Life & Times of a PM2.5 Filter: Quality Control in the Laboratory and Beyond

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Inorganic Laboratory Section
Monitoring and Laboratory Division
Air Resources Board
Field Sampling Site / Site Operator (SO)

Laboratory / Chemist
Presentation Objective

To Improve the Quality and Completeness of PM2.5 Data
Presentation Topics

* PM2.5 Mass Program Overview
* Life of a PM2.5 Filter
* Ways to Improve
* Conclusion
Presentation Topics

* PM2.5 Mass Program Overview
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PM2.5 Mass Program Overview

* Federal Regulations and Guidelines
  * CFR, Pt 50, Appendix L
  * Quality Assurance Guidance Document 2.12, 1998

* MLD-055, Standard Operating Procedure for the Determination of PM2.5 Mass in Ambient Air by Gravimetric Analysis, Revision 1.0, March 4, 2014
PM2.5 Mass Program Overview

≈7,000 Samples / 15,000 Weighings Per Year
Presentation Topics

* PM2.5 Mass Program Overview
* Life of a PM2.5 Filter
* Ways to Improve
* Conclusion
Life of a PM2.5 Filter

Topics

- PM2.5 Program and Balance Room Quality Controls
- Inspection/Pre-Weighing
- Chain of Custody (COC)
- Inspection/Post-Weighing/Documentation
- Data Management
Life of a PM2.5 Filter
Topics

* PM2.5 Program and Balance Room Quality Controls
* Inspection/Pre-Weighing
* Chain of Custody (COC)
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* Data Management
PM2.5 Program Quality Controls

- Standard Operating Procedures (SOP)
- Balance Room Controls/Calibrations
- Microbalance
- Mass Standards
- Blanks (Lot, Lab, Field and Trip)
Balance Room Quality Control: Temperature and Relative Humidity

- Average Temperature: 20-23°C (Control ±2°C)
- Average Relative Humidity: 30-40% (Control ±5%)
- 24 Hours Prior to Weighing
- Recorders Certified as NIST-Traceable Annually
- Checked Quarterly and Verified Daily
Balance Room Quality Control: What is NIST?

* The National Institute of Standards and Technology
* Non-regulatory agency of the U.S. Department of Commerce, established by Congress in 1901
* Supplies industry, academia, government, and other users with over 1,300 Standard Reference Materials (SRMs).
* SRMs--certified and used as calibration standards for measuring equipment and procedures, quality control benchmarks for industrial processes, and experimental control samples.
Balance Room Quality Control: Microbalance

- Calibrated and Certified as NIST-Traceable Annually
- Checked Quarterly
- Verified Daily with Mass Standards
Balance Room Quality Control: Mass Standards (Weights)

- Primary Weights Certified as NIST-Traceable Annually
- Working Weights are Verified Quarterly
  ---Used as Controls During Weighing Sessions
Balance Room Quality Control: Blanks

* Lot Blanks—Stability Study for Conditioning Time
* Lab Blanks—Indicates Lab Contamination
* Field Blanks—Indicates Sampler Contamination
* Trip Blanks—Indicates Shipping and Handling Contamination
Life of a PM2.5 Filter

* PM2.5 Program and Balance Room Quality Controls
* Inspection/Pre-Weighing
* Chain of Custody (COC)
* Inspection/Post-Weighing/Documentation
* Data Management
Laboratory to Field Site

- Pre-Condition Filters
- Follow Pre-Wt Schedule
- Gather Materials

- Create List in LIMS Database
- Include QC
- Weigh Filters and QC

- Document Mass, Initial, Date on COC
- Transfer Mass into LIMS

- Package and Ship Filters to Sites
- Print and Review Pre-Wt Report
What is LIMS?

- Laboratory Information Management System
- Database Storage & Management of All Samples
- From Sample Log-In to Final Reports
- Electronic Data Transfer from Instruments
- Calculations, Limit Checks and QC
Inspections

- First Inspected, then Pre-Conditioned According to Stability Study
- Inspected Prior to Pre-weigh
  - Dents, Cuts/Tears
  - Contamination
  - Discoloration
  - Uneven Thickening

Samantha Scola
Pre-Weigh

- Ensure Balance Room w/in Specifications
- Worklist Created in LIMS, Including Duplicates and Controls as QC
- Filters Assigned Barcode and ID Number ("R")
- Filters are Weighed w/QC
- Mass Results Documented And Transferred to LIMS
Life of a PM2.5 Filter

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CARB 24 Hour – FIELD SAMPLE REPORT
Delrin Cassette (FRM) PM 2.5 Samplers

Site Name: ____________________________  Cassette I. D. Number: __________
AIRS Site Number: ____________________  Scheduled Sampling Date: __________
Field Technician: ______________________  Sampler Property #: __________
Agency: ________________________________

SAMPLE SUMMARY

Start Date / Time: ____________________  Ambient Temp (°C): __________
Total Elapsed Time: __________________  Filter Temp (°C): __________
Volume: _______ M³  Pressure (mmHg): __________
Flow CV: _______ %

