

### **Lab offers training to CSM students**

The Colorado School of Mines (CSM) requires its students to enroll in an Engineering Practice Introductory Course Sequence (EPICS) Program. Students are asked to work on a "real world" project after they have completed their basic studies in mathematics, chemistry, physics, geology, and economics.

The National Water Quality Laboratory (NWQL) has participated in the EPICS program for the last two years. NWQL supplies a project and guidance to a group of students during the semester. In return, the students research the project, present an oral progress report, and produce written progress reports. At the end of the semester, a seminar is held at the School to present solutions and recommendations to the sponsors, giving students an opportunity to experience a professional work environment.

In the past two years CSM students have (1) produced specifications and requirements for the Reference Sample Library to be implemented in the Biological Assurance/Quality Control Unit; (2) produced recommendations for tracking, storage, and retrieval of Standard Operating Procedures used at the NWQL; (3) studied the use of bar codes and scanners on Reference Samples for the Biological Quality Assurance/Quality Control Unit; and (4) studied the quality-control program used to process inorganic sample analyses. Each project was worked on by a team of five to six students devoting about 100 hours to the study.

*by Sandy Turner*



**Ted Struzeski**, chemist in the Plasma Unit, experiments with high-concentration samples to determine the effects of interfering elements. (Photo by Tami Heilemann, USDOI)

### **Charity softball hits home run**

Two teams from the NWQL recently participated in a charity softball tournament that benefited Shawna and Nicole Charles, the daughters of the late Veterans Administration employee Kathy Charles.

Twelve other teams from local government agencies also participated in this tournament, which raised over \$2,100 for the two girls.

The coed teams included players from the Quality Management Program, Organic Chemistry and Inorganic Chemistry Programs, Methods Research and Development Program, Administrative Services Unit, and the Computer Services Unit.

*by Bob Brock*

## Development of HPLC/MS techniques

The Office of Water Quality is supporting new methods development at the NWQL by funding a major upgrade of mass spectrometry (MS) equipment that will be used for the analysis of polar organic compounds in aqueous samples.

The purpose of this methods development is to provide U.S. Geological Survey projects with a state-of-the-art analytical method for the identification and quantitation of polar pesticides, pesticide metabolites, surfactants, and other polar organic compounds. This new method will complement the existing NWQL Schedules 2050 and 2051, pesticides in filtered water extracted on Caropak-B solid-phase extraction cartridge and analyzed by high-performance liquid chromatography (HPLC) with ultraviolet detection.

The new method will use atmospheric pressure ionization MS to provide improved identification and sensitivity. Contact Ed Furlong (303/467-8080; [efurlong@usgs.gov](mailto:efurlong@usgs.gov)) or Mark Burkhardt (303/467-8093; [mrburk@usgs.gov](mailto:mrburk@usgs.gov)) to find out more about this project.

*by Ed Furlong*

## New on the Net

Selected field instructions, new technical memoranda, and automatic e-mail forms have been added to the NWQL Home Page this quarter. The NWQL is conducting a survey to determine what you, our customers, would like to see on our web page. Check out the suggestion form on our survey and comment page and cast your vote! Our address is

<http://www.nwql.cr.usgs.gov/>

We welcome your suggestions.

*by Sandy Turner*

## Central and Western Region District water-quality specialists meet at NWQL

The Central and Western Region District water-quality specialists held a combined meeting during the week of August 14-18, 1995, at the National Water Quality laboratory (NWQL). Dave Rickert, Chief of the Office of Water Quality, gave a welcome and provided a briefing on the current activities of his office. He talked about the technical strength of the Water Resources Division (WRD) and the support provided by U.S. Geological Survey cooperators during this difficult time of budget cuts. He also said the cooperators believe that USGS results, although excellent, are frequently slow and late. Pete Rogerson, Chief of the NWQL, provided an overview of NWQL operations, including plans for a new laboratory that are subject to congressional approval.

