

# **Pb Quality Assurance** Routine Monitoring and Pb-PEP



### **Greg Noah** USEPA, OAQPS, Ambient Air Monitoring Group



### Monitoring Requirements

- NAAQS Standard 0.15 μg/m<sup>3</sup>
- Secondary standard identified as 0.15 μg/m<sup>3</sup>
- Measured as total suspended particulate at local conditions
- Deploy low-volume PM<sub>10</sub> monitoring at NCORE sites at CBSAs with a population greater than 500,000 people
- Monitoring threshold based on emissions of 1/2 tpy for a facility (airports at 1 tpy)
- 15 Airports identified for monitoring for TSP-Pb for one year (now completed)



# Depending on the monitoring situation, two sampling methods may be used

# High volume sampling



Low volume sampling





# **Requirements for Monitoring Pb in TSP**

# The sampling and analytical requirements are found in 40 CFR Part 50:

- Appendix B sampling method (High Vol TSP)
- Appendix G analytical method (ICP-MS)
  ICP-MS is the reference method; however, you can apply for an FEM through ORD

### *The quality assurance requirements are found in 40 CFR Part 58:*

• Appendices A through E – Pb-PEP, siting, reporting, network

# High Volume TSP



# Laboratory QA Critical Criteria

### Initial Acceptance Tests: (OAQPS tests for each filter lot)

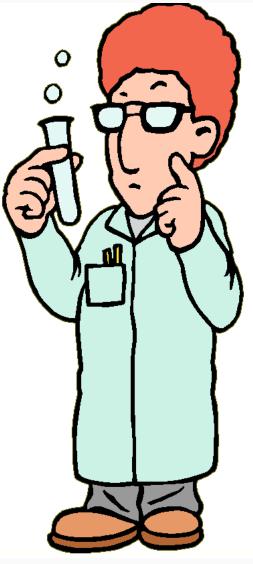
- Collection efficiency 99%
- Pressure drop 42-54 mmHg
- *pH: 6 to 10*
- *Pb content < 15ug/filter*

### Physical Examination of Filters:

• Free of defects (pin-holes, imperfections, tears)

### Calibration reproducibility checks

+/- 5% of predicted calibration curve value performed at the beginning, after every 10 samples, and at the end of each analysis







# Laboratory QA Operational Criteria

- Analysis (precision) audits 6 strips/quarter, 3 at each concentration range – 10% difference
- Field blank each quarter < LDL
- Lab blank each batch < LDL</li>
- *Reagent blank < LDL per batch*



# High Volume TSP

# Field QA Critical Criteria

### Sampling Activities:

- Sample period 1440 minutes +/- 60 minutes midnight to midnight
- Average flow rate 1.1 1.7 m<sup>3</sup>/min at local conditions following each sample run

### One point flow verification:

 +/- 7% once every 3 months (Recommend to be completed more frequently to reduce data loss)

### Sample Recovery:

 Retrieve all samples as soon as possible; best practice would be to collect samples within 24 hours



# High Volume TSP

# STATES STATES

# Field QA Operational Criteria

### Verification/Calibration:

- Leak Check, conducted prior to each flow check, not a quantitative check, leak indicated by a whistle.
- Multi-point Flow Rate Calibration /Verification, conducted after receipt, after motor maintenance or failure of 1-point check and 1/yr. Includes 5 points distributed over the flow range.

**Note:** Samplers with MFC can be calibrated in the field, VFC go to the manufacturer for calibration





# Field QA Operational Criteria

### Precision:

- Flow Audits, conducted every 6 months ensuring comparison is +/-7% of the independent audit standard.
- Collocated Samples, 15% of each method code in the PQAO collected every 12 days. Criteria is  $CV \le 20\%$ for samples that measure concentrations greater than 0.02  $\mu g/m^3$



# Field QA Operational Criteria

### Sampler Maintenance:

- Inlet Cleaning, conducted every three months
- Motor/Housing Gaskets, inspected and or replaced every ≈ 400 hours
- Blower Motor Brushes, replace every 400 -500 hours
- Manufacturer Specific Checks, different samplers may have additional checks that should be identified and addressed in agency QAPPs and SOPs.



