

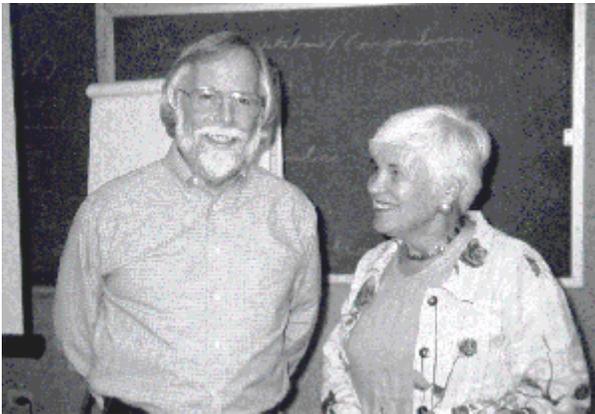
The Methods Board is made up of water-quality experts from Federal, State, and municipal agencies, Native American groups, and private-sector organizations. Specific objectives include the following:

- Set priorities for methods development,
- Produce technical guidelines for ensuring data comparability,
- Promote a performance-based methods system,
- Assist in establishing reference methods,
- Develop guidelines for methods validation, and
- Promote national laboratory accreditation and prelaboratory (field) certification programs.

An example of Methods Board collaboration involves the USGS and USEPA Drinking-Water Initiative. The drinking-water programs of both agencies have identified opportunities to collaborate on analytical methods. The Laboratory Methods Initiative includes the following:

The USGS National Water Quality Laboratory serves as a backup and reference laboratory for two analyses performed under USEPA's Information Collection Rule.

- The USGS is helping to develop a more convenient analytical method to measure trihalomethane formation potential.
- USGS and USEPA have agreed to work together on streamlining future methods development by sharing the development process and by developing methods that are acceptable to both agencies.
- Finally, USGS is field-testing several microbial methods that were recently developed by USEPA.



USGS DELEGATES – John Klein (left), assistant regional hydrologist, Sacramento, and Michalann Harthill, biologist, Reston, participated in the workgroup discussions of the Methods and Data Comparability Board, Aug. 12-14, at the Laboratory.



COLLABORATING – Charlie Peters (left), USGS hydrologist, Middleton, Wis., and Clifford G. Annis, Jr. senior quality-assurance specialist, Merck and Co., Inc., Whitehouse Station, N.J., were among some 28 delegates representing Federal, State/Tribal, and private sectors at a meeting of the Methods Board at NWQL.

Further information on the Methods and Data Comparability Board may be found at the Web site for the Intergovernmental Task Force on Monitoring Water Quality <http://water.usgs.gov/public/wicp/itfm.html>.



Long-term method detection level data collection enters new phase for high-demand water methods

The National Water Quality Laboratory (NWQL) started long-term method detection level (LT-MDL) reporting strategies with four pilot methods in 1998. These pilots were volatile organic compounds (Schedules 2020/2021), inductively coupled plasma-atomic emission spectrometry, ammonia plus organic nitrogen, and phosphorus (micro-Kjeldahl digestion) methods. The pilot methods produced LT-MDLs and new reporting levels set at the nondetection values that are presented in NWQL Technical Memorandum 98.07. The initial phase of implementing LT-MDLs at the NWQL is now complete.

For the second phase, the NWQL will start collecting LT-MDL data on October 1, 1998, for additional high-demand water methods, which are defined as those methods receiving more than 250 organic and 750 inorganic sample-analysis requests during a single year. By the end of fiscal year 1999, enough data will have been collected to determine LT-MDLs for these methods. The LT-MDLs and nondetection values (NDVs=2 x LT-MDL) will be announced by next summer to become effective October 1, 1999. Methods with fewer requests per year and nonwater matrix methods will be started in a third phase.

Ten organic methods and about 70 inorganic constituents are part of the phase 2 LT-MDL data collection. Several high-demand methods, such as drinking-water methods and methods designed to report higher concentrations, will not be included in this change of reporting conventions.

