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WATER LOGS

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U.S. Department of the Interior
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IMPROVED EXTRACTION SYSTEM—Methods Research and Development (MRDP) chemist Steve Werner loads samples into a Spark Holland Prospekt II on-line solid-phase extraction (SPE) system. This new instrument is coupled with a high-performance liquid chromatograph/mass spectrometer (HPLC/MS). The on-line SPE system couples and automates sample extraction and instrumental analysis, and is particularly well suited for automating the trace analysis of polar organic compounds. Some of the potential benefits of this technology include shorter total analysis times, improved precision through automation, and substantial reduction in the sample volume required for analysis. Initially the MRDP will be automating and evaluating the determination of human health pharmaceuticals in filtered water using the on-line SPE system.

How to get more for your analytical dollars

Customers can start saving money on their analytical work by using less expensive tests. These savings are important because cost increases are expected for some projects in the new fiscal year as the NWQL implements full-cost recovery for analyses. A review of your analytical protocols and requirements may reveal a more cost-effective approach that still meets your data needs. These increases especially affect selected schedules and lab codes for routine water-quality projects that require trace metal analysis. The NWQL Business Development Team (BDT has been working with customers to reduce analytical expenses. In some cases, we have been able to save projects hundreds of dollars per sample. The following checklist includes steps that will reduce customer costs:

- Refer to Rapi-Note 03-029: Instead of requesting trace-element analysis by graphite furnace atomic absorption spectrophotometry (GF-AAS), change to inductively coupled plasma – mass spectrometry (ICP–MS) methods to earn substantial savings. Benefits include lower reporting levels and generally better precision.

- Provide field measurements for pH and specific conductance on the analytical services request (ASR) form. The NWQL will use these measurements if the customer requests that we delete lab pH and specific conductance (lab codes 68 and 69) on the ASR, thereby saving about $19 per sample in FY04 plus the cost of a poly-ethylene bottle (RU), which may not be required.

- Review the schedules being requested for possible duplicate analyses. Examples include silica (lab codes 56 and 667). Many of the older schedules for major ions include lab code 56, a colorimetric method. When adding a schedule for trace metals that may include silica (lab code 667, an ICP method), two silica requests are added to the NWQL Laboratory Information Management System (LIMS). Because lab codes 56 and 667 are considered unique analytical requests, the customer ends up getting two silica measurements and paying two charges. Similar instances occur with manganese by ICP and ICP–MS. Silica by ICP will save about $5 per sample over the colorimetric method in FY04.

- Create unique NWQL schedules that are designed for each project to help eliminate the use of multiple schedules. These unique schedules will be easier to review and maintain as project require-ments change. Do not use schedules that are not owned by either the customer's project or the NWQL--these are subject to unannounced changes by the owner. (The owner is identified in the NWQL Catalog.) The BDT would be happy to create as many indivi-dual schedules as needed. Send a list of the tests needed for your schedule to <labhelp@ usgs. gov>, and you will receive a new schedule number usually within 2 business days.

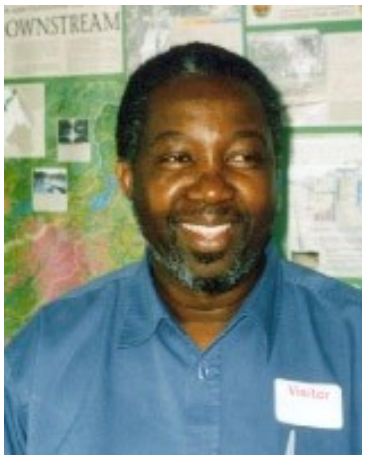
- Consult with BDT members when establishing a new project or schedule. They are up-to-date with analytical changes and prices, and are available to help customers get the most for their analytical dollars. For additional help, contact the BDT by e-mail <labhelp@usgs. gov>.

In addition, customers can use the ESTIMATOR function to see how changing Lab Codes could change the present fiscal year price for their projects. By changing schedules now, customers can save money this fiscal year.

The pricing differential between these two methods will be even greater in FY04, thereby providing greater savings next year. The FY04 pricelist is available for customers at http://nwql.cr.usgs.gov/usgs/pricelist/index.cfm. For questions, contact labhelp @usgs.gov.

If you are not sure whether your project can save money after reading this checklist, then call 1-866-ASK-NWQL (1-800-275-6975) and ask for BDT or call direct: Steve Glodt, 303-236-3721; Glenda Brown, 303-236-3160; David Macke, 303-236-3177. There is still time to save money this fiscal year if you check soon.

♦ **Steve Glodt**



DISTINGUISHED GUEST—Dr. Themba Duma, South African Department of Water Affairs and Forestry and the Institute for Water Quality Studies in Pretoria, is shown during a tour of the NWQL June 11. The 3-day visit included extensive meetings with lab personnel and orientation briefings. In return, Dr. Duma presented an overview of his laboratory in South Africa.

