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



FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION

BUREAU OF LABORATORIES

2600 BLAIR STONE ROAD

TALLAHASSEE, FLORIDA 32399

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TABLE OF CONTENTS

	Section	Page
1.	SCOPE AND APPLICATION	3
2.	SUMMARY OF METHOD	3
3.	APPARATUS AND EQUIPMENT	4
4.	REAGENTS & CHEMICALS	5
5.	EQUIPMENT AND ENVIRONMENT MAINTENANCE & SPECIFICATIONS	6
6.	GENERAL $PM_{2.5}$ LABORATORY CLEANING PROCEDURE	9
7.	$PM_{2.5}$ FILTER HANDLING PROCEDURES AND INTERFERENCES	11
8.	$PM_{2.5}$ FILTER CONDITIONING PROCEDURES AND CRITERIA	13
9.	$PM_{2.5}$ FILTER WEIGHING PROCEDURES AND CRITERIA	19
10.	FILTER SHIPPING PROCEDURES AND DOCUMENTATION	21
11.	FILTER RECEIVING PROCEDURES AND NON-CONFORMANCE REPORTING	24
12.	MASS STANDARD AND BALANCE CERTIFICATION/VERIFICATION	28
13.	QUALITY CONTROL	28
14.	FILTER STORAGE AND RECORDS MAINTENANCE/ARCHIVAL	30
15.	DATA AUTHORIZATION AND REPORTING	31
16.	HAZARDOUS WASTE AND SAFETY PROCEDURES	33
17.	METHOD VALIDATION AND PERSONNEL QUALIFICATIONS	33
18.	REFERENCES	34
19.	SOP UPDATE REVISION SUMMARY	35
20.	APPENDICES	36

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

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, handling and conditioning filters, weighing the $PM_{2.5}$ filters, shipping and receiving filters, and reporting the analytical results. The quality control requirements for each of these processes are also detailed. The procedures described within this standard operating procedure meet or exceed the criteria established in the EPA Quality Assurance Guidance Document 2.12[18.1] and EPA 40 CFR Part 50, Appendix L[18.2] for $PM_{2.5}$ measurements.

1.1. Refer to the following general Standard Operating Procedures (SOPs) for additional procedural information:

These supporting SOPs contain details that may not be fully described within this method SOP. The analyst must be completely familiar with all of these SOPs. These general SOPs are superseded by procedures provided herein.

1.1.1. *Safety and Waste Management Practices in the Organic Analysis Groups* (SOP **OG-001**).

1.1.2. *Quality Assurance/Quality Control Measurements in the Volatile Organics (VOC) Laboratory* (SOP **VO-001**).

1.1.3. *Data Review/Authorization Procedures for the Volatile Organic Laboratory* (SOP **VO-010**).

1.1.4. *Non-Conformance Reporting System* (SOP **LB-002**).

1.1.5. *Standard Operating Procedure for Records Storage and Retention* (SOP **LB-006**)

2. SUMMARY OF THE METHOD

The $PM_{2.5}$ program 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. The microbalance must have a readability and repeatability of 1 μg. Upon return of the filters from the sampling sites, they are again conditioned and post-weighed. The particulate concentration, in $\mu g/m^3$, is calculated using the pre- and post-weights and the measured volume of air drawn through the filter during sampling. However, if speciation is to be performed it is normally collected on a quartz filter, which is also described generally within this SOP. Quality control measures are employed and documented at the various stages of the program, including filter conditioning, weighing, shipment, and receipt.

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3. APPARATUS AND EQUIPMENT

3.1. Mettler-Toledo MT-5 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

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

3.6. 47-mm Quartz Filters, ashed filters for speciation

3.7. Millipore Plastic Petri-slide Dishes or equivalent

3.8. Mettler-Toledo Anti-Static Electronic U-Electrode

3.9. Fisher Nylon Forceps

3.10. Static-Free, Powder-Free Vinyl Gloves

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

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

3.13. Digi-Sense RTD Platinum NIST Traceable Digital Thermometer

3.14. Vaisala NIST Traceable HMP233 Digital Hygrometer/Thermometer (3 units) computer-interfaced

3.15. Vaisala Veriteq vLog Software Version V4.5.0.8, 2011

3.16. Ultra Cold 50 Large Igloo Coolers

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

3.18. Rubbermaid Blue Ice Packs, Large (7”x 7” x 2”) and Small (4”x 7”x 2”)

3.19. Omega OS91 IR Direct Digital NIST Traceable Thermometer

3.20. Flammable Rated Laboratory Refrigerators, Fisher IsoTemp 450R

3.21. Fisher Brand Tacky Mop™

3.22. Kappler Tyvek Lab Coats

3.23. Kappler Bonnets and Tyvek Boots

3.23. Lint-free Cloth Text Wipes

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

3.25. Solid Metal Shelves, Heavy-duty

3.26. Siemens Insight, Version 3.9.1 Electronic AC Controller Unit

3.27. Neptronic SK302M Humidifier

3.28. Munters Model HC-300 Dehumidifier

3.29. Infrared Thermometer, NIST Traceable

3.30. Emory Paper- used to remove minor scratches from the PM2.5 cassettes.

4. REAGENTS AND CHEMICALS

4.1. Staticide™, Antistatic Solution, ACL Inc.

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4.2. HPLC-Grade Methanol, Fisher Brand – While other solvents may work equally well, this alcohol was selected because it does not leave residue, evaporates quickly once used, and does not interfere with other lab operations.

5. EQUIPMENT AND ENVIRONMENT MAINTENANCE AND SPECIFICATIONS

5.1. Weighing Equipment and Supplies

5.1.1. The Analytical Microbalances:

- Must be located in a conditioned environment of 20-23 °C and 30-40% Relative Humidity.
- Must have a readability of 1 µg and repeatability of at least 1 µg.
- Routine daily calibration checks of the microbalances are mandatory.
- Operated with the leveling bubble at the level position.
- Microbalances are to be serviced by a Mettler–Toledo technician (or other qualified service provider) once a year. Additional visits by service technicians may be required if the balance is not operating within the acceptance limits of the method or is otherwise malfunctioning.
- Documentation of the instrument service must be kept on file by the laboratory.

5.1.2. Mass Reference Standards

- The *Primary Mass Reference Standards* must be certified annually by a NIST-traceable weights and measures laboratory. The primary standards are used to verify working standards. This certification is currently acquired through the Florida Department of Agriculture (Division of Standards), Troemner, or Rice Lake Weighing Systems.
- The *Working Mass Reference Standards* must be verified quarterly against the primary mass reference standards. This verification constitutes a recertification of the working standards against the NIST-certified primary reference standards. Since the lab has more than one microbalance, all working standards are verified at the same time to ensure that all gravimetric measurements are intercomparable. Working standards are used for routine filter weighing. This verification is performed by the $PM_{2.5}$ lab staff.
- Documentation of the standard weight certifications and verifications must be kept on file by the laboratory.
- Standard weights must be handled and stored properly to avoid damage. The weighs 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 LIMS software for data acquisition and reporting. The computer programs WinWedge and Excel are used for automated data collection from the microbalances. The Laboratory Computer Group is

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responsible for maintenance of the computers used in the laboratory and maintenance of the LIMS. Computer or software related problems are addressed by the Computer Group through the following Internet site http://depnet/burlabs/cgi-bin/hd/helpdesk.asp. Fill in the appropriate information regarding the computer or software problem as requested.

5.1.4. Balance Weighing Tables

The balance tables must be constructed, assembled, and located such that the weighing environment is free of interferences to the weighing process. Microbalances are located on sturdy granite or marble tables to ensure balance stability. Floor mats and isolation spacers are also used to reduce vibration. The table placement must also minimize air drafts by maintaining laminar air flow, from the ceiling to the floor. Thermal instabilities are minimized by placement of the weighing environment in a temperature-controlled environment (room within a room).

Surfaces used for handling filters must be free of paper, chemicals, oils, electrostatic charge, and particulates. To accomplish this, the surface is cleaned daily and analysts must wear caps, gowns, boots, and gloves. Balance tables must be cleaned daily with an antistatic solution before filters are processed.

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

5.2. Maintenance and Monitoring of the Air Conditioning Equipment

5.2.1. Controlled Environment Chamber - The microbalance and filter conditioning environment is recorded using ≤ 5 minute intervals and a 24-hour rolling (accumulative) average and standard deviation of the 24-hour averages. The average relative humidity must be 30-40%, with a variability not to exceed 5% RH standard deviation. The average temperature must be 20-23°C, with variability not to exceed 2 °C standard deviation. The lab will refer to 40 CFR Part 50, Appendix L of the Federal Register to establish the number of significant figures and acceptance criteria, as interpreted by the Bureau SOP **LB-024** for reporting significant figures. Filters are weighed only when environmental conditions are within these acceptance limits or will be flagged and commented (see QC section 13). Weight gain or loss 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 Condition Maintenance of the HEPA Filter and Pre-Filter - A 2” pleated pre-filter on the air conditioning unit protects the main HEPA filter from most of the particles entering the AC system and thus prolongs the life of the HEPA filter.

5.2.3. The pre-filter must be changed every 3 months or more frequently to minimize the pressure drop across the filtration system.

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5.2.4. 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.

5.2.5. The Neptronic SK302M humidifier operates almost continuously during the dry winter months, but works little during the moist summertime. In the summer, the Munters HC-300 dehumidifier operates heavily during the wet summer months, but is operational little during the dryer winter period. Both of these units require minimal upkeep, but 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. Please refer to the operator manuals for details.

5.2.6. The $PM_{2.5}$ laboratory uses Vaisala Veriteq software to monitor and record the room temperature and humidity. The Vaisala Veriteq software stores the data in daily files that are archived and can be reviewed at any time.

