



IN REPLY REFER TO:

United States Department of the Interior

U.S. GEOLOGICAL SURVEY

Box 25046 M.S. 407

Denver Federal Center

Denver, Colorado 80225

NATIONAL WATER QUALITY LABORATORY TECHNICAL MEMORANDUM 2005.02

Distribution: E
Date: January 28, 2005
Subject: Revision of the procedure for requesting laboratory matrix spikes
Effective date of change: March 1, 2005
Author: Michael Schroeder, Analytical Services Program
303-236-3270, schroede@usgs.gov
Revision: None

PURPOSE

The purpose of this technical memorandum is to revise the procedure for requesting laboratory matrix spikes of analyses at the National Water Quality Laboratory (NWQL). Laboratory matrix spikes apply primarily to organic compound determinations, although laboratory matrix spikes for inorganic and organic analyses not listed in the following table can be provided as custom analyses. See the 'Definitions' section below for a discussion of terms used in this technical memorandum.

BACKGROUND

Spikes of environmental matrix samples, whether prepared in the field or the laboratory, are part of the quality assurance program for many district projects involved in water-quality assessments. The primary use for environmental matrix spikes is to determine the effects of the environmental matrix on a method's recovery efficiency relative to recoveries obtained in laboratory quality control spikes or other reference materials. Field matrix spikes are preferred by many project chiefs because they follow the same procedural path as environmental samples, from field processing to laboratory analyses. Field matrix spikes may also provide information regarding the effects of sample storage and transport, provided a laboratory matrix spike is also analyzed. Laboratory matrix spikes may be useful as alternatives to field matrix spikes when the effects of sample storage and transport have been demonstrated to be insignificant, or when materials or facilities for preparing field matrix spikes are unavailable. Guidance on the preparation of on-site field matrix spikes and other quality-assurance samples is provided in the USGS National Field Manual (Wilde and others, 1999).

Field matrix spikes may routinely be submitted to the NWQL, with results reported to the field through standard National Water Information System (NWIS) procedures. To make an appropriate assessment of analyte recoveries, field matrix spike samples require analysis of a separate, unspiked environmental sample that has been collected at the same time. Field spiking solutions for some organic compound determinations may be purchased from the NWQL, with instructions for field

spiking included with the spike solutions. Spike solution lot numbers, solution concentrations, spiking instructions, and instructions for the calculation of spike recoveries are available from the NWQL website (<http://www.nwql.cr.usgs.gov/USGS/spike/spike.html>). Contact the NWQL at labhelp@usgs.gov for information regarding the availability of field matrix spike solutions not listed on the website.

In the past, laboratory matrix spikes have been requested in conjunction with field matrix spikes in order to evaluate potential losses of organic analytes in sample transit and storage. To facilitate identification of the replicate sample to be spiked at the laboratory, and to ensure that the same spike solution lot would be used as a laboratory spike, customers shipped an ampoule of spike solution from the field with the sample bottle to be spiked. Such concurrent shipment of field matrix spikes and replicates designated for laboratory spiking has decreased significantly, while the NWQL has received an increasing number of ad hoc requests for laboratory matrix spikes. Because a procedure for requesting laboratory matrix spikes independently of field matrix spikes has not been defined, occasional mistakes in processing have occurred. A formalized procedure for requesting laboratory matrix spikes will reduce processing errors and make information, such as spike solution concentrations and lot numbers, more readily available for data users.

SCOPE

The analyses for which laboratory matrix spikes may routinely be requested from the NWQL are listed in the table below. Laboratory matrix spikes for analyses other than those listed, including inorganic analyses, may be arranged as custom analyses by contacting the NWQL at labhelp@usgs.gov. Because of cost, mixture incompatibility, and analytical concerns, the spike solutions may consist of a subset of the analytes listed for a given analytical method. Similarly to field matrix spikes, all laboratory matrix spikes require analysis of a separate, unspiked environmental sample that has been collected at the same time. Note that concentrations detected in the unspiked sample are not subtracted from the spiked sample results prior to transmission of the analytical data by the NWQL. If laboratory matrix spikes are requested in conjunction with field matrix spikes, the NWQL cannot guarantee that the same lot of spike solution used to prepare the field matrix spike will be available for the laboratory matrix spike.