### Pb QA Systematic Criteria

- Ensure sampler meets FRM designation
- Annually evaluate if siting requirements are met
- 75% data completeness each quarter
- Measurements are reported in μg/m<sup>3</sup> at local conditions
- Data is reported truncated to three decimal places

- LDL is at least 0.07 μg Pb/m<sup>3</sup>
- Precision for single analyzer quarterly of 90% CL of CV < 20% > 0.02 μg/m<sup>3</sup>
- Bias, measured by Pb-PEP of 95% CL Absolute bias +15% > 0.02 μg/m<sup>3</sup>

# High Volume TSP



### Pb QA Systematic Criteria

### **Field Activities**

#### Flow Rate Transfer Standard

- Resolution 0.02 m3/min
- + 2% reproducibility
- Should have annual multi-point certification traceable to NIST

#### Field Thermometer

- 2° C resolution
- Should have annual multi-point certification traceable to NIST

#### Field Barometer

- + 5 mm Hg resolution
- Should have annual multi-point certification traceable to NIST

#### Clock/Timer Verification

- + 2 min/24-hour
- Should be comparable to network time (internet, cell phone, etc)

### Lab Activities

#### Reagents (HNO<sub>3</sub> and HCI)

• ACS reagent grade

#### Pb Nitrate Pb(NO<sub>3</sub>)<sub>2</sub>

 ACS reagent grade (99.0% purity)



# High Volume TSP

### These are not in CFR or the QA Handbook, but are important!

### **Temperature and BP Audits**

Recommend checking monthly and auditing quarterly to ensure temperature is +/- 2 °C and BP is +/-10 mmHg

Temperature and pressure are important in samplers that use MFCs to control flow and for samplers using VFCs calibrated under STP conditions





# The requirements for monitoring Pb in PM<sub>10</sub> are similar to PM<sub>10</sub> particulate

The requirements are found in 40 CFR Part 50:

Appendix L – sampling method

Appendix Q – analytical method (XRF FRM)

And scattered through 40 CFR Part 58

Appendices A through E – Pb-PEP, siting, reporting, network

# Low Volume PM<sub>10</sub>



Dennis covered the Appendix L lowvolume PM<sub>10</sub> method earlier

See **40 CFR Part 50, Appendix L,** the **QA Handbook** and validation Templates for more information

CEPA And Bades Construction Printed Printed In Construction
Quality Assurance Handbook for Air Pollution Measurement Systems
Volume II
Ambient Air Quality Monitoring Program



# Notable Differences from low volume PM<sub>10</sub>

- Analytical testing of filters for background Pb by OAQPS (~ 20 test filters per lot and 90% of filters < 4.8 ng Pb/cm<sup>2</sup>
- Must use an EPA approved analytical method (FRM/FEM) for Pb analysis
- Quarterly Pb filter audits (more on this later)
- Pb-PEP



### **Pb Collocation Requirements**



### Lead Collocation Requirements: 40 CFR Part 58 App A sec 3.4.4 (TSP) and 3.4.5 (PM<sub>10</sub>)

- Collocate 15% of primary monitors in PQAO (not counting non-source oriented NCore sites in PQAO).
- Have a minimum of one collocated monitor.
- Site the first collocated sampler at the highest measuring Pb site in the network.
- Monitors must be sited within 4 meters of each other and...
  - ≥ 2 meters apart (inlet to inlet) for TSP
  - $\circ \geq 1$  meter apart (inlet to inlet) for PM<sub>10</sub>
- Follow the 1 in 12 sampling frequency.







### **Cutoff concentrations for use in collocation calculation:**

**0.002 μg/m3** (Methods approved after 3/04/2010, with exception of manual equivalent method EQLA-0813-803).

**0.02 μg/m3** (Methods approved before 3/04/2010, and manual equivalent method EQLA-0813-803).

EQLA-0813-803 - Flame Atomic Absorption Spectroscopy (FAAS) Manual Equivalent Method



Pb-Performance Evaluation Program (Pb-PEP) and Pb Strips/Filters

### Pb-PEP

Independent program that evaluates total measurement system bias (field and laboratory) in the network by comparing collocated samplers with primary samplers

### **Pb Strips/Filters**

Provides a check of laboratory bias between laboratories supporting the Pb monitoring network