Local Condition Codes: (Circle One)
NO UNUSUAL CONDITIONS
High Winds  Forest Fire
Farming Nearby  Construction Nearby
Sand/Gravel Streets  Highway Construction
Roofing Operations  Prescribe Burn

Sampler Flag Codes: (Circle One, as necessary)
F. Flowrate 5-min average, out of spec
T. Filter Temp differential, 30 minutes interval out of spec
E. Elapsed sample time, out of spec

Operator Comments: ____________________________

CHAIN OF CUSTODY

<table>
<thead>
<tr>
<th>ACTION</th>
<th>DATE</th>
<th>TIME</th>
<th>FILTER TEMP °C</th>
<th>NAME</th>
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<tbody>
<tr>
<td>Sample Load</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample Removal</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample Placed in Freezer</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample Shipped to Lab</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample Received at Lab</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Start Post-conditioning</td>
<td></td>
<td></td>
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</table>

FOR LABORATORY USE ONLY

<table>
<thead>
<tr>
<th>MASS</th>
<th>DUP MASS</th>
<th>DATE</th>
<th>ANALYST</th>
</tr>
</thead>
<tbody>
<tr>
<td>PRE-WEIGHT</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>POST-WEIGHT</td>
<td></td>
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</tr>
</tbody>
</table>

Lab Comments: ____________________________
Chain of Custody (24-Hr Report) Form

- Official Data--MUST be Accurate and Complete
- Document Any Issues
- Site Operators Invalidate Samples and Schedule Make-ups ASAP, If Warranted
- Form Reviewed by Peer or Manager
- Requested by U.S. EPA during Technical Systems Audits
- Lab Staff Review and Contact SOs for Questions or Issues
If scheduled sampling day is missed or invalid, there are two ways to make it up:

1) Sample run before the next scheduled sampling day
2) Seven (7) days after the missed or invalid sampling day
Life of a PM2.5 Filter

* PM2.5 Program and Balance Room Quality Controls
* Pre-Weighing/Inspection
* Chain of Custody (COC)
* Inspection/Post-Weighing/Documentation
* Data Management
From Field Site to Lab

- Samples Removed from Sampler
- Placed in Freezer

- Filters Packaged
- Temp Strips Activated
- Shipped to Lab

- Received in Lab
- Post-Conditioned for 24 hours

- If Temp >4°C, weigh w/in 10 days of sampling
- If Temp <4°C, weigh w/in 30 days of sampling
Sample Receiving Room

Ali Adams and Samantha Scola

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Receiving PM2.5 Samples

Ali Adams
From Log-In to Post-Weigh to Archive

- Log Data from COC into LIMS
- Contact SO for Qs
- Inspect for Damage
- Post-condition for 24 hours

- Create Worksheet
- Neutralize Static Charge
- Weigh Filters w/QC
- Document Mass on COC

- Contact SO for Anomalies and Invalids
- Document on COC
- Report to ARB Management

- Transfer Data to LIMS
- Review Report
- Archive filters and COCs
Log Samples, Post-Condition 24 Hrs
Post-weigh

- Ensure Balance Room w/in Specifications
- LIMS Worksheet w/QC
- Inspect Filters
- Post-weigh
- Contact SO for Invalids/Anomalies, Document
- Transfer Data, Review Reports
- Report Issues to Mgmt
- Archive Filters and COCs

Nial Maloney
Common Examples of Invalid Samples

- **Pinhole**
- **Excessive Contamination**
- **Smudges/Fingerprints**
- **Cuts/Tears**
Life of a PM2.5 Filter

- PM2.5 Program and Balance Room Quality Controls
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Data Management

- Monthly Data Reviewed by:
  - Analyst
  - Peer Review
  - Supervisor
  - Branch Chief
- Unusual Occurrences Reported to ARB Management
- Systematic Problems Could Result in a Corrective Action Notice (CAN)
- Data Submitted to Air Quality System (AQS) as Data For Record
Presentation Topics

* Overview of PM2.5 Mass Program
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Reminders for Site Operators

* Make sure COC Forms are Complete and Accurate
* Document Problems/Invalid Samples on COC Form
* Report Issues to SO Supervisor and Lab Immediately so Make-ups Can be Scheduled and Data Not Lost
Reminders for Site Operators

- Include and Activate Temperature Strips
- Ship Samples Early
- Check Samples for Indications of Sampler Problems
- Contact Lab Immediately if Need Make-ups
- Others?
Reminders for Laboratory Staff

* Carefully Inspect Filters
* Ship Pre-Weighed Filters Early
* Respond Quickly to Site Operator Issues
* Provide Resources for Site Operators (Extra Filters, Extra Shipping Boxes)
Reminders for Laboratory Staff

* Contact Site Operators Immediately for Make-ups
* Report Anomalies/Issues Immediately to ARB Management and SOs
* Others?
Communication Between Site Operators and Lab Staff

- Preferred Method—Emails for Documentation Purposes
- Phone Calls for Immediate Needs, Follow-Up or Heads-Up
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Presentation Objective

To Improve the Quality and Completeness of PM2.5 Data
PM2.5 Program Contacts

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