3.27. Munters Model HC-300 Dehumidifier

3.28. Infrared Thermometer, NIST Traceable

4. REAGENTS AND CHEMICALS

4.1. Staticide™, Antistatic Solution, ACL Inc.

4.2. Methanol

4.3. Ethanol

5. EQUIPMENT AND ENVIRONMENT MAINTENANCE AND SPECIFICATIONS

5.1. Weighing Equipment and Supplies

5.1.1. The Analytical Microbalances:

- Located in the conditioned environment of 20-23 °C and 30-40% Relative Humidity.
- Readability of 1 µg and repeatability of at least 1 µg.
- Internally calibrated prior to weighing (see manual for instructions on auto-calibration).

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- 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 are kept on file by the laboratory.

5.1.2. Mass Reference Standards

- The *Primary Mass Reference Standards* are certified annually by a NIST-traceable weights and measures laboratory. They are used to verify working standards. The certification may be acquired through the Florida Department of Agriculture (Division of Standards), Troemner, Rice Lake Weighing Systems, or equivalent vendor.
- The *Working Mass Reference Standards* are verified quarterly against the primary mass reference standards. Working standards are generally used for routine filter weighing. This verification is performed by the $PM_{2.5}$ lab staff. The Primary Mass Reference Standards can be used for filter weighing, if necessary.
- Documentation of the standard weight certifications and verifications are stored electronically or in hard-copy.
- Standard weights must be handled and stored properly to avoid damage. The weights are handled only with nylon forceps and are placed only on clean, dry, non-metallic surfaces.
- Standard weights that have been scratched, bent, show corrosion, or no longer meet the weight specifications must not be used.

5.1.3. Computers and Associated Software

The laboratory uses WinWedge Standard 3, Microsoft Excel, and a customized Laboratory Information Management System (LIMS) software for data acquisition and reporting. The computer programs WinWedge and Excel are used for automated data collection from the microbalances. The Scientific Services and Support Program is responsible for maintenance of the computers used in the laboratory and maintenance of the LIMS. Computer or software related problems are reported through the Laboratory Help Desk.

5.1.4. Balance Weighing Tables

Microbalances are located on sturdy granite or marble tables to ensure balance stability. Floor mats and isolation spacers are used to reduce vibration. The table placement in a temperature-controlled environment (room within a room) minimizes air drafts and thermal instabilities.

Surfaces used for handling filters are kept free of paper, chemicals, oils, electrostatic charge, and particulates. The weighing table surface is cleaned before use daily with an antistatic solution.

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5.1.5. Conditioning Shelves – Industrial, heavy-duty steel shelves are used for conditioning the filters. It is important that the shelves are smooth, level, and stable.

5.2. Maintenance and Monitoring of the Air Conditioning Equipment

5.2.1. Controlled Environment - The microbalance and filter conditioning environment is recorded using ≤ 5 minute intervals and a 24-hour average and standard deviation of the 24-hour averages.

- 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 ±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 acceptance ranges. The weighing room conditions are easier to maintain if foot traffic and the number of personnel in the weighing environment is minimized.

5.2.2. Air Conditioning Maintenance – the Laboratory Engineering Specialist is responsible for performing and documenting the following:

- The air-conditioning pre-filter must be changed every 3 months or as needed to minimize the pressure drop across the filtration system. Fan speed should be adjusted seasonally.
- The HEPA filter must 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 w.g. (498 Pa).
- The Neptronic SK302M humidifier and the Munters HC-300 dehumidifier must be inspected/monitored regularly as part of the general air condition service. The humidity cartridge requires replacement 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.

5.2.3. The $PM_{2.5}$ laboratory uses Vaisala Veriteq software to monitor and record the room temperature and humidity. The Vaisala Veriteq data is archived and can be reviewed at any time.

5.2.4. The laboratory conditions are also monitored by a lab-wide Siemens Insight software.

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5.3. Forceps for Handling Filters and Standard Weights

Filters and standard weights are handled only with forceps specifically denoted for the type of handling to be performed. Nylon forceps used to handle filters and standard weights are marked on the handles with "Pre," "Post," "Weights" and "Packing" and must be used only for those purposes. Metal or Teflon-coated forceps must not be used to handle the weights or filters. Discard defective, worn forceps.

5.4. Laboratory Attire

Tyvek laboratory attire, including coats, caps, shoe covers, must be worn by anyone entering the weighing environment. The laboratory apparel is donned in the anteroom prior to entering the weighing environment. The protective clothing is disposable, but can normally be used for at least 1 week before it is soiled and 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, such as standard issue white cloth lab coats.

5.5. Refrigeration for Filter Storage

Refrigerators for archival of sampled filters must have a temperature setting 1-4 °C. The temperature is recorded either using 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.

5.6. Vaisala Digital Hygrometer & Temperature Controller and Probes

The Vaisala sensors perform several functions in the lab environment, including interface with the air condition (AC) system and recording temperature/humidity data in the conditioning/weighing lab. The AC system interface is used to regulate the temperature and humidity in the weighing room. The controller interfaces to the Vaisala Veriteq software. The lab is equipped with multiple Vaisala units for backup and verification.

- NIST certification is maintained by sending units to Vaisala for recalibration annually. The Vaisala sensor calibration criteria for accuracy is ± 0.5°C/±2%RH, and for resolution is ±0.1°C/±0.5%RH, respectively.
- Verification of the Vaisala sensors is performed on a quarterly basis with a criteria of ±2°C/±2%RH by comparing the working sensor readings to the primary sensor readings. The primary sensor unit is labeled and designated in the laboratory notebook. The verifications are documented in the Quarterly Standard Verification form in Appendix 20.8.

5.7. Filter Shipping Supplies

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5.7.1. Rupprecht & Patashnick (R & P) Magazines - Each magazine is assigned a unique magazine number. This number is stamped permanently on the bottom of the magazine using a metal number stamping kit. A barcode is also attached to the side of the magazine for easy identification and electronic scanning. The magazines must be cleaned with a Texwipe moistened with methanol or ethanol after every use. All residue, tape and particles must be removed prior to putting the magazine back into service. The condition of the magazine is also inspected and O-rings or caps are replaced if worn.

5.7.2. 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 R&P $PM_{2.5}$ Sampler since they result in equipment jams. The cassettes and screens must be cleaned with a Texwipe moistened with methanol or ethanol after every use. All residue and particles must be removed prior to putting the cassettes back into service. The condition of the screens and cassettes are also inspected for damage or corrosion. The plastic components need to be inspected closely for hairline cracks, gorges, or scratches. Special attention should be given to the top and bottom sealing surfaces as well as the "fit" of the top and bottom components. Discard worn or damaged cassettes.

5.7.3. Insulated Shipping Containers (soft side and Igloo coolers) – All shipping containers must be inspected after every use for cleanliness and damage.

5.7.4. Ice-substitute Gel Packs ("blue ice" packs) - are used to maintain the proper preservation temperature during filter transport. The ice packs are kept in sealed plastic bags, but are discarded if leaking.

