NATIONAL WATER QUALITY LABORATORY
NEWSLETTER
U.S. DEPARTMENT OF THE INTERIOR U.S. GEOLOGICAL SURVEY
Volume 4 Number 2 April 1996

Lab collaborates on Teflon cone splitter field tests

Several U.S. Geological Survey offices have collaborated to test a sample-splitting device used in on-site studies for trace elements. Joining forces in the experiment were the Office of Water Quality (OWQ), Instrumentation Committee (ICOM), National Water Quality Laboratory (NWQL), and Upper Colorado River Basin (UCOL) study unit of the National Water-Quality Assessment program.

The experiment was designed by John Gray (OWQ), Kathy Fitzgerald (OWQ), Nancy Driver (UCOL), and Bob Boulger (UCOL). Funding was provided by ICOM. Ed Zayhowski was the contact person at the NWQL.

In October 1995, the OWQ asked the UCOL study unit to test the effectiveness of the USGS field-cleaning protocol for removing trace-element contamination from the Teflon cone splitter. The splitter is designed to provide uncontaminated, representative subsamples of a composite water sample for subsequent inorganic or organic analyses.

The objective of this experiment was to split samples from the contaminated water and then determine whether the splitter could be decontaminated for trace elements using a modification of on-site cleaning procedures described by Horowitz and others (1994, p. 52-56).* The modification consisted of not disassembling the splitter during decontamination procedures.

Tests were performed at six stream sites that were impacted by metal mine drainage in the Upper Colorado River basin. Blank samples were processed before splitting the stream sample and after processing the stream sample and decontaminating the splitter. The stream sample and the after-decontamination blank were submitted for analysis to the NWQL, where Ed Zayhowski and his crew did a great job of expediting analytical data in spite of the furlough. Al Driscoll's Quality Assurance Unit coordinated timely reanalysis of selected constituents.

Results showed that the splitter could be sufficiently cleaned onsite after sampling water contaminated with trace elements. Use of this modified procedure will make on-site decontamination of the splitter more efficient. A somewhat surprising result was that all blank water is not blank! But that's a topic for another article.

Sample-Splitting Device Tested – Teflon cone splitter with protective cover and bottles.

Recommendations based on these results and other cone-splitter tests will be released shortly by the OWQ.

Hats off to those involved in this collaborative effort! All contributed to a successful experiment. For further information on these tests, contact Bob Boulger at 970/245-5257, ext 3023.

by Bob Boulger

* Horowitz, A.J., Demas, C.R., Fitzgerald, K.K., Miller, T.L., and Rickert, D.A., 1994, U.S. Geological Survey protocol for the collection and processing of surface-water samples for the subsequent determination of inorganic constituents in filtered water: U.S. Geological Survey Open-File Report 94-539, 57 p.

Park Service asks Survey to monitor air quality at Tennessee park

The U.S. Geological Survey assisted the National Park Service hazardous Materials Response Team in assessing the extent of contamination from a fuel-oil pipeline rupture near Lookout Mountain in Chattanooga National Military Park in Chattanooga, Tenn. The park was one of the first four national military parks authorized by the U.S. Congress, and in fall 1863, it was the site of some of the hardest fighting of the Civil War.

The terrain in the park is characterized by steep escarpments with Mississippian limestone riddled with fractured karst features capped by Pennsylvanian sandstone. The park was created in part because there were few other battlefields in the world that presented more formidable natural obstacles to large-scale military operations than the slope of Lookout Mountain.

After the fuel-oil spill in early February, the U.S. Geological Survey was asked by the National Park Service to assist them in determining the areal extent of contamination in the park. Bob Brock, chemist with the DODEC Laboratory Support Group, and Connor Haugh, hydrologist with the Tennessee District, went to Chattanooga in late February to monitor the air quality of some of the numerous caves near Lookout Mountain. Air quality was monitored with a portable gas chromatograph used for real-time determinations of volatile organic compounds in air samples collected from the caves. According to Brock, "The collection of air samples and the interpretation of the data were major challenges because of all the variables that had to be considered."

by Bob Brock

Instrument used to determine fluoride at low concentrations malfunctions; fix underway

The National Water Quality Laboratory (NWQL) is currently experiencing problems with regular fluoride determination. This is lab code 31, parameter code 00950B, Fluoride by Ion Selective Electrode (ISE). There is no problem with the NWQL Low-Level Fluoride determinations, lab code 1260, parameter code 00950D, Fluoride, Low Ionic Strength, by Ion Chromatography.

