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WATER LOGS

National Water Quality Laboratory Newsletter

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In this issue....

Update on audit, accreditation

The New York State Department of Health (NYSDOH) audited the NWQL the last week of July to ensure compliance with National Environmental Laboratory Accreditation Conference (NELAC) standards. The NELAC standards include criteria that address the requirements of the International Standards Organization (ISO-17025) and are widely recognized as the basis of the quality system for operating environmental laboratories. NELAC is sponsored by the U.S. Environmental Protection Agency with participants throughout Federal and State government agencies.

The NYSDOH is one of 12 current Accrediting Authorities (AA) approved to provide certification. Laboratory certification is recognized among all the AAs and by many other non-AA agencies.

The NWQL has been NELAC certified since January 2001. The current scope of accreditation has been primarily for Drinking-Water methods, but an expanded scope of accreditation for nonpotable water methods was submitted last May. The audit included a review of nonpotable water methods, the Drinking-Water methods we are currently accredited for, and the elements of our quality system.

The audit was challenging but successful. There were 20 percent fewer findings than for the NELAC audit conducted in summer 2001, and many of the findings required simple

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STUDENT INTERN—Craig Cone, physical science technician in the carbon lab, loads dissolved organic carbon samples for analysis by an ultraviolet persulfate total organic carbon analyzer. Craig is a junior at Regis University, in Denver. Donna Damrau, supervisor of the physical properties section, assists in the training program. (PHOTO BY LYNNE MONTROSE, REGIS)

corrective action. The audit findings and responses will be posted on the "Lab performance evaluations, audits, and certifications" page on the NWQL's USGS-visible webpage pending acknowledgment from NYSDOH. The direct URL to this page is http://wwwnwql.cr.usgs.gov/USGS/Performance/perf_eval.html .

Accreditation for the nonpotable water method is based on the audit findings and successful completion of two performance-testing studies. Results from the first of these studies were received in mid-October and are discussed on page 6. Refer to the URL in that article for a detailed list of the analytical procedures that are included on the requested scope of accreditation.

Participation in a second performance-testing study is planned for early winter, with the report expected in March 2004. At that time, NWQL expects accreditation for many routine nonpotable water methods. Once the NWQL obtains accreditation for the routine methods, we plan to drop accreditation for Drinking-Water methods because there is no demand for those methods.

• TOM MALONEY

EDITOR'S NOTE: An enthusiastic response to the electronic copy of our last quarterly issue of *Water Logs* was somewhat surprising but welcome news. As a result, we have decided to save postage, paper, and the costs of printing to deliver this issue—and all future issues—in electronic format. In consequence, we have also expanded our readership, no small part in efforts to improve communication with our customers and other interested parties. Drop us a note and let us know how we are doing. Your suggestions are always welcome!

GROUND-WATER SAMPLING— Students use a water-level tape to measure the depth to water in a well immediately adjacent to the National Water Quality Laboratory during a course in Field Water-Quality Methods for Ground Water and Surface Water, July 21–August 1, 2003, at the Denver Federal Center. Instructors introduced employees to USGS methodologies for collecting and processing samples for water-quality analyses. The intensive 2-week course included sample-collection and field-handling techniques, theory, methodology, and equipment.

BEATING THE FLU BUG— Shannon Taylor (right), administrative operations assistant, seems to enjoy being on the receiving end of a flu shot October 21 in the NWQL Infirmary. The vaccine was administered by Connie Riley, a nurse with the Public Health Service.

A cost-effective alternative for parent and degradate pesticides

Laboratory schedule (LS) 2003 was added to the NWQL Catalog this year as a complement to LS2001, LS2002, and LS2004. The schedule was created to meet the needs of the National Water-Quality Assessment Program (NAWQA) for the Agricultural Chemical Transport (ACT) and the Urban Land-Use Gradient (ULUG) studies. LS2003, however, is available for use by any USGS project.