6. GENERAL $PM_{2.5}$ LABORATORY CLEANING PROCEDURES

6.1. Laboratory and Equipment Cleaning

- Workbenches and filter shelves must be wiped down at least weekly using Texwipes and Staticide.
- All forceps must be cleaned before use with methanol or ethanol.
- The floor of the weighing environment and of the anteroom (heavy traffic areas) 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 laboratory entry door for custodian disposal.
- Minimize foot traffic in weighing environment to help reduce contamination.
- A tacky floor mat at the front entrance into the laboratory is used to reduce the amount of particulates brought into the weighing environment. Replace as necessary.

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- Use a 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 coolers with bleach cleaning solution and leave open to air dry. Use paper towels to wipe dry the bags with blue ice. Handles, latches, and hinges on the coolers must be intact and functional. Replace broken components using spare parts when possible.

6.3. R&P Cassette Cleaning Procedure

The filter cassettes must be cleaned after every use with Texwipes and methanol or ethanol:

- The cassette top, bottom, and screen are separated and placed on the workbench.
- Wipe each component of the cassette.
- Inspect and reassemble the cassette.
- Place the clean, assembled cassettes in a labeled plastic bag.

6.4. R&P Magazine Cleaning Procedure

The magazines must be cleaned after every use with Texwipes and methanol or ethanol:

- 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 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.

Table 6-1. $PM_{2.5}$ Laboratory Maintenance and Cleaning Schedule

Item	Responsibility	Frequency
Trash	Lab Staff	Daily
Weigh Tables and workbenches	Lab Staff	Prior to use
Microbalance Weigh Pan	Lab Staff	As needed
Cassettes and Magazines Coolers Conditioning Shelves Floors	Lab Staff	Weekly
Sticky Floor Mats	Lab Staff	As needed
AC Maintenance and HEPA Filter Maintenance	Lab Engineering Specialist	See Section 5.2.2.
Humidifier and Dehumidifier	Lab Engineering Specialist	See equipment manuals
Microbalance Calibration Service	Service Provider	Annually

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Temperature/Humidity Calibration Verification	Lab Staff	Quarterly
Working Weight Calibration Verification	Lab Staff	Quarterly

7. $PM_{2.5}$ FILTER HANDLING PROCEDURES AND INTERFERENCES

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

7.1. 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 vinyl gloves. The filters must not be removed from the lab environment unless they are protected inside a magazine or Millipore Petri-slide dish.

7.2. Handling Filters with Forceps

All filters are to be handled using appropriately labeled forceps by the filter support ring. Forceps must never touch the filter media. The filter media is very delicate and any contact will easily damage the filter surface.

7.3. Millipore Petri-slide Storage of Filters

Each filter is stored in a closed Millipore Petri-slide at all times in the laboratory except when being conditioned, weighed, and when placed into a sampling cassette and magazine for shipping. The Millipore Petri-slide has a cutaway section to make it easier to pick-up the filters with forceps.

7.4. Laboratory Interferences

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

7.4.1. 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 laboratory. To reduce these sources of contamination, personnel must wear Tyvek clothing 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.

7.4.2. 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.

7.4.3. 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.

7.4.4. 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.

7.4.5. Balance Drift and Taring – The balance should not drift or require excessive taring during the course of a typical batch of samples if the instrument is grounded and lab conditions are properly maintained. Drift may be observed if the balance has been powered-on recently. Allow the

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instrument to stabilize for 20 minutes prior to use. Continuous balance drift may be indicative of more serious electronic problems and should be reported to the lab supervisor.

8. $PM_{2.5}$ 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 handling 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.

8.1. Recording Environment Conditions

The conditioning environment for the filters must be within the acceptance criteria given in Section 5.2.1. These environmental conditions must be documented in the weigh sheets for the previous 24 hours no more than 15 minutes from the beginning of the weigh session. In addition, a plot of 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 and stored as a PDF file. 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 is collected at the end of the weigh session to verify that the weighing room was in control.

- Open the Vaisala Veriteq software program.
- Enter Name (admin) and Password (admin).
- Select “Options”/ “Locations” and select the “Trend” tab at the top of the dashboard.
- Select the working Vaisala unit. Drag the Relative Humidity and Temperature boxes into the display grid.
- Make sure the “Start Date and Time” represents the past 24 hours and click on “Apply”.
- Create a PDF file of the plots and statistics and store.
- Verify that the humidity average was maintained at 30.0-40.0% (≤ 5% SD) for the past 24-hours (or longer as required by the Filter Lot Conditioning period).
- Verify that the temperature average was 20.0-23.0°C (±2 °C SD) for the past 24-hours (or longer as required by the Filter Lot Conditioning period).
- Record the temperature/humidity minimum, maximum, and 24-hour averages to four significant figures, exactly as presented by the Veriteq software program.
- Record the temperature/humidity 24-hour standard deviations to two decimal places, exactly as is given by the Veriteq software program.
- Filters are weighed only when environmental conditions are within the temperature and humidity acceptance limits.
- The impact of temperature and humidity transients must be taken into account when weighing filters. The amount of time between the transient and the beginning of a weigh session is important when determining if a spike is significant enough to compromise the conditioning

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period. The table below summarizes acceptable transient spike periods for weighing Teflon filters. Based on lab studies, these minimum delay periods are extremely conservative. Temperature/Humidity excursions longer than 45 minutes are not considered transient. Refer to Table 8.1 for transient durations and weigh delay times. In terms of this application, a transient is defined as a brief humidity and/or temperature deviation that otherwise has an insignificant impact on the overall environmental conditions and measurements being performed as determined by the associated QC samples and mass standards. The temperature and humidity values during the transient will be included in calculation of the 24-hour criteria.

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

Table 8-1. Delay times for weighing filters after a Humidity/Temperature Spike Event.

Note: The 24-hour criteria for humidity and temperature must still be achieved after a transient spike has passed the minimum delay times given in Table 8.1.

8.2. Filter Inventory

All filters must be inventoried before use. Filters are normally received in allotments sufficient for one year from the US Environmental Protection Agency (USEPA). Each new shipment of PM2.5 filters is 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 storage shelf in the laboratory. Packs of filters are removed from storage as needed, using the oldest filters first in sequence.

8.3. Filter Lot Stability Test

A Lot Stability Test must be performed before any filters are weighed 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:

8.3.1. Obtain a $PM_{2.5}$ Lot Blank Filter Stability Test Form (See Appendix 20.1.).

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

8.3.3. Record the Lot #, Filter Box #'s, and Lab Analyst's initials.

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

8.3.5. Select three filters from each of the three boxes (nine filters total) and inspect them for damage. Filters with any defects must be cancelled in the LIMS $PM_{2.5}$ Manager (as described in Section 8.6.2. below).

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- Filter rings must be intact on both sides and not doubled.
- Filter must not have holes as viewed through with 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.

8.3.6. Place the filters in individual Millipore Petri-slides. Record the filter ID numbers for each of the nine filters on the form. Weigh the filters as described in Section 9 and record the weights for Day 1 on the form.

8.3.7. Place the Petri-slides containing the filters on the conditioning shelves. Place the Petri-slides covers such that the lids cover ¾ of the filter surface. Allow the filters to condition, 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 weighings is calculated by the analyst and entered into the Lot Stability Test Form.

8.3.9. Calculate the Lot Conditioning Period: The time required for each of the 9 filters to meet the weight difference criteria is 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.

8.4. Pre-Conditioning Filter Inspection

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

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

8.4.2. The defective filters are cancelled in the PM2.5 Manager, accompanied by a comment. Filters with very minor defects may be sent to the field as Routine Maintenance Filters (RMFs).

