

CASAC AAMM
Comments Submitted by Jay R. Turner (July 20, 2008)
Peer Review: Draft Federal Reference Method (FRM) Lead in PM₁₀ (Pb-PM₁₀)

Charge Questions

1. *What are your comments on the use of the low-volume PM_{10c} FRM sampler as the Pb-PM₁₀ FRM sampler?* The low-volume PM_{10c} FRM sampler is an appropriate choice as the Pb-PM₁₀ FRM sampler. It is an adaptation of the PM_{2.5} FRM sampler which now has nearly ten years of use and refinement, including both single-event and sequential configurations. There are also operational and cost advantages to placing measurements for multiple NAAQS on the same sampler platform. For sites specified for both PM₁₀ and Pb-PM₁₀ compliance monitoring, filter samples collected using the low-volume PM_{10c} FRM sampler could be subjected to both gravimetric analysis and Pb elemental analysis, providing compliance data for both standards from a single sample.
2. *What are your comments on the use of XRF as the Pb-PM₁₀ FRM analysis method?* I prefer the use of ICPMS (or GFAAS) as the FRM with an expectation that XRF would be given FEM status. While ICPMS does have the added complexity of a sample digestion step, it can be more easily calibrated than XRF. Our recent experience with ICPMS has demonstrated high recovery for both coal fly ash and urban particulate matter NIST Standard Reference Materials (SRM) from quartz filters using a nitric acid and hydrochloric acid extraction solution (following the NATTS PM₁₀ metals sampling and analysis protocol developed by ERG). The Pb-PM₁₀ method would use Teflon filters and ERG has also developed a protocol for this case which could be used as a starting point for the analysis method specifications.¹
3. *What are your comments on the specific analysis details of the XRF analysis method contained in the proposed Pb-PM₁₀ FRM analysis method description?* I defer to the XRF experts for a critique of the analysis method details. Given the variations in instrument hardware and software, all labs reporting compliance data based on XRF should participate in an audit program which includes analyses of samples with traceability to ICPMS.
4. *Do you think the precision, bias and MDL of the XRF method for the proposed Pb range will be adequate?* These questions are best addressed after completion of the DQO process. Perhaps the required MDL could be relaxed depending on the NAAQS concentration value, although a detection limit that is much lower than the standard is desirable to simplify the data handling for concentrations below the MDL. Precision should be determined using data with Pb concentrations above a defined threshold value since the precision reported as a percentage CV will degrade as the MDL is approached. In general, we should be prepared for both ICPMS and XRF data being reported to AQS, and these methods will have very different detection limits. This will add complexity to certain non-compliance data analyses; including trends analyses studies on Pb health effects.
5. *Are there any method interferences that we have not considered?* I defer to the XRF experts on the issue of method interferences.

¹ “Standard Operating Procedure for the Determination of Metals in Ambient Particulate Matter Analyzed by Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)”, prepared by ERG for EPA under Work Assignment 5-03, ERG No.: 0143.04.005, EPA Contract No.: 68-D-00-264, September 2005.