NATIONAL WATER QUALITY LABORATORY
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No coffee breaks for analysts handling caffeine samples

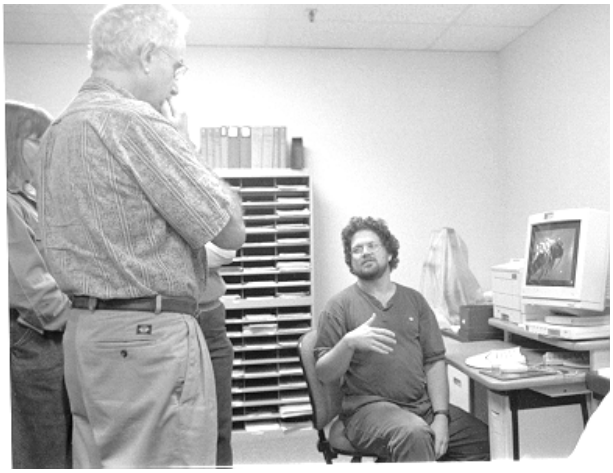
NWQL analysts have modified a method for the low-level determination of caffeine in whole-water samples. Caffeine is of interest to environmental scientists because it is a waste-water indicator, both in surface- and ground-water systems. The method was developed in response to inquiries from U.S. Geological Survey customers in Kentucky and Minnesota.

To evaluate the transport and scope of waste-water contamination, caffeine concentrations need to be measured as low as 0.01 µg/L (microgram per liter). Steve Smith and Jamie Alexander modified the analytical technique used to determine priority pollutants to focus specifically on caffeine. The result was a more efficient analysis using gas chromatography and mass spectrometry (GC/MS) and a lower detection limit than was feasible under the unmodified technique.

Provisional method recovery studies performed on 1 L (liter) of reagent water laboratory spikes have yielded recoveries ranging from 80 to 100 percent and a method detection limit of 0.02 µg/L. An expected (though still somewhat surprising) complication of the low-level determination of caffeine has been the difficulty in controlling background caffeine levels.

The ubiquitous use of caffeine in food products and medicine has required that meticulous attention be paid to the handling of samples and glassware associated with sample preparation and analysis. Caffeine junkies need to find another fix or postpone enjoyment of their favorite beverage if they plan to collect or prepare water samples for low-level caffeine analysis. Anyone interested in developing a project with this special method should contact Mike Schroeder (303-467-8200, Geomail schroede), or Ralph White (303-467-8190, wrwhite) for information regarding availability and price.

by Mike Schroeder



DIGITAL TOOLS – John Kingston, algal taxonomist in the Biological Unit, explains the digital image capture system to Dave Rickert, Chief of the Office of Water Quality. The system is used for microscopic analysis of biological specimens to keep track of taxonomy for studies by the Nation Water-Quality assessment Program.

Trace metals measured in northern Alaskan snow

Ultraclean field and laboratory procedures were used to examine trace- element concentrations in northern Alaskan snow. Sixteen soluble trace elements and total mercury were determined in snow-core samples representing the annual snowfall deposited during the 1993-94 season at two sites in the Prudhoe Bay oil field and nine sites in the Arctic National Wildlife Refuge (Arctic NWR).

Results indicate there were two distinct sources for trace elements in the Prudhoe Bay oil field-a source associated with oil and gas production and a source associated with municipal solid-waste incineration. Soluble trace- element concentrations from the Arctic NWR resembled trace-element concentrations reported elsewhere in the Arctic using clean sample-collection and processing techniques and were consistent with deposition resulting from widespread arctic atmospheric contamination.

With the exception of elements associated with sea salts, there were no orographic east-west trends observed in the Arctic NWR data nor were there any detectable influences from Prudhoe Bay oil field, probably because of the predominate easterly and northeasterly winds on the North Slope of Alaska. However, regression analysis on latitude suggested significant south-to-north increases in selected trace-element concentrations, many of which appear unrelated to the sea salt contribution. Samples were processed and analyzed at NWQL.

by John Garbarino

Bio QA Unit gets new name

From its inception in late 1993, the Biological Quality Assurance Unit was designed, funded, and partially managed by the National Water-Quality Assessment (NAWQA) Program. The intent was for the program to have strong involvement with the unit initially and then to transfer its management and operation to the NWQL. This transfer was implemented Dec. 1, 1996.