Following these introductory remarks, Tom Maloney and his Quality Management Program staff summarized quality at the NWQL from sample log-in to final quality control with help from Linda Pratt on log-in, Mike Schroeder on Organic Chemistry Program quality assurance/quality control (QA/QC), and Harold Ardourel on Inorganic Chemistry Program QC. Amy Ludtke presented the Blind Sample Program from the Branch of Technical Development and Quality Systems (BTDQS). Daily meetings ended with attempts to answer questions from these knowledgeable customers.

A tour of the NWQL followed on Tuesday. The afternoon focused on the Methods Research and Development Program. Mark Sandstrom, Program Chief, led presentations from each project to alert the specialists about advances in chemical-measurement technology that are being developed by the Program.

Meetings on Wednesday and Thursday continued to focus on QC. Quality Management Program personnel presented talks on how to use organic QA/QC data, and discussed method-detection limits and chain-of-custody issues. Field QA, radiochemical issues, a briefing on the Midwest Herbicide Program by Don Goolsby, and a description of the Biological QA/QC Unit by Stephen Porter completed the day. Thursday started with presentations from the BTDQS personnel on the Standard Reference Water Program and the external lab review process before moving into a detailed description of the Inorganic Protocol by Art Horowitz and Kathy Fitzgerald. Art, as usual, was technically correct and irreverent, and managed to keep us all entertained and informed at the same time.

Friday concluded with a presentation by Jim Blakey, Regional Hydrologist, who discussed future directions for the Central Region. We will all miss Jim's candor and insights when he retires in January.

We hope that the water-quality specialists benefited from this week as much as NWQL personnel benefited. We appreciate the opportunity to share some of our work and thoughts with you. Thank you for discussing issues with our customers. To the Northeast and Southeast water-quality specialists (and managers): Please consider having your next meeting at the NWQL.

*by Pete Rogerson*

## **New methods being developed for pesticide sample analysis at anaogram-per-liter-level**

The Methods Research and Development Program at the National Water Quality Laboratory (NWQL) is continuing to investigate methods to determine pesticides at the nanogram-per-liter (ng/L) level. Two investigations that are in the early stages are the determination of glyphosate and selected *n*-methyl-carbamates in filtered water samples.

Glyphosate is a widely used non-specific postemergent herbicide. It is the active ingredient in the commercially available herbicide Roundup. The current method (Lab Code 1834, Parameter Code 49221 A) for monitoring glyphosate in filtered water samples at the NWQL is based on U.S. Environmental Protection Agency (USEPA) Method 547, which involves the direct injection of the sample into a high-performance liquid chromatograph (HPLC).

In the new method, glyphosate is separated from environmental interferences on a Caropak-B based HPLC column with detection and quantification made by post-column reaction using a fluorescence detector. The typical sample injection volume is 250 microliters ( $\mu$ L).

The method detection limit (MDL) is 1 microgram per liter ( $\mu$ g/L). This MDL, although five times lower than the current USEPA MDL, does not meet the stated needs of the U.S. Geological Survey.

The current method (Schedule 1359) for monitoring methomyl, aldicarb, propoxur, carbofuran, carbaryl, 1-naphthol, propham, and methiocarb in water samples at the NWQL is based on the liquid-liquid extraction of the sample with methylene chloride and injection of the extract onto a HPLC.

Carbamates are separated from environmental interferences using a reverse-phase HPLC column with detection and quantification made by postcolumn reaction with ultraviolet and fluorescence detectors. The typical sample injection volume is 250  $\mu$ L. The MDLs for the selected pesticides are 500 ng/L. Aldicarb sulfoxide, aldicarb sulfone, oxamyl, and 3-OH-carbofuran were recently dropped from this schedule because of chromatographic difficulties and poor recoveries (less than 20 percent).

An internal NWQL review team decided to replace Schedule 1359 with a SPE-based method to improve recovery of the procedure, to add selected pesticides, and to complement Schedule 2050 with a low-level carbamate method.

The proposed methods currently under investigation would use SPE cartridges to concentrate a 1-L sample (or more?) for injection onto the HPLC system. Glyphosate and carbamates would be detected and quantified by using a postcolumn reaction and a fluorescence detector. Since detection by fluorescence is sensitive, expected MDLs at 1 ng/L or less are anticipated for glyphosate and carbamates. USEPA methods, along with vendor and literature research, are being investigated to optimize the separation, reaction, and quantification of the selected pesticides.

