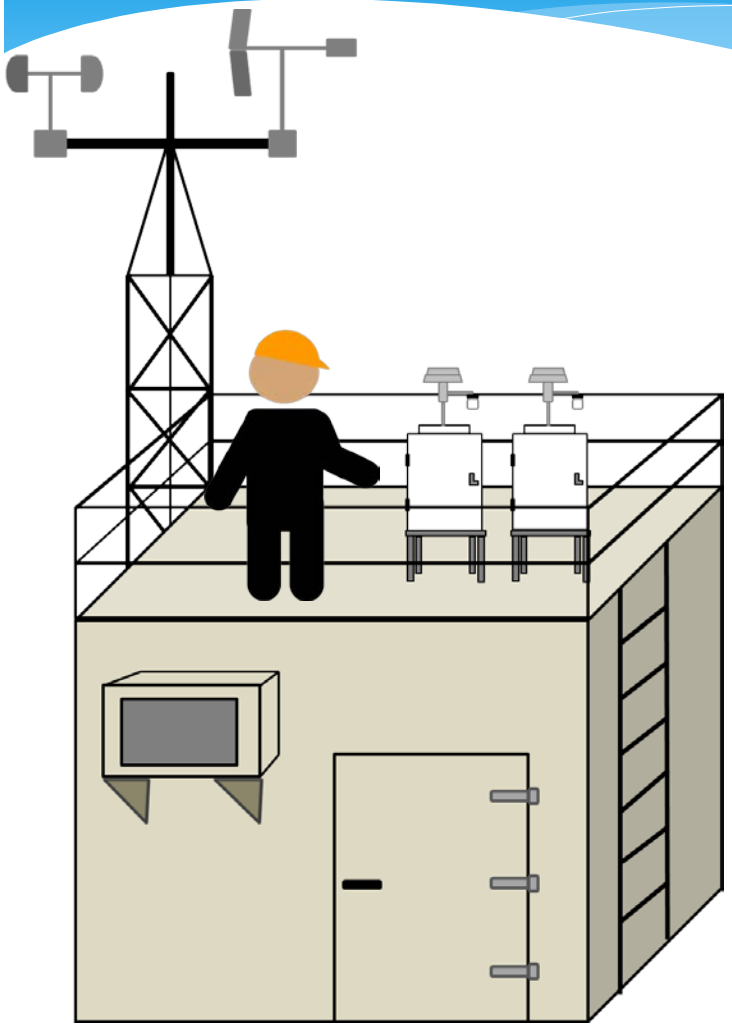
Life & Times of a PM2.5 Filter: Quality Control in the Laboratory and Beyond

Brenda Saldana, Manager
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
* Life of a PM2.5 Filter
* Ways to Improve
* Conclusion

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)
* Inspection/Post-Weighing/Documentation
* 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

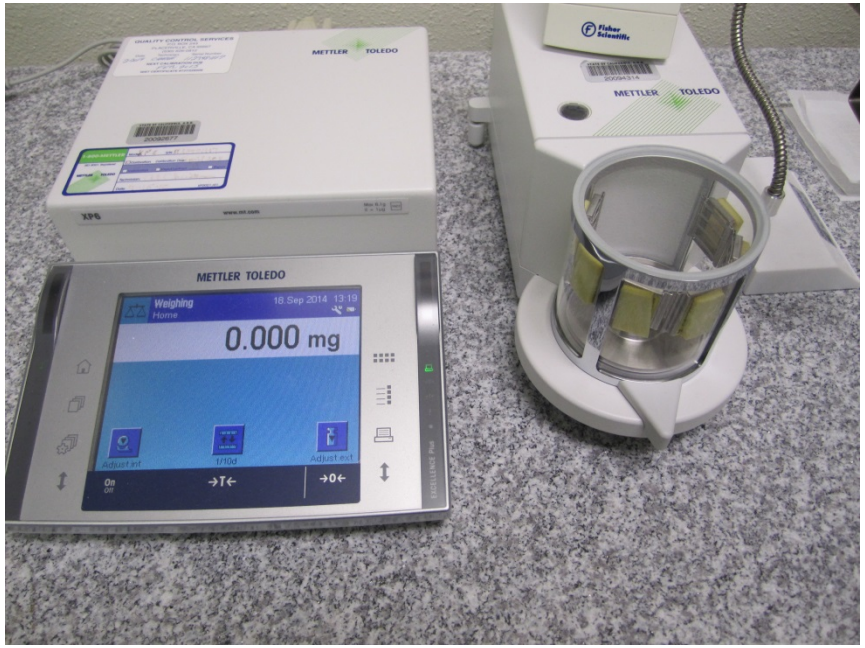
* Average Temperature: 20-23°C (Control ±2°C)
* Average Relative Humidity: 30-40% (Control ±5%)
* 24 Hours Prior to Weighing
* Recorders Certified as NIST-Traceable Annually
* Checked Quarterly and Verified Daily