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

NOTE: Transient spikes in the temperature or humidity levels will affect the value of the average and standard deviation. Do not proceed if the temperature or humidity conditions are outside of acceptance limits.

5.3. Forceps for Handling Filters and Standard Weights

Filters and standard weights are to be 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,” “Mass Ref,” and “Packaging” and must be used only for those purposes. Inspect the forceps for wear or misalignment daily. Metal or Teflon-coated forceps must not be used to handle the weights or filters since these materials have caused problems in the past. Discard defective, worn forceps.

5.4. Laboratory Attire

Tyvek laboratory attire, including coats, caps, shoe covers, and gloves, must be worn by anyone entering the weighing environment. The laboratory apparel is donned in a dressing area prior to entering the weighing environment. The protective clothing is disposable, but can normally be used for about 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 lose fibers should not worn into the weighing area, such as standard issue white cloth lab coats.

5.5. Refrigeration for Filter Storage

Refrigerators for storage and archival of sampled filters need to have a temperature setting 0-4 °C (±2 °C). These refrigerators must have a temperature log on the exterior door or an electronic data recorder where the temperature of each refrigerator is recorded daily. All archived sampled filters are stored in the refrigerator for a minimum of 1 year. The filters are maintained at 0-4 °C (±2 °C) for a minimum of one calendar year and then stored at room temperature for a total of 5 years. The filters may be disposed after this date.

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Figure 5-1. Tyvek Laboratory Attire

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 also interfaces to the Vaisala Veriteq software that stores the data in daily files that are archived and can be reviewed at any time. The lab is also equipped with a secondary Vaisala unit for backup and verification purposes. The NIST certification is maintained annually on the primary unit by sending it to Vaisala for recalibration. The Vaisala sensors meet the temperature/humidity criteria for accuracy of ± 0.5°C/±2%RH and resolution of ±0.1°C/± 0.5%RH, respectively. Verification of the Vaisala sensor calibration is to be performed on a quarterly basis with a criteria of ± 2°C/± 2%RH.



Figure 5-2. Vaisala Humidity/Temperature Units

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 Text Wipe moistened with alcohol 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 - Only beveled R&P cassettes tops are used. The older 90 degree angle tops are not suitable for the R&P $PM_{2.5}$ Sampler since they result in unacceptable equipment jams. The cassettes and screens must be cleaned with a Text wipe moistened with alcohol 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 cassettes that are worn or damaged beyond repair.

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 disposed if leaking. The Rubbermaid brand is the best we have encountered for being rugged and leak-free.

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

6.1. Daily Laboratory and Equipment Cleaning

6.1.1. Workbenches and filter shelves must be wiped down using Text Wipes and Staticide™.

6.1.2. Weighing Tables must be wiped with a Text Wipe dampened with Staticide™ solution.

6.1.3. All forceps must be cleaned with HPLC grade Methanol.

6.1.4. The floor of the weighing environment and of the anti-room (heavy traffic areas) must be rolled using the Fisher Tacky Mop™ at the end of the day. Use a Tacky Mop™ sheet to clean the floor behind the weighing tables.

6.1.5. All trash receptacles are placed outside the laboratory entry door for custodian disposal.

6.1.6. Minimize foot traffic in weighing environment to help reduce contamination.

6.1.7. A tacky floor mat at the front entrance into the laboratory is used to reduce the amount of particulates brought into the weighing environment. It must be replaced as necessary.

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Figure 6-1. PM2.5 Laboratory Cleaning Products

6.2. Weekly Laboratory and Equipment Cleaning

6.2.1. A compartment containing specialized soft bristle brushes and other balance equipment is contained on the side of the MT5 microbalances. They are available to clean the weighing pan and the surrounding area around the weighing pan. The microbalance must be cleaned each week using these supplies. This procedure must be performed with great care, permitting minimal disturbance to the sensitive weighing pan of the balance. All particles and stains must be removed from the balance surfaces.

6.2.2. Igloo and softside coolers used for carrying the filters and magazines to and from the sampling sites must be inspected for cleanliness and wear. Coolers must be free of substances that may damage or contaminate the filters. Handles, latches, and hinges on the coolers must be intact and functional. Immediately replace broken components using parts located in the storage room.

6.3. R&P Cassette Cleaning Procedure

The filter cassettes must be cleaned after every use. The analyst must wear vinyl gloves when cleaning the cassettes. The condition of the cassette top and bottom and the cassette screen must be inspected upon receipt of the cassettes. Cassettes are to be cleaned using the following procedure:

6.3.1. The cassette top, bottom, and screen are separated and placed on the workbench.

6.3.2. Dampen a Text wipe with alcohol.

6.3.3. Wipe each component of the cassette.

6.3.4. Inspect and reassemble the cassette.

6.3.5. Place the clean, assembled cassettes in a plastic Ziploc bag.

NOTE: Cassettes that are loose, scratched, or warped must not be used. Suspect cassettes must be repaired and tested before use. Minor scratches on the cassettes can be removed using Emory paper by lightly polishing the outer sealing surfaces, making sure to maintain a flat sealing surface. The testing procedure is performed to ensure that cassettes are not too worn for sampling. Leak checks may be performed using an R&P $PM_{2.5}$ Air Sampler located at the main lab with the Division of Air and Resources Management (DARM). Damaged or failing cassettes that cannot be repaired must be discarded.

6.4. R&P Magazine Cleaning Procedure

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The magazines must be cleaned after every use. Magazines are wiped with Text Wipes dampened with alcohol. The piston, magazine cap, and the magazine column are cleaned.

6.4.1. Remove the top orange cap on the magazine.

6.4.2. Wipe the underside of the orange cap.

6.4.3. Wipe the inside of the magazine column and the top the magazine piston.

6.4.4. Use the hand pump to push the piston to the top of the magazine.

6.4.5. Remove any fibers left by the cleaning process.

6.4.6. Place the orange cap on the magazine.

NOTE: Magazine pistons must be checked to ensure that the piston surface is smooth, otherwise the cassettes can be scratched as they come into contact with the piston. If the surface is not smooth, fine emery paper must be used to polish the piston until it is smooth. After polishing, the piston and magazine interior must be cleaned with a Text wipe dampened with alcohol to ensure all particles from the polishing process are removed.

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

Item	Responsibility	Frequency
Work Bench Cleaning General Surfaces Trash	Lab Staff	Daily
Cassette Cleaning Magazine Inspection Coolers Microbalance Weigh Pan	Lab Staff	Weekly
Sticky Floor Mats	Lab Staff	As required - when consumed
AC Maintenance HEPA Filter Maintenance	Lab Engineer	Monthly
Humidifier	Lab Engineer	1500 hours of operation
Microbalance Cleaning Service Calibration	Service Provider Service Provider	Annually Annually
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 minimizing interferences. These procedures require training and an initial demonstration of capability for each analyst before actual samples can be weighed.

7.1. Tyvek Laboratory Clothing

The $PM_{2.5}$ laboratory attire includes disposable Tyvek lab coats, caps, and boots that must be donned in the dressing room prior to entering the weighing area. Any person entering the weighing environment must wear proper laboratory clothing. Laboratory coats, caps, boots, and gloves must also be removed before leaving the laboratory dressing area to minimize contamination from the external environment.

7.2. Handling Filters in the Conditioning Environment

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

7.3. Handling Filters with Forceps

All filters are to be handled using appropriately labeled forceps by the filter support ring. Each filter has a protective plastic ring for protection. Forceps must never touch the filter media. The filter media is very delicate and sharp objects, such as forceps, will easily damage the filter surface.

7.4. 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. The Millipore Petri-slide has a cutaway section to make it easier to pick-up the filters with forceps. Each Millipore Petri-slide is also labeled with the filter ID number for ease of identification and storage. The Petri-slide dish is a premium storage device that normally will not open even if dropped and also has very convenient labeling and storage features. Do not use or mix with other types of filter storage containers.

7.5. Laboratory Interferences

7.5.1. 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 (coat, cap, boots, and vinyl gloves) when in the weighing room. The air in the room is purified with a HEPA filtration system. All workbenches, weighing tables, filter shelves and equipment that comes into contact with the filters must be cleaned regularly to remove particulates.

7.5.2. Static Charge – Static electrical charge is a common interference in performing microbalance measurements. The presence of static charge can adversely affect the measured weight values. To reduce this source of interference all lab equipment must be electrically grounded, working surfaces wiped with Staticide™ and a Mettler-Toledo Anti-Static Electronic U-Electrode or polonium strips are used to remove static charge from the $PM_{2.5}$ filters.

7.5.3. Environment Conditions – The stability of the lab's environmental air controls can result in interferences, such as excessive air movement or large changes in the room temperature or humidity. The room temperature and humidity must be closely regulated by the air handling system and air movement minimized with proper baffling.

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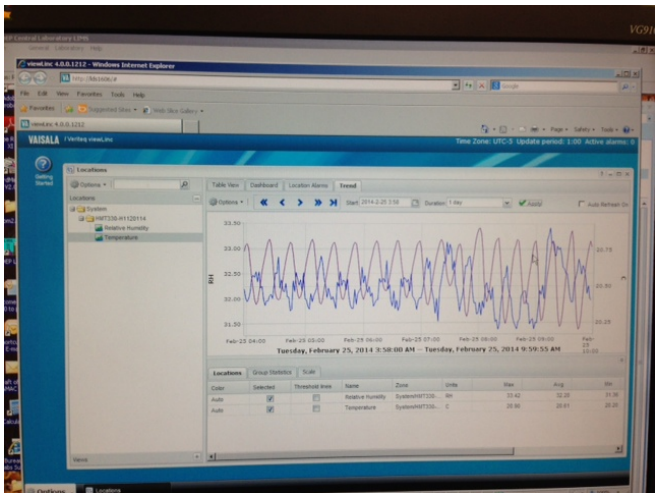


Figure 7-1. Software Screen Capture of Temp./Humidity

7.5.4. Vibrations - The lab environment must be free of vibrations. This can be accomplished by avoiding foot traffic and heavy equipment areas to place the balance. The mass of the balance bench and use of vibration absorbers can also minimize vibration interferences.