LS2003 provides parent and degradate determinations in three major pesticide classes: triazine and acetanelide herbicides, organophosphate insecticides, and specific urban land-use pesticides. Other important urban land-use compounds, including fungicides and pyrethroids, also are included. Previously, to receive parent and degradate determinations, customers would need to request LS2001 and LS2002 or LS2004. A more cost-effective and efficient alternative was developed by selecting specific high-use compounds from these schedules to create LS2003. Combining parent pesticide and degradate determinations in the same schedule enables a more complete study of the fate and transport of these pesticides.

This new schedule offers the same detection limits, sample preparation, sample analysis, quality control and quality assurance as LS2001 and LS2002, but provides a shorter compound list with a greater cost savings for customers.

For more information, contact Mark Sandstrom <sandstro@usgs.gov>, Duane Wydoski <dwydoski@usgs.gov>, or Sonja Abney <srabney@usgs.gov> or refer to the NWQL Catalog: http://nwql.cr.usgs.gov/usgs/catalog/index.cfm?a=bs&sa=s&sap=2003

• SONJA ABNEY

New publications

(NWQL authors in boldface)

Reports

Barber, L.B., **Furlong, E.T.**, Keefe, S.H., Brown, G.K., and **Cahill, J.D.**, 2003, Natural and contaminant organic compounds in the Boulder Creek Watershed, Colorado, during high-flow and low-flow conditions, 2000, chap. 5 of Murphy, S.F., Verplanck, P.L., and Barber, L.B., eds., Comprehensive water quality of the Boulder Creek Watershed, Colorado, during high-flow and low-flow conditions, 2000: U.S. Geological Survey Water-Resources Investigations Report 03-4045, p. 103–144.

Jha, V.K., and **Wydoski, D.S.,** 2003, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of organophosphate pesticides in whole water by continuous liquid-liquid extraction and capillary-column gas chromatography with flame photometric detection: U.S. Geological Survey Water-Resources Investigations Report 03-4139, 26 p.

Patton, C.J., and **Kryskalla, J.R.**, 2003, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Evaluation of alkaline persulfate digestion as an alternative to Kjeldahl digestion for determination of total and dissolved nitrogen and phosphorus in water: U.S. Geological Survey Water-Resources Investigations Report 03-4174, 33 p.

Rose, D.L., and **Sandstrom, M.W.**, 2003, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of gasoline oxygenates, selected degradates, and BTEX in water by heated purge and trap/gas chromatography/mass spectrometry: U.S. Geological Survey Water-Resources Investigations Report 03-4079, 31 p.

Sandstrom, M.W., 2003, Pesticides in the Boulder Creek Watershed, Colorado, during high-flow and low-flow conditions, 2000, chap. 6 *of* Murphy, S.F., Verplanck, P.L., and Barber, L.B., eds., Comprehensive water quality of the Boulder Creek Watershed, Colorado, during high-flow and low-flow conditions, 2000: U.S. Geological Survey Water-Resources Investigations Report 03-4045, p. 145–172.

Journal Articles

Bednar, A.J., **Garbarino, J.R.**, Ranville, J.F., and Wildeman, T.R., 2003, Arsenic speciation and redox chemistry in acid mine drainage, *in* International Conference on Acid Rock Drainage, 6th, Cairns, Australia, 2003, Proceedings: Australian Institute of Mining and Metallurgy, p. 965–968.

Ferrer, Imma, Furlong, E.T., and Thurman, E.M., Identification of homologue unknowns in wastewater by ion trap MS^n: The diagnostic-ion approach, *in* Ferrer, Imma, and Thurman, E.M., eds., Liquid chromatography/mass spectrometry, MS/MS and time of flight MS—Analysis of emerging contaminants: Washington, D.C., American Chemical Society, ACS Symposium Series 850, p. 376–393.