8.4.3. If the quantity of defective filters is greater than 15% per box, the laboratory manager must be notified.

8.4.4. Cancellation of any rejected filter is to be performed by the laboratory manager or designee. See Section 8.5.7. below.

8.5. 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.

8.5.1. Obtain 50 Millipore Petri-slides from a new container of Millipore dishes.

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8.5.2. Remove the lids from the Millipore Petri-slides.

8.5.3. 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.

8.5.4. Using the 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 dish). 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 to ensure that filters with duplicate or ambiguous ID numbers will be identified.

8.5.5. The filters are then 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 PM2.5 Manager. Create "New Filters" by following the instructions below. The Pre- and Post- filters are not weighed together in a given weigh session.
  - 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.

8.5.6. At this time, any damaged filters marked "TBC" should be cancelled in the PM2.5 Manager. Appropriate cancellation comments are the following:

- Filter damaged during pre-conditioning.
- Filter ring separated from filter media.
- Filter surface damaged.
- Filter surface torn.
- Foreign object observed on filter surface (flakes, particles).
- Pinhole observed in filter surface.
- Filter weight exceeds limits (Enter weight). See the Filter box for the acceptable weight range for that lot.

8.5.8. 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.

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- Select "Add All".
- Enter Analyst Name, Date, and Time (when filter layout was completed).
- Click "OK" when finished.

8.5.9. End Pre-Conditioning in LIMS - To end Pre-conditioning, follow the instructions above except select End Conditioning instead of Start Conditioning.

8.6. 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 ºC). The reasons for returning filters to cold storage must be documented. Prior to weighing, filters must be re-conditioned for a minimum of 24 hours, but weighing should be completed within 72 hours total, including the initial conditioning time before filters were returned to cold storage. The conditioning starts when the layout is completed. The date and time is recorded on the layout sheet and entered into LIMS (see 8.5.8.).
The instructions to start and end Post-Conditioning are identical to the Pre-Conditioning procedures provided above, except there are separate sections in the $PM_{2.5}$ LIMS module for uploading the Pre and Post results. Also, the filter layout configuration for post-conditioning is in rows of 7 filters.
Note - This is different from the layout configuration for Pre-conditioning, which is in rows of five filters. The reason for this difference is due to the fact that 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 is optimized for space.

9. $PM_{2.5}$ FILTER WEIGHING PROCEDURES AND CRITERIA

9.1. Clean the weighing surface area

The weigh table must be wiped with a Texwipe dampened with Staticide before the balance is used.

9.2. Perform microbalance automatic calibration

- Exercise the microbalance by opening and closing the draft shield of the microbalance five times.
- See balance manual for auto-calibration instructions. Auto-calibrate the balance and wait for the balance reading to return to zero.
- Document the balance auto-calibration on the weigh sheet.

9.3. Manual Weighing Filter Procedure

- Open the WinWedge software program.

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- Open the Excel template for data collection (see Appendix 20.6.) and save as a new weigh sheet with the filename format MMDDYYYY-Balance ID-#, where balance ID is either A-XP6 or B-MT5, and # is the letter A, B, etc indicating the 1st, 2nd, etc sheet of the day.
- Complete the weigh sheet information (note that only cells highlighted in yellow can be modified):
  - PRE or POST
  - Analyst Name
  - Vaisala Unit Serial Number (SN)
  - Conditioning Start and End Date/Time
  - 24-Hour Filter Conditioning Values taken from the Vaisala software:
    - Humidity Average, Minimum, Maximum, and Standard Deviation.
    - Temperature Average, Minimum, Maximum, and Standard Deviation.
  - Balance ID (after entering, the balance SN and Working Weights values and SN's will automatically update)
  - Enter the Filter ID numbers. It may be convenient to add filter ID's to the weigh sheet in sets of ten as the weigh session progresses.
  - Be sure to enable macros so that the mass data transferred from the balance is time-stamped correctly.
- Expose the 300 mg mass reference standard weight between the U-shaped electrode or between a pair of Polonium-210 strips installed in a U-frame holder assembly for a minimum of 30 seconds to discharge any static charge build-up on the weight. Open the draft shield, place the weight 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. Press the Print key once a stable reading is achieved. The WinWedge software will automatically import the mass data. Verify that the mass value is recorded in the correct cell of the Excel weigh sheet and that the timestamp is correct. Close the draft shield on the microbalance.
- Repeat the process above for the 500 mg mass reference standard weight. The weight of each of the mass reference standard weights must not exceed ±3 μg of the certified value. If either of the mass reference standard weight values fail, then the bracketed filters must be reanalyzed.
- Open the first Millipore Petri-slide and remove the filter using the appropriate forceps. Discharge the static on the filter as described above.
- While the filter is being discharged, observe that the balance returns to zero. Manually tare the balance to zero if necessary.
- 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

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- Open the draft shield, remove the filter with forceps, and place it back into the Millipore Petri-slide on the rack with the lid covering it completely.

NOTE: A batch of filters is defined as the filters weighed on one 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 every 10 filters. Excel worksheets are stored as Excel files and Text (Tab Delimited) files on the $PM_{2.5}$ network drive. The Tab Delimited files are used to upload data into the LIMS.

9.4. Batch Duplicate (BD) 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 weighing of the batch duplicate filter must be within ±15µg . If the criteria are not met, then the entire batch must be reweighed.

9.5. Lab Blank Filter Sample (QC Check Parameter)

9.5.1. 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 damaged, then the next acceptable filter is assigned as a lab blank for that filter box.

9.5.2. The laboratory 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 ±15 µg between weighings. If the lab blank weight change exceeds ±15 µg, implement appropriate troubleshooting and corrective actions.

9.5.3. 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 filter layout sheet and later archived in sequence with sampled filters.

9.5.4. 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.

9.6. Upload Filter Weight Data into LIMS

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

- Open the $PM_{2.5}$ Manager in LIMS.
- Under Filters, select "Pre" or "Post" and then select "Weigh".
- Under File, select "Open".

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- Select the Text Tab Delimited File that contains the weights to be uploaded.
- Highlight filters and the associated Lab Blanks and Batch Duplicate.
- Under File, select "Assign Batch ID" (type in a previous batch ID or select New to assign the next available number.) Then select "Upload".

10. 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 Igloo coolers.

10.1. Filter Scheduling in LIMS

- Requests for filters are received from clients and are scheduled by the Laboratory Supervisor (or designee) in the LIMS. Typically, each client will submit a quarterly filter request in advance.
- Filter shipments are packed according to the requests received.

10.2. 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 choose the appropriate magazine and double click or scan it in with the bar code reader. 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 steps 4 and 5 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.

NOTE: It is imperative that the filter order in the magazines agree with the order on the magazine loading sheet and that each of the magazine and filter ID numbers is recorded accurately. Filters are given a final inspection and loaded into the magazine one at a time. Additional filters are added to the magazine by placing the next filter cassette directly on top of the first cassette and then pushing down gently on the piston until the filter is flush with the top of the magazine. 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.

10.3. Cassette Loading Instructions

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

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- 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 appropriate forceps.
- Place the filter, with the ID number facing upward, on the cassette screen using non-metallic forceps labeled "packing."
- Finally, place the cassette top on the filter. Press firmly to ensure the cassette top and bottom makes a tight seal.