### **Pb-Performance Evaluation Program (Pb-PEP)**

Nationally implemented program; however, an implementation option is available for SLTs that can demonstrate independence and adequacy

### The Pb-PEP has two parts:

### Independent collocated audits

- National program run by ESAT contractors or SLT implementers
- An external group sets up and runs an independent sampler beside the SLT routine sampler and uses an independent lab for analysis

### Extra SLT collocations

- At their collocated site, the SLTs (preferably the QA group) runs an extra collocated sample using their existing samplers on an off-run day
- The primary sampler filter goes to the routine state lab, and the collocated sample goes to the Pb-PEP lab



### **Pb-PEP Details per PQAO**

# 15% of all sites audited per year minimum with all sites audited in 6 years. Must audit at least one of each monitor type each year.

- If 5 sites or less, 5 audits per year
- If >5 sites, 8 audits per year

### This translates into...

### 5 audits per year

- I collocation with an independent PEP sampler
- 4 filters collected from network collocated sampler

8 sites per year

- 2 collocations with an independent PEP sampler
- 6 filters collected from network collocated sampler

### National Pb QA Programs



### **Pb-PEP Data**

Pb-PEP Audits **begin** and **end** at the AIRQA Website

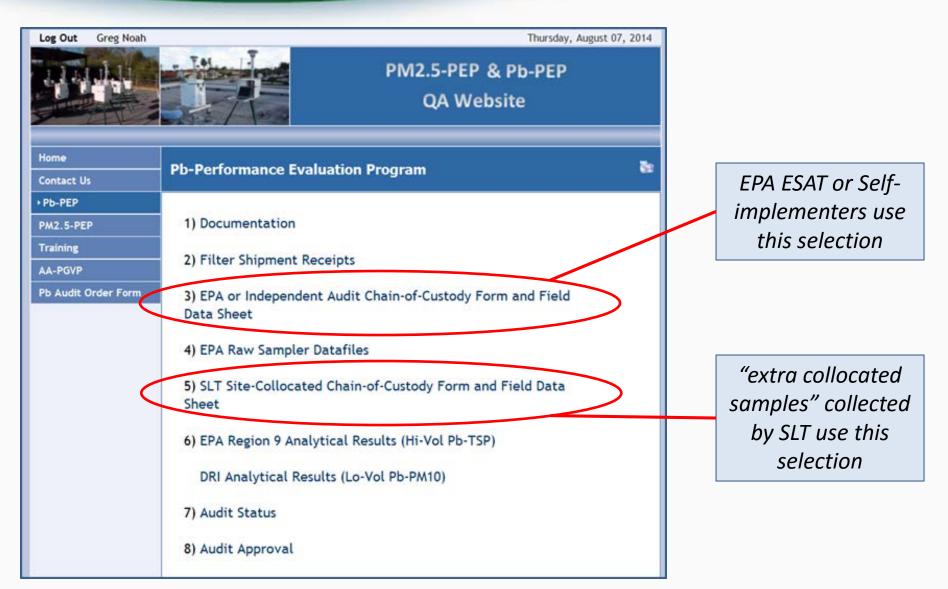
### Summary of work flow:

- ✓ Print Field Data/Chain of Custody sheets
- ✓ Enter field data on FDS/COC sheet
- ✓ Enter field data into AIRQA
- ✓ Send filters to R9 PEP Lab
- ✓ Laboratory uploads data
- ✓ Link lab and field data
- ✓ Generate concentration and QA checks
- ✓ Validation and approval decisions
- ✓ Upload to AQS

og Out Greg Noah	Thursday, August 07, 2014
and and all	PM2.5-PEP & Pb-PEP
	QA Website
ome	Pb-Performance Evaluation Program
ontact Us	
%-PEP M2.5-PEP	1) Documentation
aining	2) Filter Shipment Receipts
A-PGVP	
b Audit Order Form	3) EPA or Independent Audit Chain-of-Custody Form and Field Data Sheet
	4) EPA Raw Sampler Datafiles
	5) SLT Site-Collocated Chain-of-Custody Form and Field Data Sheet
	6) EPA Region 9 Analytical Results (Hi-Vol Pb-TSP)
	DRI Analytical Results (Lo-Vol Pb-PM10)
	7) Audit Status
	8) Audit Approval