Furlong, E.T., **Ferrer, Imma**, **Gates, P.M.**, **Cahill, J.D.**, and Thurman, E.M, 2003, Identification of labile polar organic contaminants by atmospheric-pressure ionization tandem mass spectrometry, *in* Ferrer, Imma, and Thurman, E.M., eds., Liquid chromatography/mass spectrometry, MS/MS and time of flight MS—Analysis of emerging contaminants: Washington, D.C., American Chemical Society, ACS Symposium Series 850, p. 175–187.

Ulrich, E.M., Helsel, D.R., and **Foreman, W.T.**, 2003, Complications with using ratios for environmental data: Comparing enantiomeric ratios (ERs) and enantiomer fractions (EFs): Chemosphere, v. 53, p. 531–538.

SAMPLE PREP—Chris Kanagy, chemist, prepares sediment samples during the extraction phase for schedule 1325 using a hexane-acetone solvent in a wrist-action shaker. The procedure is used to determine organochlorine pesticides and polychlorinated biphenyls (PCBs).

SETTING HIGH STANDARDS—Angeleen Newberg, physical science technician in the Carbon Unit, makes standards for phenol analysis.

For copies of these NWQL publications, contact Diana Rime by e-mail at dcrime@usgs.gov or by telephone (303-236-3714). NWQL published reports and journal articles are listed on the Web at URL http://nwql.usgs.gov/Public/pubs-public.html

Frequently asked questions

New water-quality analytical method available to determine gasoline oxygenates, degradates, and benzene, toluene, ethylbenzene, and xylenes (BTEX) in water.

What are the features of the new method? The method analyzes surface- and ground-water samples for gasoline oxygenates, selected degradates, and BTEX (benzene, toluene, ethylbenzene, and xylenes) compounds using heated purge-and-trap gas chromatography/mass spectrometry (GC/MS) at low concentrations [<5 micrograms per liter (µg/L) in whole-water samples]. Sample preservation at pH 2 is not recommended for these analytes because published studies have indicated the potential formation of *tert*-butyl alcohol from *tert*-butyl methyl ether. However, the new method report (Rose and Sandstrom, 2003) describes data from a holding-time study indicating that samples may be acid preserved to pH 2 with no changes in these analytes (except methyl acetate) for up to 46 days. Methyl acetate is only stable for 7 days at pH 2. All of the analytes are stable at pH 7 for at least 46 days.

Why is a new method for gasoline oxygenates needed? Gasoline oxygenates have been in the NWQL's Schedule 2020 method since 1997, but there was no way to determine the degradates of the gasoline additives. The degradates, specifically *tert*-butyl alcohol, *tert*-amyl alcohol and methyl acetate, require different extraction conditions for analysis. A separate method was needed for their determination. The new method, which used a heated extraction, provides determination of these degradates as well as oxygenates at environmentally relevant concentrations. The BTEX compounds were also included because they are major components of gasoline that can be found in water samples.

How sensitive is the new method? The short-term method detection limits (MDL) range from 0.035 to 0.052 µg/L for the gasoline oxygenates, 0.216 to 0.62 µg/L for the oxygenated degradates, and 0.005 to 0.036 µg/L for BTEX. The long-term method detection limit (LT-MDL) has not been assessed for this method. When at least 30 replicate laboratory-reporting-level (LRL) spikes are analyzed, the LT-MDL will be calculated.

What is the new method number? The USGS method number is O-4024-03 and may be requested through the NWQL as Schedule 4024 for unacidified samples and Schedule 4025 for acidified (pH 2) samples.

May any District use the new schedules? Yes. The Office of Water Quality approved Schedules 4024 and 4025 on 5 November 2002 for all projects and programs.

What analytes are determined in Schedules 4024 and 4025? A list of the gasoline oxygenates, degradates, and BTEX compounds is available on the NWQL USGS-Visible website at http://wwwnwql.cr.usgs.gov/USGS. Click on LIMS catalog and request Schedule 4024 (unacidified) or 4025 (acidified).