Although originally intended to be a quality assurance unit, invertebrate samples now are processed in-house, and species are identified and counted at the NWQL. Reference materials are confirmed via contracts and purchase orders with experts around the country. Plans are underway within the unit to process initial algal samples, prepare slides, and identify and count species as well. Access to algal specialists will be provided under arrangements similar to those used for invertebrate specimens.

Since the unit's mission has expanded to include data production as well as quality assurance, it will now be called the Biological Unit.

by Allison Brigham

New instrument set up for oxygen demand

New instrumentation for chemical oxygen demand (COD) was set up December 9, 1996, at the National Water Quality Laboratory. The equipment uses identical chemistry to the existing equipment* but with updated technology. The new lab code is 2144, and the WATSTORE (Water Data Storage and Retrieval System) code changes to 340C. The method reporting limit remains at 10 milligrams per liter, and the price stays at $21.22 per sample. The bottle type and preservation remain the same (125-milliliter glass bottle preserved with sulfuric acid to pH less than 2, chill sample).

The previous COD equipment used 30-year-old technology. The old equipment has been replaced with a Hach COD digester and colorimeter system. The new system is considerably safer to use and produces much less hazardous waste than the old instrument. Because of instrumentation problems, any samples received by the laboratory after October 23 were analyzed by the new system. The new instrument also will be used for reanalyses.

Data quality for the replacement instrument is equivalent to or better than the old system. More details about the new COD instrument will be released soon as a NWQL technical memorandum. For questions, contact Glenda Brown (303)467-8122 or gebrown@usgs.gov

by Glenda Brown

* Reference
Fishman, M.J., and Friedman, L.C., eds., 1989, Methods for determination of inorganic substances in water and fluvial sediments (3d ed.): U.S. Geological Survey Techniques of Water-Resources Investigations, book 5, chap. A1, p. 357.

LAB EARNS SAFETY AWARDS *– Pete Rogerson (right), Chief, Branch of Analytical Services, congratulates Carlos Arozarena, Safety Officer, after the National Water Quality Laboratory was honored with the U.S. Department of Interior's Special Achievement Award for Safety and Occupational Health Excellence. NWQL's use of a "team approach" has assured safety program employee involvement and integration at every organizational level. Robert M. Hirsch, Chief Hydrologist, said this team approach has resulted in minimizing or eliminating environmental or employee hazard exposure and substantially reduced hazardous material use. In addition, Roger Smith, Chemical Hygiene Officer at NWQL, was honored with the USGS Safety Management Award for his efforts to ensure that the Laboratory complies with numerous Federal and State regulations for managing hazardous waste. The DOI plaque was installed inside the front door of the West Lab.*

Biological Unit's Web site records increasing "hits"

The Biological Unit (BU) launched a home page on the World Wide Web (WWW) in February 1996 to provide a user-friendly method of obtaining data associated with algal and benthic invertebrate samples. These samples have been submitted for processing by biologists with the National Water-Quality Assessment (NAWQA) Program.

All transactions from the initial entry of a sample-processing request (Analytical Services Request) to the release of final data occur electronically via the BU's Web site. Information on the status of sample processing is updated daily. The site is heavily used by the BU and NAWQA as a source of relevant sample data, receiving at present about 300 "hits" or inquiries a day.

Password-protected information is entered initially by field biologists through WWW forms on the Internet; data may be updated by the biologists themselves or by BU staff. The processing data reside on an Ingres data base that is downloaded to the Web site for retrieval or manipulation. Algal, benthic invertebrate, and site data are immediately available as individual reports in several formats, the most popular being a dynamically generated FrameMaker summary as a spreadsheet. This report is useful as a shipping log or to edit or update data. Data are manipulated easily, and FrameMaker reports can be produced regardless of the platform available to the user; with a Web browser and FrameMaker software, access is available on Data General, PC, or Macintosh computers.

