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Association of
Metropolitan
Sewerage Agencies

February 23, 2001

William Telliard
Chief, Analytical Methods Branch
U.S. Environmental Protection Agency
Engineering and Analysis Division
Ariel Rios Building (MC 4303)
1200 Pennsylvania Ave., N.W.
Washington, DC 20460

Dear Bill:

AMSA greatly appreciates the EPA initiative and prompt implementation of the validation study for *Method 245.7 Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry*. As you know, the use of this method for low-level mercury analysis will greatly benefit the regulated community, with the savings of approximately $17,000,000 per year nationwide.

However, as the samples were distributed to the participating laboratories and the validation work begun, a serious omission was discovered by one of the AMSA member laboratories in the latest, November 2000 version of Draft Method 245.7. We are extremely concerned that this error may affect the final outcome of the study and jeopardize the final method validation.

The latest draft of the Method, in Section 10.1 regarding standard preparation directs the user to prepare the standards in reagent water only, without acidification. All previous versions of the method have the standards matrix matched with the samples, which included addition of hydrochloric acid and the bromate/bromide solutions at the same concentrations as in the samples. The sentence addressing the acid and bromate/bromide addition was omitted in the latest version. An addition of bromate/bromide may not be necessary for the standards, however, as any chemist performing metals analyses knows, metals standards must be acidified to be stable in the solution. As the above mentioned

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laboratory discovered, without the addition of acid to the calibration standards, obtaining good calibration and good performance of the method becomes impossible.

We believe that the omission of reagents in section 10.1 was unintentional, since the discrepancy was found in the following section (10.1.1.3). This section directs the user to add hydroxylamine solution to the standards to remove the yellow color caused by the addition of the bromate/bromide. Obviously, since this reagent was not added to the standards, the yellow color would not exist.

The DynCorp personnel in charge of the validation study have been contacted about this error, but directions they gave to the lab were: "continue using the method as written." We were informed that at least two of the participating laboratories have decided to go ahead with adding acid to the standards anyway, and are planning to note this deviation from the method in the data report

It is our extreme concern that the remaining laboratories may attempt to conduct the study, using the method "as written" and therefore their data, based on the improperly made and unstable calibration standards may adversely affect the outcome of the validation process.

The deadline for data submission is fast approaching (March 5, 2001) and most likely the laboratories are currently in the process of analyzing validation samples. We believe it is imperative that those laboratories be immediately notified of the error and be given an opportunity to correctly handle calibration standards.

We urge EPA to take an immediate action and notify participating laboratories without delay, of this inadvertent error and advise them on proper standard preparation procedure.

Sincerely,

Mark P. Hoeke
Director, Government Affairs

cc: Maria Gomez-Taylor, U.S. EPA