Since starting the four pilot methods, additional experience was gained with significant figures in the statistical evaluation of LT-MDLs. The standard number of significant figures reported for concentrations near the LT-MDL might need to be adjusted, especially since these concentrations are estimated. Starting with the new high-demand water methods in fiscal year 1999, the NWQL will limit significant figures for NDVs to the digit represented by the standard deviation used to calculate the LT-MDL and NDV. For example, for an LT-MDL of <1.213 with a standard deviation of 0.135, the NDV will be limited to the tenths place, that is, <1.2.

Concentrations of detected constituents will include three significant figures within the analytical range of the method. Concentrations that are remarked with an estimated "E" code, which will include all those less than the NDV, should be limited to two significant figures or the least significant digit.

The NWQL presented the LT-MDL and NDV policy at the National Monitoring Council's Interagency Methods and Data Comparability Board meeting held at the NWQL in August. The Board discussed a variety of important issues, including laboratory accreditation (see related article on page 1 of this Newsletter). This group of distinguished scientists acknowledged the potential value of the LT-MDL and NDV approach, and looked forward to publication of the new LT-MDL procedure and reporting conventions. A journal article and Office of Water Quality technical memorandum detailing the procedure and use of the LT-MDL and NDV concept in data reporting currently are being drafted.



*by Brooke Connor, Tom Maloney,
and Bill Foreman*

Scientists brainstorm in the Rockies

Seven scientists from the National Water Quality Laboratory spent a few days in Estes Park, Colo., last month, brainstorming with other U.S. Geological Survey scientists who are interested in identifying emerging environmental contaminants. Mike Thurman, Kansas District, coordinated the meeting September 16-18.

Ed Furlong, Mark Burkhardt, Bill Foreman, Tom Leiker, Mark Sandstrom, and Bob Green, all with the Methods Research and Development Program, along with Steve Zaugg, Organic Chemistry Program, were part of a working group assembled by the Toxic Substances Hydrology Program. A central goal of the meeting was to develop a list of compounds for future studies and to collaborate on new research. The scientists--drawn from throughout the USGS--are seen as a powerful resource for advising the Office of Water Quality regarding emerging issues.

Some of the emerging issues relate water pollution to public health. For example, some chemicals have been implicated in endocrine disruption or birth abnormalities. This development represents a paradigm shift in understanding how to target future research because of the additional complexity. Some of the compounds discussed at Estes Park included pharmaceuticals, food additives, herbicides, insecticides and their metabolites.

The sharing of plans and ideas is essential to making the most of research dollars and developing useful contributions on a national scale. A summary of the results of the brainstorming session will appear in the next issue of the NWQL Newsletter.



by Bob Green

The NWQL has an oversupply of the following report should our readers need a copy for their bookshelf or reference library:

Fishman, M.J., and Friedman, L.C., eds., 1989, Methods for determination of inorganic substances in water and fluvial sediments: U.S. Geological Survey Techniques in Water-Resources Investigations, book 5, chapter A1.

Nutrient preservation report approved

A U.S. Geological Survey report assessing the effects of different sample-processing treatments on the stability of nutrient species in water samples during storage was approved and is now in press. The authors are Charles Patton, NWQL analytical chemist, and Edward Gilroy, Branch of Quality Systems statistician (ret.).

The report is the result of a cooperative effort started in 1992 between the NWQL and the Office of Water Quality. Samples and analytical data were collected to produce a valid statistical comparison between the then-current USGS (mercury amendment) and U.S. Environmental Protection Agency (USEPA) (sulfuric acid amendment) nutrient sample preservation protocols. Effectiveness of these USGS and USEPA protocols were also compared with a third protocol involving field filtration and chilling, or in the case of whole-water samples, chilling only.

The experimental design for this study required collection and processing--splitting, filtering, adding amendments--of surface-, ground-, and municipal-supply water samples at 15 widely distributed sites within the continental United States. These samples were shipped to the NWQL for first analyses within 24 hours of collection.

Upon receipt at the NWQL, processed samples from each collection site were analyzed in quadruplicate for ammonium, nitrate, nitrite, dissolved Kjeldahl nitrogen, orthophosphate, dissolved phosphorus, total Kjeldahl nitrogen, and total phosphorus. Quadruplicate analyses of splits from each collection site were performed five more times at set intervals up to 35 days after collection.

