Welcome to Pb QA!

Pb Quality Assurance
Routine Monitoring and Pb-PEP

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Monitoring Requirements

- **NAAQS Standard - 0.15 µg/m³**
- **Secondary standard identified as 0.15 µg/m³**
- **Measured as total suspended particulate at local conditions**
- Deploy low-volume PM$_{10}$ 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
- Reagent blank < LDL per batch
Field QA Critical Criteria

Sampling Activities:
- Sample period - 1440 minutes +/- 60 minutes midnight to midnight
- Average flow rate - 1.1 – 1.7 $m^3/min$ 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
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 ≤ 20% for samples that measure concentrations greater than 0.02 µg/m³
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 $\mu g/m^3$ at *local conditions*
- Data is reported truncated to three decimal places

- LDL is at least 0.07 $\mu g$ Pb/$m^3$
- Precision for single analyzer quarterly of 90% CL of CV < 20% > 0.02 $\mu g/m^3$
- Bias, measured by Pb-PEP of 95% CL Absolute bias +15% > 0.02 $\mu g/m^3$
**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 (HNO₃ and HCl)**
- ACS reagent grade

**Pb Nitrate Pb(NO₃)₂**
- 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$_{10}$ are similar to PM$_{10}$ 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
Dennis covered the Appendix L low-volume $PM_{10}$ method earlier.

See 40 CFR Part 50, Appendix L, the QA Handbook and validation Templates for more information.
Notable Differences from low volume PM$_{10}$

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

$\approx$ PM$_{10}$
Lead Collocation Requirements:
40 CFR Part 58 App A sec 3.4.4 (TSP) and 3.4.5 (PM$_{10}$)

- 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
  - ≥ 1 meter apart (inlet to inlet) for PM$_{10}$
- 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).

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
- 1 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
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
National Pb QA Programs

- Documentation
- Filter Shipment Receipts
- EPA or Independent Audit Chain-of-Custody Form and Field Data Sheet
- EPA Raw Sampler Datafiles
- SLT Site-Collocated Chain-of-Custody Form and Field Data Sheet
- EPA Region 9 Analytical Results (Hi-Vol Pb-TSP)
- DRI Analytical Results (Lo-Vol Pb-PM10)
- Audit Status
- Audit Approval

EPA ESAT or Self-implementers use this selection

“extra collocated samples” collected by SLT use this selection
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
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