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.

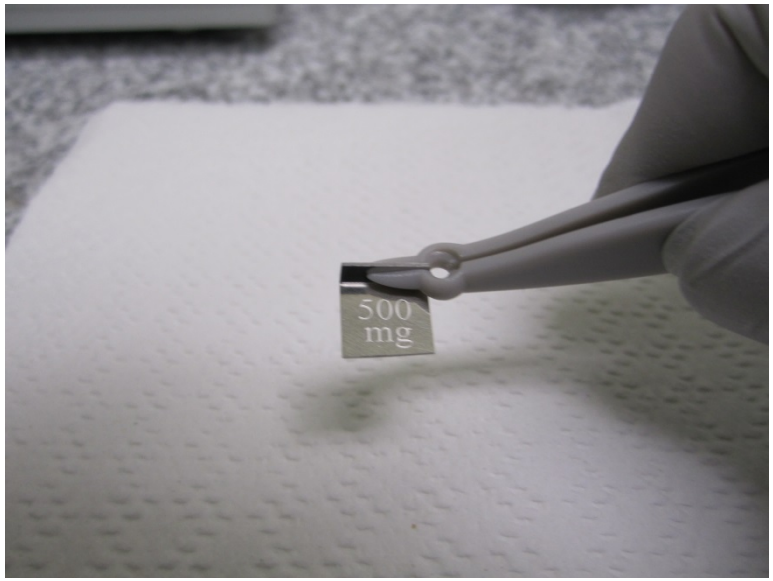
Balance Room Quality Control: Microbalance

* Calibrated and Certified as NIST-Traceable Annually
* Checked Quarterly
* Verified Daily with Mass Standards



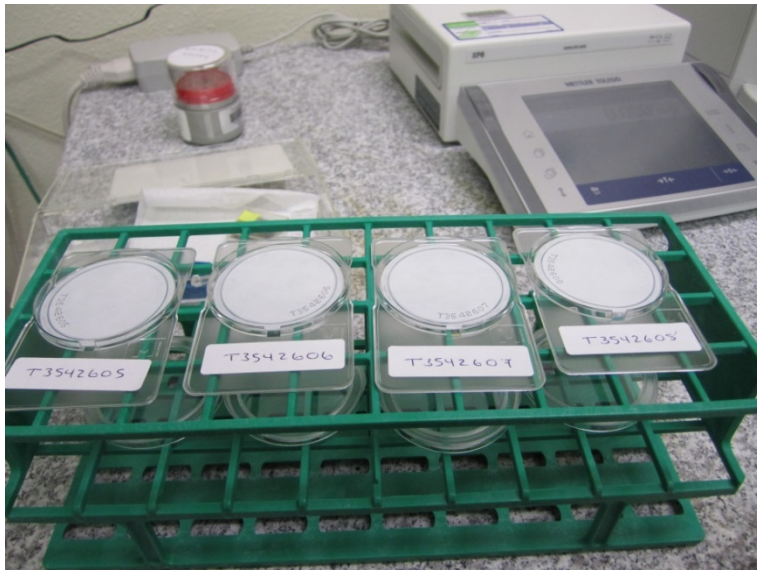
Balance Room Quality Control: Mass Standards (Weights)

