NATIONAL WATER QUALITY LABORATORY
NEWSLETTER
U.S. DEPARTMENT OF THE INTERIOR U.S. GEOLOGICAL SURVEY
Volume 2 Number 3 July 1994

Six new analytical methods developed at Lab to meet NAWQA Program requirements

During the past 2 to 3 years, six new analytical methods have been developed and implemented at the NWQL to meet the needs of the reconnaissance and monitoring phase of the National Water-Quality Assessment (NAWQA) Program. In general, these methods offer increased number of analytes, lower detection limits, greater efficiency, and improved quality-control procedures compared to older methods.
The new methods are as follows:

1. Determination of pesticides in water by solid-phase extraction and gas chromatography/mass spectrometry (GC/MS) (Schedules 2001/2010)
2. Determination of pesticides in water by solid-phase extraction and high-performance liquid chromatography/ultraviolet-diode array detector (Schedules 2050/2051)
3. Determination of chlorinated organic compounds in aquatic biota by gas chromatography/electron capture detector (GC/ECD) (Schedule 2100)
4. Determination of trace organic semivolatile compounds in bed sediment by GC/MS (Schedule 2502)
5. Determination of trace chlorinated organic compounds in bed sediment by GC/ECD (Schedule 2501)
6. Determination of trace metals in aquatic biota (Schedule 2200)

These methods will provide accurate and reliable data at a significantly lower cost to the Water Resources Division (WRD). For example, 2,714 samples were analyzed by Schedules 2001/2010 during 1993 at a total cost of $530,100. Using comparable older analytical methods would have cost $2,632,580, a saving of over $2 million to NAWQA.

The new methods will be approved by the U.S. Geological Survey (USGS), published as USGS Open-File Reports, and made available to the WRD in the coming months. As these methods are approved and published, a technical memorandum will report their availability, along with announcements in future editions of the *Newsletter*.

For information about these methods, please contact Mark Sandstrom, Chief, Methods Research and Development Program (telephone 303-467-8086).

by Mark Sandstrom

Income estimates lagging

The April reporting period for estimates of District income for the National Water Quality Laboratory was not as successful as the request for the January period. Most estimates were tardy and several Districts did not respond, according to Deborah Treseder, Administrative Officer.

NWQL management relies on the accuracy and timeliness of the estimated quarterly income to keep Headquarters and Regional staff informed. Planning for fiscal-year expenditures requires up-to-date information.

Treseder applauded the Western Region for being the first Region to respond in its entirety. She also reminds the Districts that the deadline for the next response is July 18. Once again, she asks for totals for the entire fiscal year, not just from July through September.

New class-100 clean room

Jerry Hoffman, chemist in the Methods Research and Development Program, has recently overseen the completion of a modular type class-100 clean room facility at the National Water Quality Laboratory.

This new facility has been designed to lower ambient atmospheric particulate concentrations for trace metals equivalent to a class-100 clean room as described in Federal Standards. The clean room is a controlled-environment facility in which all incoming air passes through filters capable of removing a minimum of 99.97 percent of all particles 0.3 micrometer or larger. The clean room is maintained at a positive pressure relative to the ambient pressure immediately outside to prevent leakage of nonfiltered air into the clean room. In addition, the use of metal components in construction has been minimized, and, where used, they have been covered with vinyl or painted with polyurethane.

Gerry Hoffman uses the class-100 clean bench to dry acid-cleaned plastic ware. Note the HEPA-filter units in the ceiling.

The new clean room will be used to prepare samples for trace-metal determinations at nanogram-per-liter concentrations and to clean apparatus and containers used for water sampling and instrumental analysis.

The clean room is available for custom service for WRD Districts that need special sample handling. A District program requiring special cleaning for sample bottles, sampling equipment, or nonroutine sample preparation can obtain these services by contacting Jerry Hoffman (303-467-8082) or Merle Shockey (303-467-8101) at the NWQL.

by Debbie Vaught

More insights into sample handling and analysis at the NWQL

What happens to the bottle after you put it in the mail?

By Ed Furlong
Methods Research and Development

Editor's note: This column contains questions that originally were part of the Water Resources Division (WRD) training class "Water-Quality Principles." Through this question and answer format, Paul Capel (Minnesota District) and Ed Furlong (Methods Research and Development Program) answer some of the more common questions that WRD personnel ask the NWQL. If you have a question about the Laboratory, please send it via EDOC to EFURLONG. Selected questions and answers will be printed in future columns.

