Preliminary Comments from Mr. Bart Croes on EPA's White Paper, *Approach for the Development of a New Federal Reference Method (FRM) for Lead in Total Suspended Particulates (Pb-TSP)*

Staff should be commended for taking a systematic approach towards implementation of the revised National Ambient Air Quality Standard (NAAQS) for lead (Pb) in total suspended particulates (TSP). I appreciate the opportunity to comment during several stages of the process, and agree with the general approach taken by U.S. EPA toward extraction and analysis as described in the white paper. My comments reflect input from California Air Resources Board (ARB) staff responsible for implementing U.S. EPA monitoring requirements and using the data in source apportionment and health studies.

Charge Questions:

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?*

   Both methods are acceptable for extraction of Pb from TSP for NAAQS compliance purposes. Since SLT agencies are already extracting Pb as part of source apportionment and air toxics monitoring networks, and want to conserve resources, this flexibility is desirable. Pb is easy to extract, but it would be desirable to have performance standards for extraction efficiency in order to demonstrate equivalency for data from other existing networks.

2. *What are the panel's views on ICP-MS as the analysis method for Pb-TSP?*

   ICP-MS is preferred as the FRM, but other analytical methods besides should be allowed. The ARB air toxics network (Xontech 924, low volume sampler TSP, Teflon filter) uses ICP-MS for toxic metals, including Pb, because of high accuracy and precision across many species (in a single scan) over the entire ambient concentration range.

   The Flame Atomic Absorption Spectroscopy (FAAS) method is used for the existing high volume sampler TSP-Pb network by many SLT agencies, and also has good precision and accuracy. The FAAS method should be retained as an FEM. ARB's high volume sampler TSP-Pb network uses Atomic Absorption Spectroscopy (AAS) with a graphite furnace and has a 1.0 ng/m^3 (0.0010 µg/m^3) limit of detection (LOD), far below the new NAAQS of 0.15 µg/m^3. A cursory look at the last few months of data from two FRM sites (Calexico and Tijuana) showed all results to be well above this LOD. The AAS with a graphite furnace method should be retained as an FEM.

   Will this issue seems to be beyond the scope of the current CASAC AAMM Subcommittee review, modern low volume samplers offer advantages over the high volume samplers used as an FRM. They are much more sophisticated with micro processors, temperature and

pressure sensors, automated filter loading, and serial data communications. ARB's ICP-MS LOD for TSP-Pb by low volume sampler Teflon filters is 1.5 ng/m^3 (0.0015 μg/m^3), far below 0.15 μg/m^3. While the XRF method is not appropriate for quartz fiber filters, because the strong Si peak (from the quartz filter matrix) wipes out the spectra of other elements, it does work for Teflon filters. While XRF provides an efficient method of analysis and requires less sample preparation than other analytical methods, it is destructive and has problems with non-uniform deposits. However, ARB's XRF LOD for TSP-Pb by low volume sampler Teflon filters is 3.0 ng/m^3 (0.0030 μg/m^3), again far below 0.15 μg/m^3.

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?*

   With the inclusion of the AAS methods discussed above, the approach described for evaluating and testing the proposed analytical method for a Pb-TSP FRM appears adequate, especially the range of spiked and ambient samples and number of proposed facilities (four) for the inter-laboratory comparison. The criteria that each laboratory will achieve bias (accuracy) ≤10% and precision ≤15% for the spiked filter strips seems loose and could be tightened. Typically, the ARB laboratory sees ≤5% bias and ≤5% precision for metals analyses, but perhaps ≤10% for both is a more realistic expectation.

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?*

   An inter-laboratory bias (accuracy) ≤10% and precision ≤15% for the group of measurements that make up a 3-month average is a reasonable expectation. The inter-laboratory comparison should be weighted toward the ambient samples and spiked samples that are near the level of the NAAQS.