POTENTIAL SAFETY HAZARD IN METHOD 101A

INTRODUCTION

The potential exists in Method 101A for chlorine evolution with accompanying pressure increase in the recovered sample containers due to the 8 N HCl rinse during sample recovery.

SUMMARY

Method 101A was designed for mercury testing at combustion sources and was subsequently applied to sewage sludge incinerators. The brown product formed from $KMnO_4$ reduction during sampling is removed from the impinger walls with an 8 N HCl rinsing that is added to the recovered sample container.

Hydrochloric acid reacts with $KMnO_4$ to liberate chlorine gas. Although this is a minimal concern when small quantities of HCl (5-10 ml) are used in the impinger rinse, a potential safety hazard may still exist. At sources that emit higher concentrations of oxidizable materials such as power plants, more HCl may be required to remove the larger amounts of brown deposit formed in the impingers. In such cases, the potential more likely exists for safety hazards through sample pressurization due to addition of the larger HCl rinse to the recovered sample. These concerns are eliminated by storing and analyzing the HCl impinger wash separately from the permanganate impinger sample.

CONCLUSION

Section 7.2.1 of Method 101A should be modified as follows after the 250 to 400-ml $KMnO_4$ rinse:

> To remove any precipitated material and any residual brown deposits on the glassware following the permanganate rinse, rinse with approximately 100 ml of deionized distilled water, and add this water rinse to the permanganate Container No. 1. If no visible deposits remain after this water rinse, do not rinse with 8 N HCl. However, if deposits do remain on the glassware after this water rinse, wash the impinger surfaces with 25 ml of 8 N HCl, and place the wash in a separate sample container labeled Container No. 1A as follows:
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> Place 200 ml of water in a sample container labeled Container No. 1A. Wash the impinger walls and stem with the HCl by turning the

impinger on its side and rotating it so that the HCl contacts all inside surfaces. Pour the HCl wash carefully with stirring into Container No. 1A.

Analyze the HCl rinse separately by diluting the contents of Container No. 1A with stirring to 500 ml with deionized distilled water. Filter (if necessary) through Whatman 40 filter paper, and analyze for mercury according to Section 7.4, except limit the aliquot size to a maximum of 10 ml. Prepare a water-diluted 8 N HCl blank using the same procedure as used for Container No. 1A, except add 5 ml 8 N HCl with stirring to 40 ml water, and dilute to 100 ml with water. Then analyze as instructed for the sample from Container No. 1A.

Because the previous separate permanganate solution rinse (Section 7.2.1) and water rinse (as modified by this document) have the capability to recover a very high percentage of the mercury from the permanganate impingers, the amount of mercury in the HCl rinse may be very small, possibly even insignificantly small. However, add the total of any mercury analyzed for the HCl rinse sample (Container No. 1A) to that calculated for the mercury sample from Section 7.3.2 for calculation of the total sample mercury concentration.

REFERENCE

1. Memorandum from William J. Mitchell to Roger T. Shigehara discussing the potential safety hazard in Section 7.2 of Method 101A. February 28, 1990.