Before we send in some kinds of water samples, we add nasty chemicals, but not to others. Are you trying to get rid of some of your analysts back in the lab?
No, in fact we need to bring in more analysts rather than get rid of them. We do add chemicals (mercuric chloride, nitric acid, sodium thiosulfate) to preserve some analytes that Laboratory staff knows will rapidly decompose or otherwise change concentration during transit, even using chilling, overnight shipping, and properly cleaned containers.

I send in a bottle of water and some money and get a piece of paper with some numbers (concentrations of analytes) in exchange. Can you explain to me what goes on in between? Say I want to get a chloride analysis? . . . How about a DDT analysis?
Both determinations: The sample is logged in at the NWQL and given a laboratory identification (I.D.) number. The DDT sample is placed in a refrigerator. Individual samples (and associated paperwork) are grouped into sets of samples that require the same analysis and are picked up by an analyst. The analyst sorts samples by I.D. and verifies them with the paperwork.
Chloride determination: Samples are dispensed into autosampler cups. There are no analyte-concentration or isolation steps. An ion chromatography method is used, which is based on anion exchange. An aliquot of the sample is injected onto an anion exchange resin column. Anions, including chloride, are retained by the column. The column is then rinsed with a carbonate/bicarbonate solution that remobilizes the anions. The different anions are eluted (washed off) the column in a specific, reproducible order. The liquid stream flows through a conductivity detector that generates a signal as the anions pass through. The observed signal--recorded as a near-Gaussian peak--is proportional to the increase in conductivity resulting from the ions present. The area under the peak is proportional to the anion concentration in the sample. The exact proportions of peak area to analyte concentration are determined from a calibration curve derived from standard solutions.

DDT determination: Samples are extracted within 48 hours of receipt by shaking the 1-liter water sample with hexane three times. The hexane aliquots are collected, residual water removed, and reduced to a volume of 1 milliliter. The crude extract is separated into several fractions by column chromatography with alumina and silica gel sorbents. DDT is isolated in a single fraction, but since DDT is only one component of the schedule, several fractions are collected, reduced to 1 milliliter, and analyzed. The analysis is done by fused silica capillary column gas chromatography with electron capture detection. Two different capillary columns are used, each coated internally with a different polymer. DDT elutes (comes out) at different times on each column, and the two columns are used to verify DDT's identity. The peak area for DDT (proportional to the DDT mass) is measured for each column. The measured concentration using each column is calculated, the two values compared, and the lower of the two values reported, assuming that the higher value may indicate the presence of a coeluting interference. The data are tabulated, entered into the Laboratory Information Management System (LIMS), and then transmitted to the Prime.

What if I try to trick you and send in the exact same water for the same analyses, but call it two different samples? What kind of results do you think I'll get back? Does anybody ever do such a silly thing?
Occasionally people send in field duplicates. There are difficulties in collecting a good field duplicate, but assuming this is done correctly, your results will depend on the type of analysis you request, the constituent of interest, and the sample matrix. Further assume that the constituent concentrations are at least five times the method reporting limit. Then for dissolved chloride in water, the results should be pretty close, within 5 to 10 percent. For DDT in a bed-sediment sample, the result could vary as much as 50 to 100 percent. Field duplicates are valuable because they provide a sense of how precise the analytical method is for samples from your study area.

There are so many choices in your Services Catalog. How am I supposed to decide which analysis to get done?
Call the NWQL! Both the Organic and Inorganic Chemistry Programs of the NWQL are happy to assist you in deciding which analyses will best help you to achieve your study objectives. If you have a custom request, call the Methods Research and Development Program (Mark Sandstrom, Program Chief, 303-467-8086) at the NWQL. These programs work together to provide District hydrologists with the best analytical services possible. In addition, if we do not provide a particular analysis, dioxins for example, then we will contract out for the analytical services you need. Finally, the new NWQL Services Catalog (Timme 1994) is just off the press; it provides much information in a user-friendly form.

References
Pritt, J.W., and Raese, J.W., 1992, Quality assurance/quality control manual: U.S. Geological Survey Open-File Report 92-495, 33 p.

Timme, P.J., 1994, National Water Quality Laboratory 1994 services catalog: U.S. Geological Survey Open-File Report 94-304, 104 p.

Somewhere the sun was shining . . .

The word of the day was irony. What more could the Water Lab want for a picnic than rain? And what did we expect for a Friday the 13th in May? Irony. Yet irony is sometimes positive; we gathered at Pioneer Park in Arvada which has a huge covered pavilion. It held all 125 of us. Happy irony. So, though the skies were steely (irony?), so was our iron will to cheerfully ignore them and party on. We had plenty of excellent food, the usual comraderie around the keg, and a bit of entertainment provided by a few die-hard softballers who disdained the drizzle. In retrospect, the highlight of the day had to be our chefs cooking under aluminum foil hats. Ironically, no one will admit to praying for lightning!