7.5.5. 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 and lab conditions are properly maintained. Drift may be observed if the balance has been powered-on recently. Allow the 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 always be within the acceptance criteria given in Section 5. These environmental conditions must be entered in the Temperature and Relative Humidity Sections of the WinWedge data collection software (Excel spreadsheet) template before each weighing session. In addition, a plot of the previous 24-hour temperature and humidity data from the Vaisala Veriteq software (including temperature and humidity minimum/maximum/average and standard deviation) must be recorded within 15 minutes of the beginning of the first weigh session of the day and stored with the daily filter batches as a PDF file. All filters processed during a given weigh session must be completed within 12 hours of the assigned filter conditioning end time.

8.1.1. Open the Vaisala Verteq software program.

8.1.2. Enter Name (admin) and Password (admin).

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8.1.3. Select “Options”/ “Locations” and select the “Trend” tab at the top of the dashboard.

8.1.4. Drag the Relative Humidity and Temperature boxes into the display grid.

8.1.5. Make sure the “Start Date and Time” represents the past 24 hours and click on “Apply”.

8.1.6. Click on the “Group Statistic” tab at the bottom to show statistical information.

8.1.7. Create a PDF file of the plots and statistics and store with the daily data batches.

8.1.8. Verify that the humidity average was maintained at 30-40% (≤ 5% SD) for the past 24-hours or longer (if a 48-hour or longer Lot conditioning time is required).

8.1.9. Verify that the temperature average was 20-23°C (±2 °C) for the past 24-hours or longer (as required by the Filter Lot Conditioning period).

8.1.10. Record the temperature/humidity minimum, maximum, and 24-hour averages to four significant figures, exactly as presented by the Verteq software program.

8.1.11. Record the temperature/humidity 24-hour standard deviations to two significant figures, exactly as is given by the Verteq software program.

8.1.12. Filters are weighed only when environmental conditions are within the temperature and humidity acceptance limits.

8.1.13. 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 weighing session is important when determining if a spike is significant enough to compromise the conditioning 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.

Temp/Humidity Spike Duration	Minimum Delay for Weighing
< 10 minutes	30 minutes
10-30 minutes	60 minutes
30-60 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 even 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 are removed from the original shipping box and externally inspected to ensure the 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.

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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 Appendices 20.3.).

8.3.2. Select 3 boxes of filters 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, as described below, must be voided in the LIMS $PM_{2.5}$ module.

8.3.5.1. Filter rings must be intact on both sides and not doubled.

8.3.5.2. Filter must not have holes as viewed through with a good light source.

8.3.5.3. Filter must not have discoloration or water marks.

8.3.5.4. Filters must not have scratches or dents in the Teflon surface.

8.3.5.5. Filter number must be present, legible, and unique.

8.3.6. Place the filters in individual Millipore Petri-slides.

8.3.7. Place the Petri-slides containing the filters on the conditioning shelves.

8.3.8. Place the Petri-slides covers such that the lids cover ¾ of the filter surface.

8.3.9. Record the filter ID numbers for each of the nine filters on the form.

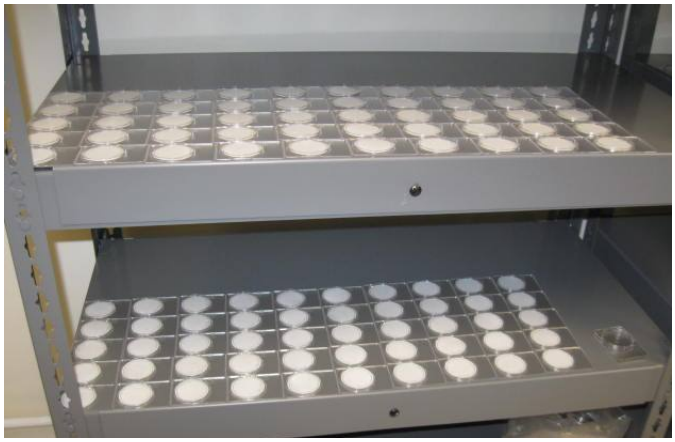


Figure 8-1. PM2.5 Filters Shown on Conditioning Shelves

8.3.10. Allow the filters to condition for 24 hours and then weigh the filters. The 300 mg and 500 mg mass reference standards must be weighed before and after the weighing of the filters in order to bracket the analytical batch. The weight of each of the mass reference standards must not exceed ±3 μg of the certified value for the mass reference standard. The weight values are

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recorded on the Lot Stability Test forms in the rows indicated by "300mg" and "500mg". If any of the standard mass values fail, then the entire batch must be reanalyzed.

8.3.11. Weigh 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. The weight difference between successive 24-hour weighing is calculated by the analyst and entered into the Lot Stability Test Form.

8.3.12. Calculate the Lot Preconditioning 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 days, is used as the minimum pre-conditioning time for all subsequent filters conditioned from the same lot.

NOTE: The filters may continue to lose weight over several days, however the weight loss must be less than 15 μg per day. This meets the criteria described above although does not constitute a stabilization of the filter. In case of continued daily weight loss that is on the order of 15 μg per day, consult the laboratory supervisor. Consecutive weight measurements for a stable filter do not vary typically by more than ± 3μg.

8.4. Pre-Conditioning Lot Exposure Stability Test

A Lot Exposure Blank Stability Test must be performed each time a set of filters is laid out for pre-conditioning. This test is designed to ensure that the filters have been conditioned properly after the pre-conditioning period is complete. The procedure for this test is outlined below.

8.4.1. Obtain a Pre-Sampling Filter Information and Lab Blank Stability Form (See Appendices 20.1.).

8.4.2. Record the Filter Box Number and Lab Analysts in the appropriate blanks.

8.4.3. Record the environmental conditions on the form.

8.4.4. Select 3 filters from those laid out for conditioning.

8.4.5. Record the current date in the appropriate space.

8.4.6. The filter ID numbers are recorded in the column titled "Filter ID" and the date is entered above the column in which the filter weights will be entered on the form.

8.4.7. Weigh the filters according to the section titled "Filter Weighing Procedures." The weights of the filters are recorded on the Pre-Sampling Filter Information and Lab Blank Stability Form next to the appropriate filter ID number.

8.4.8. The three filters are returned to the Millipore Petri-slides, placed back on the conditioning shelf and covered ¾ of the way with the lids.

8.4.9. After 24 hours, the same three filters are weighed again. The weight difference for each of the three filters from the previous weighing is calculated and entered into the Pre-Sampling Filter Information and Lab Blank Stability Form.

8.4.10. Calculate the average for the three weight difference values from the previous form entry. If this average is ≤5 μg, then the remainder of the filters can be weighed.

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8.4.11. If the average value of the weight differences is greater than ±5 μg, then the three filters are placed back into the Petri-slides with the lids covering them by 3/4. After another 24 hours, the three filters are weighed again, in the same manner as before, and the values recorded on the Pre-Sampling Filter Information and Lab Blank Stability Form. The weight difference values between the last two consecutive 24 hour measurements are averaged. If the average value is ≤5 μg, then the remainder of the filters can now be weighed. If the value is still greater than ±5 μg, then this procedure is repeated until the average value between successive readings is ≤5 μg.

8.4.12. Once the 3 test filters are determined to be at the end of the conditioning period, the whole group of filters is then moved from Pre condition status to End condition status in the LIMs. The filters that were laid out are now ready to be weighed.

8.5. 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.

8.5.1. 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.

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

8.5.3. Obtain the Pre-Sampling Filter Information and Lab Blank Stability Form and fill in the # of filters accepted for that box.

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

8.5.5. Cancellation of any rejected filter is to be performed by the laboratory manager or designee.

8.5.6. 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).

NOTE: A filter suitable for sampling will typically have a weight between 350 and 400 mg.

8.6. Filter Pre-Conditioning

Each filter must be conditioned for the specified amount of time set by $PM_{2.5}$ Lot Exposure Blank Filter Stability Test. The conditioning procedure for pre-sampled filters is outlined below. Only filters that pass inspection are conditioned.

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

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

8.6.3. Place the Petri-slides on the Pre-conditioning shelf in rows of 5. For a box of 50 filters there will be 10 rows of five on the shelf.

8.6.4. 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 not placed into Petri-slides on the Pre-conditioning shelf. Use an empty Petri-slide where they would be located as a place holder. Each filter position is maintained to ensure that filters with duplicate or ambiguous ID numbers will be identified.

8.6.5. 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.

8.6.6. The filters laid out for the current exposure lot must be entered into the LIMS as follows in the preceding instructions.

8.6.7. Create New Filter in LIMS by following the outline below:

8.6.7.1. Open the $PM_{2.5}$ Manager in LIMS.

8.6.7.2. Under Filters, select "New Filters".

8.6.7.3. In the "Start Serial Number", type the ID of the first filter of the box that is to be added.

8.6.7.4. In "Number to Add" type in the total number of filters (typically *200*).

8.6.7.5. Type in the Lot # and Exposure #. The Lot and Exposure numbers are used to keep track of associated filters. In rare cases, a given Lot # may have multiple Exposure tests performed, so they must be differentiated.

8.6.7.6. Click "Add".

8.6.7.7. Click "OK" when finished.

8.6.7.8. Start Pre-Conditioning in LIMS:

8.6.7.7.1. Open the $PM_{2.5}$ Manager in LIMS.

8.6.7.7.2. Under Filters, select "Pre".

8.6.7.7.3. Select "Start Conditioning".

8.6.7.7.4. A window will appear listing filters available for pre-conditioning.

8.6.7.7.5. Select "Add All".

8.6.7.7.6. Enter Analyst Name, Date, and Time.

8.6.7.7.7. Click "OK" when finished.

8.6.7.7.8. End Pre-Conditioning in LIMS.

8.6.8. End Pre-Conditioning in LIMS:

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

8.6.9. Once complete, the filter Petri-slides remain tightly closed. The Petri-slides are only opened during conditioning, inspection, weighing, or transfer to/from the magazine cassettes.