Biologists may download final algal and invertebrate data in relational data-base (RDB) format from the Web site by a simple point-and-click; data also load directly into a spread-sheet. Fish data are retrieved in a format created for loading into the Biological Data Analysis System (BDAS). Algal and invertebrate data soon will be available in the BDAS format as well.

The Web site will be enhanced this year. These enhancements include the following: addition of preference/priority designations by sample for processing, billing information by study unit or project, links to other biological data sites, downloading/uploading features for creating large sample requests, and availability of digital images of specimens for viewing by specialists as part of the ongoing quality-control activities of the BU.

The biologists of the NAWQA Program's fiscal year 1991 study units participated as the "test group" by providing sample information and data for nearly 6,000 samples collected from 1993 through 1995. Their efforts contributed to developing an automated system to handle all aspects of sample and data management, which is the foundation of the BU's ongoing activities and ensures user-friendly access to provisional and final sample data.

by Sandy Turner

Home Page features new pricelist with search capability

The NWQL has a new pricelist for procedures at "http://wwwnwql.cr.usgs.gov/USGS/pricelist_main.html"

This is a World Wide Web form that provides a way to search analytical categories, labcodes, schedules, or NWQL codes for an item of interest. The information provided by this page is updated daily.

The pricelist is linked to NWQL's Home page ("http://wwwnwql.cr.usgs.gov/USGS") under the heading "Pricing-Searchable Version."

by Chris Lindley

New titles in print

Hoffman, G.L., 1996, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory-Preparation procedure for aquatic biological material determined for trace metals: U.S. Geological Survey Open-File Report 96-362, 42 p.

Gates, P.M., Furlong, E.T., Dorsey, T.F., and Burkhardt, M.R., 1996, Determination of nitroaromatic explosives and their degradation products in unsaturated-zone water samples by high-performance liquid chromatography with photodiode-array, mass spectrometric, and tandem mass spectrometric detection: Trends in Analytical Chemistry, v. 15, no. 8, p. 319-325.

Copies of these publications or any other published methods and articles are available from the NWQL by contacting Korey Williams [kcwill] by Geomail, telephone 303/467-8006, or fax 303/467-8240.

Biological Unit takes part in Windy Peak open house

Some members of the Biological Unit participated in a day-long open house Oct. 19 at the Windy Peak Outdoor Education Laboratory School near Bailey, Colo. Jefferson County elementary school students and their families took part in activities to acquaint students with potential curricula such as forestry, astronomy, pioneer living, fly tying, fishing, and entomology. Steve Moulton, Joe Slusark, and Dave Stagliano provided hands-on instruction on the identification and ecology of local stream and pond macroinvertebrates (worms, snails, crustaceans, and insects). Participants observed living invertebrates and had the opportunity to preserve specimens for show-and-tell at their school.

by Steve Moulton

New unit set up for analytical contracting

The Radiochemistry and Department of Defense Environmental Conservation (DODEC) Units of the NWQL have been merged into the Analytical Contracting Unit (ACU). This new unit consolidates the analytical contracting expertise of the NWQL into a single unit to provide improved backup on contracting issues and efficient use of personnel.

The unit will focus on providing radiochemical, inorganic, organic, and stable isotope analyses through contract laboratories. However, the radiochemistry laboratory, which provides gross alpha and beta analyses, radon, and uranium by laser, will remain part of this unit. Support of the relational data base developed for use by DODEC projects in the U.S. Geological Survey also will be provided by the ACU. The goals of the unit will be to offer a high level of customer service and to provide quality data in a reasonable time to NWQL customers.
Contracts administered by the unit include the following:

- Inorganic and organic analyses-Quanterra, Denver and Sacramento
- Radiochemical analyses-Quanterra, Richland, Wash.
- Data validation-Dames and Moore, Linthicum, Md.
- Carbon 14 and carbon 13/carbon 12-University of Waterloo, Ontario
- Nitrogen 15/nitrogen 14-Global Geochemistry
- Low-level tritium-University of Miami, Coral Gables, Fla.
- Tritium and helium-Columbia University, New York

In addition, the ACU will monitor agreements with the following:

