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Chain of custody at NWQL standardized, strengthened

For several years, the NWQL has offered three levels of sample chain-of-custody (COC) procedures to meet the needs of U.S. Geological Survey customers requiring high levels of sample accountability and security.

The first level is routine, the security level used for most samples logged into the NWQL. No special handling or paperwork is required because the NWQL is a restricted-access facility that uses a proximity card system for employee entry.

The second level involves COC record forms that accompany samples to be filled out and returned to the sender. This procedure documented receipt by the NWQL and the condition of the sample bottles when received, but it did not actually imply that COC procedures were followed. The NWQL set up a new Sample Receipt Notification System in June 2002 that provides e-mail to customers regarding the status of samples logged in that day. This new notification system has made obsolete the use of COC forms to document sample receipt by the NWQL.

The third COC level has been expanded to eliminate the second-level COC and is soon to be the only COC procedure offered by the NWQL. Under COC rules, all COC samples are kept in someone's custody (physical possession or view) at all times or kept in a designated locked, limited-access room or refrigerator until needed. At no time are the samples left unattended unless they are locked away. All transfers of custody are documented and the time/date for each analyst who removes an aliquot from a bottle also is documented.

To access these samples, analysts must arrange for a sample custodian to give them the COC samples or witness the analysts' handling of the sample. Detailed documentation is kept for all handling of COC samples. These records are available to the project chief as defined in the project proposal (see below).

All customer projects requiring COC must be arranged through the NWQL prior to the submission of samples. USGS district project chiefs must contact the NWQL Business Development Team at *labhelp@usgs.gov* or phone 1-866-ASK-NWQL (1-866-275-6975) to begin COC arrangements. A chemist whose background is appropriate to the project will be appointed as liaison between the project leader and the NWQL. The NWQL chemist will write a "project proposal," detailing the specifics of the COC agreement between the NWQL and district personnel.

NWQL will assess a handling cost for COC procedures commensurate with the number of samples involved and the number of individual analytes requested. These costs will account for the additional security and individual attention required by the COC procedures.

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These COC procedures will be implemented after review by the U.S. Department of the Interior's Office of the Solicitor, because that office will be defending NWQL data in the event of litigation. COC processing is recommended for projects that may be associated with future litigation and for projects that are expected to have a high degree of scientific and public scrutiny.

For more information, see the NWQL chain-of-custody web page at *http://wwwnwql.cr.usgs.gov/USGS/COC/coc.html* or contact Al Driscoll (*driscoll@usgs.gov* or call 303-236-3470).

• AL DRISCOLL

Green accepts detail to teach at OPM training center

Greg Mohrman, NWQL Chief, has announced that Robert Green will take part in a special program with the Office of Personnel Management (OPM). Mohrman said that the chief of Methods Research and Development will be working for the Executive-in-Residence program at the OPM training center in Aurora, Colorado. Green's initial assignment will be developing a project management curriculum for Federal managers.

Green's tour of duty will be for a minimum of 1 year and could extend to 2 years. His last day at the Laboratory was January 8.

Ed Furlong, research chemist, will serve as acting program chief for the first 6 months starting January 12, followed by John Garbarino, also a research chemist, who will hold the position for the second 6 months.

Mohrman added that Green would continue to assist the Evaluation Team, which is interviewing candidates for the chief of Analytical Services.

NWQL helps World Bank study arsenic exposure during pregnancy and birth outcomes in Bangladesh

Bangladesh is facing widespread arsenic contamination of drinking water obtained from shallow tube-wells. About 97 percent of the rural population relies on shallow tube-wells for drinking water, and an estimated 25 percent of such well water is contaminated with hazardous levels of arsenic (greater than or equal to 50 parts per billion) from geological sources[1].

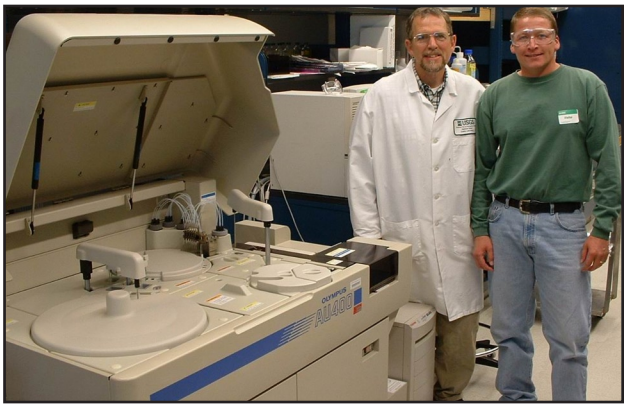
The World Bank has sponsored a study to determine the relations between arsenic exposure during pregnancy and birth outcomes. This research involves record reviews of women who attended nutrition clinics during pregnancy, interviews of the women after the end of the pregnancy, and assessment of the total inorganic arsenic content of their drinking water.