Data on which this report is based, including nutrient concentrations in synthetic reference samples determined concurrently with those in real samples, resulted from more than 20,000 analytical determinations and are published separately in Patton and Truitt (1995). Patton said that even 10 years ago, the field and laboratory logistics required for an experiment of this scope and magnitude would have been prohibitive. Recent advances in USGS at-site sample-collection equipment and methods helped to make this study possible, including instrumentation, computer hardware and software for data acquisition and processing, and near-universal access to overnight shipping service.



by Charles J. Patton

References cited:

Patton, C.J., and Gilroy, E.J., in press, U.S. Geological Survey nutrient preservation experiment--Experimental design, statistical analysis, and interpretation of analytical results: U.S. Geological Survey Water-Resources Investigations Report 98-4118.

Patton, C.J., and Truitt, E.P., 1995, U.S. Geological Survey nutrient preservation experiment: Nutrient concentration data for surface-, ground-, and municipal-supply water samples and quality-assurance samples: U.S. Geological Survey Open-File Report 95-141, 140 p.

Procedure outlined for NWQL to certify organic blank water

A recent lot of NWQL organic blank water (OBW) had low-level detections of total organic carbon (TOC) and dissolved organic carbon (DOC). The NWQL notified its customers regarding the contamination. In response to this notice, the NWQL was asked to outline the procedures used for certifying OBW.

The NWQL contacts vendors and specifies the grade of water, acceptable concentrations of contaminants, and quantities needed. This decision is complicated since the NWQL would like to order enough supply to keep the cost down while also considering shelf life.

After a vendor is selected, the lot is sequestered for testing and certification. The vendor sends the NWQL a case of water, and the Quality Assurance Unit prepares the water by pouring aliquots into bottles for analysis.

Two grades of water are certified through the NWQL: pesticide and volatile. The pesticide grade water is analyzed as supplied by the manufacturer. The volatile grade water is purged at the NWQL with ultrapure nitrogen for several hours prior to analysis. The pesticide grade OBW is then analyzed using the following schedules:

Lab code 52 - Phenols; Lab code 113 - DOC; Lab code 114 - TOC; Schedule 79 - Herbicides; Schedule 1379 - Triazines; Schedule 1385 - BNA; Schedule 1608 - Organochlorines (EPA 608); Schedule 1399 - Low-level Organophosphates and Organochlorines; Schedule 2001 - Multiclass pesticides; and Schedule 2050 - Multiclass pesticides.

The pesticide grade (nonpurged) water must meet the following guidelines: DOC less than or equal to 0.1 mg/L (milligram per liter) and TOC less than or equal to 0.2 mg/L. Detections in all schedules must be at or less than the current method reporting level and the water must not have significant background interferences.

The volatile grade (nitrogen purged) water must meet the aforementioned criteria and also be analyzed by the low-level Volatile Organic Compound (VOC) Schedule 2020. No more than five compounds may be present, and each concentration must be less than the current reporting level of the method.

If the test water meets all the criteria, then the NWQL proceeds with the order. If the criteria are not met, then the requisition process begins again. Contact either Jeff Pritt (geomail jwpritt@usgs.gov) or Kim Pirkey (geomail kdpirkey@usgs.gov) with any questions regarding the certification of organic blank water.



by Kim Pirkey

USGS customer feedback

The National Water Quality Laboratory (NWQL) fulfills analytical requirements of the U.S. Geological Survey (USGS) by analyzing environmental samples for inorganic, organic, and radiochemical constituents and for biological assemblages. The NWQL strives to provide high-quality results in a timely, cost-effective manner. The Laboratory provides project planning and data-interpretation assistance to meet established quality objectives and to support USGS water-quality investigations. The Laboratory also develops new analytical methods and sample-collection procedures as needed by the USGS.

The NWQL will not stand pat. "We want to improve the aforementioned services," said Bob Williams, chief, NWQL. Our readers' suggestions to the following questions will help us to strengthen services in response to customer needs:

- What do you--our USGS customer or potential customer--feel is your greatest need?
- What are you doing to satisfy this need?
- If you were looking for a new service to satisfy this need, what kind of service would you be seeking?
- What could the NWQL do for you? What advice can you give our management team?