* 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

→

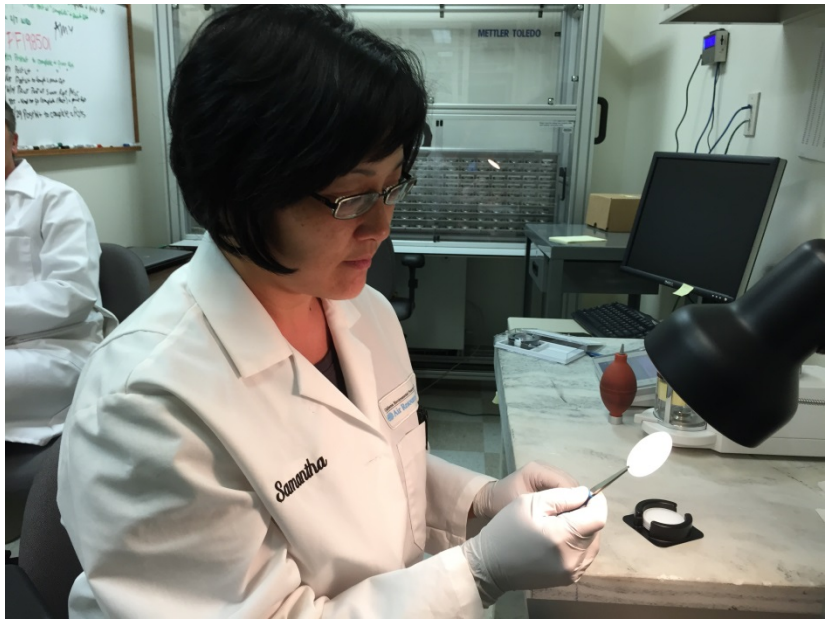
- Package and Ship Filters to Sites
- Print and Review Pre-Wt Report

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

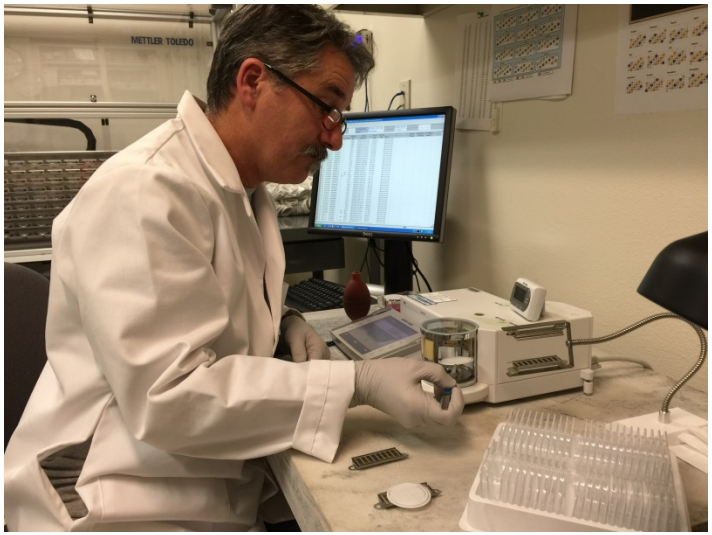
* First Inspected, then Pre-Conditioned According to Stability Study
* Inspected Prior to Pre-weigh
  * Dents, Cuts/Tears
  * Contamination
  * Discoloration
  * Uneven Thickening



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



Nial Maloney

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

Chain of Custody (24-Hr Report) Form

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

Bar Code:

LIMS Sample ID:

Site Name: _____
AIRS Site Number: _____
Field Technician: _____
Agency: _____

Cassette I. D. Number: R
Scheduled Sampling Date: _____
Sampler Property #: _____

SAMPLE SUMMARY

Start Date / Time: _____ / _____
Total Elapsed Time: _____ Hr:min
Volume: _____ M^3
Flow CV: _____ %

	MIN	AVG	MAX
Ambient Temp(°C):			
Filter Temp (°C):			
Pressure (mmHg):			

Local Condition Codes: (Circle One)

NO UNUSUAL CONDITIONS
High Winds — Forest Fire
Farming Nearby — Construction Nearby
Sanding/Salting 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

ACTION	DATE	TIME	FILTER TEMP °C	NAME
Sample Load				
Sample Removal				
Sample Placed in Freezer				
Sample Shipped to Lab				
Sample Received at Lab				
Start Post-conditioning				

