Review of Approach for Development of New Federal Reference Method (FRM) for Pb-TSP

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Current FRM for Pb-TSP

• Overall method for Pb-TSP consists of the sampling method and the analysis method combined
  – Sampling method based on high-volume TSP per 40 CFR Part 50, Appendix B
  – Analysis method based on Atomic Absorption Spectroscopy (AAS) per 40 CFR Part 50, Appendix G
Need for a new FRM for Pb-TSP

• Advances in new analytical measurement technologies warrant a revision and update to the existing AAS FRM analysis method
  – Improved analytical sensitivity (detection limits) to support much lower NAAQS
  – Improved and more efficient extraction methods
  – Wider use and availability of newer analytical instrumentation

• Focus of new FRM is on the analysis method only and will not address sampling
Proposed FRM for Pb-TSP

• Extraction Methods
  – Heated ultrasonic water bath – EQL-0510-191
    • 80 ± 5°C with 1.02M nitric/2.23M hydrochloric
  – Hot block digestion – EQL-0710-192
    • 95 ± 5°C with 3.5% nitric (v/v)

• Analysis Method
  – Inductively-coupled mass spectrometry (ICP-MS)
  – Advantages:
    • Very low method detection limits (MDLs)
      – On the order of 0.2 ng/m³ or less for high-volume samples
    • Commonly used
FRM and Method Performance

Based on following guidance documents and references:

- EQL-0510-191, Determination of Lead in TSP by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) with Heated Ultrasonic Nitric and Hydrochloric Acid Filter Extraction.

- EQL-0710-192, Heated Nitric Acid Hot Block Digestion and ICP-MS Analysis for Lead(Pb) on TSP High-Volume Filters.

- Validation and Peer Review of U.S. Environmental Protection Agency Chemical Methods of Analysis, prepared for The EPA Forum on Environmental Measurements (FEM); FEM Document Number 2005-01, October 14, 2005.

- Guidance for Methods Development and Methods Validation for the RCRA Program; Development and Validation of SW-846 Methods Phase 2: Formal Validation, April 6, 1992.


Method Performance

- Analysis of Standard Reference Materials (SRMs)
- Analysis of filters spiked with NIST-traceable Pb salts solutions
- Effects of Interferences and filter matrix effects
- Determination of MDL (40 CFR part 136, appendix B)
- Intra-laboratory method performance
  - Testing to assess bias and precision
    - Bias 10% and Precision 15%
  - Evaluation of glass and quartz filter matrices
  - Analysis of spiked filter strips, SRMs and real world samples
  - Test small variations in extraction temperature and time
  - Evaluate extract storage stability
Method Performance

• Inter-laboratory (between-lab) method performance
  – Purpose to assess the performance of the method when applied by other laboratories
  – Need at least four laboratories to participate
  – Labs will be asked to analyze filter samples and spiked filter strips using both extraction options and the ICP-MS analysis method
  – The coefficient of variation (CV) will be used to determine the variability at the 95% confidence interval
Charge Questions

• What are the panel’s views on the two extraction method options of heated ultrasonic with nitric/hydrochloric acids and graphitic hot block with nitric acid for the extraction of Pb from TSP?

• What are the panel’s views on ICP-MS as the analysis method for Pb-TSP as the FRM?

• What are the panel’s views on the approach described for evaluating the performance the method prior to proposing it through the rule making process as a new FRM for Pb-TSP?

• Inter-laboratory testing will be done to assess between-laboratory variability. What are the panel’s views on a reasonable level of variability (CV) at the 95% confidence interval?
Next Steps

- Consider and incorporate advice
- Complete intra-laboratory performance tests
- Complete inter-laboratory performance tests
- Proposed and Final Rule within 18 months