To: Fred Butterfield, Designated Federal Officer
EPA SAB, Clean Air Scientific Advisory Committee (CASAC)
Ambient Air Monitoring and Methods Subcommittee (AAMM)

From: George Allen, AAMM subcommittee member, July 17, 2008

Re: Peer review of the Draft Federal Reference Method (FRM) Lead in PM10 (Pb-PM10)

The following are written comments based on the Charge Questions in the EPA OAQPS memo to the SAB dated June 15, 2008. These comments also reflect discussion during the July 14 teleconference AAMM meeting on a peer review of the Draft Federal Reference Method (FRM) Lead in PM10 (Pb-PM10). A copy of these comments is also being sent to Dr. Ted Russell, CASAC AAMM Subcommittee Chair.

Peer Review Charge Questions in **Bold**:

1. What are your comments on the use of the low-volume PMl0c FRM sampler as the Pb-PM10 FRM sampler?

The existing PM10c sampler is an obvious choice for a sampler since it is well characterized and commercially available from several vendors. Sequential (automated) PM10 samplers should also be allowed, either as FRM or FEM samplers. The dichotomous sampler is another obvious candidate for an FRM or FEM sampler for PM-10 lead.

2. What are your comments on the use of XRF as the Pb-PM10 FRM analysis method?

XRF is sufficient for routine analysis, but for the FRM, a more sensitive and specific technique should be used, such as ICPMS. If XRF is used, the method should be an FEM. If XRF remains the FRM analysis method, there are concerns of uniform deposit on the filter that may differ with different sizes (coarse vs. fine mode) of particles. Appropriate filter deposition testing would have to be done prior to promulgation of XRF as the FRM analysis method. There are also concerns regarding different XRF analytical methods and calibration techniques across different laboratories, the lack of a NIST thin-film XRF Pb reference standard, possible issues with heavy filter loading, the difficulty of generating spiked samples, and the possibility of interferences. ICPMS does not have any of these concerns.

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?

The XRF analysis method description proposed here is well written and takes into account most of the issues raised above. It does not resolve the issues of non-uniform deposition or the lack of

a NIST thin-film standard for Pb. A PM10 filter can appear visually to have a uniform deposit, but in urban areas the visual appearance is often driven by fine-mode aerosol which may not reflect the deposition pattern of coarse mode Pb. Thus, visual inspection is only a crude first test for uniform deposition of PM-10 Pb. The issue of filter blanks needs more attention; blank values can vary by manufacturing lot. Thus, the blanks used for a set of samples must be from the same lot. The method description does not use field blanks; it is important to have 5% of filters used as field blanks. The method needs to include a section on how levels below the method's LOD or LOQ will be handled. I suggest reporting the blank-corrected data as measured (even if it is slightly negative), but flagging it as below the LOD.

4. Do you think the precision, bias and MDL of the XRF method for the proposed Pb range will be adequate?

The XRF MDL for Pb will be a function of XRF method and blank levels and variability. Although the MDL noted in this method description (1 ng/m3 one-sigma) is adequate, it may or may not be achieved in the real world, since the MDL is a function of many things, including the number and stability of lab and field blank filters and the length of XRF analysis time. The bias and method detection limits in this draft are appropriate. I would suggest that the FEM precision be tightened from 15% to 10%.

5. Are there any method interferences that we have not considered?

Not that I am aware of.