10.4. Magazine Loading Instructions

- Cassettes are packed in serialized and bar-coded magazines.
- Obtain a manual hand pump and attach it to the coupling 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 ID facing down. To load the cassettes into the magazine, place the first 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.
- Place the orange cap on the magazine after all the cassettes are loaded in the magazine.
- Place a yellow or black protective cap on the magazine pump coupling.
- Pack the magazine in a soft-side cooler. Store the soft-side coolers in the anteroom until ready for shipment.

10.5. Shipping Documentation

- Greyhound will send shipping labels to be printed and attached to the igloo coolers. Copies of the shipping invoices provided by Greyhound will be archived electronically.
- In the PM2.5 Manager generate a Magazine Custody Sheet for each customer, time-stamped according to the date and time the coolers are picked up by the Greyhound courier.
- The lab analyst must electronically sign the Magazine Custody Sheet and send to the customer via e-mail and save a copy to the appropriate file on the network.

10.6. Cooler Packing for Shipment

- Move the packed soft-side coolers from the weighing lab to the hallway.
- Obtain a large Igloo cooler with the name of the district or local customer receiving the shipment.
- Place large and/or small blue ice packs (room temperature for outgoing shipments) in the soft-side cooler to secure the magazines.
- Place the soft-side cooler in the Igloo cooler. Center the soft-side cooler in the larger Igloo cooler, then place room temperature blue ice packs on both sides to secure the soft-side cooler. (These blue

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ice packs are provided to the customers to freeze at their facility for adequate cooling of the return shipment of sampled filters.)

- Close the Igloo cooler and secure the lid with packaging tape.
- Attach the Greyhound shipping label to the cooler

10.7. Shipping a Cooler

Coolers are routinely shipped by commercial carrier, currently Greyhound. FedEx may be utilized for unscheduled shipments. Procedures for using the Greyhound carrier shipping service are provided in the Air Division's Standard Operating Procedure DEP 03-14 (REV NO 0:12/11).

11. FILTER RECEIVING PROCEDURES AND NON-CONFORMANCE REPORTING

11.1. Cooler Inspection

11.1.1. 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.

11.1.2. Measure the current temperature on the surface of a magazine inside the soft-side cooler using an IR electronic thermometer and record the temperature on the Cooler Receiving Log (Appendix 20.2.).

11.1.3. If a cooler is received above 4.0 ºC, the qualifier "ISP" (Inadequate Sample Preservation) and a comment stating the filter magazine receipt temperature will be added to the associated filter IDs in the LIMS system.

11.1.4. Temperatures greater than 25ºC will result in data qualification with the qualifier "SIS" (Sample Integrity Suspect).

11.1.5. PM2.5 Filter Holding Time Determination - Per 40 CFR Part 50, Appendix L, Section 8.3.6, a 30-day holding time is applicable (for samples collected at > 4 ºC) if the filters are maintained at temperatures below the average ambient sampling temperature; per 2.12 Guidance, a 30-day holding time is allowable if samples are transported at < 4 ºC. Since the laboratory does not have access to the average sampling temperatures and field storage temperatures, the FDEP Division of Air Resource Management (DARM), or designee, will determine the appropriate holding time for all filters and will apply appropriate data null or qualifier codes. Furthermore, the DARM will qualify filters as exceeding the holding time requirement if their average sampling temperature is less than the receiving temperature 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 18-17) for additional details.

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11.1.6. The signed Cooler Receiving Log with cooler receipt temperatures, and any irregularities are added to the weekly folder with Filter Field logs, the filter layout sheet, and copies of the Vaisala data for the weigh session.

11.1.7. Remove and inspect the magazines from the cooler.

11.1.8. Place the magazines and paperwork together on a cart in the anteroom leading to the weighing lab. This anteroom is conditioned the same as the weighing room and allows the magazines to come to equilibrium in a controlled environment before unpacking. Typically the magazines are received in the morning and unpacked that afternoon.

11.2. Non-Conformance Reporting

- Non-conformances (NCRs) are recorded on the Cooler Receiving Log, the filter layout sheet, or in the LIMS Receiving module. 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 when reporting the data in the LIMS.

11.3. 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 to remove filters one-by-one. Place the filters on the receiving desk in the same order they are removed.
- Gently open the cassette to expose the filter. Visually inspect the filter for damage. Make a note of any filter damage. These comment notes will be used to qualify and comment the data in the final report. Recommended comments and their associated flags for damage, voided filters, or holding times are as follows:

Data Qualifier	Code Flag	Recommended Comments
Sample Integrity Suspect	SIS	Filter damage observed upon receipt (ex: pinhole). Filter upside down in magazine upon receipt in lab. Filter was received without a magazine. Foreign object on filter surface (ex: particle, fiber).
Lab Accident	LAC	Laboratory accident or damage.
Possible Shipment Damage	PSD	Filter damaged during shipment.
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.

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Improper Sample Preservation	ISP	Filter Magazine received above 4 ºC.

- Using the Filter layout sheet (Appendix 20.4.), list the customer name, the 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 is visible through the top of the Millipore Petri-slide.
- Place the filter on the post-conditioning shelf with the slide cover ¾ over the filter.

  NOTE: The order of the filters on the layout sheet mimics 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 8.6.)

11.5. 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 field sheet (which is saved in text/tab delimited format on the server).
- Select the date on which the filters arrived into the lab.
- Select "Upload" and the filters will automatically upload.
- Check the list to ensure the correct filter ID's and magazines were selected. Add comments from the layout sheet (see Section 11.3.).
- Select "Analyst ID"
- Add the cooler temperature as recorded on the Cooler Receiving Log.
- Select "Finish" to upload data to LIMS

12. MASS STANDARD AND BALANCE CERTIFICATIONS/VERIFICATIONS

12.1. Annual NIST Traceable Certifications

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

- Primary Mass Standard Annual Certifications – Are purchased new each year or are recertified by Troemner, the Florida Dept. of Agriculture, Rice Lake Weighing Systems, or an equivalent vendor.
- Primary Vaisala Humidity and Temperature Units – Are certified annually by Vaisala Inc.

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- Certification documents are stored electronically on the computer data server and in hard-copy in the $PM_{2.5}$ Laboratory.

12.2. Quarterly Verification of Working Mass Standards

Working mass standards must be verified against the primary mass standards on a quarterly basis using the following procedures.

12.2.1. Use the Mass Reference Standard Verification Form (see Appendix 20.8).

12.2.2. Inspect each mass reference standard for cleanliness and wear. The standard mass weights may be cleaned by rinsing with methanol or ethanol and air dried on a Texwipe, but it may take at least an hour to obtain a stable weight after this cleaning.

12.2.3. Fill out the form with the Balance ID, Analyst Name, Date, and Time. The 24-hour Humidity and Temperature data are also recorded.

12.2.4. Weigh the 300 mg weights following the order listed on the form. Then repeat with the 500 mg weights. The WinWedge program can transfer the mass data from the balance into the selected cells on the form.

12.2.5. Calculate the Working Standard Mass Correction (C_w) values for each weight.

12.2.6. The acceptance criteria for the quarterly verification of the working weights C_w are ± 2 µg compared to the previously determined C_w. If the acceptance criteria are not met, then the working weight must be removed from service and labeled "Do not use" or discarded. Notify the supervisor of verification failures.

