

Relocation planning intensifies as new Laboratory moves forward

As the occupancy date for the new Laboratory draws closer, planning for the move of the NWQL has intensified. The NWQL has been meeting with the General Services Administration, USGS Operations staff, the U.S. Environmental Protection Agency, and the State of Colorado to work out the details for occupying the new building. The proposed occupancy date is March 1999.

A relocation of this scope and complexity requires that the NWQL execute a well-planned task schedule and timeline of moving events as far in advance as is feasible.

The first step in the planning process is to get a comprehensive inventory of equipment and property at the NWQL, which must be completed by mid- to late summer.

To help with the planning and logistics, the NWQL has requested and received a proposal from a move-management company. The company will create a bar-coded inventory of the entire Laboratory and will also plan and schedule moving all equipment and furniture.



NEW LAB TAKING SHAPE – This view of the construction site shows progress on the south wing of the National Water Quality Laboratory as of May 9 on the Denver Federal Center campus.

The Laboratory Operations Program will be working on a Web page to keep NWQL personnel updated on timelines for move-related tasks.



by Pat Timme

Taxonomic initiative receives Hammer Award

Ten USGS scientists have received Vice President Al Gore's Hammer Award for governmental reinvention for their contributions to the Integrated Taxonomic Information System (ITIS) <http://itis.usda.gov/itis/> at a ceremony in Washington, D.C. The award consists of certificates for each recipient signed by Vice President Gore and a \$6 hammer tied with a red ribbon, which is the Vice President's answer to the \$600 hammer of yesterday's government.

Water Resources Division employees Allison Brigham (NWQL), Nelson Williams (North Carolina District), and Tom Yorke (Office of Surface Water, Reston) were recognized with about 40 other Federal employees, who have participated in the development and support of the interagency partnership over the past 6 years.

The ITIS is a comprehensive, standardized reference for scientific names, including synonyms and common names, of the flora and fauna of North America and surrounding oceans; it is a fundamental component of the National Biological Information Infrastructure (NBII) <http://www.nbii.gov/>.



by Allison Brigham and Gary Waggoner

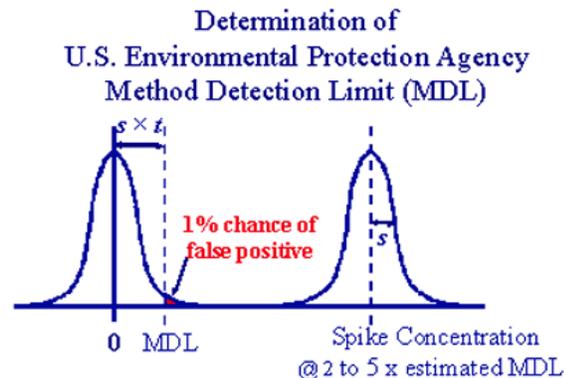
Method detection level (MDL) and reporting conventions change

The focus of much of the analytical work at the National Water Quality Laboratory is to address requirements of U.S. Geological Survey investigations that assess ambient levels of chemical constituents in water. The NWQL uses analytical methodology specifically tuned to assess trace concentrations of these constituents. Since the focus of NWQL methods is on the low-concentration range of the methods, we must document the quality of analytical work in this range.

Typically, laboratories use assessments of reference standards or other quality-control data at the low end of the analytical range to establish detection and reporting levels. Numerous articles have been written on procedures to establish criteria, which have led to a proliferation of terms and a general state of confusion in the analytical industry on how to address these issues.

Several years ago, the NWQL adopted the U.S. Environmental Protection Agency (1992) (USEPA) procedure as the way to establish criteria for new analytical methods. The statistical logic behind determination of a method detection limit using the USEPA procedure is shown in the accompanying illustration.

The USEPA requires that this procedure be used for all regulatory methods for compliance monitoring. However, analysts raised concerns regarding limitations of the procedure, including dubious method detection limit assessments when additional variables need to be accounted for. Examples of additional variables are multiple instruments, multiple analysts, and seasonal variability--common issues for analytical methods offered by the NWQL. Another concern is that the USEPA procedure provides guidance for establishing method detection limits but offers no guidance on setting reporting levels.



- Determined from analysis of n (≥ 7) replicate samples in a given matrix containing the compounds and constituents of interest
 - Matrix: reagent water, clean sand, sodium sulfate,
 - Iterative process: typically performed once or twice over a short time.
- $MDL = s \times t$
 - s = standard deviation of n replicate, and
 - t = Student's t -value at 99% confidence level and $n-1$ degrees of freedom.