FOR LABORATORY USE ONLY

	MASS	DUP MASS	DATE	ANALYST
PRE-WEIGHT				
POST-WEIGHT				

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

Make-Up Rules

* 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

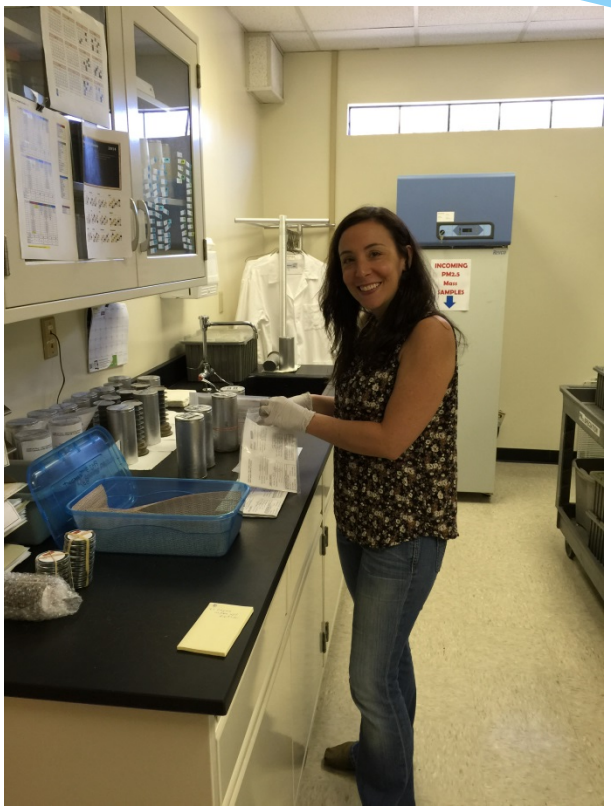
→

- 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

Receiving PM2.5 Samples



Ali Adams

From Log-In to Post-Weigh to Archive

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

→

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

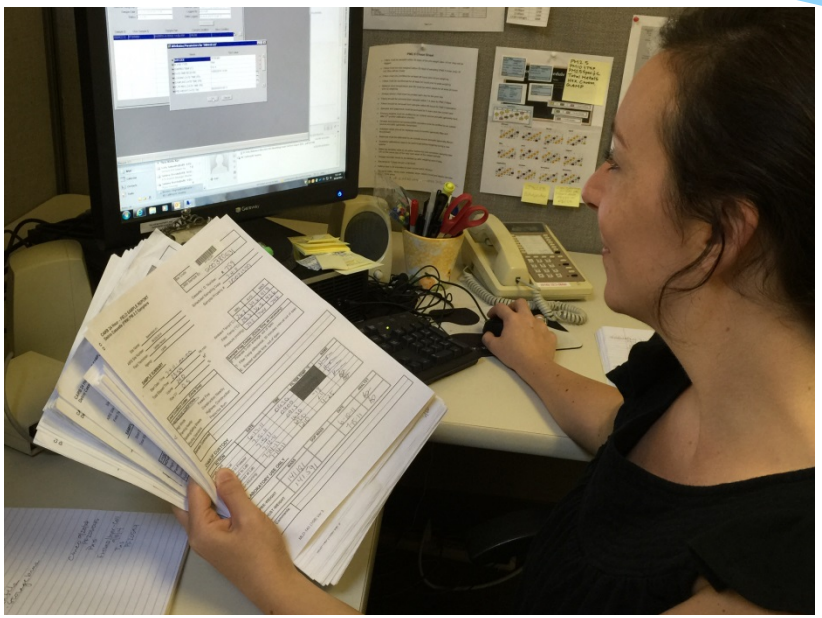
→

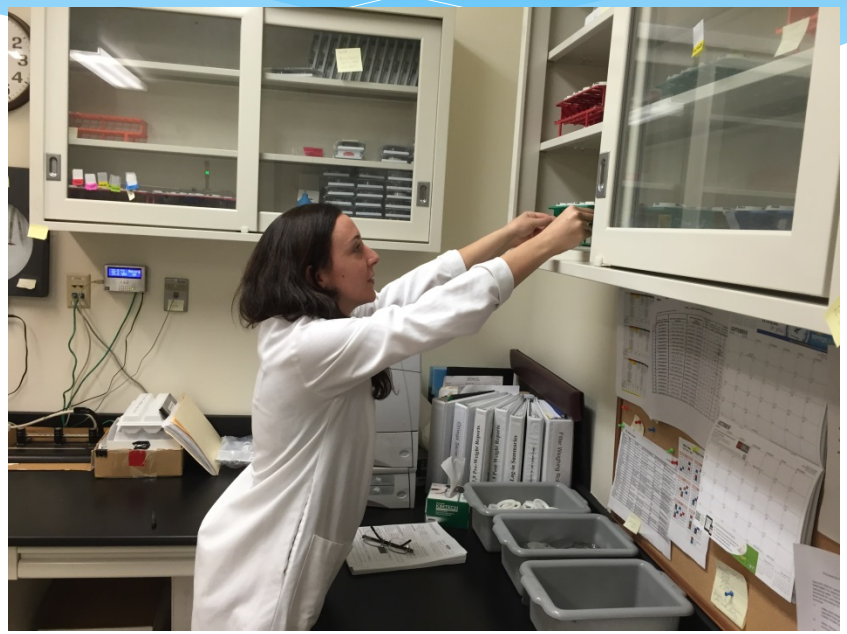
- Contact SO for Anomalies and Invalids
- Document on COC
- Report to ARB Management