12.2.7. Submit the calibration verification to the laboratory supervisor. Maintain records in the $PM_{2.5}$ certificates folder.

12.2.8. Upon acceptable verification, the working weights may be used for routine weigh sessions.

12.3. Annual Microbalance Maintenance Service and Calibration

- A Mettler-Toledo technician will complete this service annually or more often if required.
- Certification documents are stored on a server and a paper copy in the $PM_{2.5}$ Laboratory.

13. QUALITY CONTROL

The quality control procedures discussed below are described in detail within the text of this document, but are summarized here. Table 13-1 provides qualifiers and comments for failed quality control. A summary of all automated LIMS verification checks are provided in Appendix 20.10.

13.1. Conditioning Environment Records

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 ±5.0 % and temperature does not exceed ±2.0 °C.
- Use Table 13-1 for LIMS flagging of holding times and temperature/humidity control.

13.2. Filter Lot Stability Test

The Lot Stability Test filters are weighed, then conditioned for a minimum of 24 hours. The weight difference between successive 24-hour measurements is made until the difference between the last two measurements is less than 15 µg. The time required to reach the criteria for the Lot Stability Test determines the required pre-conditioning time for the specific lot of filters.

13.3. Lab Filter Blanks

Lab blanks are weighed with every batch of filters and must meet the criteria of ±15 µg, compared to the original mass measurement made when the blank filter was first weighed.

NOTE: The laboratory does not verify the frequency or mass of Field Blanks (FB). However the FB results are checked to verify that the weight change does not exceed ±30 µg when identified as blanks on field sheets. If the information is available on the field sheets, then customers are notified for values exceeding the 30 µg specification.

Table 13-1: Automated LIMS Flags and Comments for Holding Time and Temp/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 at ≤4°C or ≤ 10 days at ambient temperature	HTE	Weighed more than [X] 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 Std. Dev. ≤ 2.0°C	FLT	Lab temperature 24-hour Std. Dev. criterion exceeded.
Lab Conditioning Humidity Avg. 30.0-40.0% RH	FLH	Lab average humidity criterion exceeded.
Lab Conditioning Humidity 24-hour Std. Dev. ≤ 5.0%	FLH	Lab humidity 24-hour Std. Dev. criterion exceeded.

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Pre-Post-Sample Conditioning Humidity Difference does not exceed ± 5.0% RH	FLH	Pre-/Post-Sample Humidity Average Difference of ± 5.0 % RH exceeded.

13.4. Working Standard QC checks

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

13.5. Batch Duplicate Filter Weighing

A duplicate filter is weighed with each batch of filters. The first filter on any weigh sheet is denoted as the Batch Duplicate (BD). The weight difference of the filter must be ±15µg.

14. FILTER STORAGE AND RECORDS MAINTENANCE/ARCHIVAL

14.1. Filter Storage and Archival

- Upon completion of filter sampling, post-weighing and reporting, the filters are stored in Millipore Petri-slides and labeled with the filter ID number.
- 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 box, along with any filters in the sequence that were cancelled. A label denoting the series of filters contained in each box will be placed on the exterior of the box. Date and initial the label before placing the box in the storage 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.

14.2. Data Records Maintenance and Archival

- Store all paper and electronic records for a minimum of five years.
- The $PM_{2.5}$ data are stored in MS Excel, Adobe, and/or Text Tab-delimited sheets.
- 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 electronic data is archived by year/month in the $PM_{2.5}$ directory.
- 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. For additional details see SOP LB-006 for "Records Maintenance and Storage".

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14.3. Log Book and Other Supporting Document Storage

- Service Reports - Maintenance reports are kept in folders for each of our laboratory instruments in the $PM_{2.5}$ filing cabinet. The contents of the folders detail maintenance performed and includes the date and signature of the person performing the service.
- Shipping & Receiving Documentation - Shipping and Receiving documents are electronically stored in the $PM_{2.5}$ directory on a network server. These folders contain detailed information regarding all coolers sent and received by the laboratory for each client. Information obtained for each shipment includes the following: Date, Time, Shipping/Receiving Cooler, Customer, Analyst, contents, and any comments.
- Weekly Filter layout sheet - This document lists all the post-filters weighed in the laboratory for the week and provides detailed information on the location and status of the filter upon receipt into the lab. The filter layout sheets are stored in the $PM_{2.5}$ folders.
- LIMS Electronic Logbook – The $PM_{2.5}$ laboratory maintains an electronic notebook for recording issues regarding the laboratory equipment maintenance, lab condition controls, cleaning activities, and other $PM_{2.5}$ laboratory activities.

15. DATA AUTHORIZATION AND REPORTING

The procedures provided in this section detail the data review process, responsible parties, and final report authorization necessary for reporting results in the $PM_{2.5}$ laboratory.

15.1. Data Review and Authorization

The primary responsible parties for reviewing the $PM_{2.5}$ data are the Analyst and Analyst Supervisor. They have the responsibility to review all documents relevant to $PM_{2.5}$ filter preparation, weighing, and reporting.

15.1.1. Open the $PM_{2.5}$ Manager module in the LIMS.

15.1.2. Under Filters, select Review/Authorize.

15.1.3. Select 09-Weight-2-Complete. Filters in this category are ready for review. In this portion of the $PM_{2.5}$ LIMS one can view data associated with any filter by searching for a filter by its ID, selecting a filter by what stage it is in, or by the assigned sampling site. This is the most useful aspect of the $PM_{2.5}$ LIMS because it displays all the available information for a filter. Corrections or comments to data may be performed in this module and documented in the Receiving or Post-Analysis Documents and LIMS.

15.1.4. The following documents must be included in the $PM_{2.5}$ Post Sampling data review:

- Weighing Data Acquisition Excel Form - Must be accurate and complete.
- Field Filter Log Forms - Review for accuracy and date consistency.

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- Cooler Receiving Log and Filter layout sheets - Information must be consistent with the Field and LIMS data.
- Customer Chain-of Custody documents - must be complete and saved.

15.1.5. The LIMS reports the Pre-weight, Post-Weight and Delta values with 3 significant figures.

15.1.6. Review the uploaded results in the LIMS and reweigh samples with Delta values less than -10 µg or more than 500 µg, for confirmation. Also, reweigh any field blank filters where the Post-Weight is more than ±30 µg of the Pre-Weight. Any reweighed filters must meet all the conditioning and weighing criteria for Post-Weighing. Add a comment to the re-weighed filters indicating that the results were confirmed.

15.1.7. The LIMS is designed to automatically add some qualifiers and comments (see Table 13-1), others must be entered manually.

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

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

15.1.10. Then highlight the filters and select Authorize Selected.

15.2. $PM_{2.5}$ 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.
- Then select "Add".
- At the top are options to Preview the report or Generate New Serial #. Choose the appropriate one. 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.
- Once a filter is reported, it is no longer listed as an available filter to report.
- The final $PM_{2.5}$ report will automatically print at the Main Lab's designated printer. The paper report is filed in the Lab Report cabinets located in Building A, 3rd floor Chemistry Section.
- An example PM2.5 report is provided in Appendix 20.9.
- The lab has a Paper-Free Policy for reporting data to our clients. Upon authorization, an electronic copy of the final report is automatically sent to each client.

