MEMORANDUM

DEPARTMENT OF ENVIRONMENTAL QUALITY
OFFICE OF WATER PERMIT PROGRAMS

SUBJECT: Guidance Memo No. 00-2006
Spiking Requirements for Metals Analysis

TO: Regional Directors

FROM: Larry Lawson, P.E.

DATE: March 23, 2000

COPIES: Regional Compliance and Enforcement Managers, Regional Technical Services Managers, Martin Ferguson, Richard Ayers, Bill Purcell, Karen Sismour, Howard Freelander, Maria Williams, and Betsy Ziomek

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SUMMARY: The purpose of this memo is to provide guidance to VPDES permittees to ensure that appropriate spiking requirements for metals analysis are met when citing Standard Methods and Methods for Chemical Analysis of Water and Wastes.

In Virginia, spiking of all metals samples analyzed by graphite furnace atomic absorption (GFAA) using Standard Methods or the 200 series included in Methods for Chemical Analysis of Water and Wastes has been required only for drinking water samples. Beginning June 1, 2000, spiking will be required on all VPDES compliance samples analyzed by GFAA using either of these methods. This change is due to a recent EPA inspection conducted in Virginia. At that time it was pointed out that the term 'matrix', as used in the methods, refers to an individual outfall, thus requiring spiking on 100% of samples.

> Standard Methods, 18th edition, Part 3020, pg. 3-3, states: "To determine whether matrix effects exist, make known additions to samples before any digestion." Part 3113B 4.d, further states: "Analyze all samples except those demonstrated to be free of matrix interferences (based on recoveries of 85%-115% for known additions) using the method of standard additions."
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> Methods for Chemical Analysis of Water and Wastes, in the "Notes" listed for each Method 2__.2 states: "For every sample matrix analyzed, verification is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption methods section of this manual)." When this method is cited, 10% of samples should be pre-digested spikes. The remaining samples may be post-digested spikes.

Laboratories have the option of using EPA Method 200.9, "Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption, Revision 2.2." EPA Region III has endorsed the approval of this method as an interim alternate test procedure. This method requires 10% of samples to be spiked prior to digestion, with a 70-130% recovery range. For labs to obtain approval to use this method for VPDES reporting, they must send a letter of request to Betsy Ziomek, DMR-QA Coordinator, VA DEQ, P. O. Box 10009, Richmond, VA 23240. The letter must include the method ID, parameters to be tested, and the permit number(s) for which the method will be used. Also, include a statement that Section 9.2 (Initial Demonstration of Performance) of the method has been completed and is being kept on file at the laboratory.

In an attempt to lessen the burden on laboratories performing GFAA, labs may request approval from DEQ's Waste Division to use EPA Method 200.9 for groundwater analyses associated with SW846. This gives laboratories the option of having one digestion method and one analytical method to satisfy three regulatory programs (VPDES/VPA, SW846 and Drinking Water). To apply for approval to use EPA Method 200.9 for SW846, the facility must contact Howard Freelander, DEQ, P. O. Box 10009, Richmond, VA 23240.

Questions or comments regarding this topic can be directed to Betsy Ziomek via telephone at (804) 698-4181, FAX at (804) 698-4032, or e-mail at esziomek@deq.state.va.us.

DISCLAIMER

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