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Custom analysis: A winning proposition for customers and the National Laboratory

The National Water Quality Laboratory has been providing custom analytical work for the past 20 years, including inorganic, organic, and radiochemical analyses. In addition, the NWQL can respond now to requests for custom biological taxonomic work.

What is a “custom” analysis?—An analysis is considered “custom” if it is used to determine a specific chemical compound or compound class that is not in a USGS-approved method. For example, a custom method might include a new compound that is being added to an approved method, or a specified compound, which is not available using USGS-approved methods but needs to be developed. A custom analysis, on the other hand, does not include deleting a compound or set of compounds from an approved method.

Custom analytical proposals have been used to handle special reporting levels, atypical matrix or sample type. In addition, the development of new analytical methods usually starts with a custom analysis.

These custom projects, which require close communication between customers and the NWQL, have been successful in advancing science, and at the same time provide customers with information useful for scientific and policy decisions. The following are examples of ongoing or completed custom analyses:

Fipronil and its degradates—The Louisiana District, Aventis (the manufacturer), and the NWQL investigated the capability of analysts to detect fipronil and its degradates in irrigation water. Fipronil and three of its degradates are going to be added to Schedule 2001 as approved compounds based on the results of this custom project.

Methyl *tert*-butyl ether (MTBE)—MTBE was identified first by analysts at the NWQL. The National Water-Quality Assessment Program (NAWQA) Volatile Organic Compound Synthesis Team was informed and investigations ensued. As a result of this proposal, MTBE was included in Schedule 2020; and a new method for gasoline oxygenates was developed by NWQL (an ongoing research project).

Wastewater indicators and pharmaceuticals—These NWQL proposals have resulted in the determination of compounds that have not been identified systematically in the United States. The results have been reported widely in the media and emphasize the role of the USGS as a leader in water-quality analysis.

Sediment method—The NWQL and the Texas District have collaborated to develop a sediment analytical method for selected organic contaminants. The results include the development of a USGS-approved method that lowers analytical costs.

Additional projects—These projects include saltwater intrusion in New Jersey, metal speciation, and snow and fish analysis. The NWQL currently (2002) has about 20 ongoing custom projects.

All custom analyses require a proposal. The proposal is a formal agreement between the NWQL and the customer detailing the entire analytical work, including the price, data transmittal method, reporting levels, and billing method. Contact Gary Cottrell (cottrell@usgs.gov) for additional information about specific projects or the custom analysis process.

• Mark Burkhardt

Instructions changed for sample shipments

The Laboratory recently received sample shipments that were sent on Friday for arrival at the NWQL on Saturday. The Fed Ex airbill on these shipments did not indicate to Hold for Pickup or Saturday Delivery. The coolers did not arrive at the NWQL until the next business day, which was Monday or Tuesday, in the case of a Federal holiday. As a result of the delay in receipt, the samples arrived warm, and customers were informed that the samples did not meet the minimum temperature requirements for login and analysis.

To avoid this problem, updated instructions from the local Fed Ex Office require the airbill to be completed as follows:

- Section 4a – Mark the shipment for “PRIORITY OVERNIGHT.”
- Section 6 – Mark for “SATURDAY DELIVERY.”

The coolers will be held at the local Fed Ex office on Saturday for pickup by an NWQL employee. These instructions apply when shipping samples priority overnight on Friday for arrival at the NWQL on Saturday. A \$10.00 processing fee will be added by Federal Express to the shipping charge for handling on Saturday.

SERVICE VETERANS—Merilee Bennett, administrative officer for the National Water Quality Laboratory, recently received a 30-year certificate from Tom Casadevall, USGS Central Region Director. Mike Werito, physical science technician in the Safety Office, also received a 30-year certificate.



Summary sheet listing NWQL capabilities, services enclosed with *Water Logs*

Newsletter readers will find a special insert in the July issue of *Water Logs* that lists the unique capabilities of the National Water Quality Laboratory.

The summary accomplishes the following:

- Responds to requests from NWQL customers;
- Documents the Laboratory’s analytical and research credentials and capabilities;
- May be used by customers to develop cooperative agreements that include environmental analyses; and
- Increases the customers’ awareness of the complexity of producing and maintaining defensible data of known quality.

The summary is being posted to the Laboratory’s website for downloading and printing additional copies. A Rapi-Note will be issued shortly to identify the website for easy access. Please feel free to send us your comments and suggestions regarding the use and effectiveness of this publication; address your comments to Allison Brigham (303-236-3465, abrigham@usgs.gov). And, if we can provide multiple copies for District use and cooperators, get in touch with Diana Rime (303-236-3502, dcrime@usgs.gov).

LIMS celebrates first birthday; new applications underway

The new Laboratory Information Management System (LIMS) was launched May 21, 2001. To celebrate, the Information Technology group hosted a birthday party with traditional cake and ice cream.