15.3. Turn-Around Time

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The lab's typical turn-around time for $PM_{2.5}$ data is between 7 to 12 days. However, the lab has a policy of reporting routine samples within 28 days.

16. HAZARDOUS WASTE & SAFETY PROCEDURES

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

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

17. METHOD VALIDATION AND PERSONNEL QUALIFICATIONS

The laboratory must perform a formal validation of the $PM_{2.5}$ Method prior to using the method to generate reportable data. Also, all laboratory personnel using the method must be trained on the laboratory procedures and successfully demonstrate their ability to perform the analysis in a Demonstration of Capability and Annual Assessments thereafter.

17.1. 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 initial demonstration of capability for the method involves measuring the mass of a set of standard weights and demonstrating that the instrumentation and personnel can meet all of the method criteria.

17.2. Personnel Qualifications

All personnel who generate reportable data must first successfully perform an Initial Demonstration of Capability (IDOC) and then an Annual Proficiency Evaluation thereafter. It is imperative that the staff be part of a continuing education and training program to insure that their skills and knowledge stay up-to-date. As indicated, some of the training items listed below are required for either analysts or supervisors, while some is recommended only for supervisors. Training completion is documented in the LIMS Training module.

17.2.1 Method IDOC Training (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. This training also includes demonstration of the ability to handle sample filters within the laboratory workflow, to perform calibration

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procedures, to assess and document environmental conditions, and to maintain a clean working environment.

17.2.2. SOP Training

- $PM_{2.5}$ SOP PM-001 (required)
- Document Archival SOP LB-006 (recommended)

17.2.3. Laboratory Training (required)

- Quality Manual for State of Florida Department of Environmental Protection Bureau of Laboratories, FDOH Certification Number E31780

17.2.4. Regulatory Training

- 40 CFR Part 50 Appendix L (required)
- 40 CFR Part 58 (recommended)
- Quality Assurance Guidance Document 2.12 Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods, November 1998 (recommended)
- Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Quality Monitoring Program, EPA-454/B-13-03, May 2013 (recommended)

17.2.5. 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 $Matter_{2.5}$ Monitoring Program
- APTI SI-422 Air Pollution Control Orientation Course
- APTI SI-434 Introduction to Ambient Air Monitoring

18. REFERENCES

18.1. 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.

18.2. Reference Method for the Determination of Fine Particulate Matter as PM2.5 in the Atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 50, Appendix L.

18.3. Quality Assurance Handbook for Air Pollution Measurement Systems Volume II, U.S. EPA, Ambient Air Quality Monitoring Program, Research Triangle Park, NC, May, 2013.

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

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

18.6. 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, 02/29/2002.

18.7. Mettler Microbalance Operation Manuals.

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18.8. WinWedge Version 3.4.3 Standard Edition Software, 2014 User Manual.

18.9. Florida Department of Environmental Protection, Standard Operating Procedure LB-006, *Standard Operating Procedure for Records Storage and Retention*.

18.10. Florida Department of Environmental Protection, Bureau of Laboratories, "Comprehensive Quality Assurance Plan" (2015).

18.11. Florida Department of Environmental Protection, Bureau of Air Monitoring & Mobile Sources, DEP 03-14, "Standard Operating Procedure - $PM_{2.5}$ Filter Cooler Shipping", Rev. No. 0:12/11.

19. SOP UPDATE REVISION SUMMARY

19.1. 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 SESD's August 3, 2015 review of the laboratory SOP.
- Updated holding time determination per DARM's Statewide Data Handling and Data Validation SOP (DEP 18-17), Section 12.2.2. The laboratory 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 laboratory data

19.2. Subsequent revisions will be listed by date:

06/03/2016 Made updates as requested in EPA SESD's February 12, 2016 review of the laboratory SOP.

20. APPENDICES

The Appendices contain examples of the referenced forms or documents. The current version of each form is maintained in the Laboratory's Document Control system.

20.1. Example Filter Lot Stability Test:

Lot Number: _____ Filter Box Numbers: _____ Analyst Initials: _____

		Date		Date		Date		Date		Date	Total Days	Average
	Filter ID	Day 1	Difference	Day 2	Difference	Day 3	Difference	Day 4	Difference	Day 5		
QC1 300mg												
QC2 500mg												
1												
2												
3												
4												
5												
6												
7												
8												
9												
QC1 300mg												
QC2 500mg												

		Temperature	Humidity
DAY 1	Average		
	Minimum		
	Maximum		
		Temperature	Humidity
DAY 2	Average		
	Minimum		
	Maximum		
		Temperature	Humidity
DAY 3	Average		
	Minimum		
	Maximum		
		Temperature	Humidity
DAY 4	Average		
	Minimum		
	Maximum		
		Temperature	Humidity
DAY 5	Average		
	Minimum		
	Maximum		

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20.2. Example Cooler Receiving Log

Effective Date: 12/16/2015
Revision Date: 12/15/2015
Revision Author(s): C. Rakowski, A. Lomasney, B. Steinbock

PM-RECEIVINGLOG-1.0

FDEP PM 2.5 Cooler Receiving Log				
Infrared Thermometer SN:			Calibration Date:	
County/District	Received (Y/N)	Comments	Received Temp (°C)	Method of Delivery
Broward County				
Central District				
Duval County				
North East District				
North West District				
Orange County				
Miami-Dade County				
Pinellas County				
Hillsborough County				
Palm Beach County				
Tallahassee				
Sarasota County				
South District				
South West District				
Ambient Air				
General Comments:				
Relinquished By: _____ Date and Time: _____ Received By: _____ Date and Time: _____				

1 of 1

PM-RECEIVINGLOG-1.0.xlsx

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20.3. Example Field Filter Log

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

INCOMING				FILTER RUN DATA				FILTER STORAGE DATA				OUTGOING		
REC.			OPER	START		STOP		SAMPLER REMOVAL		Frozen Blue Ice	OPER	SHIP		LAB FILTER
DATE	MAG #	FILT. #	IN	DATE	TIME	DATE	TIME	DATE	TIME	Yes/No	IN	DATE	MAG #	COMMENTS
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

	Completed By:	A. Humphrey	Date:	5/17/2016
	QA/QC By:	N. Rabanal	Date:	5/17/2016

Operator's and a supervisor's initials with their respective dates, are required before filing away Form DEP 03-8-2A in PDF

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20.4. Example Filter Layout Sheet

PM 2.5 Laboratory **PM-POSTLAYOUT-1.1**

Effective Date: 02/24/2016
Revision Date: 02/22/2016
Revision Authors: C. Rakowski, A. Lomasney, B. Steinbock

Chart for Post Sampled Filters

T556	T556	T556	T556	T556	T556	T556
T556	T556	T556	T556	T556	T556	T556
T556	T556	T556	T556	T556	T556	T556
T556	T556	T556	T556	T556	T556	T556
T556	T556	T556	T556	T556	T556	T556
T556	T556	T556	T556	T556	T556	T556
T556	T556	T556	T556	T556	T556	T556
T556	T556	T556	T556	T556	T556	T556
T556	T556	T556	T556	T556	T556	T556
T556	T556	T556	T556	T556	T556	T556

