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OFFICE OF
SOLID WASTE AND
EMERGENCY RESPONSE

MEMORANDUM

SUBJECT: Use of Hydrochloric Acid (HCl) in digests for ICP-MS analysis

FROM: Schatzi Fitz-James, Chief, Waste Characterization Branch/s/
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Office of Resource Conservation and Recovery, USEPA

TO: EPA Regional Laboratory Directors I-X
EPA Regional QA Officers I-X
User Community for the SW-846 Manual

This letter is to inform you that the Office of Resource Conservation and Recovery (ORCR, formally OSW) is currently evaluating several issues with Method 3050 (Acid Digestion of Sediment, Sludges, and Soils). While we plan to address most of these issues as part of Update VI, there is one that we believe should be addressed before Update VI is published. Currently, two different digestion procedures are being used to prepare samples for analysis by inductively coupled plasma-atomic emission spectrometry (ICP-AES) method (i.e., Method 6010) or inductively coupled plasma-mass spectrometry (ICP-MS) (i.e., Method 6020).

SW-846 Methods 3050 A and B describe two different digestion procedures. The choice between them depends on whether the digest is to be analyzed by ICP-AES or ICP-MS. The main difference between these two procedures is that hydrochloric acid is included in the ICP-AES procedure, but not in the ICP-MS procedure. The historical reason for this difference is that high chloride matrices can result in the formation of molecular interferences in ICP-MS, especially for arsenic (from $^{40}\text{Ar}^{35}\text{Cl}^+$) and vanadium (from $^{35}\text{Cl}^{16}\text{O}^+$).

Unfortunately, lack of HCl in the digestion acid mix in 3050 B frequently results in poor recovery of antimony and silver (and potentially tin and zirconium) due to the formation of insoluble precipitates that would not be formed if HCL was present.¹ ICP-MS technology has improved since the last revision of Method 3050B in Update IV (2004). There are now several effective techniques available for removing molecular interferences, including the use of collision and/or reaction cells. Therefore, if it can be demonstrated that molecular interferences are under control (by using either collision and/or reaction cell and demonstrated using appropriate quality control samples), the use of hydrochloric acid in the digestion acid mix for samples to be analyzed by ICP-MS is allowed, and is preferred if silver and/or antimony are analytes of interest. The use of hydrochloric acid is also allowed for other target analytes, assuming that molecular interferences are well controlled.

Based on the same reasoning, sample digests prepared using methods 3005A and 3010A may be analyzed by ICP-MS, assuming that procedures to control molecular interferences are demonstrated and documented.

At this time, ORCR will continue to evaluate and revise method 3050 to incorporate other improvements. Until the Update VI is published, ORCR recommends that you follow the approach described in this note.

Should you have questions regarding this issue, please contact Kim Kirkland at (703) 308-0490.

cc: Kim Kirkland, MRWMD
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Charles Sellers, MRWMD

¹ D. D. Link, P. J. Walter, H. M. Kingston, "Wastewater Standards and Extraction Chemistry in Validation of Microwave-Assisted EPA Method 3015A," ES&T, Vol. 33, p. 2469-2473, 1999.