### National Pb QA Programs





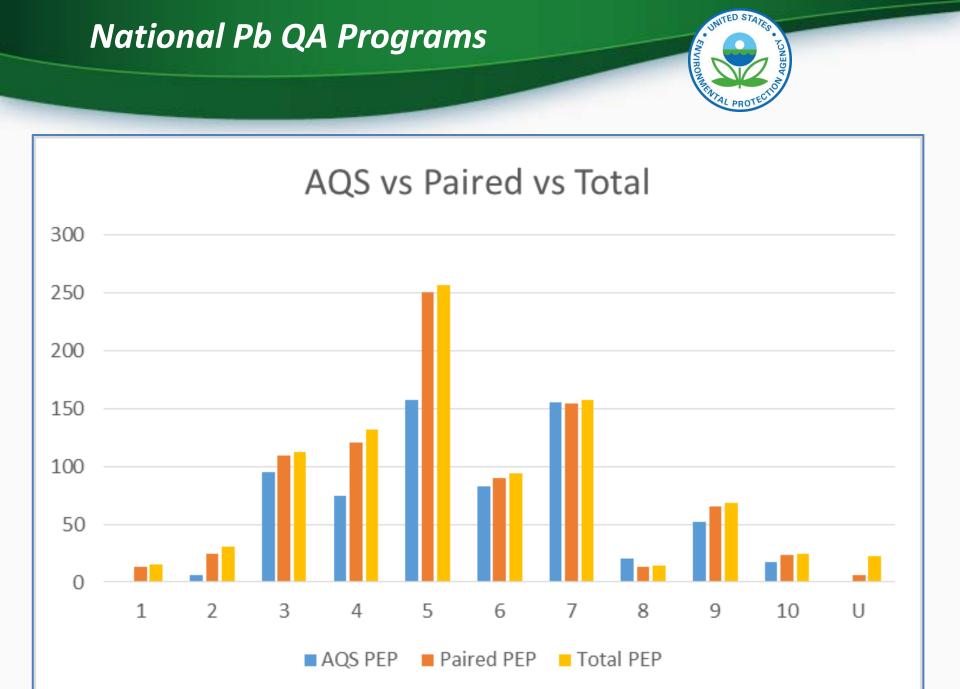


### **Pb-PEP Data Issues**

- All field data is not being entered into AIRQA; therefore it is very difficult to pair with lab data
- Data is not being approved on AIRQA in a timely manner

# What Have We Done to Help?

- Revised the Pb-PEP website to eliminate entry errors
- Reviewed the data to discover weaknesses and errors
- Identified what data we have that is not paired.
- Distributed this data to the Pb-PEP regional contacts





## **Pb-Strips/Filters**

### For each laboratory analyzing for Pb NAAQS:

6 strips/filters must be analyzed quarterly (24 annually)

The 6 filters will have certified values split between two ranges:

- 3 at low range (30-100% of the NAAQS)
- 3 at high range (200-300% of the NAAQS)

The check must be within **10% difference** of the certified value of the strip or filter

ICP-MS is a destructive analysis so 24 strips are required XRF is not destructive so only 6 Teflon filters are required



### Need Help Getting the Data into AQS

### **Pb-PEP Coordinators**

- I have submitted a couple spreadsheets of audits that need attention address those and get those audits corrected
- Completed audits must be approved before uploading the data into AQS

**Going forward**, we have used the LEAN process to re-invent this program to:

- Improve efficiency
- Speed data upload to AQS
- Eliminate confusion
- Improve the review and approval process

- Reduce cost
- Reduce contract support
- Better utilize technology
- Eliminate errors

### National Pb QA Programs



### **Pb-Strips/Filters Ordering Directions**

- Mike sends out a notice every year that he is ordering audit filters (about February concluding in May)
- When you get the email, order the filters
- Here's the web link to AIRQA: https://www.sdas.battelle.org/airqa/
- If you do not order, you will get automated reminders
- Only one POC in each agency gets the email, make sure it is the right contact and let us know if a change is needed.
- Fill out the form and tell us how many filters you need

