

**Charge Questions for the Clean Air Scientific Advisory Committee's
(CASAC) Ambient Air Monitoring & Methods Subcommittee Peer Review on
the Approach for the Development of a New Federal Reference Method
(FRM) for Lead (Pb) in Total Suspended Particulates (TSP)
September 15, 2010**

Purpose of the Peer Review

The need for lowered sensitivity (detection limits) in response to the much lowered Lead (Pb) NAAQS; advances in measurement technology that have occurred since promulgation of the original Pb-TSP Federal Reference Method (FRM); and new methods that are now available with improved precision, detection limits, and extraction efficiency support the need for a new Pb-TSP FRM. This new FRM is intended for use by analytical laboratories performing the analysis of Pb-TSP filters to support the NAAQS. The analytical method will be evaluated with multiple matrices for bias, precision, and repeatability. Sampling procedures for NAAQS-related data collection will continue to be performed in accordance with the FRM described in 40 CFR part 50, Appendix B and therefore are not included as part of this new FRM or the peer review. EPA is seeking CASAC advice in order to better plan its approach to the task of establishing a new FRM for Pb-TSP.

Charge Questions

The approach for developing a new FRM for Lead in TSP (Pb-TSP) is described in the accompanying white paper. Two candidate methods have been identified for extracting lead from filters for subsequent analysis by inductively coupled plasma mass spectrometry (ICP-MS). The extraction methods are based on 1) EQL-0510-191 which uses a heated ($80 \pm 5^\circ\text{C}$) ultrasonic water bath with 1.02M nitric/2.23M hydrochloric acids and 2) EQL-0710-192 which uses a heated ($95 \pm 5^\circ\text{C}$) graphite hot block with 3.5% (v/v) nitric acid. These methods use equipment that is commonly used by many laboratories, is more practical (use of a single vessel for the entire extraction process and storage), and have improved throughput that make them more efficient and cost effective than the current FRM. ICP-MS has been chosen as the analytical technique because it has much improved sensitivity, selectivity, linear range, and is much more readily available than FAAS in laboratories today.

1. What are the panel's views on the two extraction method options of heated ultrasonic and graphitic hot block with nitric/hydrochloric acid for the extraction of Pb from TSP?
2. What are the panel's views on ICP-MS as the analysis method for Pb-TSP?

The proposed FRM and the approach for evaluating method performance are provided in the whitepaper and based on the guidance documents and references provided below. The approach includes the analysis of Standard Reference Materials (SRMs) and NIST-traceable Pb salt solutions; assessment of method detection limits (MDLs), bias, precision; and inter-laboratory testing to assess between laboratory variability.

3. What are the panel's views on the approach described for evaluating and testing the method prior to proposal as a new FRM for Pb-TSP?
4. Inter-laboratory testing of the method will be done to assess between-laboratory variability (CV) at the 95% confidence interval. What are the panel's views on a reasonable level of inter-laboratory variability?

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. <http://www.epa.gov/ttn/amtic/files/ambient/pb/EQL-0510-191.pdf>

EQL-0710-192, Heated Nitric Acid Hot Block Digestion and ICP-MS Analysis for Lead (Pb) on TSP High-Volume Filters. <http://www.epa.gov/ttn/amtic/files/ambient/pb/EQL-0710-192.pdf>

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. http://www.epa.gov/fem/pdfs/FEM_MV_doc_final_10-14-2005.pdf

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. <http://www.epa.gov/osw/hazard/testmethods/pdfs/methdev.pdf>

EPA's Office of Solid Waste (OSW) compendium of sampling and analytical methods for Evaluating Solid Waste, SW-846 Method 6020A, "Inductively Coupled Plasma-Mass Spectrometry", Revision 1, February 2007. <http://www.epa.gov/wastes/hazard/testmethods/sw846/pdfs/6020a.pdf>

Harper et al., (1983). Simplex Optimization of Multielement Ultrasonic Extraction of Atmospheric Particulates; Anal. Chem., 55 (9), 1553-1557.

Long et al., (1979). Lead Analysis of Ambient Air Particulates: Interlaboratory Evaluation of EPA Lead Reference Method; APCA Journal, 29, 28-31.