- Oxygen 18/oxygen 16, deuterium, sulfur 34/surfur 32-National Research Program (NRP), Reston
- Tritium-NRP, Menlo Park

Ann Mullin will supervise the ACU and will continue to report to Tom Maloney, Quality Management Program Chief. Bob Brock will serve as Mullin's primary backup. The other members in the ACU include Jim Lewis, Bruce Darnel, Jeanne Hatcher, and Mark Cree.

by Tom Maloney

SUPERIOR SERVICE AWARD *– Robert S. (Bob) Williams, Jr. was presented with the U.S. Department of the Interior Superior Service Award by Gordon P. Eaton, Director, U.S. Geological Survey, in August. Pete Rogerson, NWQL Chief, is shown congratulating Williams in recognition of "outstanding contributions to management" of the Laboratory. Eaton cited Williams for bringing "a district perspective for resolving issues unique to the NWQL environment" and for establishing guidelines for clear communications that "increased trust between district and Laboratory personnel." Williams recently accepted a new assignment for the Office of Water Quality (OWQ) at NWQL. The joint position with OWQ, NWQL, and the National Water-Quality Assessment Program will focus on liaison and interface among these organizations.*

Short course slated on environmental chemistry

The training course "Environmental Chemistry of Organic Pollutants I-Controlling Processes" (G0482) is scheduled to be held March 10-14 at the U.S. Geological Survey's National Training Center in Denver. Registration deadline has been extended to February 21. Bill Foreman, NWQL research chemist, is coordinating the 1-week course. Topics include environmental processing of organic chemicals, primarily semivolatile organic pollutants (for example, pesticides, PCBs, and polycyclic aromatic hydrocarbons). Students will be introduced to the major chemical classes of pollutants.

THE LAB AS A BENCHMARK *– A delegation from Cairo toured the NWQL last month to glean ideas for a new laboratory under construction for the National Water Research Center of Egypt. The chemists and managers represented the Environment and Climate Research Institute and the Drainage Research Institute of Cairo. Gary Cottrell (left), NWQL chemist, gave the visitors a tour emphasizing good laboratory practice at the U.S. Geological Survey.*

DDT analysis is not always straightforward

For three decades, the insecticide *p,p'*-DDT (DDT) was heavily used in agriculture and to control the spread of vector-borne human diseases, especially malaria. Recognition of DDT's detrimental impact on animals led to use bans in many countries in the 1970s, including a U.S. ban in 1972, although heavy DDT use continues even today in some countries. DDT and its two primary environmental degradation products, *p,p'*-DDD and *p,p'*-DDE, are persistent compounds which, through long-range atmospheric transport, have become global pollutants that are bioaccumulating in flora and fauna in even the most remote regions of Earth.

The ability to measure DDT and its primary degradation products accurately in various matrices is critical to understanding their transport, fate, and ecological impact. For example, ratios of DDT to

DDE or DDD concentrations have been used to infer inputs of old versus new DDT residues or to attempt to characterize the importance of various environmental degradation pathways. The DDT family also has been implicated in various aspects of endocrine system disruption, with the degradation products (for example, *p,p'*-DDE) more effective than the parent DDT in disrupting some hormonal pathways.

Nearly all laboratories worldwide (including the NWQL) rely on conventional analysis of DDT in environmental samples using gas chromatography coupled with electron capture (GC/ECD) or mass spectrometric (GC/MS) detection. Standard methods (including USGS and most U.S. Environmental Protection Agency methods) require monitoring for degradation of this insecticide by the hot GC injection system. GC degradation is indicated by formation of the breakdown products *p,p'*-DDD and *p,p'*-DDE, both also primary environmental degradation products.

Research chemist Bill Foreman and chemist Paul Gates of the NWQL's Methods Research and Development Program have shown that traditional techniques used to monitor GC-derived degradation of DDT may be inadequate. They observed sample-specific matrix-enhanced degradation of DDT by the GC system and devised a new technique that provides for sample-specific monitoring of this breakdown problem when conducting DDT analysis by GC/MS. Their findings are detailed in a paper titled "Matrix-Enhanced Degradation of *p,p'*-DDT During Gas Chromatographic Analysis-A Consideration" which is in press in *Environmental Science and Technology*. For more information, contact Bill Foreman (wforeman@usgs.gov).

by Bill Foreman

Mailbag

I thought the October issue [v. 4, no. 4, 1996] of the NWQL Newsletter was particularly informative. I was especially interested in articles by Pirkey, Glodt, Maloney, Sandstrom, and the notice of availability of new publications. Nice job.