The first year was a challenge. Not only was the new LIMS launched this past year, but the National Water Information System (NWIS) revised its software. Between the two new releases of software and the increased security measures, it has been

difficult for the user to identify the source of data problems. The LIMS group meets bi-weekly with the Phoenix group of NWIS to identify problems and sort out inconsistencies. The NWQL requests that all problems be reported to labhelp@usgs.gov. We will make sure the problem report gets to the appropriate system personnel.

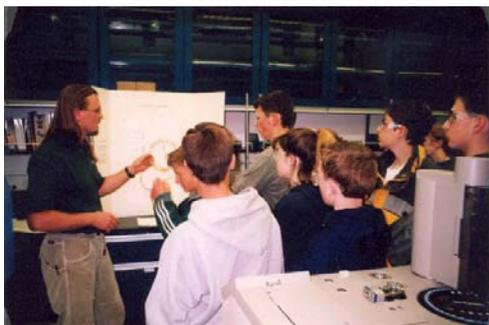
Only a few samples remain on the old Laboratory Analytical Data System (LADS). These samples will be converted to the new LIMS in the near future. The major emphasis for the LIMS group has changed from implementation and error correction to enhancements and new applications. All of the analyses that were supported on the LADS are now supported by the LIMS. Many of these analyses have been changed from manual data entry of results to electronic transfer of data. In addition, quality-control results are maintained by the system.

As the analysts learn the power of the new LIMS, they are requesting new applications and enhancements. End-of-day reports are automatically generated for the Login Section, and customers are sent e-mail when their samples are logged in. Paper copies of Analytical Services Requests (ASRs) are scanned into the system to allow immediate access without hunting through files of paper forms. An electronic Analytical Services Request (eASR) with barcode has been developed and is in production testing now.

An NWQL proposal module was developed to support low-demand and custom analyses. A special number is assigned to the proposal when it is approved. This number is entered on the ASR to activate the proposal for the specified samples. The proposal can allow a special price to be associated with the samples as well as limit the use of the proposal by time frame or by number of samples. Automatic e-mail is sent to the personnel designated by the proposal at the beginning and when the proposal is nearing completion. The proposal can then be extended, if necessary.

We think the LIMS will skip the "Terrible Two's" and enjoy a long and productive life!

• Sandy Turner



INTRODUCTION TO CHEMISTRY—Chris Kanagy, chemist in Analytical Services, served as one of the tour guides April 25 during Bring Your Child To Work Day at the NWQL.

New publications (NWQL authors in boldface)

REPORTS

Jha, V.K., and Wydoski, D.S., 2002, *Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of organophosphate pesticides in filtered water by gas chromatography with flame photometric detection: U.S. Geological Survey Water-Resources Investigations Report 02-4071, 29 p.*

JOURNAL ARTICLES

Bednar, A.J., Garbarino, J.R., Ranville, J.F., and Wildeman, T.R., 2002, Preserving the distribution of inorganic arsenic species in groundwater and acid mine drainage samples: *Environmental Science & Technology*, v. 36, no. 10, p. 2213–2218.

SAMPLE PREP—April Dittrich, physical science aid in Analytical Services, elutes a pesticide sample from the C-18 carbon cartridge. The analytical process is called "vialing." After elution, the sample is sent to a chemist for compound identification.



Letters, faxes, and e-mail

To all my friends at USGS:

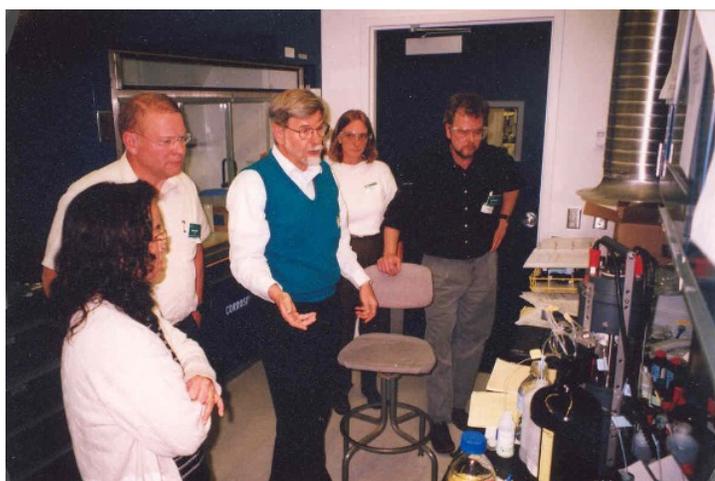
Your love, share, gifts, thoughts, prayers, cards, concerns, and laughs, along with warm memories, have meant more than I can convey in a letter. Several of you have asked how I'm doing? I have completed 35 radiation treatments at 35 minutes each. The radiation program is completed. The chemotherapy program is continuing, and I will enter the seventh cycle this week.

I know I have not thanked everyone enough, but the *NWQL Water Logs* is a start. Nobody knows the difference that kind words can make in a person's life. I know because they have made a difference in my life. Thank you all so much for helping me to keep the glass half-full with a positive attitude.