*by Mark Burkhardt*

## **Chlorophyll analysis methods compared**

In cooperation with the National Water-Quality Assessment (NAWQA) Program, the NWQL is comparing chlorophyll analysis methods. This project will provide a quantitative comparison between USGS-approved methods and other methods used by academic researchers and government agencies.

NAWQA and NWQL staff will jointly collect samples of periphyton from at least two NAWQA study units.

Personnel of the Methods Research and Development, Inorganic Chemistry, and Organic Chemistry Programs from the NWQL will then conduct experiments to determine differences between several extraction and analysis treatments. Contact Ed Furlong (303/467-8080; [efurlong@usgs.gov](mailto:efurlong@usgs.gov)) to find out more about this project.

*by Ed Furlong*



**Donna Rose**, chemist in the Volatile Organic Carbon Unit, prepares standard solutions for a sample analysis. (Photo by Tami Heilemann, USDO)

## **New data-handling system focuses on customer involvement, accessibility**

The National Water Quality Laboratory (NWQL) is moving all released data from the Laboratory Information Management System (LIMS) and PRIME computer to the more versatile and powerful UNIX-based Data General and Hewlett-Packard systems. The NWQL Computer Services Unit (CSU) has designed and developed the new system for compatibility with the National Water Information System II (NWIS-II).

Historically, U.S. Geological Survey (USGS) staffers have developed and maintained isolated pockets of data using a variety of software (Lotus 1-2-3, ENABLE, Word Perfect, and INFO). These data often were inaccessible or unknown, or both, to other USGS personnel. Even when others discovered the data, it was difficult to retrieve the information. The development of NWIS-II should provide easy access to data throughout USGS and for its cooperators.

The new system is modeled on a relational data base. The data are stored in simple tables with basic relations among data expressed as references to values in other tables. By using a relational model, large amounts of data can be manipulated easily and quickly. The concept provides flexibility but not without a penalty—the data base is complex to design and implement.

In addition to on-going development of NWIS-II, efforts are underway in the CSU to produce computer programs for operation with NWIS-II and data transmission from the PRIME and LIMS to the Data General and Hewlett-Packard systems. The NWQL approach involves small steps at first while analytical results are integrated into the data base.

Under the present system, analytical request data is manually entered into the computer system. The new NWIS-II design focuses on using Electronic Analytical Services Requests (EASR). Districts will be responsible for entering sample information into their computers and electronically transmitting that information to the NWQL's computers via the EASR system. As samples arrive at the NWQL, log-in staff will scan the samples into the computer; theoretically, the sample data will automatically link with the existing EASR information, thus eliminating several potential data-entry errors.

In addition, new software has been installed that will give NWQL personnel easy access to archived data. As a result, the NWQL Administrative Services Group will be able to track custom samples and cost-production data more efficiently than with the old system. There are also plans to provide greater access to quality-control data and charts by developing new software. Moreover, a World Wide Web page has been developed by CSU that includes information such as NWQL contact list and technical memoranda. The Universal Resource Locator is <http://www.nwql.cr.usgs.gov/>.

What does all this change mean for our customers, users, and system developers? It means changes in attitudes, development cycles, and increasing and varied amounts of accessible information. It represents change in how we develop the software that presents the data produced by NWQL, resulting in closer cooperation and more customer involvement than in the past.

*by John Crisci and Robert Mayer*

## **New titles in print**

Sandstrom, M.W., 1995, Filtration of water-sediment samples for the determination of organic compounds: U.S. Geological Survey Water-Resources Investigations Report 95-4105, 13 p.

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

Zaugg, S.D., Sandstrom, M.W., Smith, S.G., and Fehlberg, K.M., 1995, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory-Determination of pesticides in water by C-18 solid-phase extraction and capillary-column gas chromatography/mass spectrometry with selected-ion monitoring: U.S. Geological Survey Open-File Report 95-181, 49 p.

Please contact the NWQL by telephone or Geomail (jwraese) if you need copies of these titles or any other reports prepared by Laboratory authors.

## **Newsletter Staff**

Jon Raese, Editor

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