Because of a malfunction in the instrument, recent fluoride determinations of concentrations of 0.2 mg/L and less were reported as 0.2 mg/L. Concentrations greater than 0.2 mg/L were reported correctly. The NWQL failed to catch this malfunction because it only affected data near the detection limit where a larger amount of variance is normally expected. NWQL customers spotted this problem by noticing that all lower concentration fluoride determinations, including clean blanks, were being returned as 0.2 mg/L. The NWQL is grateful for its customers having discovered the error. Thanks and we'll do our best to fix it.

The fluoride instrument was recently repaired by the manufacturer and will be sent back for additional work. In the meantime, the Methods Research and Development Program and Production Program personnel are working to devise an alternate, but equivalent, ISE control device. All suspect fluoride determinations will be reanalyzed as soon as possible after the instrument is back in operation.

The NWQL apologizes to its customers for this unfortunate malfunction. Please check any recent fluoride determinations for multiple 0.2-mg/L measurements when lower concentrations may have been expected. Those may be in error and will be reanalyzed. Higher concentrations are unaffected by this malfunction. For more information, contact Gary Cottrell (cottrell), (303) 467-8248.

by Pete Rogerson

Methods Research staff coordinate courses at National Training Center

NWQL research chemist Bill Foreman recently finished coordinating the training course "Environmental Processing of Organic Chemicals" (G0482) at the National Training Center (NTC) in Denver. The 1-week course (March 11-15) covered aspects of processing semivolatile organic contaminants, including major chemical classes and their physical/chemical properties.

Also discussed were sorption/partitioning, air-water exchange, hydrolysis, oxidation, photolysis, bioaccumulation, and other fate and transport mechanisms. Foreman said the course was designed to give the students "a firm understanding of the movement of organic contaminants in geochemical cycles with particular emphasis on transport processes that influence water quality."

Ed Furlong and Mark Sandstrom are coordinating the course "Sampling and Analysis of Organic Contaminants in Surface and Ground Water (G0242)," scheduled to meet April 22-26 at the NTC. The course is designed for hydrologists involved in projects that emphasize organic contaminants. Instructors will explain on-site sampling methods and laboratory analytical procedures for determining organic contaminants in samples of surface water, ground water, and bed sediment.

In addition, instructors will demonstrate the use of quality-assurance (QA) samples during on-site sampling and provide guidance on the interpretation of QA sample analytical results. The course is intended for personnel working in water-quality projects.

Home, but not alone, on the Web

The National Water Quality laboratory invites its customers to check out our home page at

<URL:http://wwwnwql.cr.usgs.gov/>.

We are continuing to develop information services at our Web site for the use of USGS readers. Please check us out. We welcome your comments.

Duties shifted in Quality Management Program

For the past year, Ann Watterson has been the primary customer contact for reanalysis requests. These are the requests that are forwarded to DENQC Geomail mailbox. Ann has since assumed responsibility for coordinating NWQL's participation in Performance Evaluation Studies, quality assurance and quality control of field supplies (sample bottles, nitric acid, and other materials and accompanying certificates of analysis), and oversight of Standard Operating Procedures within the Laboratory.

Kathy Bryant is now the DENQC contact for processing requests for reanalysis. Kathy is also developing a process to conduct internal audits of NWQL analytical methods. Personnel throughout the Laboratory will be involved in the audit process. The NWQL plans to begin audits in the spring.

by Tom Maloney

Central Region schedules outreach events in April

The USGS Central Region is participating in several outreach events in April, including the KCNC TV News 4 Education Expo and the USGS Open House.