Really, all of us only have today, so let's make it special. You have made my life special during these difficult and trying times.

W. Ralph White, Supervisory Chemist
June 5, 2002



TOUR DE FORCE—Pete Rogerson (center), senior chemist for the Office of Water Quality, guides members of the USGS Upper Midwest Environmental Sciences Center (UMESC) through the nutrients research lab May 20 at the National Water Quality Laboratory in Denver. Left to right are Shirley Yuan, chemist from La Crosse, Wisconsin; David Soballe, chief, Long-Term Resource Monitoring Program; Rogerson; Janice Ward, assistant regional hydrologist, Central Region Water Programs; and William Richardson, program chief, Nitrogen Cycling in the Upper Mississippi Basin. The UMESC documents the effects of chemicals and drugs used in public fish hatcheries. It also studies the population and ecosystem effects of invasive, nonnative species in the Upper Mississippi River and Great Lakes Basins.

The UMESC, in La Crosse, is one of 18 USGS biological research and technology centers in the United States.

Client survey underway

The NWQL is asking its customers to evaluate the Laboratory's services. With the assistance of the Regional Water-Quality Specialists, the Laboratory is distributing a client survey to an initial core-respondent group of about 100 persons. These individuals have an interest in, and considerable experience with, the Laboratory. Following the initial release of the survey to the test group in mid-June and a preliminary review of the results in early July, the survey will be made available to all of the NWQL's customers.

Customers are asked to rate the NWQL based on their experiences over the past several years. Space is provided throughout the survey for respondents to include additional information if they feel that the survey has not asked the right question(s). Questions deal with products and services that customers would like to see offered or improved at the NWQL. Customers are encouraged to describe other laboratories that provide superior services, customer support, value, and quality. The NWQL wants to set benchmarks that can be used for planning and evaluating progress.

The NWQL's commitment to its customers for taking the time to respond to the survey is two-fold. First, survey results will be made available on the Web and elsewhere. Second, the Laboratory will use the information to make substantive changes and improvements, and continue to enhance its products and services.

Frequently asked questions

New water-quality analytical method available to determine 20 organophosphate pesticides and 5 degradates in filtered water

What is the new method number?

The USGS method number is O-1402-01 and may be requested through the NWQL as Schedule 1402.

Where may I find information on the compounds included in Schedule 1402?

A list of 20 parent pesticides and 5 degradates, and associated laboratory and parameter codes is available on the NWQL USGS-Visible website at <http://www.nwql.cr.usgs.gov/USGS>. Click on LIMS catalog and request Schedule 1402.

May any District use the new schedules?

Yes. The Office of Water Quality approved Schedule 1402 on 1 February 2002 for all projects and programs.

What are the features of the new method?

The filtered-water method is most applicable for compounds that are (1) primarily in the dissolved phase in a water sample, (2) efficiently isolated from the sample matrix and sorbed onto a C-18 solid-phase extraction (SPE) column, (3) efficiently displaced from the SPE column by an ethyl acetate elution solvent, (4) chromatographically resolved and identified using a gas chromatograph equipped with flame photometric detectors, and (5) sufficiently stable to chemical or thermal degradation to allow accurate quantification.

Seven compounds will be routinely reported with the "E" data qualifier, signifying that although the compounds are qualitatively identified as present, their reported concentrations have greater uncertainty because of poor recovery, high variance, or short holding times.

Is there an advantage to using Schedule 1402?

This method is a low-cost complement to other filtered-water methods, such as Schedules 2001 (pesticides) and 2060 (polar pesticides and pesticide metabolites). Schedule 1402 is an alternative to Schedule 1319 (organophosphates in whole water by gas chromatography with flame photometric detection). Analyte concentrations reported from Schedule 1402 are only for filtered water. The total concentration of some compounds that sorb to suspended matter in the water may be underestimated.

What bottle type, treatment, and preservation are necessary?

Water samples should be filtered using the procedure described by Wilde and others (1999)* in the USGS Water Resources National Field Manual, available at <http://water.usgs.gov/owq/FieldManual/>. Do not rinse bottle. Samples are collected in precleaned (baked at 450 °C by laboratory) 1-liter amber glass bottles available through 1-Stop Shopping (<http://1stop.usgs.gov/>). Do not fill bottle beyond shoulder because reagents must be added at the NWQL before analysis. Chill and maintain at 4 °C. Ship at once.

How sensitive is the new method?

Single-operator method detection limits (MDLs) determined and combined in three sample matrices (reagent water, surface water, and groundwater) range from 0.0004 to 0.012 microgram per liter (µg/L).

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://www.nwql.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:

Jha, V.K., and Wydoski, D.S., 2002, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of organophosphate pesticides in filtered water by gas chromatography with flame photometric detection: U.S. Geological Survey Water-Resources Investigations Report 02-4071, 29 p.

*Detailed references are provided in the published method report.

• Duane Wydoski and
Allison Brigham

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