→

- Transfer Data to LIMS
- Review Report
- Archive filters and COCs

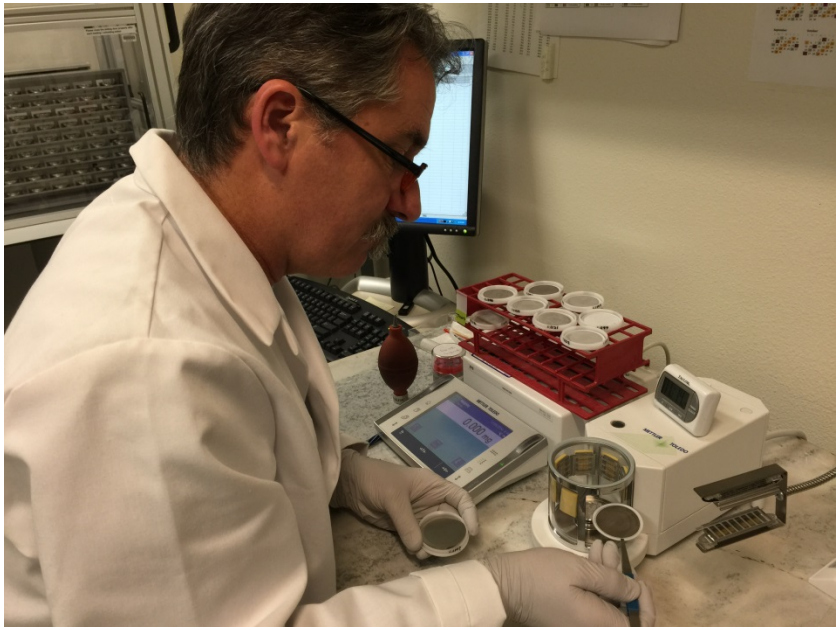
Log Samples, Post-Condition 24 Hrs





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

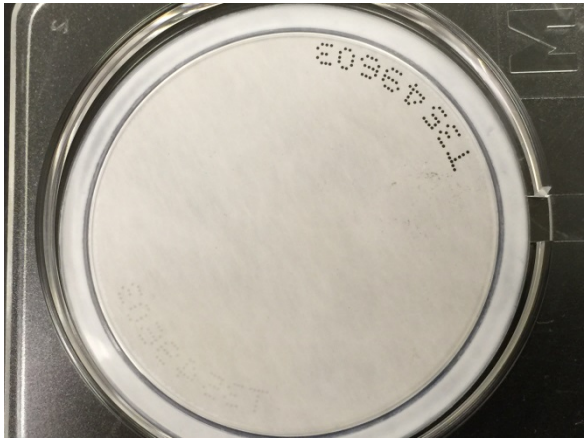


Nial Maloney

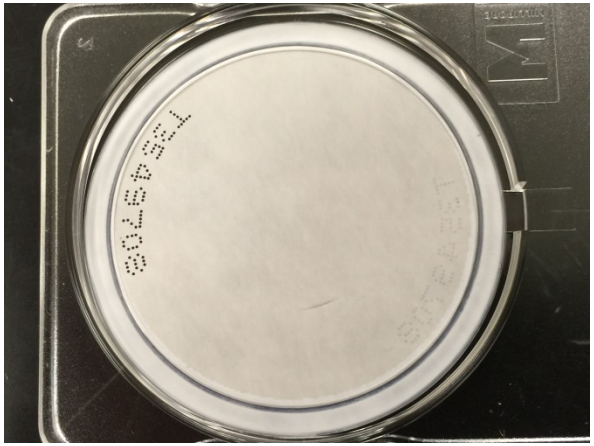
Common Examples of Invalid Samples

Pinhole

Excessive Contamination



Smudges/Fingerprints



Cuts/Tears

Life of a PM2.5 Filter

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

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

Michael Werst, Chief