8.7. <u>Filter Post-Conditioning</u>

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Once filters are received from the field, they must condition for the specified time set by the Post-Sampling Filter Stability Test (see Appendices 20.5.). The instructions to start and end Post-Conditioning are identical to the Pre-Conditioning procedures provided above. The only difference is that Post is selected instead of Pre in the LIMS.

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

9.1. Clean the weighing surface

9.1.1. The weighing area must be wiped with a Text wipe dampened with Staticide™ before the balance is used each day. This is done to minimize sources of contamination.

9.2. Perform microbalance automatic calibration

9.2.1. Close the draft shield of the microbalance.

9.2.2. Press “Menu” on the balance keypad. The screen will read “CAL Int”.

9.2.3. Press “Re-Zero”.

9.2.4. The balance will then calibrate itself. This process will take approximately 2 minutes.



Figure 9-1. Mettler MT5 Microbalance and Antistatic U-Electrode

9.3. Manual Weighing Filter Procedure

All filters, regardless of their status, are to be weighed in the following manner.

9.3.1. Select a batch of about 10 to 30 filters to weigh.

9.3.2. Open the WinWedge software program and open the PM2.5 Balance method. This will automatically bring up the Excel template for data collection (see Appendices 20.7).

9.3.3. Fill in the Excel template for:

- Analyst Name
- Date (MM/DD/YYYY) and Time – both are automatically added by the instrument.
- File Name – Date sensitive (MMDDYY plus incrementing letter A to Z)
- 300 and 500 mg Standard Mass Values
- Filter 24-Hour Conditioning Values taken directly from the Vaisala software:

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- Temperature Minimum, Maximum, Average, and Standard Deviation
- Humidity Minimum, Maximum, Average, and Standard Deviation

- Lab Blank Values
- Enter the Filter ID numbers

9.3.4. Close the draft shield on the microbalance and zero the balance.

9.3.5. Place the 300 mg Working Standard on the balance pan and close the draft shield. Press the Collect Weight key in the WinWedge program once a stable reading is achieved. The software will automatically put the data in the correct column.

9.3.6. Repeat the process in the previous step for the 500 mg Working Standard.

9.3.7. Open the first Millipore Petri-slide and remove the filter using the appropriate forceps.

9.3.8. Expose the filter between the U-shaped Electrode. All filters are to be exposed to the U-Shaped electrode for a minimum of 30 seconds to eliminate any static charge build-up on the filter.

9.3.9. While the filter is being discharged, close the draft shield on the microbalance and zero the balance.

9.3.10. Once the balance indicates that a stable zero reading has been obtained, remove the filter from the electrode exposure, place it on the microbalance pan, and close the draft shield.

9.3.11. Wait until the balance indicates that a stable reading has been obtained. A stable reading has been obtained once the indicating circle in the top left corner of the balance disappears.

9.3.12. Press the "Collect" Weight key in the data collection program and the mass value will automatically be recorded in the Excel worksheet.

9.3.13. 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: Filters are weighed in batches of 10 - 30 sample filters, 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 Filter Sample (QC Check Parameter)

The first sample filter in each batch of 10-30 filters is designated as the batch duplicate (BD) filter. This filter is the first and last sample filter weighed in each batch. The second weighing of the batch duplicate filter must be ±15µg from the first weight. 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 one of its filters assigned as a lab blank. The lab blank filter is normally the first filter (filters ending in "01" or "51"). If the first filter is damaged, then the next available filter is assigned as the lab blank for that filter box.

9.5.2. When determining the weight of a lab blank, the filter is weighed three successive times with each weight not exceeding ±15µg from the first weight. These values are recorded on the Lab Blank Weight Determination Form (see Appendices 20.1). If either the second or third weight

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is outside of this range, a fourth measurement must be made to replace the invalid value. Use the average value as the lab blank mass.

9.5.3. Once the weight of the Lab Blank is determined, the filter is placed in a Millipore Petri-slide. 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 it is needed. The lab blank must be left in the "Condition" state with the samples until all associated filters have been post-weighed and reported.

9.5.3. Further measurements made with the lab blank in subsequent batches also must not exceed ±15µg of the initial value determined. If subsequent values for a lab blank are more than ±15µg from the previously determined value, it must be re-weighed. This procedure is to be performed twice, as required. If after two additional measurements, the value is still greater than ±15µg from the determined value, begin troubleshooting and consult the lab manager.

9.6. Upload Filter Weight Data into LIMS

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

9.6.1. Open the $PM_{2.5}$ Manager in LIMS.

9.6.2. Under Filters, select "Pre" or "Post" and then select "Weigh".

9.6.3. Under File, select "Open".

9.6.4. Select the Text Tab Delimited File that contains the weights to be uploaded.

9.6.5. Highlight filters and the associated Lab Blank.

9.6.6. Under File, select "Assign Batch ID" (type in a previous batch ID or select New to assign it to the next available number.) Then select "Upload".

NOTE: A batch containing filters from multiple boxes may require more than one Lab Blank. In this instance, multiple batch ID's are assigned.

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 shipment temperature. To achieve this, the filters are placed in cassettes, sequentially stacked in magazines, and packaged in soft-side coolers within the weighing environment. Packaging of the soft side coolers into larger coolers is completed in the shipping room, located just outside the weighing environment.

10.1. Filter Scheduling in LIMS

10.1.1. Requests for filters are received from clients and are scheduled by the Laboratory Supervisor (or designee) in the LIMS. Typically, each of the clients will submit a monthly filter request by e-mail one month in advance.

10.1.2. Filter shipments are packed according to the requests received.

10.2. LIMS Program Instructions for Filter Packing

10.2.1. Open the $PM_{2.5}$ Manager in LIMS.

10.2.2. Under Filter, select "Send".

10.2.3. Select the Schedule for a Customer.

10.2.4. 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.

10.2.5. 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). A window will appear prompting for the cassette ID to be entered. Type in the cassette ID and click OK when finished. This procedure is done until all the filters for that magazine have been selected and their cassette ID entered. If another magazine is needed to fill the request, repeat steps 4 and 5 until the request is complete.

10.2.6. Once a request is complete, select the Analyst name at the top of the screen and select the Finished button.

10.2.7. 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 order of the filters in the magazines agrees with the order specified on the magazine loading sheet and that each of the ID numbers (magazine, cassette and filter) 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 they may damage the filters above or below them.

10.3. Shipping Documentation

All filter shipments must include shipping documentation.

10.3.1. At the top of the screen are options to print address labels and magazine chain-of-custody.

10.3.2. Once the Finish button is selected, there will be prompts to print the address label, chain-of-custody form, and a magazine loading sheet. De-select the address label and print the Magazine Chain-of-Custody form (see Appendices 20.7.).

10.3.3. The lab analyst must electronically sign and include the Greyhound shipping invoice on the Magazine Chain-of-Custody form and send to the customer via e-mail and save a copy to the appropriate file on the network. (A paper copy is not necessary if an electronic copy is permanently maintained in the $PM_{2.5}$ network directory.)

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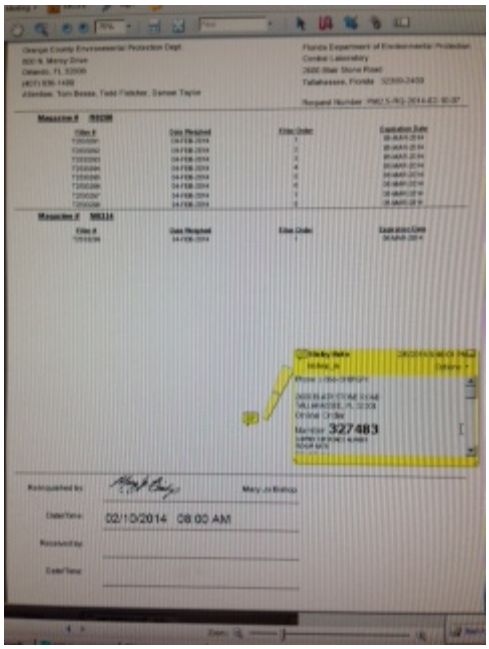


Figure 10-1. Magazine Electronic Custody Form

10.4. Cassette Loading Instructions

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

10.4.1. Place the cassette bottom on the workbench.

10.4.2. Place the cassette screen, with the screen ID down, in the cassette bottom.

10.4.3. Remove the filter from a Millipore Petri-Slide with the appropriate forceps.

10.4.4. Place the filter, with the ID number facing upward, on the cassette screen using non-metallic forceps labeled “packing.”

10.4.5. Finally, place the cassette top on the filter. Press firmly to ensure the cassette top and bottom makes a tight seal.

10.5. Magazine Loading Instructions

10.5.1. Cassettes are packed in serialized and bar-coded magazines.

10.5.2. 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.

10.5.3. 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 then 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.

10.5.4. Place the orange cap on the magazine after all the cassettes are loaded in the magazine.

10.5.5. Place a yellow or black protective cap on the magazine pump coupling.

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10.5.6. Pack the magazine in a soft-side cooler. Store the soft-side coolers in the weighing environment until ready for shipment in the large Igloo coolers.

10.6. Cooler Packing for Shipment

10.6.1. Move the packed soft-side coolers from the weighing lab to the main shipping room.

10.6.2. Obtain a large Igloo cooler.

10.6.3. Place 7-9 large blue ice packs and 2 small blue ice packs in the Igloo cooler.

10.6.4. Place the soft-side cooler in the Igloo cooler according to the diagram.

10.6.5. Zip the soft-side cooler closed.

10.6.6. The analyst packing the cooler must electronically sign and date the form on the space provided.

10.6.7. Close the Igloo cooler and secure the lid with packaging tape on both ends.

10.6.8. Label the cooler on each surface with the name of the district or local customer receiving the shipment. (The coolers may be marked with permanent site identification.)