An NWQL team evaluated the issues involved with assessing detection levels and reporting procedures that would address the needs of the USGS with specific attention to analytical concerns. The team recommended standardized procedures in the laboratory. The NWQL recognized that the recommendations would involve changes in the way data are reported and evaluated. The Laboratory has consulted with the Office of Water Quality, District, and National Water-Quality Assessment (NAWQA) Program personnel to address the impact these changes will have on data users.

At the Water-Quality Specialists Meeting in April, several presenters discussed method detection level (MDL) development to date and answered questions on the topic. Bill Foreman and Tom Maloney, from the NWQL,

- Explained the confusion that the analytical industry is facing in setting standards for establishing detection and reporting levels;
- Described the benefits of the new Long-Term MDL procedures being implemented at the NWQL; and
- Discussed the logic behind proposed new reporting procedures.

In addition, Callie Childress, North Carolina District, made a presentation on impacts and benefits that could be expected in reporting and assessing analytical data. And, Dennis Helsel, NAWQA National Synthesis Project, Colorado, discussed issues relating to impacts on statistical assessments. Viewgraphs for the presentations by Foreman, Maloney, and Childress are posted on the NWQL Home Page at the following URL: <http://www.nwql.cr.usgs.gov/USGS/lvegas.html>. The NWQL will provide additional information on this topic in future issues of the Newsletter, through technical memoranda, electronic mail, and other venues. For questions or comments, contact Tom Maloney (tmaloney / 303-467-8041) or Bill Foreman (wforeman / 303-467-8079).



by Tom Maloney and Brooke Connor

Reference:

U.S. Environmental Protection Agency, 1992, Guidelines establishing test procedures for the analysis of pollutants (Part 136, Appendix B. Definition and Procedure for the Determination of the Method Detection Limit--Revision 1.11): U.S. Code of Federal Regulations, Title 40, revised as of July 1, 1992, p. 565-567.

Customers access reanalysis data via Home Page Web site

The National Water Quality Laboratory (NWQL) has upgraded the District reanalysis data base to an Ingres-based application with a Web interface. This upgrade was necessary because the software used to maintain the former system was no longer supported by the manufacturer.

The Web interface is accessed through the NWQL Home Page and will eventually replace the electronic mail that is currently used to request reanalysis of samples. District customers are encouraged to use the Web version; electronic mail requests for reanalysis will be accepted through the end of the 1998 water year (Sept. 30). If the Web version is not an option for your District, contact Kathy Bryant (kmbryant) to make other arrangements.

District personnel can access the NWQL home page at <http://www.nwql.cr.usgs.gov/USGS> and select the District rerun (reanalysis) request form listed under Services.

The application uses station, date, time (include seconds with time--default is 00), WATSTORE (Water Data Storage and Retrieval System) code, and method code to process the reanalysis request. Entry in these fields is mandatory and the required information can be obtained from the Watlist reports that are prepared by NWIS when chemical analyses are entered. If mandatory fields do not have entries, users will receive an error message on the screen. In the "Type of Request" field, please specify either a District rerun or District verification.

The NWQL retains the major ion and trace metal sample bottles for 6 months and the nutrient sample bottles for 30 days. Samples still in storage at the NWQL can be reanalyzed; after samples are discarded, the results will be verified. Tab through the fields after each entry, and then submit the request by selecting the "Submit Request" key in the lower left of the form. To access the form again for subsequent entries, select the "back" option in the menu bar. Any discrepancies between the information supplied by the customer and data in the NWQL data base will be communicated to the District requester via electronic mail. Completed results will also be communicated via electronic mail.

Under "Optional Information" on the form, the comment field can be used by the requester to specify a high or low value when the value differs from the site history, to provide additional information to the analyst, and to expand the NWQL "Reason for Request" list. The "Other Email Notification Recipients" section of the form allows three copies to be entered with the request, and completed reanalyses will be sent to these individuals.

Future plans for the application include providing the status of reanalysis requests and completed results on the NWQL Web page. Contact Bryant (kmbryant) or (denqc) at 303-467-8043 or Tom Bushly (tjbushly) at 303-467-8103 for assistance or questions.



by Kathy Bryant

Personnel briefs

James Montoya has been selected as the system administrator in the Computer Services Unit of the Laboratory Operations Program. He started work May 26. Montoya recently worked as a computer specialist in the National Mapping Division. He replaces Eric Mahlowiz, who went to work in the private sector.