PROCEDURE

The suggested procedure for requesting laboratory matrix spikes is as follows:

- A separate analytical services request (ASR) form needs to be completed for each laboratory schedule for which a laboratory matrix spike is requested.
- The medium code of the sample to be spiked should be R (surface water) or S (ground water) and the sample type code should be "1" (spike)
- A sampling-time offset (e.g., add 1 minute) from the actual environmental sampling time should be used for each laboratory schedule for which a laboratory matrix spike is requested.
- In the appropriate spaces on the ASR, enter laboratory matrix spike lab code 4000, as well as the number of the laboratory schedule to be spiked. The price for laboratory environmental matrix spiking per lab code 4000 will be the same for all analyses.
- To ensure that the appropriate sample will be spiked upon arrival at the NWQL, the sample bottle to be spiked should be conspicuously labeled as "NWQL SPIKE XXXX", where "XXXX" is the laboratory schedule number.

When a laboratory matrix spike is requested per lab code 4000 and the appropriate laboratory schedule, analytical results and descriptive data will be transmitted to the NWIS database. Information stored in NWIS will include the following:

- The NWIS spike solution lot number (parameter code 99104).
- Spike type code (parameter code 99106). The spike type code will have a value of 20 to indicate laboratory spike.
- Spike source code (parameter code 99107). The spike source code will have a value of 10 to indicate the source as NWQL.
- Spike volume, in milliliters (parameter code 99108).
- Date of sample spiking at NWQL (parameter code 99874).

For further information, it may be helpful to refer to specific coding instructions found on the National Water-Quality Assessment Program (NAWQA) web page:

<http://ar.water.usgs.gov/nawqa/sample-coding/outline.html#defQCroutine>.

Instructions for requesting laboratory matrix spikes similar to those described here have been provided to NAWQA users of volatile organic compound (VOC) analysis schedules. VOC analysis users should continue to follow the specific instructions for laboratory spiking of VOC environmental samples for NAWQA; however, lab code 4000 should be used in place of lab code 8140 for requesting laboratory matrix spikes (see NAWQA Memorandum dated June 13, 2002 available at: <http://water.usgs.gov/nawqa-only/ftsup/CycleII.VOC.QAQCGuidance.doc>).

The NWIS spike solution lot number may be used to look up the analyte concentrations, as well as other useful information on the NWQL 'Organic Spike Lot Numbers and Certificates' website: <http://wwwnwql.cr.usgs.gov/USGS/spike/spike.html>. The information in the website may be used for documentation purposes and to calculate analyte recoveries with the same procedures used to calculate field matrix spike recoveries found at <http://wwwnwql.cr.usgs.gov/USGS/SpikeCalc.html>.

Following these procedures will enhance NWQL's ability to ensure that samples will be properly spiked at the NWQL according to customer's wishes. Please contact labhelp@usgs.gov, or phone 1-866-ASK-NWQL (1-866-275-6975) for questions or comments.

NWQL Analyses available for routine laboratory matrix spiking. Further information on these analyses may be found in the NWQL catalog at: http://wwwnwql.cr.usgs.gov/servlets_u/Catalac

Laboratory Schedule	Description
1380	Volatile organic compounds at minimum reporting levels, whole water
1383	Semivolatile organic compounds, base/neutral/acid extractable, whole water
1433	Waste water compounds, filtered water
2001	Pesticides, filtered water