Alkaline persulfate digestion proposed as alternative to Kjeldahl digestion

Two new alkaline persulfate digestion methods have been approved by the USGS Office of Water Quality for determining total and dissolved nitrogen and phosphorus in water samples. The report that documents Methods I-2650-03 for determining dissolved nitrogen and phosphorus in filtered water samples and I-4650-03 for determining total nitrogen and phosphorus in whole-water acidified samples by alkaline persulfate digestion has been submitted to the Regional Executive for Water for approval.

The report includes details of the alkaline persulfate digestion procedure, interference studies, recovery of various nitrogen- and phosphorus-containing compounds, and other analytical figures of merit. The automated air-segmented continuous flow methods developed to determine nitrate and orthophosphate in the alkaline persulfate digests also are described.

In comparison to previously published studies of alkaline persulfate and Kjeldahl digestion methods for nitrogen and phosphorus determinations, this one is based on a much larger (about 2,100 paired results) and geographically diverse sample population collected during high-flow and low-flow conditions. In these respects, "it may be the most comprehensive study to date supporting adoption of the alkaline persulfate digestion method as the preferred alternative to the time-honored, but operationally flawed, Kjeldahl digestion method," according to the principal investigator, Charles J. Patton, research chemist at NWQL.

A Frequently Asked Questions column describing the two new methods will be featured in an upcoming issue of *Water Logs*.

QA internal audits started

During spring 2002, the Quality Assurance (QA) Section of the NWQL developed and launched an internal audit program to verify NWQL's compliance with approved standard operating procedures (SOPs) and published literature. In the last year the audit team, consisting of Kathy Bryant, Jim Lewis, and Al Driscoll, conducted 17 internal audits. The audits included procedures of the Analytical Services Section, laboratory implementation of secondary data review, the Support Services Section, and in-depth audits of the phenol and oil and grease procedures.

The NWQL audit team prepares for each audit by reviewing approved SOPs, published method reports, the NWQL Quality Management System, previous external and internal audit reports, Blind Blank information, and results from the Inorganic and Organic Blind Sample Programs. The audit team also interviews analysts to examine critical aspects of the procedure, reviews instrumentation and standard preparation logbooks, as well as calibration and quality-assurance procedures, and examines randomly selected test samples.

Audit reports, which include observed deviations, ancillary issues, and a follow-up of previous external and internal audit issues, are distributed after each audit to the appropriate supervisor. The supervisor provides a response to the audit team within 30 days, outlining the corrective actions needed to resolve deviations and also includes an estimated time for resolving problems. The audit team monitors progress for corrective action.

The audits have been helpful in preparing the NWQL for the National Environmental Laboratory Accreditation Program audit in July 2003. For questions related to NWQL internal audits, contact Kathy Bryant.

Kathy Bryant

INTERNATIONAL EXCHANGE PROGRAM—Ms. Olushola Sodeko (center), a Humphrey Fellow at Rutgers University and Chief Scientific Officer with the Federal Ministry of Water Resources, in Abuja, Nigeria, is briefed in the Login Section by Patricia Alex and Phil Grano. The Humphrey fellowship program is sponsored by the U.S. Department of State to foster leadership skills. Sodeko is focusing on water-quality management and sanitation. She visited the NWQL in mid-May to learn about general laboratory operations.

Laboratory schedule 2050 for pesticides to be eliminated

Laboratory schedule (LS) 2050 will be replaced with LS 2060 to provide results for more compounds, to lower detection levels, and to reduce costs to customers.

LS 2060 provides 28 additional compounds compared to LS 2050. In addition, LS 2060 laboratory reporting levels are about one order of magnitude lower than those of LS 2050. Moreover, LS 2060 uses a mass spectrometer, which is less subject to matrix interferences than is the photodiode array detection system that is used for LS 2050.

The date for the change is October 1. For further information about LS 2060, see Furlong and others (2001),* and Rapi-Note 03-031, at http://wwwnwql.cr.usgs.gov/ USGS/rapi-note.html. Refer questions to labhelp@usgs.gov.

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*Furlong, E.T., Anderson, B.D., Werner, S.L., Soliven, P.P., Coffey, L.J., and Burkhardt, M.R., 2001, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of pesticides in water by graphitized carbon-based solid-phase extraction and high-performance liquid chromatography: U.S. Geological Survey Water-Resources Investigations Report 01-4134, 73 p

WEED CONTROL—The General Services Administration (GSA) at the Denver Federal Center sprayed for weeds June 4 on the grounds surrounding the National Water Quality Laboratory in building 95. The product sprayed was Momentum, containing clopyralid, 2,4-D, and triclopyr. GSA notifies the NWQL in advance of spraying to preclude conflicts with analytical work.