Sherry Oman, secretary in the Quality Management Program, is training half-time as a computer specialist for the Laboratory Operations Program.

Bob Brock, former chemist in the Analytical Contracting Unit, is now working for the U.S. Environmental Protection Agency, Region 8, in Denver.

New titles in print

Andrle, V.A.S., and Kingston, J.C., 1998, Planothidium species in rivers and lakes of the United States [abs.], *in* Modreski, P.J., compiler, Collected abstracts of selected poster papers presented at scientific meetings: U.S. Geological Survey Open-File Report 98-209, p.1.

Glodt, S.R., and Pirkey, K.D., 1998, Participation in performance-evaluation studies by the U.S. Geological Survey National Water Quality Laboratory: U.S. Geological Survey Fact Sheet FS-023-98, 6 p.

Lusby, J.P., 1998, Qualitative techniques for the processing of periphyton samples containing sediments [abs.], *in* Modreski, P.J., compiler, Collected abstracts of selected poster papers presented at scientific meetings: U.S. Geological Survey Open-File Report 98-209, p. 2.

Marr, L.J., 1998, Notes on some commonly misidentified and problematic diatom taxa of the genus *Cymbella* in U.S. rivers [abs.], *in* Modreski, P.J., compiler, Collected abstracts of selected poster papers presented at scientific meetings: U.S. Geological Survey Open-File Report 98-209, p. 3.

Pirkey, K.D., and Glodt, S.R., 1998, Quality control at the U.S. Geological Survey National Water Quality Laboratory: U.S. Geological Survey Fact Sheet FS-026-98, 4 p.

Copies of these publications are available from NWQL by contacting Korey Williams (kcwill) by electronic mail, telephone 303/467-8006, or fax 303/467-8240.

Seminar scheduled

"Pesticide Enantiomers as Environmental Tracers," Dr. Renee L. Falconer, Chemistry Department, Youngstown State University, Youngstown, Ohio
10:30 a.m. Thursday, July 2

Denver Metro area visitors from USGS branches and District offices are invited to stop by the Laboratory conference room and join the staff for the NWQL Water-Quality Colloquia. If interested in delivering an address, contact Jon Raese (jwraese) for arrangements.

Laboratory Information Management System (LIMS) placed on-line in May

The NWQL began logging samples into the "new" Laboratory Information Management System (LIMS) on May 11. It has been nearly 10 years since the NWQL installed the "old" Perkin-Elmer LIMS--a long time for any computer system to be in place. The new Laboratory will only be equipped for office- environment computers. Hence the need to decommission the Perkin-Elmer, which requires a specialized type of air-conditioned room.

Parts of the "new" LIMS are not really new. The data-base structure was derived from the National Water Information System (NWIS-II) design. All sample archive data have been stored in the data base for 3 years. The Sample Update, Data Release, and all reference list maintenance applications have been active for almost as long. The operational part of the LIMS came on-line in May. This operation includes bottle login, Analytical Services Request (ASR) login, worksheet production, batch and on-line quality control, various query and update applications, as well as automatic and user-selected reports.

Some immediate advantages have been gained by implementing LIMS in place of the Perkin-Elmer system. For example, the relational design allows for more flexibility to accommodate changing requirements.

All applications use a Graphical User Interface to interact with the data base. In addition, the data base supports a robust system of backup (checkpointing) that allows an up-to-the-second restoration in case of a hardware failure. Moreover, there are no limits

to how many users can be on the system at any time. And samples can be accessed in the system within 15 minutes after data entry.

Physical samples are now entered and compared with the schedule and lab code requests, and exception reports are mailed to lab personnel. For the first time, all sample data are in one data base. The new LIMS will help NWQL to evaluate the benefits of a commercial system.

There are many unknowns in acquiring a commercial LIMS product. For example, it took 3 years to implement the Perkin-Elmer system after it was purchased. For these reasons, it was not feasible to rely on a commercial LIMS to be in operation before we move to the new lab.

Currently, the Computer Services Unit staff is focused on maintaining sample flow and providing a solid operational base for the new LIMS. Applications will be developed to take advantage of the new system.



by Richard Husband



AVALANCHE SAFETY TRAINING – Armin Burdick, physical science aide in the Organic Chemistry Program, peers through a hand lens to examine snowflakes March 29 on a spring field trip to Berthoud Pass, west of Denver. About 30 participants dug snow pits, practiced searching for buried victims with transceivers (beepers), and learned the basics of identifying avalanche danger signals. Other participants from the National Water Quality Laboratory were Jeff Cahill and Jon Raese.