Layout Initials/Date/Time:_____
Shelf ID:_____ of _____
Reviewer Initials/Date:_____

PM-POSTLAYOUT-1.1

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20.5. Example Chain-of-Custody Form

PM2.5 Filter Custody Log

Page 1 of 1

Hillsborough County Environmental Protection Commission
Air Monitoring Division
3629 Queen Palm Drive
Tampa, FL 33619
(813) 627-2600
Attention: Tom Tamanini

Florida Department of Environmental Protection
Central Laboratory
2600 Blair Stone Road
Tallahassee, Florida 32399-2400

Request Number: PM2.5-RQ-2006-05-22-12

Magazine # M0007

Filter #	Cassette#	DateWeighed	FilterOrder	ExpirationDate
T4033363	RP40802	17-MAY-2006	1	16-JUN-2006
T4033364	RP041879	17-MAY-2006	2	16-JUN-2006
T4033365	RP002468	17-MAY-2006	3	16-JUN-2006
T4033366	RP024821	17-MAY-2006	4	16-JUN-2006

Magazine # M0200

Filter #	Cassette#	DateWeighed	FilterOrder	ExpirationDate
T4033367	RP053967	17-MAY-2006	1	16-JUN-2006
T4033368	RP006423	17-MAY-2006	2	16-JUN-2006
T4033369	RP48088	17-MAY-2006	3	16-JUN-2006
T4033370	RP49519	17-MAY-2006	4	16-JUN-2006

Magazine # M0246

Filter #	Cassette#	DateWeighed	FilterOrder	ExpirationDate
T4033371	RP40890	17-MAY-2006	1	16-JUN-2006
T4033372	RP49435	17-MAY-2006	2	16-JUN-2006
T4033373	RP003500	17-MAY-2006	3	16-JUN-2006
T4033374	RP48064	17-MAY-2006	4	16-JUN-2006

Magazine # M0318

Filter #	Cassette#	DateWeighed	FilterOrder	ExpirationDate
T4033375	RP49206	17-MAY-2006	1	16-JUN-2006
T4033376	RP005691	17-MAY-2006	2	16-JUN-2006
T4033377	RP042068	17-MAY-2006	3	16-JUN-2006
T4033378	RP48058	17-MAY-2006	4	16-JUN-2006

Magazine # M0339

Filter #	Cassette#	DateWeighed	FilterOrder	ExpirationDate
T4033379	RP48798	17-MAY-2006	1	16-JUN-2006

Relinquished by: _____

Date/Time: _____

Received By: _____

Date/Time: _____

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20.6. Example PM2.5 Data Acquisition Weigh Template

Effective Date: 02/12/2016
Revision Date: 02/11/2016
Revision Author(s): A. Lomasney

FDEP PM 2.5 Laboratory Weigh Sheet

PM-002-1.4

'RE/POST:		Vaisala Unit SN:			Environmental Conditions Control	Document Control
		Humidity:		Temperature:	Conditioning Start Date/Time:	Form: PM-002-1.4
Analyst:		24 h Avg:		24 h Avg:	Conditioning End Date/Time:	DEP SOP: PM-001-3.15
		Minimum:		Minimum:		
'erified By:		Maximum:		Maximum:	NOTE: *Leave Filter ID blank for unused rows. Only yellow fields are e*	
		Std. Dev.:		Std. Dev.:		

File Name:	PM25-Weigh-Template-version 1.4.xlsm			
Balance ID:	B-MT5	Working Weights		S/N
alance S/N:	1117513806	300 mg:	300.001	1000111252
		500 mg:	500.001	1000123032

MR: Mass reference - these go to mass reference QC table
SF: Single Filter
LB: Lab Blank - these go to the "filter QC table"
BD: Batch Duplicate - duplicate of first SF

Filter ID	Filter Type	Weight	Date/Time	Difference	Acc/Rej	Balance ID		Stability Test Weight	Stability Test Qualifiers	Comments
300 mg	MR		############			B-MT5				
500 mg	MR					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
300 mg	MR					B-MT5				
500 mg	MR					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
300 mg	MR					B-MT5				
500 mg	MR					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	SF					B-MT5				
	LB					B-MT5				
	LB					B-MT5				
	LB					B-MT5				

Page 1 of 2

PM25-Weigh-Template-version 1.4

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20.7. Example Quarterly Standard Mass Reference Verification Form

Florida Department of Environmental Protection

$PM_{2.5}$ Quarterly Working Masses and Environmental Verifications

Balance ID: _____ **Test No.:** _____ **Date:** _____ **Time:** _____

Analyst : _____

Primary Standards:

Standard ID	Serial Number	Calibration Due Date	Primary Nominal Mass (N_p) (mg)	Primary Mass Correction (C_w) (mg)
300 mg (P_{300})	1000111252	7/21/2016	300	0.0006
500 mg (P_{500})	1000111462	7/25/2016	500	0.0002

Measurements of Working vs. Primary Standard Masses:

300 mg Test Results:

Measurement	Standard ID	Weight (mg)
O_1	W_{300}	300.002
O_2	P_{300}	300.003
O_3	P_{300}	300.003
O_4	W_{300}	300.002

500 mg Test Results:

Measurement	Standard ID	Weight (mg)
O_5	W_{500}	500.000
O_6	P_{500}	500.001
O_7	P_{500}	500.000
O_8	W_{500}	499.999

Working Standard Verification Results:

Standard ID	Serial Number	Working Nominal Mass (N_w) (mg)	Calculated Working Mass Correction (C_w) (mg)
300 mg (W_{300})	1000033955	300	-0.0004
500 mg (W_{500})	1000034105	500	-0.0008

Criteria for working masses: C_w must be less than 0.002 mg. Pass Criteria: ☐ N ☒ Y

Environment Conditions:

Measurement	Minimum	Maximum	24-hour Average	Standard Deviation
Temperature (°C)	20.30	21.16	20.78	0.19
Humidity (%RH)	31.86	36.17	33.33	1.00

Conventional Mass Calculations:

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

Where C_w = Primary standard mass correction; C_r = Working standard mass correction; O_i = Observed weight; N_p = Primary nominal mass; N_w = Working nominal mass.

For the 300 mg Test: $C_w = 0.0006 + [(300.002-300.003-300.003+300.002)/2] + 300 - 300 = -0.0004$

For the 500 mg Test: $C_w = 0.0002 + [(500.000-500.001-500.000+499.999)/2] + 500 - 500 = -0.0008$

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20.8. Example Quarterly Temperature -Humidity Verification Form

Florida Department of Environmental Protection

Quarterly Temperature-Humidity Calibration Verifications 2016

Unit Serial Number	Date/Time	Humidity* (%RH)	Temperature* (^{0}C)	Certification Date	Installation Point
H1120114	01/05/2016 11:35 am	32.41	20.99	08/05/2015	ViewLinc Recorder
L0930143	01/05/2016 11:35 am	32.10	21.32	02/26/2015	ViewLinc Recorder
L1350144	01/05/2016 11:35 am	32.00	21.37	03/27/2015	ViewLinc Recorder
U1620002	01/05/2016 11:35 am	32.2	21.2		Seimens Recorder
Pass/Fail		Pass	Pass		

*The humidity and temperature criteria is ± 2 %RH and ± 2^{0}C, respectively. Instantaneous measurements. By ARL

Measurement Comments:

The unit H1120114 was sent out for recalibration on July 13th, 2015 and returned into service in the PM2.5 weighing lab on August 17th, 2015. In the calibration certification report, the "as found" and "final adjusted" conditions for the H1120114 sensor were both within the PM2.5 weighing criteria of ± 2 %RH and ± 2^{0}C. The "as found" condition for humidity however, exceeded the 1% RH criteria established by Vaisala and was adjusted accordingly.