10.7. Shipping a Cooler

Coolers are routinely shipped by the Air Division courier or by commercial carrier. The commercial carrier, currently Greyhound, is used to deliver to the coolers and FedEx for emergency or unscheduled shipments. Procedures for using the Greyhound carrier shipping station are provided in the Air Division's Standard Operating Procedure **DEP 03-14 (REV NO 0:12/11)** and are summarized below:

10.7.1. Turn on the Carrier computer monitor. (Only the monitor is turned on because the computer and printer must always be on to allow the Carrier to update their information as necessary.) The Carrier Online screen will appear.

10.7.2. Make sure the printer is on and the paper is ready.

10.7.3. Select "Shipping" from the top left corner.

10.7.4. "Set Pickup Date" will appear. Choose the current date and press "OK."

10.7.5. Choose Customer ID.

10.7.6. Place cooler on a zeroed balance.

10.7.7. Wait for the final weight to appear and select "Ship It.". The shipping label will print. A Carrier pick-up label will be printed and is should be attached to the cooler for shipping.

11. FILTER RECEIVING PROCEDURES AND NON-CONFORMANCE REPORTING

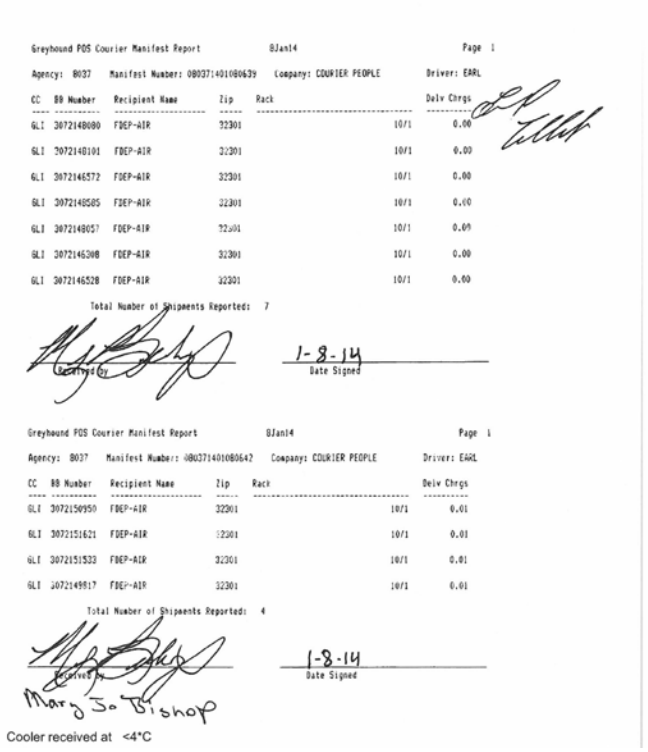
11.1. Cooler Inspection

11.1.1. When a cooler is received from the field, it must be inspected to make sure it is sealed properly.

11.1.2. The analyst breaks the seal on the cooler.

11.1.3. Measure the current temperature inside the softside filter-storage cooler using an IR electronic thermometer and record the temperature on the received Manifest documents. The temperature must be ≤ 4 °C in order to have 30 days between the sample collection (defined as the time sample is collected for refrigeration) and the start of the post-collection conditioning period.

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Greyhound POS Courier Manifest Report BJan14 Page 1

Agency: 8037 Manifest Number: 08037140108063 9 Company: COURIER PEOPLE Driver: EARL

CC	BB Number	Recipient Name	Zip	Rack	Delv Chrgs
GLI	3072148080	FDEP-AIR	32301	10/1	0.00
GLI	3072148101	FDEP-AIR	32301	10/1	0.00
GLI	3072146572	FDEP-AIR	32301	10/1	0.00
GLI	3072148585	FDEP-AIR	32301	10/1	0.00
GLI	3072148057	FDEP-AIR	32301	10/1	0.00
GLI	3072146308	FDEP-AIR	32301	10/1	0.00
GLI	3072146528	FDEP-AIR	32301	10/1	0.00

Total Number of Shipments Reported: 7

Relinquished By 1-8-14 Date Signed

Greyhound POS Courier Manifest Report BJan14 Page 1

Agency: 8037 Manifest Number: 08037140108064 2 Company: COURIER PEOPLE Driver: EARL

CC	BB Number	Recipient Name	Zip	Rack	Delv Chrgs
GLI	3072150950	FDEP-AIR	32301	10/1	0.01
GLI	3072151621	FDEP-AIR	32301	10/1	0.01
GLI	3072151533	FDEP-AIR	32301	10/1	0.01
GLI	3072149917	FDEP-AIR	32301	10/1	0.01

Total Number of Shipments Reported: 4

Relinquished By 1-8-14 Date Signed

Mary Jo Bishop

Cooler received at <4°C

Figure 11-1. Example of a Carrier Shipping Manifest

11.1.4. If a cooler is received above 4 ºC, use the Filter Holding Time Temperature Sliding Scale Calculation to determine the holding time for post weighing. The calculation is:

$$\mathbf{D = 34 - T_{rec}}$$

D = days of holding time from the end of the sampling period (were $10 \leq \mathbf{D} \leq 30$)

$\mathbf{T_{rec}}$ **= T**emperature of the sample upon receipt

This scale provides a more conservative holding time than the EPA guidance which uses an average shipping temperature. The receiving temperature will be equal or higher than the average shipping temperature, so the calculation will provide a slightly more conservative holding time. Refer to *EPA Additional Guidance on PM2.5 Cassette Handling and Transportation* (Jan. 2000) for additional details.

11.1.5. Temperatures greater than 25ºC will result in data qualification with the qualifier SIS.

11.1.6. Verify that the cooler temperatures are written on the Custody documents.

11.1.7. Inspect the cooler condition and make note of any damage or irregularities on the Greyhound manifest.

11.1.8. The signed Greyhound manifest, documented cooler receipt temperatures, and any irregularities are scanned into a PDF file and stored in the PM2.5 network drive under the Greyhound Custody directory.

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11.2. Cooler Paperwork and Contents

11.2.1. Remove and inspect the magazines from the cooler.

11.2.2. Place the magazines and paperwork together on the cabinets in the interlock room leading to the weighing lab. This interlock room is conditioned the same as the lab and allows the magazines to come to equilibrium in a controlled environment for at least 4 hours before unpacking. Typically the magazines are received in the afternoon and unpacked the following morning.

11.4. Non-Conformance Reporting

11.4.1. Non-conformances (NCRs) are recorded on the Greyhound manifest and in the LIMS Non-Conformance module. It is important to note abnormalities associated with shipments (i.e. broken or damaged magazines, missing items, improper packing or paperwork).

11.4.2. These records will be used to add appropriate qualifiers when reporting the data in the LIMS.

11.4.3. See **SOP LB-002** for additional information on NCR reporting within the laboratory.

11.5. Filter Removal from Magazines

11.5.1. Once the magazines are at room temperature, move them into the conditioned weighing environment.

11.5.2. Select the first sampling site filters to receive into the lab and remove the orange cap from one of the magazines.

11.5.3. 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.

11.5.4. Gently open the cassette to expose the filter. Visually inspect the filter for damage. Make a note on the receiving sheets 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 the following:

- Filter damage observed upon receipt (pinhole)……. SIS
- Filter upside down in magazine upon receipt in lab…….. SIS
- Filter was not received in magazine…….. SIS
- Foreign object on filter surface (particle, fiber)……..... SIS
- Laboratory accident or damage……..…….. LAC
- Filter damaged during shipment……..... PSD
- Post-weighed more than 30 days after sampling…….HTE
- Holding time in field exceeded 177 hours……. HTE
- Sampled more than 30 days after pre-weight…….. HTE
- Filter voided by customer…….. VOD
- Exceeded temperature parameters in lab……... FLT
- Exceeded humidity parameters in lab……..... FLH

11.5.5. Using the Filter Archival Chart, list the customer name, the order # of the filter, the filter ID, the magazine ID, and the date of shipment receipt (see Appendices 20.5.).

11.5.6. Remove the filter with the appropriate forceps taking extra precautions not to disturb the filter media and the particulate matter on the filter.

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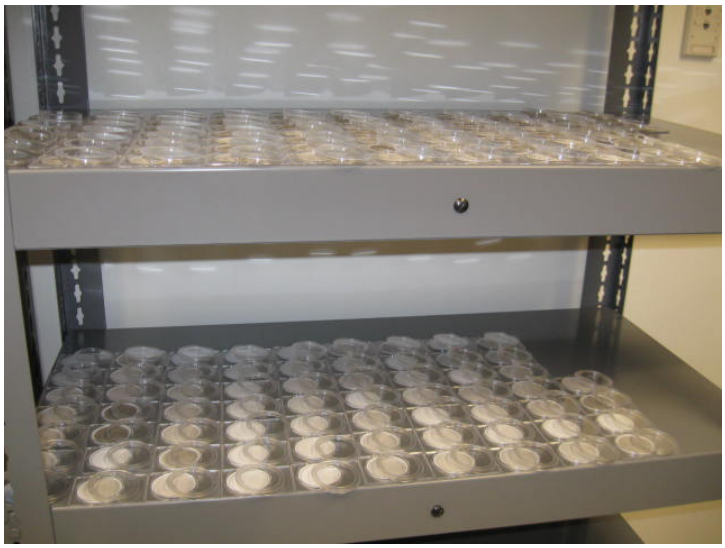


Figure 11-1. PM2.5 Filters Conditioning on Shelves

11.5.7. Place the filter, ID number facing up, in a Millipore Petri-slide.

11.5.8. The filter ID is visible through the top of the Millipore Petri-slide.

11.5.9. Place the filter on the appropriate conditioning shelf with the slide cover ¾ over the filter.

NOTE: The order of the filters on the archival sheet mimics the order of the filters on the shelves.