What bottle type, treatment, and preservation are necessary? Water samples should be collected using the procedure described in section 5.6.1 of the USGS Water-Resources National Field Manual, available at http://water.usgs.gov/owq/FieldManual/. Samples for the analysis of volatile organic compounds (VOC) are collected in triplicate (ground-water samples) or quadruplicate (surface-water samples) in clean 40-milliliter (mL) borosilicate amber vials (VOC vials) with Teflon-faced silicone septa. Supplies are available through 1-Stop Shopping (http://1stop. usgs.gov). No preservative is required for samples for Schedule 4024. Samples submitted for Schedule 4025 that may be degraded by bacterial action must be acidified to pH 2 using 1:1 NWQL quality-controlled hydrochloric acid (HCl). The vials are filled to overflowing, capped immediately, and labeled using the supplied label and a ball-point pen. Samples must be chilled and maintained at 4 °C ± 2 °C. Ship samples immediately on ice; do not use dry ice or samples will freeze.

Are blanks and spikes required? Field equipment, trip, and source-solution blanks may be submitted with the environmental samples. The field equipment blank confirms whether or not the equipment used to collect the samples is a source of contamination. Trip blanks are used to determine if contamination is caused by transportation. Source solution blanks evaluate the quality of the VOC-grade water to differentiate between contaminants present in the water itself and contaminants in the equipment. If the samples are preserved with HCL, then the source solution blank should also be acidified.

How do I submit samples for laboratory spikes? Three additional vials of an environmental sample are submitted on a separate Analytical Services Request (ASR) form, requesting both Schedule 4024 and lab code 8140. The sample time is offset from the environmental sample. Upon receipt, the sample will be spiked by an analyst, held for 5 to 7 days, and then analyzed.

May I have an environmental sample spiked for both Schedules 4024 and 2020? Yes. Six additional vials of the environmental sample are submitted. Three vials, unpreserved, are submitted as described above for laboratory spikes. Three additional vials, preserved to pH 2 with 1:1 HCI, are submitted for Schedule 2020 and lab code 8140, with the time offset from the environmental sample and the spike for Schedule 4024.

How are samples stored and analyzed? The NWQL stores VOC samples in the dark at 4 °C and analyzes them within 14 days of collection. Samples received within 4 days of sampling will be analyzed within 10 days of receipt in the order of arrival at the laboratory, unless

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special arrangements have been made in advance. Direct inquiries to LabHelp@usgs.gov.

How do I obtain a copy of the new method? A copy of the report may be downloaded from the NWQL USGS-Visible website (http://wwwnwql.cr.usgs.gov/USGS/pubs.html), requested by E-mail to the NWQL Technical Editor (jwraese@usgs.gov) or LabHelp@usgs.gov, or calling 1-866-ASK-NWQL. The citation follows:

Rose, D.L., and Sandstrom, M.W., 2003, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of gasoline oxygenates, selected degradates, and BTEX in water by heated purge and trap/gas chromatography/mass spectrometry: U.S. Geological Survey Water-Resources Investigations Report 03-4079, 31 p.

• Donna Rose, Mark Sandstrom, and Allison Brigham

TOOLS OF THE TRADE—USGS students attended a Data Toolbox class October 6–10 at NWQL in Denver to gain proficiency in using tools to evaluate data from the National Water Information System (NWIS). Class picture, front row (left to right): Susan Hartley, Atlanta, Ga.; Patricia Ladd, Helena, Mont.; Jon Dillow, Baltimore, Md.; Ronald Seanor, Ruston, La.; David Naftz, West Valley City, Ut.; Karen Thomas, Carson City, Nev.; Frank Rinella, Portland, Ore.; Cherie Miller (instructor), Baltimore, Md.; Robert Kent (instructor), San Diego, Calif.; Robert Broshears (instructor), Central Region, Denver. Back row: Kathy Neitzert, Rapid City, S.D.; Dwain Curtis and Virginia McGuire, Lincoln, Neb.; Shaun Wicklein, Altamonte Springs, Fla.; William Hughes and Andrew Hickey, Atlanta, Ga.; Lynn Taylor (NWIS instructor) and Timothy Rowe, Carson City, Nev.; Glenda Brown, NWQL; Roland Tollett and Stan Skrobialowski, Baton Rouge, La.; and Thomas Bushly (instructor), NWQL.