Jeffrey Martin, Hydrologist, Indianapolis

New capabilities developed for users of LADS data base

The LADS (Laboratory Analytical Data System) went on-line in October 1995. All completed analytical data from 1992 to the present are stored in the LADS data base.

This data base was originally designed to be the laboratory component of the data base for the National Information System (NWIS-II); however, numerous delays in the NWIS-II project caused the NWQL to proceed with implementing the LADS data base. The Laboratory has been in need of improved data integrity and data-base capabilities that were only available using a relational structure. The LADS was successfully implemented in the past year, as noted in the following examples:

Storage of nonpreferred measurements. This capability was not possible with the old PRIME computer. The NWQL can now store all measurements produced by an analysis, not simply what is reported to the customer.

Data integrity. The relational design of the LADS data base allows NWQL to identify and correct mistakes in the data base. The Computer Services Unit (CSU) spent considerable time correcting data that were moved from the PRIME. The NWQL will no longer produce "inconsistent" data.

Release of data. The CSU can now verify that it is not releasing incorrect data. Measurements are being checked for correct format, and mail is sent to section chiefs when incorrect measurements are detected.

Release of individual concentrations. Until recently, CSU could only release data in a group (by subsample), even though only one analytical result was needed by a user. CSU can now release single measurements when necessary.
Data accessibility. NWQL computer users can now pull data reports based on a set of variable criteria using a special application. Special data retrievals can be handled by request through CSU personnel.

On-going projects. CSU is developing new applications for the LADS data base, including graphical interfaces. By implementing a relational data base, the NWQL seeks to improve its ability to store, retrieve, and analyze data.

by Richard Husband

Changing and overwriting data

The NWQL routinely uses multiple techniques or methods to determine constituents in a given sample matrix. Internally, each method has a unique lab code and method character matched to an appropriate parameter code.

For example, fluoride, in filtered water, has a parameter code of 00950. In the fluoride example, the laboratory has an ion-chromatography method and an ion-specific electrode method, each with a different reporting level. The ion-chromatography method would be sent to the customer as 00950D, whereas the ion-specific electrode method would be sent as 00950B.

The NWIS-I (National Water Information System) can only store a single result for each parameter code, regardless of the technique used. The last result sent to the customer will overwrite the previous one if the customer requests multiple schedules with overlapping constituents that are analyzed using multiple methods. In some cases in organic analyses, especially with the volatile organic compounds (VOCs) and semivolatiles, there may be 1 or 2 orders of magnitude difference between the reporting levels (for example, dichlorobenzene).

When requesting multiple schedules, the customer should look to see if there are any overlapping compounds or constituents, and do one of the following:

- If inorganic constituents, select the labcode of the method that best matches the requirements of the project and delete the labcode that is not needed on the Analytical Services Request (ASR) form;
- If organic schedules are used, submit separate ASRs for each schedule with the time offset by 1 minute. This procedure will allow storage of both results in the customer's data base.

by Steve Glodt

Lab Chief home from hospital, recovering from brain surgery

Pete Rogerson, NWQL Chief, is resting comfortably at home following brain surgery to remove a benign tumor December 28 at a Denver hospital. After bandages were removed January 6, Pete said he was "feeling good and recovering from his adventure. I don't think they took out anything I can't live without," he added.

Some of us were wondering if surgery resulted in a complete personality change, but Pete said not to count on it. He was quite happy to receive all the cards and letters. Recovery is expected to take 4 to 6 weeks. Send Geomail to <BonnyRoger@aol.com>. Address cards to Pete Rogerson, 1895 Foothills Drive South, Golden, CO 80401.

Acting NWQL Chief in Pete's absence is Merle Shockey, head of the Production Program.

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.