Northern Laboratory Branch

Presentation Topics

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

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

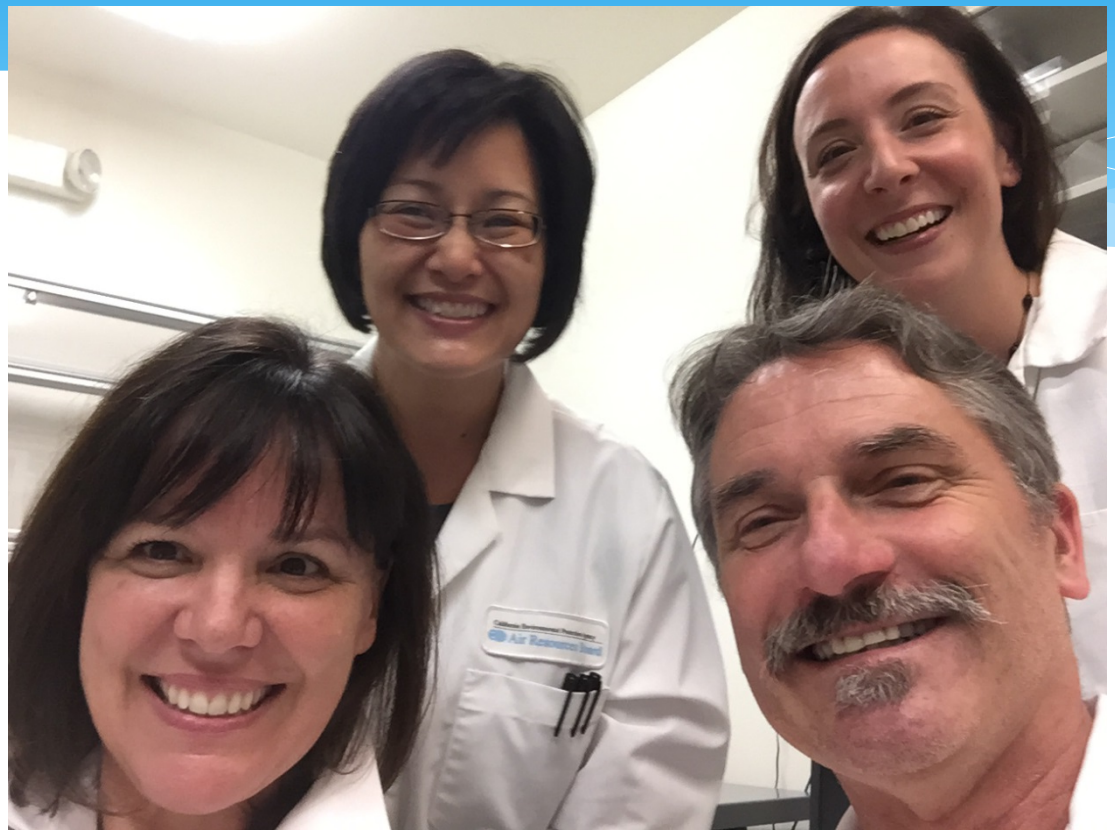
Presentation Topics

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

Presentation Objective

To Improve the Quality and Completeness of PM2.5 Data

PM2.5 Program Contacts



* Nial Maloney, (916) 327-2729 nial.maloney@arb.ca.gov
* Ali Adams, (916) 323-0668 alicia.adams@arb.ca.gov
* Samantha Scola, (916) 322-0220 sscola@arb.ca.gov
* Brenda Saldana, (916) 322-0222 bsaldana@arb.ca.gov