Unflappable softball player – Clarence Nichols, Safety Office, catches for switch-hitter Linda Pratt, Laboratory Operations Program

Jeff Pritt, Quality Management Program

Al Driscoll, Quality Management Program takes on Betty McLain, Inorganics Program, in the cribbage finals.

Ralph White, Quality Management Program and his son Jordan

Maggie Page, Mary Flansberg, and Nancy Warner, Administrative Services

Ron Berg, Laboratory Operations Program, with Pete Cinotto and Jeff Deacon, Organics Program

Kailin Terry, Quality Management Program.

Suranne Horodyski and daughter Ashley with Kim Pirkey, Quality Management Program.

Barbara Kemp, Methods Research and Development Program and Pete Rogerson, NWQL Chief.

Mark Sandstrom (left), Kevin Fehlberg, and Ed Furlong, Methods Research and Development Program.

We wish to especially thank Jeff Deacon, Jim Kammer, Mike McGinley, George Herrera, and Nancy Warner for orchestrating this shebang--Nancy literally dodged trains to get the food to the park on time; Debbie Vaught, Suzy Ahrendts, and Barbara Kemp for jumping in to save the day setting up; and Tracye Meyer, Donna de Avera, Diane Moffett, and others for making sure the park was sparkling when we left. And to all, thanks for the memory.

by Karlin Allen

Laboratory chemists developing new methods for suspended-sediment samples

U.S. Geological Survey chemists John Garbarino and Tom Leiker, Methods Research and Development Program, are part of a research team determining the spatial distribution of herbicides in the Mississippi River in May-June 1994. The upriver trip started May 31 below New Orleans, La., and was scheduled to end about June 11 at St. Paul, Minn., a distance of some 1,700 river miles.

The objective of this investigation is to determine the spatial distribution of the concentrations and flux of organic contaminants and trace metals in the major tributaries and the main stem of the Mississippi River during spring runoff. Previous work has shown that the distribution of atrazine along the Mississippi River main stem is "patchy." It has been hypothesized that this distribution results from "slugs" of water containing high concentrations of atrazine which originate in various tributary basins and which enter the Mississippi River on differing time scales.

The two National Water Quality Laboratory chemists are evaluating new methods for the extraction and determination of organic and inorganic constituents associated with suspended sediment from samples of natural water. Attempts will be made to identify the source of contamination plumes by collecting whole-water samples every 10 miles.

Major herbicides being studied include atrazine, cyanazine, metolachlor, alachlor, the alachlor metabolite ESA, and a new herbicide--acetochlor. No data have previously been collected for acetochlor.

Brown bag seminars--past and present--announced

Bill Steinkamph, Water Resources Division, "Yucca Mountain--Sample Collection," April 7; Mark Sandstrom, NWQL, "Field and Laboratory Quality-Control Samples for Low-Level Pesticide Analysis," April 13; Ed Furlong, NWQL, "Field Applications of Membrane Solid-Phase Extraction," May 11; Jerry Hoffman, NWQL, "Everything You Ever Wanted To Know About Silver Determinations But Were Afraid to Ask," May 18.

Charles Patton, NWQL, "How Effective Are Methods Used by the U.S. Geological Survey and U.S. Environmental Protection Agency To Preserve Samples?" June 1; Bill Foreman, NWQL, Course lectures from "Environmental Processing of Organic Chemicals, I--Vapor Pressure," June 8; John Garbarino, NWQL, "Where Have All the Heavy Metals Gone in the Mississippi River?" June 15; Tom Leiker, NWQL, "Determination of Chlorinated Pesticides by NCI Mass Spectrometry," June 22.

Seminars begin at 11:00 a.m. in the conference room on the second floor of the East Laboratory.

New reports off the press

New titles published by the National Water Quality Laboratory (NWQL) are as follows:

Lindley, C.E., Burkhardt, M.R., and DeRusseau, S.N., 1994, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory--Extraction of nitroaromatic compounds from water by polystyrene divinylbenzene cartridge and determination by high-performance liquid chromatography: U.S. Geological Survey Open-File Report 94-62, 15 p.

Timme, P.J., 1994, National Water Quality Laboratory 1994 services catalog: U.S. Geological Survey Open-File Report 94-304, 104 p.

For copies of these reports, write directly to the NWQL or send electronic mail to KIALLEN.

Newsletter Staff

Jon Raese, Editor

The National Water Quality Laboratory Newsletter, is published quarterly by the National Water Quality Laboratory, U.S. Geological Survey, Box 25046, MS-407, Denver Federal Center, Denver, CO 80225-0046. For copies, call Jon W. Raese (303) 236-3464.