The News 4 Education Expo is scheduled from about 8 a.m. to 5 p.m., April 12 and 13, at the Colorado Convention Center, 700 14th Street, Denver. The event is expected to attract some 20,000 visitors throughout the two days. The USGS will have three booths, including one for the Water Resources Division. The Expo provides a USGS showcase for educators in a four-state area.

The second event is the USGS Central Region Open House on April 27 at the National Earthquake Information Center, 1711 Illinois Street, in Golden, Colorado. The Open House is slated to run from 9 a.m. to 3 p.m. The general public is invited.

Bottle mix-ups decline at Lab

Over the last several years, the Log-in Unit at the NWQL has worked very hard to reduce the number of bottle mix-ups that find their way into the workflow. Bottles are mixed when an error is made between sample collection and analysis of the sample, resulting in analytical measurements being assigned to the wrong station identifier, date, and time of collection.

Bottle mix-ups are typically caught at the NWQL when samples fail quality-control (QC) checks for anion/cation calculations or field/laboratory comparisons. Occasionally, District customers also may spot an anomaly in their results that indicates a mix-up. All suspected bottle mix-ups are investigated by the NWQL Quality Assurance Unit (QAU), and both the log-in procedure and analytical measurements are verified or reanalyzed when appropriate. Resolution of bottle mix-ups is necessarily a complex and time-consuming process.

To reduce the frequency of bottle mix-ups, the Log-in Unit has modified the way samples are unpacked, lined up, and logged-in so as to minimize the chance of errors at NWQL. In addition, the NWQL has established a policy of calling District customers whenever a question arises at log-in so that it can be cleared up right away. Also, problem reports have been produced for District customers to inform them if samples were received with improper or incorrect log-in information. This report was designed to allow customers to identify those sample-submission practices that could be improved to minimize the chances of errors. Unfortunately, some customers chose to use these reports as performance-monitoring tools rather than as process-improvement tools. This resulted in some negative reactions to NWQL efforts to improve the log-in process. The NWQL regrets this unanticipated outcome of efforts to improve the process.

The good news is that the error rate of bottle mix-ups has markedly decreased over the last year. In calendar year 1994, the NWQL experienced 130 identified bottle mix-ups for an error rate of 0.185 percent. Because of improved NWQL log-in procedures and great cooperation from District customers, the number of identified bottle mix-ups decreased to 46 in calendar year 1995, for an error rate of 0.084 percent. Of those 46, 20 have been identified as a single set of log-in errors at NWQL that resulted in another modification to the log-in procedures. The NWQL continues to work with its internal procedures to reduce all sources of errors and with District customers to minimize errors and confusion at receipt.

Thanks to all District customers who have worked with the NWQL to improve the flow of log-in information so that the process goes smoothly, and to the Log-in staff for their continued efforts to strive for perfection at log-in.

by Pete Rogerson

Quality Council appoints team to improve Lab tours

Historically, tours at the NWQL have received mixed reviews from District staff and the general public. At the request of the Quality Council, a team has been assigned to review how tours are arranged and conducted and to recommend improvements.

The NWQL Quality Council placed a March 31, 1996, deadline for a report outlining the team's recommendations. The team members are Pat Alex, Gary Cottrell, Lee Duray, Suranne Horodyski, Dennis Markovchick, Diane Moffett, Steve Moulton, and Ralph White. Armin Burdick represents the Quality Council as facilitator for the group.

by Dennis Markovchick

Inorganic Chemistry Program reorganizes

The Inorganic Chemistry Program was reorganized from five to four units, effective March 3. The program was reorganized to improve efficiency for inorganic analyses.

The four units and their supervisors are as follows: Plasma (Ed Zayhowski), Majors (Glenda Brown), Nutrients (Juan Vasquez), and Atomic Absorption (Betty McLain). Questions concerning any of these units should be addressed to the supervisor. Harold Ardourel is acting assistant chief, Inorganic Chemistry Program, and Merle Shockey is still program chief.