11.5.10. Repeat the above steps until all filters are removed from the magazine.

11.6. Receive Shipments into the LIMS

11.6.1. Open the $PM_{2.5}$ Manager in LIMS.

11.6.2. Under Filter, select "Receive".

11.6.3. Select a Customer and then choose "Next".

11.6.4. Select "Load".

11.6.5. Select the appropriate customer's field sheet (which is saved in Excel format on the server).

11.6.6. Select the date in which the filters arrived into the lab.

11.6.7. Select "Upload" and the filters will automatically upload.

11.6.8. Check the list to ensure the correct filter ID's and magazines were selected.

11.6.9. Select "Analyst ID"

11.6.10. Select "Finish" to upload filter to LIMS

NOTE: After the filters are received, they must begin the post-conditioning process. This is identical to the preconditioning procedure outlined in the Filter Pre-Conditioning section.

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.

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12.1.1. Primary Mass Standard Annual Certifications – Are purchased new each year or are recertified by the Florida Dept. of Agriculture or Rice Lake Weighing Systems.

12.1.2. Primary Vaisala Humidity and Temperature Units – Are certified annually by Vaisala Inc.

12.1.3. Certification documents are stored on the computer data server and paper 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 Appendices 20.8).

12.2.2. Inspect each mass reference standard for cleanliness and wear. The standard mass weights may be cleaned by rinsing with alcohol and air dried on a Text Wipe.

12.2.3. Fill out the form with the Analyst Name, Date, and Time. The Humidity and Temperature readings for the weighing room are also recorded.

12.2.4. Weigh the 500 mg weights following the order listed on the form. Then proceed with the 300 mg weight.

12.2.5. Submit the calibration verification to the laboratory supervisor. Maintain records in the $PM_{2.5}$ lab calibration and verification folder.

12.2.6. Certification documents are stored on the computer data server.

12.3. Annual Microbalance Maintenance Service and Calibration

12.3.1 A Mettler-Toledo technician will complete this service annually or more often if required.

12.3.2. 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. Also refer to SOP **VO-001** for additional QA/QC details.

13.1. Conditioning Environment Records

The average, minimum, maximum, and standard deviation of the temperature and relative humidity values are recorded with each weighing session. The conditioning room 24-hour average temperature must be 20-23 °C and the Relative Humidity (RH) in the range of 30-40 %. The following temperature and humidity checks are also performed by the LIMS. Permanent digital records of the temperature and humidity are maintained.

13.1.1. Verify and record the preceding 24-hour average temperature (20-23 °C) with a variability not to exceed 2°C standard deviation.

13.1.2. Verify and record the preceding 24-hour average Relative Humidity (30-40 % RH) with a variability not to exceed 5% RH standard deviation.

13.1.3. Verify that the difference in the Pre- and Post- relative humidity does not exceed ±5 %.

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13.1.4. Use Table 13-1 for LIMs flagging of holding times and temperature/humidity control. The instrument significant figures are rounded to those given in 40 CFR Part 50, Appendix L. For example, for the purpose of assessing the need for data qualifiers, a temperature reading of 19.61 °C rounds to 20°C (2 significant figures) or a relative humidity difference of 5.3% rounds to 5% (1 significant figure).

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 20-23°C	FLT	Lab temperature average criterion exceeded.
Lab Temperature Control 24-hour Std. Dev. ≤ 2°C	FLT	Lab temperature 24-hour Std. Dev. criterion exceeded.
Lab Conditioning Humidity Avg. 30-40% RH	FLH	Lab humidity average criterion exceeded.
Lab Conditioning Humidity 24-hour Std. Dev. ≤ 5%	FLH	Lab humidity 24-hour Std. Dev. criterion exceeded.
Pre-Post-Sample Conditioning Humidity Difference does not exceed ± 5% RH	FLH	Pre-/Post-Sample Humidity Average Difference of ± 5 % RH exceeded.

13.2. Lot Filter Exposure Test

The Lot Exposure Test filters are 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 Exposure Test determines the required conditioning time for the specific lot of filters.

13.3. Lab Filter Blanks

Lab filter 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 pre-conditioned.

NOTE: The laboratory does not verify the frequency or mass of Field Blanks (FB). However the FB results are checked to verify that the delta does not exceed ±30 µg when denoted as such on field sheets. If the information is available on the field sheets, then the Field Blank information is downloaded into the LIMS electronically and verified in the data review process. Field customers are notified for values exceeding the 30 µg specification.

13.4. Working Standard QC checks

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Mass references of 300 mg and 500 mg are weighed at the beginning and end of each weighing session and must not exceed ±3 µg. Mass reference standard values are recorded on the Excel weigh spreadsheets with each weighing session.

13.5. Batch Duplicate Filter Weighing

A duplicate filter is weighed with each batch of 10 filters. The first filter of any weighing session 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

14.1.1. Upon completion of filter sampling, post-weighing and reporting, the filters are stored in Millipore Petri-slides and labeled with the filter ID number.

14.1.2. Filters are stored in numerical order in Petri-slide boxes.

14.1.3. When all the filters have been post-weighed from a box of 50 filters, the associated Lab Blank must be placed in the Petri-slide box. If a filter is missing because it was cancelled earlier in the process, place an empty Petri-slide in its place.

14.1.4. 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 0-4°C ±2 °C.

14.1.5. 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. The storage area for the second and third year must be free from excessive heat, humidity, and contamination.

14.2. Data Records Maintenance and Archival

14.2.1. Store all paper and electronic records for a minimum of five years.

14.2.2. The $PM_{2.5}$ data are stored in MS Excel, Adobe, and Text Tab-delimited sheets with file names composed of six numbers and one letter. For example, for the file name “041513B”, the numbers represent the date April 15, 2013, and the letter denotes it is the second file created that day.

14.2.3. The Excel and Adobe spreadsheets containing the Pre- and Post-Weight data are saved as Document and 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.

14.2.4. The electronic data is archived by year/month in the $PM_{2.5}$ directory on the TLHLAB3 server.

14.2.5. 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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File Name: 030314E

PRE/POST:	POST	Humidity:		Temperature:						
		24 h Avg:	33.21	24 h Avg:	20.66	HINT: Ctrl 0 to stamp date/time				
		Minimum:	31.95	Minimum:	20.42					
Analyst:	COTTON_K	Maximum:	34.47	Maximum:	20.91					
Balance ID:	A					MR	Mass reference - these go to mass reference QC table			
300 mg:	300.001					SF	Single Filter			
500 mg:	500.002					LB	Lab Blank - these go to the "filter QC table"			
LAB BLANK						BD	Batch Duplicate - these go to the "filter QC table"			

Filter ID	Filter Type	Weight	Date/Time	Difference	Acc/Rej	Batch ID	Lab Blanks	Weight	Qualifiers	Comments
300mg	MR	300.004	03/03/2014 12:00	0.003	Accept	A	T2532951	390.487		
500 mg	MR	500.005	03/03/2014 12:01	0.003	Accept	A	T2533101	371.968		
T2532971	SF	379.564	03/03/2014 12:02		Accept	A	T2533501	386.732		
T2532972	SF	375.84	03/03/2014 12:03		Accept	A				
T2532976	SF	377.487	03/03/2014 12:04		Accept	A				
T2532977	SF	397.531	03/03/2014 12:05		Accept	A				
T2532978	SF	385.511	03/03/2014 12:06		Accept	A				
T2532979	SF	393.115	03/03/2014 12:07		Accept	A				
T2532984	SF	395.019	03/03/2014 12:08		Accept	A				
T2532985	SF	383.344	03/03/2014 12:09		Accept	A				
T2532986	SF	389.824	03/03/2014 12:10		Accept	A				
T2532987	SF	398.306	03/03/2014 12:11		Accept	A				
300mg	MR	300.004	03/03/2014 12:12	0.003	Accept	A				
500 mg	MR	500.004	03/03/2014 12:13	0.002	Accept	A				
T2532988	SF	376.312	03/03/2014 12:14		Accept	A				
T2533144	SF	389.743	03/03/2014 12:15		Accept	A				
T2533145	SF	374.978	03/03/2014 12:16		Accept	A				
T2533146	SF	375.724	03/03/2014 12:17		Accept	A				
T2533147	SF	392.869	03/03/2014 12:18		Accept	A				
T2533148	SF	372.282	03/03/2014 12:19		Accept	A				
T2533149	SF	377.179	03/03/2014 12:20		Accept	A				
T2533150	SF	372.569	03/03/2014 12:21		Accept	A				
T2533502	SF	389.551	03/03/2014 12:22		Accept	A				
T2533531	SF	390.835	03/03/2014 12:23		Accept	A				
300mg	MR	300.002	03/03/2014 12:24	0.001	Accept	A				
500 mg	MR	500.005	03/03/2014 12:25	0.003	Accept	A				
T2533532	SF	385.676	03/03/2014 12:26		Accept	A				
T2533534	SF	385.77	03/03/2014 12:27		Accept	A				
T2533535	SF	388.504	03/03/2014 12:28		Accept	A				
T2532951	LB	390.488	03/03/2014 12:29	0.001	Accept	A				
T2533101	LB	371.968	03/03/2014 12:30	0	Accept	A				
T2533501	LB	386.731	03/03/2014 12:31	-0.001	Accept	A				
T2532971	BD	379.563	03/03/2014 12:32	-0.001	Accept	A				
300mg	MR	300.002	03/03/2014 12:33	0.001	Accept	A				
500 mg	MR	500.004	03/03/2014 12:34	0.002	Accept	A				

Figure 14-1. Example of an Electronic Weighing Form

14.3. Log Book and Other Supporting Document Storage

14.3.1. 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.