Send your comments and suggestions to the [editor](#) via electronic mail (jwraese@usgs.gov), FAX (303-467-8240), or letter (Jon Raese, USGS/NWQL, 5293 Ward Rd., Arvada, CO 80002).

Thank you.

Construction picks up pace

Construction of the new laboratory at the Denver Federal Center continues at a rapid pace. NWQL personnel have been touring the new facility, and excitement is growing now that individual offices and laboratories are definable.



West Façade of the NWQL at Denver Federal Center shown in various stages of completion at end of August.



Laboratories in the south wing, for example, are being tiled and casework installed. In the north wing, walls are going up and windows are being installed. Although some problems have been encountered, the overall progress on construction has been smooth and remains on schedule for occupancy to begin in March.

BTG, a move management company, has been contracted to inventory the old laboratory on Ward Road. BTG arrived on-site August 17 and proceeded to set up furniture and equipment standards. On August 24, the company started its inventory and estimated 6 to 8 weeks to complete the process.

The data base assembled by BTG will provide a detailed inventory of equipment and an inventory control program. BTG will also provide the on-site movers with customized orders that show where furniture and equipment are located in the old facility and where it should be placed in the new laboratory.

Preliminary planning to restore the Ward Road facility is underway. The General Services Administration, USGS Office of Program Support, and the building owner have met, but restoration agreements have not been defined.

Visit the NWQL's [Relocation](http://www.nwql.cr.usgs.gov/USGS) Web site on the USGS-visible page (<http://www.nwql.cr.usgs.gov/USGS>) to view the construction progress (this Web site cannot be accessed by the public). The Laboratory Operations Program changes move-related information on this page as events occur.



by Pat Timme

New titles in print

Brigham, A.R., and Kingston, J.C., 1998, Consistency, accuracy and accessibility-- Promoting cooperation among governmental agencies, museums and taxonomists [abs.]: *Journal of Phycology*, supplement to v. 34, no. 3, June 1998, Abstracts of papers of the Phycological Society of America Annual Meeting, 52nd, Flagstaff, Ariz., 1998, p. 9.

Sandberg, J.B., and Szczytko, S.W., 1997, Life cycle of *Isoperla lata* (Plecoptera: Perlodidae) in a central Wisconsin trout stream: *The Great Lakes Entomologist*, v. 30, no. 4, p. 143-159.

Ultrax nitric acid to be packaged in polypropylene vials

The Office of Water Quality (OWQ), National Water Quality Laboratory (NWQL), and the Quality of Water Service Unit (QWSU) have evaluated and approved polypropylene vials as an alternative to the glass ampules and Teflon vials for Ultrax-grade nitric acid preservative. The Teflon vials are expensive (a box of 24 costs about \$480 compared to \$108 for the polypropylene vials), and the glass ampules could add aluminum, barium, boron, silica, chromium, and zinc, contaminating the field blanks and samples.

In recent tests, the samples that used the polypropylene vials and Ultrax acid were equal in quality to the Teflon ampules and much better than the Ultrax ampules in glass. At Newsletter deadline in mid-September, the tests were completed, and an effective date for the change was scheduled to be announced in a technical memo.

Although concentrated 15 N nitric acid is not recommended for storage in polypropylene, a diluted concentration of 7.6 N HNO₃ apparently has no effect on the vials. The polypropylene vials would be provided with 2 milliliters of 7.6 N HNO₃ per vial, which should acidify most of the field samples to a pH less than 2. These vials are easier to use than the Teflon vials and the glass ampules.



by Ann Watterson

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.

The purpose of the *National Water Quality Laboratory Newsletter* is to improve communications on water-quality issues in the U.S. Geological Survey. The Newsletter is for administrative use only. It should not be quoted or cited as a publication. The use of trade, product, or firms names in this publication is for descriptive purposes only and does not imply endorsement by the U.S. Geological Survey. Visit the NWQL Home Page Web site at <http://wwwnwql.cr.usgs.gov/USGS>.