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U.S. Department of the Interior  
U.S. Geological Survey



SUNRISE at Mono Lake, California, in mid-October 2004, by Christopher Lindley, USGS chemist at the National Water Quality Laboratory.

Chris uses a Canon D60 with exquisite results. In his log, Chris wrote about his first visit to Mono Lake: “What a treat. As the sun rose, I realized that I was among some very spectacular geology. Tufa formations (in this South Tufa Area) rose several feet above my head. Interesting composition could be made almost anywhere.”

## USGS method for glyphosate from Kansas Lab set up at NWQL in new approach for services

The National Water Quality Laboratory has implemented a method for the analysis of two herbicides, glyphosate and glufosinate, and the primary degradate of glyphosate, aminomethylphosphonic acid (AMPA).

Glyphosate [N-(phosphonomethyl) glycine] is a broad-spectrum, nonselective, postemergence herbicide that is extensively used in agricultural and landscape management, and is the active ingredient in Roundup herbicide. Glufosinate [ammonium DL-homoalanin-4-(methyl)phosphinate] is

similar to glyphosate in chemical structure and herbicidal activity and is also applied for weed control. Glufosinate is the active ingredient in Rely, Finale, and Liberty pesticide formulations.

The method was developed at the USGS Kansas Organic Geochemistry Research Laboratory as an official method with unrestricted use for all USGS projects and programs. The method report, U.S. Geological Survey Open-File Report 01-454, is available

*(continued on following page)*

at <http://ks.water.usgs.gov/Kansas/pubs/reports/ofr:01-454.html>.

The method uses derivatization of the sample and on-line solid-phase extraction (SPE) to isolate glyphosate, glufosinate, and AMPA from the water-sample matrix. The sample concentrate then is directly eluted into a high-performance liquid chromatograph/mass spectrometer (HPLC/MS), where the three compounds are separated, identified, and quantified.

The minimum reporting levels (MRLs) for glyphosate, glufosinate, and AMPA are 0.06, 0.12, and 0.16 microgram per liter, respectively. The method detection limits (MDLs), calculated using the U.S. Environmental Protection Agency's method (promulgated in volume 40 of the Code of Federal Regulations, part 163, appendix B), are 0.03, 0.06, and 0.08 microgram per liter for glyphosate, glufosinate, and AMPA, respectively. No concentrations less than the MRL will be reported.

The parameter and associated method codes for glyphosate, glufosinate, and AMPA are 62722A, 62721A, and 62649A, respectively.

**NEW METHOD IMPLEMENTED—**Laura Coffey, chemist in the Liquid Chromatography Unit, loads water samples in the automated sample tray in preparation for operating the new high-performance liquid chromatograph/mass spectrometer (HPLC/MS) for the determination of glyphosate, glufosinate, and the glyphosate degradation product aminomethylphosphonic acid (AMPA).



Reporting information for the method follows:

**Schedule 2052 (also Labcode 12052)—Glyphosate, aminomethylphosphonic acid (AMPA), and glufosinate, water, filtered, recoverable, µg/L, SPE, HPLC/MS**

| Test ID | MDL  | MRL  | Analyte   |
|---------|------|------|---|
| 62649A  | 0.08 | 0.16 | AMPA, water, filtered, recoverable, µg/L        |
| 62721A  | 0.06 | 0.12 | Glufosinate, water, filtered, recoverable, µg/L |
| 62722A  | 0.03 | 0.06 | Glyphosate, water, filtered, recoverable, µg/L  |

**Schedule description in NWQL catalog—Glyphosate, AMPA, and glufosinate, filtered water, SPE, HPLC/MS**

Description of sampling requirements (from the NWQL Catalog)—The water sample is filtered through a nominal 0.7-µm glass-fiber filter. Filters are preconditioned with about 200 mL of sample prior to filtration of the sample. The filtrate is collected in a baked, 125-mL amber glass bottle

with Teflon-lined lid. **DO NOT RINSE BOTTLE.** Do not fill bottle beyond shoulder. Chill sample and maintain at 4 deg C, ship immediately. Sample **MUST** be received at lab within 3 days of collection. Note: This method may not be suitable for treated/finished (chlorinated) water.

This method was implemented at the behest of the National Water-Quality Assessment Program (NAWQA),

*(continued on following page)*



**KANSAS LAB PROVIDES SUPPORT—**Laura Coffey loads on-line solid-phase extraction cartridges into the HPLC/MS in preparation for testing and calibration. View shows the instrumentation used to analyze water samples for the two herbicides and their primary degradate. The USGS Kansas Organic Geochemistry Research Laboratory developed the glyphosate method now being used at the NWQL, and assisted with support, training, and insight into applying the new method.

and the capacity the NWQL has developed for analyzing samples submitted for this schedule is based on sample estimates provided by NAWQA. Other USGS programs and projects that would like to use this method are requested to contact Gary Cottrell of the NWQL Business Development Team (cottrell@usgs.gov) and provide estimates of the number of samples planned and the approximate time period for submission, so as to ensure that the NWQL's capacity for this new analysis is not exceeded.

The implementation of a method across organizational units within the USGS is a new approach to providing cutting-edge analytical technologies to the widest range of USGS projects and programs. The NWQL thanks the Kansas Organic Geochemistry Research Laboratory for providing support, training, and insight into the practical application of this method. It is hoped that this new approach to providing the latest analytical techniques to USGS scientists will continue to be fruitful.