NWIS Toolbox class held at the NWQL

The NWQL hosted the "QW Data Toolbox for NWIS Users" October 6–10, in Denver. The class is offered to provide an assortment of tools to manage water-quality data in the National Water Information System (NWIS).

The NWQL generally participates in the Toolbox class to provide an overview of laboratory processes and Laboratory Information Management System (LIMS) software and its interface with NWIS. The location of the class at the NWQL allowed students to tour the Laboratory and participate in an annual luncheon.

The NWQL has its own copy of NWIS with test data for testing changes in the data release and to verify how data values are received and loaded into NWIS. Representatives of the NWQL Business Development Team (BDT) took the Toolbox training to increase understanding of customer needs.

General topics included structure of NWIS and USGS data-base policies; documentation of data; interface and use of software, such as Excel, S-Plus, QW switchyard, and SAS; data review and interpretation; and publication and archiving of USGS data. For information about future NWIS Toolbox classes, contact the class coordinator, Cherie Miller (cvmiller@usgs.gov or 410-238-4254).

• Sandy Turner

MANAGING DATA—David Naftz (left to right), Virginia McGuire, and Glenda Brown concentrate on the Toolbox course October 7 in the NWQL training room.

NEW INSTRUMENT—Steve Werner (seated, from left), NWQL chemist, Colleen Rostad, research chemist, National Research Program, John Fitzpatrick, technician with Applied Biosystems, and Ed Furlong (standing), NWQL research chemist, install a new triple quadrupole mass spectrometer, October 22, at the NWQL. The new instrument (shown in the background) will be used to develop specific and sensitive methods for determining hormones, pharmaceuticals, and other emerging organic contaminants.

EDUCATIONAL PROJECT—Five local middle-school students working on a photo essay toured the NWQL October 1 as part of a program to learn about civics and photojournalism. The Home of the Free™ program is an original project that provides 7th and 8th graders with a chance to work with Pulitzer prize-winning photographers as they chronicle the daily activities of public servants, community leaders, and government officials. The best work will be chosen for a national exhibition in Washington, D.C. next year. Kneeling in front is Sophia Pezoa; standing (left to right) are Jeffrey Poland (mentor with Cherry Creek Schools, Denver), Alexis Berkow, Kayla Ezell, Kelly Rawlins, James Marchino (mentor from Washington Mutual), and Josh Scott. For more details, visit www.wamuhomeofthefree.com.

Excellent ratings posted by the NWQL on recent performance tests for USGS analytical methods

This past summer, the NWQL took part in the first of two performance-testing studies administered through the New York State Department of Health (NYSDOH). The performance tests were part of the NWQL's effort to achieve accreditation by the National Environmental Laboratory Accreditation Conference (NELAC) for USGS methods on environmental water samples. Samples were logged in using regular USGS schedules and lab codes. For the most part, the NWQL requested accreditation for specific analytes by using more than one USGS method. [An analyte is the substance being determined in an analysis.] The NWQL successfully competed in nearly 96 percent of all attempted tests.

The results of the NYSDOH nonpotable water study conducted last summer are posted on the Web at URL http://nwql.usgs.gov/Public/Performance/publicny0303.html . This site is best viewed with Internet Explorer.

A second round of performance testing will follow in January 2004 with results available by late April. After completion of the second study, the NWQL will receive accreditation for all method-specific analytes that received two successive satisfactory ratings. The NWQL will maintain accreditation by taking part in semiannual nonpotable studies administered by NYSDOH.

• **Mary Cast**

Newsletter Staff

Jon Raese, Editor
Diana Rime, Editorial Assistant

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