The Plasma Unit has responsibility for all inductively coupled plasma, inductively coupled plasma/mass spectrometry, direct-current plasma, and tissue-preparation activities. The Atomic Absorption Unit provides analyses by flame, hydride, cold vapor mercury, and graphite furnace atomic absorption spectrophotometric methods. The Nutrients Unit provides analyses of all nutrient parameters, preparation of bottom materials, colorimetric silica analysis, and sulfide. The Majors Unit has responsibility for potassium, anions (including Cl, SO4, F, Br, I, and cyanide), physical property analyses (color, turbidity, residue on evaporation, suspended solids, and pH/conductance), alkalinity, and acidity.

by Glenda Brown

Mail Basket

Stratton pleased with services

Mr. Rogerson,

This is just a quick note to thank lab personnel for a recent out-of-the-ordinary request. I am the water-quality data-base manager in Minnesota. The other day I received a request to have some water-quality samples "returned" to us. One of the cooperators on a project here was interested in using the excess water. This was a new one on me. So I called Kathy Bryant in the Quality Assurance Unit, and she said, "I'll see what we can do."

This request was made on Friday, January 26. By Tuesday, January 30, I received a message saying that the samples were in the mail. This kind of request could have taken me hours searching for the correct samples. I want to thank Kathy, Al Driscoll, Will Lanier, and all the people involved in this request for their quick action. I often deal with Kathy, Carmen Reed-Parker, and others in more "ordinary" data requests such as reloads and reruns. I rely on that group to the point where I may be taking their efficient dedication to work for granted. I know that I can count on them and I do.

I guess what I am saying is that because of the way in which the lab, in general, and some personnel, in particular, go about their duties, it saves me time and worry. I can count on the lab, and I wanted to thank you and everyone at the lab. On a regular basis, I commend and recommend the lab and its services to my colleagues. Keep up the good work!

Greg Stratton, Mounds View, Minn.

Custom method developed to determine hydrocarbon metabolites in fish bile

The NWQL Methods Research and Development Program is using a new custom method to analyze fish bile for polyaromatic hydrocarbon (PAH) metabolites as benzo[*a*]pyrene equivalents. A custom method requires special arrangements with the NWQL.

Briefly, water and sediment near urban centers can contain large concentrations of PAHs. Aquatic organisms accumulate these compounds and metabolize some to toxic and carcinogenic products that might be more harmful than the parent compounds. However, routine analysis of tissue samples from these aquatic organisms often show only trace levels of PAHs. Since PAH and PAH metabolites accumulate in bile for metabolism and removal, determining the concentration of these compounds in the bile is a better estimate of the short-term exposure than the tissue analysis.

The method requires a high-performance liquid chromatograph (HPLC) equipped with a fluorescence detector. A gradient using methanol and acetic acid acidified water is used to separate the PAH metabolites on a reverse-phase HPLC analytical column. Each sample is analyzed three times and the average fluorescent area count is compared to an average benzo[*a*]pyrene area count. The standard deviation of the three analyses, converted to milligrams per liter (mg/L), is provided as an estimate of the precision. A set spike and a blank are analyzed at the beginning and ending of each sample batch. In addition, three individual PAH metabolites (4-phenylphenol, 2-hydroxybiphenyl, and 9-phenanthrol) have been tested and measured in fish bile using the described method.

The method has been demonstrated on environmental samples. Bile samples from eleven channel catfish were analyzed. The average recoveries and standard deviation from spiked samples range from 89 $\pm$ 11 percent to 98 $\pm$ 2 percent. Between 11 and 103 mg/L PAH and PAH metabolites (as benzo[a] pyrene equivalence) were found in the bile samples. Three samples contained 4-phenylphenol, while two samples contained 2-hydroxybiphenyl. Contact Mark Burkhardt (303/467-8093; mrburk@usgs.gov) to find out more about this method and to arrange sample analysis before samples are sent to the laboratory.

by Mark Burkhardt

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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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