14.3.2. 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.

14.3.3. Weekly Filter Archival Chart - 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 Archival Charts are electronically stored in the $PM_{2.5}$ directory on a network server.

14.3.4. LIMS Electronic Logbook – The PM2.5 laboratory maintains an electronic notebook for recording issues regarding the laboratory equipment maintenance, lab condition controls, cleaning activities, and other PM2.5 laboratory activities.

15. DATA AUTHORIZATION AND REPORTING

Review and authorization of data are duties performed by the Laboratory Administrator (or designee) using the following procedures:

15.1. Data Review and Authorization

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

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

15.1.5. 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.

15.1.6. The LIMS is designed to automatically add qualifiers and comments (see Table 13-1).

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

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

15.1.9. Then highlight the filters and select Authorize Selected.

15.2. PM2.5 Analysis Reports

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

15.2.2. Under “Utilities”, select “Build Final Reports”.

15.2.3. Select “New Reports”.

15.2.4. Choose a “Customer”. All filters ready for reporting will appear in the Available Filters column.

15.2.5. Choose the filters that are to be reported by highlighting each one.

15.2.6. Then select “Add”.

15.2.7. 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.

15.2.8. Once a filter is reported, it is no longer listed as an available filter to report.

15.2.9. 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.

15.2.10. 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

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.

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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. Please refer to the **SOP OG-001** for details on *Lab Safety and Waste Management Practices*.

16.1. 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.

16.2. Use care when using solvents. Use gloves when recommended and wash hands thoroughly after use.

16.3. The lab is located within a facility which has a Sonitrol Security System. Training on the operation of the security system is provided by the facility management.

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 on a set of standard weights and demonstrating that the instrumentation and personnel can meet all of the method criteria.

17.1.1. Record the Temperature and Humidity.

17.1.2. Auto-calibrate the MT5 microbalance.

17.1.3. Tare the balance initially and between each measurement.

17.1.4. Measure the mass of the 300 µg and 500 µg standard weights a minimum of 4 times each and record the value in the WinWedge software program.

17.1.5. Use the software program to determine the accuracy and precision of the mass measurements to insure that the values are in compliance of the method requirements.

17.1.5. Repeat this process on every analytical balance that will be used to collect data and for every new analyst.

17.2. Personnel Qualifications

All personnel who generate reportable data must first successfully perform a Demonstration of Capability 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.

17.2.1. Demonstrate the ability to operate the MT5 Microbalances and anti-static devices. This is demonstrated by performing an initial Demonstration of Capability and annual Performance Evaluations, both of which involve taking multiple measurements of standard weights. The

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acceptance criteria for the accuracy and precision of the measurements are established by the method.

17.2.2. Demonstrate ability to handle sample filters within the laboratory.

17.2.3. Demonstrate ability to perform calibration procedures.

17.2.4. Demonstrate ability to maintain a clean working environment.

17.2.5. Pass the Annual Performance Evaluation. This annual performance evaluation is documented in the LIMS Training module.

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, November 1988.

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 MT5 Microbalance and Electronic U-electrode Operation Manuals.

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

18.9. Florida Department of Environmental Protection, Standard Operating Procedure **LB-002** Non-Conformance Reporting System.

18.10. Florida Department of Environmental Protection, Standard Operating Procedure **LB-027** SOP for Reporting Qualified Data.

18.11. Florida Department of Environmental Protection, Standard Operating Procedure **LB-024** Significant Figures: Policy of the DEP Bureau of Laboratories.

18.12. Florida Department of Environmental Protection, Standard Operating Procedure **VO-001**, Quality Assurance Documentation and Quality Control Measures in the Volatile Organics Laboratory.

18.13. Florida Department of Environmental Protection, Standard Operating Procedure **VO-010**, Data Review/Authorization Procedures for the Volatile Organic Laboratory.

18.14. Florida Department of Environmental Protection, Standard Operating Procedure **OG-001** Safety and Waste Management Practices in the Organic Analysis Groups.

18.15. Florida Department of Environmental Protection, Bureau of Laboratories, "Comprehensive Quality Assurance Plan" (2014).

18.16. 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

The following revisions were made to SOP revision PM-001-3.14 from the prior version PM-001-3.13:

1. Incremented the SOP number.
2. Added signature page.
3. Added Table 6-1 for lab maintenance schedule.
4. Clearly described the requirements for recording cooler temperatures upon receipt into the lab.
5. Added Table 8-1 to describe appropriate delay times for weighing after a temperature or humidity transient.
6. Added standard deviation requirement for percent relative humidity.
7. Added Table 13-1 which describes and defines automated LIMS qualifiers.
8. Corrected a few minor typos and clarifications.
9. Updated References.
10. Added Automated LIMS QA Verification Check Table to the appendix.

20. APPENDICES

20.1. Pre-sampling Filter Information and Lab Blank Stability Test Form:

Pre-Sampling Filter Information and Stability Test

Analyst Initials: KC Date Inspected: 01/03/05

Lot Number	Filter ID Number Ranges From	To	Number Accepted	LIMS Exposure #
2085027	T3300126	T3300150	25	193
2085027	T3300151	T3300200	50	193
2085027	T3300201	T3300250	50	193
2085027	T3300251	T3300300	50	193
4251001	P4032326	P4032350	25	280
4251001	P4032351	P4032375	25	280

Comments: _____

	Date 01/04/05		Date 01/05/05		Date	
	Day 1	Difference	Day 2	Difference	Day 3	Difference
QC1 100mg	99.995		99.994			
QC2 200mg	199.998		199.998			
T3298801	146.477		146.477			
T3298851	141.555		141.553			
T3298901	144.589		144.590			
QC1 100mg	99.993		99.993			
QC2 200mg	199.998		199.998			

	11/16/04	Temperature	Humidity
DAY 1	Average	20.51	34.0
	Minimum	19.78	32.9
	Maximum	20.79	35.1

	11/17/04	Temperature	Humidity
DAY 2	Average	20.54	34.0
	Minimum	19.75	33.7
	Maximum	21.31	35.5

		Temperature	Humidity
DAY 3	Average		
	Minimum		
	Maximum		

Malday/10-25-04 FDEP SOP PM-001

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20.2. Lot Blank Filter Stability Test:

PM2.5 Lot Blank Filter 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												

DAY 1	02/28/13	Temperature	Humidity
	Average		
	Minimum		
	Maximum		

DAY 2	03/01/13	Temperature	Humidity
	Average		
	Minimum		
	Maximum		

DAY 3		Temperature	Humidity
	Average		
	Minimum		
	Maximum		

DAY 4		Temperature	Humidity
	Average		
	Minimum		
	Maximum		

DAY 5		Temperature	Humidity
	Average		
	Minimum		
	Maximum		

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20.3 Field Filter Log Forms:

Duval County Filter Log - Mandarin Road

INCOMING REC. DATE	INCOMING MAG SER #	FILT. #	Operator In	FILTER RUN DATA START DATE	START TIME	STOP DATE	STOP TIME	FILTER STORAGE DATA FILTER REFRIG. DATE	FILTER REFRIG. TIME	REFRIG TEMP (° C) MIN	REFRIG TEMP (° C) MAX	OUTGOING SHIP DATE	OUTGOING MAG #	FILTER COMMENTS	Operator Out
12/31/13	M0046	T2532449	AH	01/14/14	00:00	01/15/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0046		AH
01/07/14	M0157	T2532607	AH	01/15/14	00:00	01/15/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0046	FIELD BLANK	AH
01/07/14	M0157	T2532608	AH	01/15/14	00:00	01/16/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0046		AH
01/07/14	M0157	T2532609	AH	01/16/14	00:00	01/17/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0046		AH
01/07/14	M0157	T2532610	AH	01/17/14	00:00	01/18/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0046		AH
01/07/14	M0157	T2532611	AH	01/18/14	00:00	01/19/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0046		AH
01/07/14	M0157	T2532612	AH	01/19/14	00:00	01/20/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0046		AH
01/07/14	M0157	T2532613	AH	01/20/14	00:00	01/21/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0046		AH
12/31/13	M0366	T2532458	AH	01/14/14	00:00	01/15/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0366		AH
01/07/14	M0239	T2532615	AH	01/15/14	00:00	01/15/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0366	FIELD BLANK	AH
01/07/14	M0239	T2532616	AH	01/15/14	00:00	01/16/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0366		AH
01/07/14	M0239	T2532617	AH	01/16/14	00:00	01/17/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0366		AH
01/07/14	M0239	T2532618	AH	01/17/14	00:00	01/18/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0366		AH
01/07/14	M0239	T2532619	AH	01/18/14	00:00	01/19/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0366		AH
01/07/14	M0239	T2532620	AH	01/19/14	00:00	01/20/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0366		AH
01/07/14	M0239	T2532621	AH	01/20/14	00:00	01/21/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0366		AH
12/31/13	M0392	T2532466	AH	01/14/14	00:00	01/15/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0392		AH
01/07/14	M0368	T2532623	AH	01/15/14	00:00	01/15/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0392	FIELD BLANK	AH
01/07/14	M0368	T2532624	AH	01/15/14	00:00	01/16/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0392		AH
01/07/14	M0368	T2532625	AH	01/16/14	00:00	01/17/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0392		AH
01/07/14	M0368	T2532626	AH	01/17/14	00:00	01/18/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0392		AH
01/07/14	M0368	T2532627	AH	01/18/14	00:00	01/19/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0392		AH
01/07/14	M0368	T2532628	AH	01/19/14	00:00	01/20/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0392		AH
01/07/14	M0368	T2532629	AH	01/20/14	00:00	01/21/14	00:00	01/21/14	14:30	-4	5	01/21/14	M0392		AH

Received by: Mary Jo Bishop

Digitally signed by Mary Jo Bishop
DN: cn=Mary Jo Bishop, o=Florida Department of Environmental Protection, ou, email=maryjo.bishop@dep.state.fl.us, c=US
Date: 2014.01.27 20:14:51 -06'00'

BROWARD COUNTY DPEP **Super Site 31**

FILTER LOG File # 08/02 QFL.