SNOW JOB – Dr. Bill Hotchkiss, Scientist Emeritus with the USGS Water Resources Division, lectures students from a snow pit during a Snow Avalanche Safety Training Seminar, March 29, on Berthoud Pass. Hotchkiss, a member of the National Ski Patrol and co-founder of the National Avalanche School, discussed snow metamorphism and stratigraphy, avalanche dynamics, and safe winter travel in the backcountry. He has given avalanche seminars throughout the Rocky Mountains and is regarded as an expert on the subject.

Analytical services priced for FY1999

The Office of Water Quality has approved a 2-percent price increase for chemical-analytical services offered by the National Water Quality Laboratory (NWQL) for fiscal year 1999. These prices will be effective October 1. The new prices--along with old prices--are now listed on the NWQL Home Page. See masthead for Website address and click on "Price List."

Only in-house analytical-chemistry prices are being raised by 2 percent from 1998 prices. Prices for all other analytical work--such as analyses offered by the Geologic Division, contract analyses for radiochemical parameters and the Department of Defense Environmental Conservation Program, and Biological Unit analyses--will be announced as soon as they are available.

Tech memos in production

The following unapproved technical memos are in various stages of preparation. They are listed here for information purposes only. After approval, NWQL technical memos can be retrieved at the Laboratory's Home Page Web site which is listed in the Newsletter's masthead.

"Method change for the analysis of phytoplankton biomass," by Ron Brenton.

"Change in reporting unit for relative abundances of helium isotopes," by Leonard Plummer and Ann Mullin.

"Polar pesticide analysis by liquid chromatography/mass spectrometry," by Mark Burkhardt.

Supersensitive ICP-MS instruments to be placed online

The NWQL is replacing 10-year-old inductively coupled plasma-mass spectrometers (ICP-MS) with state-of-the-art instrumentation. The new instruments are tentatively scheduled to be placed online in the first quarter of the new fiscal year.

The instruments will provide method detection limits that are 4 to 50 times lower than the instruments they replace (less than 100 nanograms per liter for most elements). This increase in sensitivity provides significantly lower variability at ambient trace-metal concentrations. Schedules that currently use ICP-MS methods are 1050, 2200, and 2703. In addition, the recently approved method by Garbarino and Struzeski (in press) for the determination of trace elements in whole-water digests will use the new ICP-MS.

We are also planning to add elements to the ICP-MS method--namely, arsenic, boron, lithium, selenium, strontium, thallium, and vanadium. Some of these elements are already being determined in certain sample types, however, selected elements will be added to other sample types for consistency.

NWQL will issue a technical memorandum before the new instrument is added to the production line. At that time, information will be provided regarding instrument performance and possible impacts on water-quality data bases.



by John Garbarino

Reference: Garbarino, J.R., and Struzeski, T.M., in press, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory--Determination of elements in whole-water digests using inductively coupled plasma-optical emission spectrometry and inductively coupled plasma-mass spectrometry: U.S. Geological Survey Open-File Report 98-165.

New method being developed for determining polar organic pesticides

A new liquid chromatography/mass spectrometry (LC/MS) method for determining polar organic pesticides and pesticide metabolites in water samples is expected to be available by spring 1999 from the National Water Quality Laboratory (NWQL). This new method is designed to replace the existing Schedule 2050, but to ensure consistent data quality, Schedule 2050 will remain available until the new method is fully developed and successfully implemented.

The NWQL Methods Research and Development Program is designing this new method with knowledge gained from efforts to incrementally improve Schedule 2050. Mark Burkhardt, co-lead chemist on the project, said that the efforts in developing an interim method over the past 18 months have resulted in the following improvements:

- A polymer-based solid-phase extraction cartridge demonstrates higher extraction efficiencies of selected compounds than the graphitized carbon cartridges used in Schedule 2050.
- The use of acidified acetone instead of acidified methylene chloride/methanol as the elution solvent improves the recovery of selected compounds. It also reduces concentration-step time by about 25 minutes.
- A mass spectrometer successfully resolves small concentrations of selected compounds from background interference caused by dissolved organic compounds (DOCs), which was not possible with the photodiode-array detection system used in the present method.

Burkhardt said that in addition to these technical improvements, the extraction process will be automated and several new compounds would be added to the schedule. The new method is expected to be more rugged, accurate, and precise than the method it will replace. The new design is being drafted and will be reviewed by a joint team from NWQL and the National Water-Quality Assessment Program (NAWQA) prior to final approval.

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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.

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