An integral part of this study is to test drinking-water samples to determine total inorganic arsenic concentration. Initial samples were analyzed in a Bangladesh laboratory using hydride generation–atomic absorption spectrophotometry. In December 2003, the World Bank solicited the help of the U.S. Geological Survey National Water Quality Laboratory in providing an independent verification of these results for 60 water samples.

Craig Stapert, physical science technician at NWQL, analyzed the samples using graphite furnace–atomic absorption spectrophotometry. Stapert's results compared favorably to the original results with a linear regression correlation coefficient of 0.97.

• JOHN GARBARINO

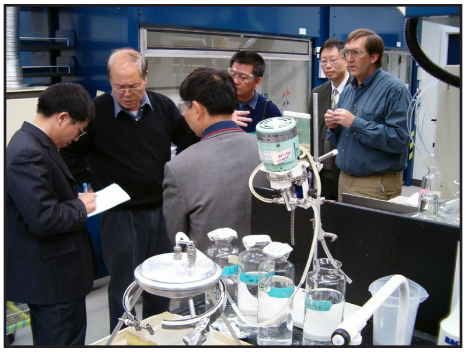
[1]Department of Public Health Engineering, Government of the People's Republic of Bangladesh; Department for International Development, United Kingdom; British Geological Survey, United Kingdom, 2000, Groundwater studies of arsenic contamination in Bangladesh—Final report summary: 16 p. The report is available at URL *http://www.bgs.ac.uk/arsenic/bangladesh/Reports/SummaryWithFigs.pdf*



ROBOTIC CLINICAL ANALYZER TESTED—Kris Fischer (right), water-quality analyst for the Klamath Tribes in south-central Oregon, and Charles Patton, Methods Research and Development Program, recently discussed using robotic, random-access clinical analyzers (instrument in left foreground) for dissolved nutrient determinations in water samples.

Patton, along with NWQL colleagues Colleen Gupta and Jennifer Kryskalla, began evaluating this technology last July to replace air-segmented continuous flow nutrient analyzers. Compared to continuous flow analyzers, robotic, random-access clinical analyzers are easier to operate and maintain, use less sample and reagents, and require fewer consumable supplies. The concentrations of hazardous waste are reduced by a factor of 4. In addition, bar-code recognition hardware and software—standard features of clinical analyzers—have the potential to eliminate sample-handling errors and substantially reduce sample-turnaround time. This potential for increased performance and productivity, however, is offset by limited analytical method flexibility and user interfaces that lack functionality and data-processing software that environmental analysts need and expect. Workarounds for these deficiencies are being explored and should be complete by midsummer 2004.

SCIENTISTS FROM CHINA VISIT LAB—A delegation of scientists from the People's Republic of China visited the NWQL December 17 to exchange program and project ideas and developments in technology with USGS National Research Program (NRP) scientists. Cary Chiou (second from left), resident NRP researcher in building 95, accompanied the lab tour. Gary Cottrell (far right), chief of the NWQL Business Development Team, led the tour. The group, representing the China Academy of Geological Sciences, voiced a special interest in water-quality contaminants, such as metals, agricultural



run-off nutrients, pesticides, and sewage. A Chinese-American businessman, James Xu (second from right), with U-Linx Corp., accompanied the delegation.

Workshop held for pipet and balance training

An overview of standard operating procedures for using pipets and balances was taught in four sessions on January 14 and 21 at the NWQL.

Technical staff, primarily from Analytical Services, Methods Research and Development, and Quality Assurance, attended the training sessions. Class instructor was Kane Aldinger.

FISH TISSUE ANALYSIS—Lisa Greaser, chemist with the Methods Research and Development Program, prepares to extract fish tissue with an Accelerated Solvent Extractor. The samples will be analyzed to determine organochlorine pesticides, polychlorinated biphenyls, and polybromide diphenylethers.

New publications

NWQL authors are listed in boldface.

Reports

Mast, M.A., Campbell, D.H., Ingersoll, G.P., **Foreman, W.T.**, and Krabbenhoft, D.P., 2003, Atmospheric deposition of nutrients, pesticides, and mercury in Rocky Mountain National Park, Colorado, 2002: U.S. Geological Survey Water-Resources Investigations Report 03-4241, 15 p.

Journal Articles

Bednar, A.J., Garbarino, J.R., Burkhardt, M.R., Ranville, J.F., and Wildeman, T.R., 2003, Field and laboratory arsenic speciation methods and their application to natural-water analysis: Water Research, v. 38, no. 2, p. 355–364.