ASTM recognizes Lab for long-term support

The American Society for Testing and Materials (ASTM) has presented an award to the NWQL for many years of dedicated service on various committees.

Present for the award June 19 were former NWQL employees, including Dave Erdmann, Doug Manigold, and Marv Fishman. Others who have contributed or continue to contribute their expertise include Donna Damrau, Ann Cleveland, Mike Schroeder, Jana Iverson, and the late Ed Zayhowski.

ASTM said the organizational award was made in recognition of NWQL's long-standing support of the Society and its mission. ASTM participation is voluntary, and all costs are borne by the participating agencies.

Greg Mohrman, NWQL Chief, thanked past and present scientists from the NWQL for the award, and said it "demonstrates a leadership role for our Laboratory."

USEPA honors Water Resources Discipline

The U.S. Environmental Protection Agency (USEPA) has presented the Assistant Administrator's Partner Award to the entire Water Resources Discipline of the U.S. Geological Survey (USGS) in recognition of contributions to public health and environmental protection. The USEPA specifically cited the collection, analysis, and dissemination of high-quality data.

The award was accepted June 25 by Robert Hirsch, USGS Associate Director for Water, on behalf of the USGS.

The USEPA said that USGS "has made a deliberate commitment to become the principal source of high quality, accessible and useful data on the nature, location, and characteristics of our Nation's water." In pursuing this goal, USGS has "adopted a strong customer service ethic and demonstrated initiative and creativity in identifying and meeting information needs."

The announcement cited a number of partnership activities, including peer reviews, sharing expertise, and joint project development. Specific examples included "development and refinement of better, faster, and cheaper analytical methods." The USGS also was cited for providing data and analysis for drinking-water standards for arsenic and MTBE (methyl *tert*-butyl ether), among others.

MONITORING WELL—One of the 200 monitoring wells for water and soil samples is shown adjacent to the National Water Quality Laboratory in building 95 at the Denver Federal Center. CH2M-HILL is under contract with the General Services Administration to drill the wells, as mandated by the Colorado Department of Public Health and Environment, to investigate and clean up contaminated sites from World War II ordnance plants.

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Recently published NWQL reports, journal articles

(NWQL authors in boldface)

REPORTS

Raese, J.W., 2003, National Water Quality Laboratory annual report, fiscal years 2001 and 2002: U.S. Geological Survey Open-File Report 03-14, 16 p.

JOURNAL ARTICLES

Andraski, B.J., **Sandstrom, M.W.,** Michel, R.L., Radyk, J.C., Stonestrom, D.A., Johnson, M.J., and Mayers, C.J., 2003, Simplified method for detecting tritium contamination in plants and soil: *Journal of Environmental Quality,* v. 32, no. 3, p. 988–995.

Garbarino, J.R., Bednar, A.J., Rutherford, D.W., Beyer, R.S., and Wershaw, R.L., 2003, Environmental fate of roxarsone in poultry litter. Part I. Degradation of roxarsone during composting: *Environmental Science & Technology*, v. 37, no. 8, p. 1509–1514.

Garrison, V.H., Shinn, E.A., **Foreman, W.T.,** Griffin, D.W., Holmes, C.W., Kellogg, C.A., Majewski, M.S., Richardson, L.L., Ritchie, K.B., and Smith, G.W., 2003, African and Asian dust: From desert soils to coral reefs: *BioScience*, v. 53, no. 5, p. 469–480.

Leenheer, J.A., **Ferrer, Imma, Furlong, E.T.,** and Rostad, C.E., 2003, Charge characteristics and fragmentation of polycarboxylic acids by electrospray ionization—Multistage tandem mass spectrometry, *in* American Chemical Society Symposium Series 850, chap. 18, p. 312–324.

Lerch, R.N., **Ferrer, Imma,** Thurman, E.M., and Zablotowicz, R.M., 2003, Identification of trifluralin metabolites in soil using ion-trap LC/MS/MS, *in* American Chemical Society Symposium Series 850, chap. 17, p. 291–310.

Rutherford, D.W., **Bednar, A.J., Garbarino, J.R.,** Needham, R., Staver, K.W., and Wershaw, R.L., 2003, Environmental fate of roxarsone in poultry litter. Part II. Mobility of arsenic in soils amended with poultry litter: *Environmental Science & Technology*, v. 37, no. 8, p. 1515–1520.

Wershaw, R.L., Rutherford, D.W., Rostad, C.E., **Garbarino, J.R., Ferrer, Imma**, Kennedy, K.R., Momplaisir, George-Marie, and Grange, Andrew, 2003, Mass spectrometric identification of an azobenzene derivative produced by smectite-catalyzed conversion of 3-amino-4-hydroxyphenylarsonic acid: *Talanta*, v. 59, p. 1219–1226.

Copies of these NWQL publications are available by contacting Diana Rime by e-mail at dcrime@usgs.gov or by telephone (303-236-3714).