FRONT A QUARTZ SN 20125

DATE RCVD	FILTER NUMBER	MAGAZINE NUMBER	FILTER RUN DATA On Date	On Time	Off Date	Off Time	FILTER STORAGE DATA Filter in refrig. Date	Time	Refrig T (°C) Max	Min	Date Shipped	Collection Mag Number
07/05/05	Q 6848	M-0424	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0424
07/12/05	Q 6921	M-0416	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0424
07/12/05	Q 6922	M-0416		FIELD BLANK			########	15:30	7.0	-4.0	########	M-0424
07/12/05	Q 6923	M-0416	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0424
07/12/05	Q 6924	M-0416	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0424
07/12/05	Q 6925	M-0416	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0424
07/12/05	Q 6926	M-0416	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0424
07/12/05	Q 6927	M-0416	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0424
									*			
* PLease see the note on Broward County s												
07/12/05	Q 6928	M-0416										

FILTER LOG

REAR B QUARTZ

DATE RCVD	FILTER NUMBER	MAGAZINE NUMBER	FILTER RUN DATA On Date	On Time	Off Date	Off Time	FILTER STORAGE DATA Filter in refrig. Date	Time	Refrig T (°C) Max	Min	Date Shipped	Collection Mag Number
07/05/05	Q 6856	M-0448	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0448
07/12/05	Q 6929	M-0437	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0448
07/12/05	Q 6930	M-0437		FIELD BLANK			########	15:30	7.0	-4.0	########	M-0448
07/12/05	Q 6931	M-0437	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0448
07/12/05	Q 6932	M-0437	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0448
07/12/05	Q 6933	M-0437	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0448
07/12/05	Q 6934	M-0437	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0448
07/12/05	Q 6935	M-0437	########	07:00	########	07:00	########	15:30	7.0	-4.0	########	M-0448
07/12/05	Q 6936	M-0437										

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20.4 Post-Sampling Filter Stability Test:

City of Jacksonville Environmental & Compliance Dept.
214 N. Hogan Street
Suite 500
Jacksonville, FL 32202
(904) 255-7207
Attention: Alvin Humphrey

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

Request Number: PM2.5-RQ-2014-02-03-06

Magazine # M0191

Filter #	Date Weighed	Filter Order	Expiration Date
T2533226	27-JAN-2014	1	26-FEB-2014
T2533227	28-JAN-2014	2	27-FEB-2014
T2533228	28-JAN-2014	3	27-FEB-2014
T2533229	28-JAN-2014	4	27-FEB-2014
T2533230	28-JAN-2014	5	27-FEB-2014
T2533231	28-JAN-2014	6	27-FEB-2014
T2533232	28-JAN-2014	7	27-FEB-2014
T2533233	28-JAN-2014	8	27-FEB-2014

Magazine # M0304

Filter #	Date Weighed	Filter Order	Expiration Date
T2533234	28-JAN-2014	1	27-FEB-2014
T2533235	28-JAN-2014	2	27-FEB-2014
T2533236	28-JAN-2014	3	27-FEB-2014
T2533237	28-JAN-2014	4	27-FEB-2014
T2533238	28-JAN-2014	5	27-FEB-2014
T2533239	28-JAN-2014	6	27-FEB-2014
T2533240	28-JAN-2014	7	27-FEB-2014
T2533241	28-JAN-2014	8	27-FEB-2014

Magazine # M0431

Filter #	Date Weighed	Filter Order	Expiration Date
T2533242	28-JAN-2014	1	27-FEB-2014
T2533243	28-JAN-2014	2	27-FEB-2014
T2533244	28-JAN-2014	3	27-FEB-2014
T2533245	28-JAN-2014	4	27-FEB-2014
T2533246	28-JAN-2014	5	27-FEB-2014
T2533247	28-JAN-2014	6	27-FEB-2014
T2533248	28-JAN-2014	7	27-FEB-2014
T2533249	28-JAN-2014	8	27-FEB-2014



Relinquished by: Mary Jo Bishop

Date/Time: 02/03/2014 08:00 AM

Received by: V. Alvin Humphrey

Digitally signed by V. Alvin Humphrey
DN: cn=V. Alvin Humphrey, o=City of Jacksonville, ou=ESD, email=vahumphrey@coj.net, c=US
Date: 2014.02.07 07:47:45 -05'00'

Date/Time:

20.5. Filter Archival Chart

PM 2.5 Laboratory*
Chart for Post Sampled Filters

Analyst Initials_____
Shelf ID_____
Date completed_____

☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966
☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966
☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966
☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966
☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966
☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966
☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966
☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966
☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966
☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966	☐ T966

*Includes customer ID
Magazine number
Filter ID number, order received into the lab and NCRS.

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20.6 Magazine 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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20.7 PM2.5 Data Acquisition Template

Analyst: BRAKE_M
Date: 101701
Status:

File Name: 101701A

Lab Blank ID #

PRE/POST:	POST	Humidity:		Temperature:						
		24 h Avg:	34.1	24 h Avg:	20.6	HINT: Ctrl D to stamp date/time				
		Minimum:	32.9	Minimum:	19.5					
Analyst:	BRAKE_M	Maximum:	35.2	Maximum:	20.96					
Balance ID:	A					MR	Mass reference - these go to mass reference QC table			
100 mg:	99.994					SF	Single Filter			
200 mg:	199.999					LB	Lab Blank - these go to the "filter QC table"			
LAB BLANK						BD	Batch Duplicate - these go to the "filter QC table"			

Filter ID	Filter Type	Weight	Date/Time	Difference	Acc/Rej	Batch ID	Lab Blanks	Weight	Qualifiers	Comments
100 mg	MR	99.994	101701 8:32:40	0	Accept	A	T1244151	147.312		
200 mg	MR	200	101701 8:32:41	0.001	Accept	A				
T1244157	SF	144.08	101701 8:32:42		Accept	A				
T1244158	SF	146.2	101701 8:32:43		Accept	A				
T1244159	SF	144.847	101701 8:32:44		Accept	A				
T1244161	SF	145.414	101701 8:32:45		Accept	A				
T1244162	SF	145.766	101701 8:32:46		Accept	A				
T1244163	SF	146.038	101701 8:32:47		Accept	A				
T1244164	SF	143.353	101701 8:32:48		Accept	A				
T1244174	SF	152.122	101701 8:32:49		Accept	A				
T1244175	SF	151.149	101701 8:32:50		Accept	A				
T1244176	SF	140.251	101701 8:32:51		Accept	A				
T1244177	SF	140.91	101701 8:32:52		Accept	A				
T1244151	LB	147.318	101701 8:32:53	0.006	Accept	A				
T1244157	BD	144.981	101701 8:32:54	0.001	Accept	A				
100 mg	MR	99.994	101701 8:32:55	0	Accept	A				
200 mg	MR	200.001	101701 8:32:56	0.002	Accept	A				

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20.8. Quarterly Standard Verification Form for Mass References:

Florida Department of Environmental Protection

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

Balance ID: A-MT5 , S/N 1117481501 **Test No.:** 129 **Date:** March 12,2015 **Time:** 9:00 AM

Analyst : M. Bishop

Working Standard ID:

300 mg (W300)	S/N 33956	Nw = 300.001 mg Cw = calculated below
500 mg (W500)	S/N 34106	Nw = 500.002 mg Cw = calculated below

Primary Standard ID:

300 mg (P300)	S/N 1000095907	Np = 300.001 mg Cp = 0.0016 mg
500 mg (P500)	S/N 1000095933	Np = 500.002 mg Cp = 0.0016 mg

300 mg Test Results:

Measurement	Weight ID	Value (mg)
O1	W300	300.001
O2	P300	300.001
O3	P300	300.000
O4	W300	300.000

500 mg Test Results:

Measurement	Weight ID	Value (mg)
O1	W500	500.001
O2	P500	500.000
O3	P500	500.001
O4	W500	500.001

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Environment Conditions:

Measurement	Minimum	Maximum	24 hour Average	Standard Deviation
Temperature (°C)	20.59	21.44	21.07	0.18
Humidity (%RH)	31.2	36.17	32.73	0.80

Calculations:

Formula: Cw = Cp [(01-02-03+04)/2]+Np-Nw

For the 300 mg Test:

Cw = 0.0005 [(300.001-300.001-300.000+300.000)/2]+300.001-300.001 = **.000**

For the 500 mg Test:

Cw = 0.0017 [(500.001-500.000-500.001+500.001)/2]+500.001-500.001 = **0.0005**

Vaisala Humidity and Temperature Quarterly Calibration Verification

Vaisala Sensor S/N	Humidity (%RH)	Temperature (°C)	Certified Unit
U1620002	33.3	21.3	
T4530017	33.5	21.5	Good to 12/30/1015
H1120114	33.3	21.3	

Acceptance criteria: Temperature must be within ± 2°C and the Humidity must be within ± 2%RH of the certified standard.

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20.9. Refrigerator Daily Temperature Log:

FDEP CHEMISTRY LAB VOC WORKGROUP

Refrigerator ID: 103841 **Thermometer ID: PM-001**

Thermometer Bias (°C) : 0.0** **Calibration: Oct. 1, 2013**

Accepted Temperature Range (°C): ≤ 4.0 **Expiration: July 6, 2014**

Date January 2006	*Time AM/PM*	*Temperature* (°C) TO + B ** *Bias Corrected*	*Staff Initials*	*Comments*
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
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30				
31				

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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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