2002	Moderate use pesticides and degradates, filtered water
2003	NAWQA, ACT/ULUG selected pesticides and degradates, filtered water
2020	Volatile organic compounds, NAWQA, plus tentatively identified compounds, whole water
2021	Volatile organic compounds, NAWQA, whole water
2060	Polar pesticides and degradates, filtered water
4024	Gasoline oxygenates and degradates, unacidified, water, unfiltered
4025	Gasoline oxygenates, degradates, and BTEX, acidified, water, unfiltered
4054	Volatile organic compounds at minimum reporting levels, plus tentatively identified compounds, whole water

Effect on Data Base: None

/signed/

Gregory B. Mohrman, Chief
National Water Quality Laboratory
Branch of Analytical Services

Supersedes: None

Key words: Spike, Organic, Matrix,

Distribution: E and <http://www.nwql.cr.usgs.gov/USGS>

DEFINITIONS

Custom analysis: A term applied to any analysis that requires a proposal. This includes modifications to an approved, operating U.S. Geological Survey National Water Quality Laboratory (NWQL) method or implementation of a non-NWQL approved method. Also included are analyses using methods that have not been completely evaluated applying USGS guidelines for developing approved methods and for which the proveout has not been completed. Proposals are required for all nonroutine analyses, including new method development, method modifications, and other projects requiring time and resources of NWQL staff beyond what is routinely available (Maloney, 2004).

Laboratory code (lab code): A four-digit code that uniquely represents a parameter determined by a particular method of chemical analysis (Timme, 1995)

Laboratory Schedule: A four-digit number that denotes a group of laboratory codes determined as a unit (Timme, 1995).

Matrix: The substrate of a test sample (National Environmental Laboratory Accreditation Conference, 2001). A matrix may consist of natural or synthetic material that is liquid, solid, or gas, or a combination of these phases.

Minimum reporting level (MRL): The smallest measured concentration of a constituent that may be reliably reported using a given analytical method (Timme, 1995).

Spike: A known mass of selected analyte added to a blank sample or subsample; used to determine recovery efficiency or for other quality-control purposes (National Environmental Laboratory Accreditation Conference, 2001). "Spike" and associated terms used in this memorandum refer to the practice of adding a known quantity of analyte to an environmental or laboratory matrix, for the purpose of evaluating analyte recoveries from the sample matrix using a given analytical procedure.

Laboratory matrix spike: An environmental sample fortified in the laboratory with known concentrations of all, or a representative selection of, the method analytes. The spikes are usually added in the laboratory immediately before sample preparation and analysis. Matrix spikes generally are considered project-specific QC samples because information about bias from a sample matrix is specific to a particular project, not to the performance of the method. Consequently, laboratory matrix-spike samples need to be submitted by project personnel as part of their QC samples. (Sandstrom, 1994)

Field matrix spike: An environmental sample fortified in the field with known concentrations of all, or a representative selection of, the method analytes.

REFERENCES

Maloney, T.J., 2004, National Water Quality Laboratory Quality Management System, accessed September 22, 2004 at <http://www.nwql.cr.usgs.gov/USGS/qms.html>

National Environmental Laboratory Accreditation Conference (NELAC), 2001 NELAC Standards, Chapter 1: Program policy and structure standard, accessed September 22, 2004, at <http://www.epa.gov/nerlesd1/land-sci/nelac/index.html>

Sandstrom, M.W., 1994, National Water Quality Laboratory Technical Memorandum 94-07, "Description and use by districts of Laboratory QC sample information in organic determinations," accessed September 22, 2004, at: http://nwql.usgs.gov/Public/tech_memos/nwql.94-07.html

Timme, P.J., 1995, National Water Quality Laboratory 1995 services catalog: U.S. Geological Survey Open-File Report 95-352, 120 p.

Wilde, F.D., Radtke, D.B., Gibs, Jacob, and Iwatsubo, R.T., 1999, Spike samples (section 4.3.3), *in* National field manual for the collection of water-quality data: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A1, accessed September 22, 2004, 2002, at http://water.usgs.gov/owq/FieldManual/chapter4/html/4.3_contents.html