The working unit L0930143 passes the criteria for humidity and temperature against the recently calibrated primary unit H1120114.

The Seimens Recorder certificate is out of date, but was verified and passing against the certified Vaisala units.

System Descriptions:

1) Installation points are side-by-side in the PM2.5 weighing/conditioning room.
2) The data transferred into the LIMS are from the Vaisala ViewLinc Recorder unit (S/N L0930143).
3) Data from recorders were compiled on 01/05/2016 at 11:35 am by Anna Lomasney.

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20.9. Example $PM_{2.5}$ Laboratory Report Format

PM2.5 Analysis Report

Florida Department of Environmental Protection
Central Laboratory
2600 Blair Stone Road
Tallahassee, Florida 32399-2400

Dept. of Environmental Protection
2600 Blair Stone Road
Building, MS #5510
Tallahassee, FL 32399-2400
850-717-9060
Attention: Tammy Eagan, Mike Revell,Jim Magoon

For Additional Information Contact:
Dr. Liang Lin
Dr. Kerry Tate
Phone (850) 245-8085

Printed: 9/11/2015 1:44:39 PM

Abbreviations and Data Remark Codes

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

Filters Received in Magazine: M0254

T4611258	Weight (mg)/Delta (ug)	Date	Qualifier	Comment
PRE	362.327	07-JUL-2015 09:04		
POST	362.541	03-AUG-2015 09:15		
DELTA	214	NA		

T4611259	Weight (mg)/Delta (ug)	Date	Qualifier	Comment
PRE	364.101	07-JUL-2015 09:05		
POST	364.258	03-AUG-2015 09:11		
DELTA	157	NA		

Filters Received in Magazine: M0257

T4611260	Weight (mg)/Delta (ug)	Date	Qualifier	Comment
PRE	365.736	07-JUL-2015 09:06		
POST	365.852	03-AUG-2015 09:19		
DELTA	116	NA		

--- End of Report ---

Serial # 13095 **Page 1 of 1**

20.10. $PM_{2.5}$ Laboratory Automated LIMS QA Verification Checks

Check	Purpose	Action on Failure	Qual.	Comment	New
Weigh sheet: Analytical low mass ± 3 µg at start of batch	Verify balance calibration	Flag Accept/Reject in sheet			N
Weigh sheet: Analytical high mass ± 3 µg at start of batch	Verify balance calibration	Flag Accept/Reject in sheet			N
Weigh sheet: Analytical low mass ± 3 µg at end of batch	Verify balance calibration	Flag Accept/Reject in sheet			N
Weigh sheet: Analytical high mass ± 3 µg at end of batch	Verify balance calibration	Flag Accept/Reject in sheet			N
Weigh sheet: Filters in batch bracketed by passing analytical masses	Verify balance calibration bracketing	Flag Accept/Reject in sheet			N
Weigh sheet: Lab blank(s) within ± 15 µg of original mass	Verify blank mass stability	Flag Accept/Reject in sheet			N
Weigh sheet: Filter duplicate within ± 15 µg of original mass	Verify filter duplicate mass stability	Flag Accept/Reject in sheet			N
Weigh sheet upload: Filter in correct status	Verify filter is in the correct status to process	Prevent data upload			N
Weigh sheet upload: Filter ID exists	Verify the filter ID exists in the database	Prevent data upload			N
Weigh sheet upload: Analyst name exists	Verify that the analyst name is a valid user	Prevent data upload			N
Weigh sheet upload: Balance ID is not blank	Verify that the weigh sheet includes a balance ID	Prevent data upload			N
Weigh sheet upload: Weight is a valid number	Verify that weight is a valid non-negative number	Prevent data upload			N
Weigh sheet upload: Dates are valid values	Verify that dates are formatted correctly	Prevent data upload			N
Weigh sheet upload: Pre-condition start date < Pre-condition end date	Verify date order	Prevent data upload			Y
Weigh sheet upload: Pre-condition end date ≤ Pre-weigh date	Verify date order	Prevent data upload			Y
Weigh sheet upload: Pre-weigh date < Sample date	Verify date order	Prevent data upload			Y
Weigh sheet upload: Sample date < Post-condition start date	Verify date order	Prevent data upload			Y
Weigh sheet upload: Post-condition start date < Post-condition end date	Verify date order	Prevent data upload			Y
Weigh sheet upload: Post-condition end date ≤ Post weigh date	Verify date order	Prevent data upload			Y
Weigh sheet upload: Pre-condition end date – Pre-condition start date ≥ 24 hours	Verify minimum conditioning period	Prevent data upload			Y
Weigh sheet upload: Post-condition end date – Post-condition start date ≥ 24 hours	Verify minimum conditioning period	Prevent data upload			Y
Weigh sheet upload: Pre-weigh date – Pre-condition end date ≤ 12 hours	Verify weighed within allowed time since conditioning	Prevent data upload			Y
Weigh sheet upload: Post-weigh date – Post-condition end date ≤ 12 hours	Verify weighed within allowed time since conditioning	Prevent data upload			Y
Receive: Magazine ID exists	Verify the magazine ID exists in the database	Error message in interface			N

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$PM_{2.5}$ Laboratory Automated LIMS QA Verification Checks (continued)

Check	Purpose	Action on Failure	Qual.	Comment	New
Receive: Filter ID exists	Verify the filter ID exists in the database	Error message in interface			N
Receive: Collected date - Sample date ≤ 177 hours	Verify holding time after field sampling	Flag filter in interface			N
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.	N
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.	N
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.	N
Review: Received temp 5 - 23 °C → Post weigh date - Sample date ≤ [X] days	Verify variable hold time [11 - 29 days based on received temp]	Qualify/comment record	HTE	Weighed more than [X] days (based on received temp) after sampling.	N
Review: Received temp ≥ 24 °C → Post weigh date - Sample date ≤ 10 days	Verify 10 day hold time (based on received temp)	Qualify/comment record	HTE	Weighed more than 10 days (based on received temp) after sampling.	N
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.	N
Review: Pre-conditioning Temperature Average 20 – 23 °C	Verify conditioning period temperature criteria	Qualify/comment record	FLT	Lab temperature average criterion exceeded.	N
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.	Y
Review: Pre-conditioning Humidity Average 30 – 40 % RH	Verify conditioning period humidity criteria	Qualify/comment record	FLH	Lab humidity average criterion exceeded.	N
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.	Y
Review: Post-conditioning Temperature Average 20 – 23 °C	Verify conditioning period temperature criteria	Qualify/comment record	FLT	Lab temperature average criterion exceeded.	N
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.	Y
Review: Post-conditioning Humidity Average 30 to 40 % RH	Verify conditioning period humidity criteria	Qualify/comment record	FLH	Lab humidity average criterion exceeded.	N
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.	Y
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.	N

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