UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF SOLID WASTE
AND EMERGENCY RESPONSE

AUG 5 2010

MEMORANDUM

SUBJECT: Spiking (Prior To vs. After Sample Drying) Issue in SW-846 Organic Extraction Methods

FROM: James Michael, Chief, Waste Characterization Branch
Materials Recovery and Waste Management Division (MC-5304P)
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 Methods Manual

This letter is to inform you that the Office of Resource Conservation and Recovery (ORCR, formally OSW) is currently evaluating a quality control (QC) spiking inconsistency that commercial and EPA regional laboratories raised for the extraction of semivolatile and nonvolatile organic compounds in solids. In the interim, we are recommending that you not follow the cautionary notes in three SW-846 methods, specifically the notes in Section 9.3.1 of Method 3500C (for organic extraction and sample preparation), and Section 11.5 of Method 3545A (pressurized fluid extraction) and Section 11.3 of Method 3550C (ultrasonic extraction).

These cautionary notes were inserted into three Update IV SW-846 methods that were published on January 3, 2008 (73 FR 486-489). These notes state that, "it is CRITICAL that any compounds added to a sample, including surrogates, are added to the sample aliquot PRIOR TO any additional processing steps. This means that the surrogates and matrix spike compounds should be added to the sample PRIOR TO adding drying agents such as sodium sulfate to solid samples." The note in Method 3500C further states that, "As each 3500 series extraction procedure is revised, the order of the procedural steps will be made consistent with this note. However, until such time as all those methods are revised, the instructions in this note SUPERSEDE those in each extraction method."

Commercial and EPA Regional laboratories have pointed out that adding surrogates and other spiked compounds to environmental and QC samples prior to mixing with drying agents may cause major recovery issues depending on the analyte and/or the matrix. Additional instructions requiring evaporation of the solvent from the surrogate and spiking solutions compound the problem. For example, when the spike solutions are added to a clay sample, they may roll off without absorption to the matrix. The spiking solvent could evaporate quickly, before the solutions can be effectively mixed with the sample. Spiking in this manner has been

shown to cause considerable losses of the more volatile and light-sensitive compounds, resulting in poor recovery. One study showed recovery for more than 1/3 of the semivolatile analyte list can easily drop 50-100%.

In addition, we found that there is no such cautionary note in two other organic extraction methods that were published at the same time as the three above mentioned methods. These two methods, specifically Methods 3546 (microwave extraction) and 3562 (super critical fluid extraction), recommend spiking surrogates and other compounds after the sample drying procedure.

At this time ORCR is evaluating the information and records that were used in support of said spiking procedural change; communicating with commercial labs; and reviewing Department of Defense and Contract Laboratory Program protocols to verify what spiking protocols the analytical community is following. We plan on working with commercial and EPA Regional laboratories to acquire data for ORCR's consideration to resolve this spiking inconsistency and revise those three methods as necessary. Until that time, ORCR recommends that you not follow the language in these notes.

Should you have question regarding this issue, please contact Shen-yi Yang, of my staff at (703) 308-0437.

cc: Shen-yi Yang, MRWMD
Kim Kirkland, MRWMD
Charles Sellers, MRWMD
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