Ferrer, Imma, Schröeder, H.F., and **Furlong E.T.,** 2003, Atmospheric pressure ionization mass spectrometry—LC–MS

analyses of cationic surfactants: Methods and applications, *in* Knepper, T.P., Barceló, D., and De Voogt, P., eds., Analysis and fate of surfactants in the aquatic environment: Amsterdam, The Netherlands, Elsevier, v. 40, p. 353–383.

Petty, J.D., Huckins, J.N., Alvarez, D.A., Brumbaugh, W.G., Cranor, W.L., Gale, R.W., Rastall, A.C., Jones-Lepp, T.L., **Leiker, T.J.,** Rostad, C.E., and **Furlong, E.T.,** 2004, A holistic passive integrative sampling approach for assessing the presence and potential impacts of waterborne environmental contaminants: Chemosphere, v. 54, p. 695–705.

Frequently asked questions

New water-quality analytical method available to determine total and dissolved nitrogen and phosphorus in water following alkaline persulfate digestion.

Why is a new digestion method needed for total nitrogen and phosphorus?

Alkaline persulfate digestion was evaluated and validated as a more sensitive, accurate, environmentally benign, and less toxic alternative to Kjeldahl digestion for routine determination of total nitrogen (N) and total phosphorus (P) in surface- and ground-water samples.

Existing batch Kjeldahl digestion methods used at the U.S. Geological Survey (USGS) National Water Quality Laboratory (NWQL) had serious drawbacks, including:

- health and safety risks posed by concentrated acids, toxic reagents, such as mercury, and high temperatures (370°C);
- environmental effects and cost associated with processing and disposing of the waste containing mercury;

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- propensity of acidic digests to trap and become contaminated by ammonia vapors in ambient laboratory air; and
- higher laboratory reporting levels (LRL) that limit the precision of mass-balance estimates.

How will data from the new method compare to data from the Kjeldahl digestion?

Patton and Kryskalla (2003) compared the new method to traditional Kjeldahl digestion procedures for total N and total P.

Results from alkaline persulfate digestion represent total N, including nitrate plus nitrite N and ammonium and organic N. Kjeldahl N is only ammonium and organic N. Subtracting nitrate plus nitrite N from the persulfate digestion N produced results that were significantly different from Kjeldahl N at p = 0.05 from 2,066 paired results. The difference resulted from both positive and negative interferences of nitrate on the results of Kjeldahl digestion, especially in the presence of high nitrate concentrations.

Total P was statistically equivalent at the p = 0.05 level. There was no difference from a paired t-test for 2,093 paired results.

Are special treatments, preservation, or bottle types required?

The alkaline persulfate digestion may be performed on unfiltered and 0.45-micrometer (µm) filtered water. Filtered samples may be left unacidified or amended with 1 milliliter (mL) of 4.5 *N* sulfuric acid (H_2SO_4) (USGS water-quality field supply number Q438FLD) per 120 mL of sample. Standard 125-mL brown polyethylene bottles are specified for filtered samples, and 125-mL translucent polyethylene bottles for unfiltered samples.

Samples must be chilled to 4°C and shipped to the NWQL immediately.

Additional information is available on the NWQL USGS-Visible website at *http://wwwnwql.cr.usgs.gov/USGS*. Click on "LIMS Catalog" and search for the alkaline persulfate digestions using lab codes 2754 (filtered), 2755 (filtered, acidified), and 2756 (whole water) for total nitrogen (ammonia, nitrite, nitrate, organic) or 2757 (filtered), 2758 (filtered, acidified), and 2759 (whole water) for total phosphorus.

What are the new method numbers?

The USGS method numbers are I-4650-03 for whole water N and P, and I-2650-03 for filtered water N and P.

How sensitive is the new method?

The short-term method detection limit (MDL) for nitrogen is 0.015 mg-N/L (analytical range, 0.03 to 5.00 mg/L as N) and for phosphorus is 0.007 mg-P/L (analytical range, 0.01 to 2.00 mg/L as P). The LRL will be about twice the MDL concentrations.

A long-term method detection limit (LT–MDL) has not been assessed for this method.

Total N and total P concentrations determined for quality-control check solutions by the alkaline persulfate digestion method were tightly centered around published most probable values (MPV) and well within published control limits.

May any District use the new method?

Yes. The Office of Water Quality approved the new method on 2 July 2003 for all projects and programs.

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://wwwnwql.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:

Patton, C.J., and Kryskalla, J.R., 2003, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Evaluation of alkaline persulfate digestion as an alternative to Kjeldahl digestion for determination of total and dissolved nitrogen and phosphorus in water: U.S. Geological Survey Water-Resources Investigations Report 03-4174, 33 p.

- CHARLES PATTON, JENNIFER KRYSKALLA, AND ALLISON BRIGHAM

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