• ED FURLONG

## Green returns from executive-in-residence detail

Bob Green, chief, Methods Research and Development Program, returns this month from a 1-year detail to the Office of Personnel Management's Western Management Development Center (WMDC) in Aurora, Colo. As an executive-in-residence, Green was involved with developing and implementing an integrated project management curriculum.

This assignment was driven by a growing demand for project management skills in the Federal Government and increasing requirements for training to support professional certification. The curriculum, which was developed by a WMDC team, including Green, is being offered for the first time in fiscal year 2005 and includes three core courses, two specialty courses, and a preparation course for the certification exam.

Green took the lead in submitting the WMDC's application for Registered Education Provider status to the Project Management Institute, a certifying body for professional project managers. The Institute's

approval process involved a thorough evaluation of the curriculum, including content, alignment with the Institute's best practices, course delivery methods and faculty. Registered Education Provider status was awarded to the WMDC in June 2004.

Green also has been the program manager for several residential and on-site WMDC courses. In addition, he was involved with the Colorado Leadership Development Program, a year-long program of assessment and training for Colorado Federal and State agency employees.

Green commented that the executive-in-residence experience was particularly timely because of the NWQL's increasing emphasis on effective project management. He thanks the WMDC for allowing him to participate in many aspects of the Center's operation and for giving him the opportunity to achieve an ambitious agenda in a short time. Green also appreciates the support of the USGS Office of Water Quality and the NWQL, which allowed him to accept the detail.



**RECOGNIZING SERVICE**—Dennis Markovchick, physical science technician in the Liquid Chromatography/Sediment Unit, is pleased to receive his 20-year service award October 7 from Greg Mohrman, NWQL chief.

## Meeting and course calendar, 2005

- First Annual Conference on Environmental Sampling for Bio-Threat Agents, U.S. Department of Defense Earth Science Program, **January 27 and 28**, Baltimore, Maryland
- Major Accomplishments and Future Directions in Public Health Microbiology Workshop, USGS Office of Water Quality, **February 15–18**, Columbus, Ohio
- 8th Federal Interagency Sedimentation Conference and 3rd Federal Interagency Hydrologic Modeling Conference: Interdisciplinary Solutions for Watershed Sustainability, **April 2–6**, Reno, Nevada

## Reminder for sample LogIn, LabHelp

Questions and answers regarding how to ship samples to the National Water Quality Laboratory are found on the NWQL website at <http://www-nwql.cr.usgs.gov/USGS/faqLogin.html>. Contact labhelp@usgs.gov or phone 1-866-ASK-NWQL (1-866-275-6975) for questions or comments regarding NWQL services.

## Lab news briefs

**Mark Burkhardt**, research chemist, and **Ann Cleveland**, chemist, audited Eberline Laboratory October 3–6, in California. Eberline holds a contract for analyzing USGS radchem samples.

**Ed Furlong**, research chemist, spoke at the 4th International Conference on Pharmaceuticals and Endocrine Disrupting Chemicals in Water, October 12–15, in Minneapolis.

Chemist **Tom Leiker** took part in a joint planning meeting on funded proposals regarding cooperative work on Lake Mead studies, October 13–15, in Las Vegas, Nevada.

\* \* \*

**Furlong** spoke at the ASA/CSSA/SSSA International Annual Meetings with the Canadian Society of Soil Science, October 31–November 4, in Seattle.

**Mark Sandstrom**, research chemist, participated in the ACT watershed visits November 1–5, in Mississippi, Tennessee, and Iowa.

\* \* \*

**Furlong** and **Sandstrom** attended the Annual Meeting of the Geological Society of America, November 8–10, in Denver.

**Bill Foreman**, **Leiker**, **Sandstrom**, and **Serena Skaates** attended the 4th Society of Environmental Toxicology and Chemistry Annual Meeting, November 14–18, in Portland, Oregon.

\* \* \*

**Ed Furlong**, USGS research chemist, is quoted in an article found at MSNBC.com entitled, “Livestock antibiotics found in waterways,” at URL <http://msnbc.msn.com/id/6299642/>. A research team from Colorado State University discovered antibiotics and other chemicals near farms along Colorado’s Cache la Poudre River. Furlong, who worked on the 2002 study, said the findings help to narrow possible sources of contamination



**PAVELICH RETIRES**—Mary P. (Pat) Pavelich, chemist in the metals unit, retired December 10 after 17 years at the NWQL. Through the years, she operated almost every inorganic chemistry analysis. Pat was a major contributor to recent decisions regarding turbidometric measurements. Pat’s future plans include travel, starting with a cruise with her husband early in 2005.



**BRENTON HANGS UP SPURS**—Ron Brenton, chief of the liquid chromatography/sediment unit, retired December 31. Brenton graduated from the University of Northern Colorado with a bachelor of arts degree in chemistry in 1963. After a short stint in the Army, he went to work for the Great Western Sugar Company in its Research and Process Development Laboratory, where he became a project leader working with environmental monitoring and research. He left the sugar company after 17 years and eventually went to work for the NWQL in 1987. Brenton started with the sample preparation unit, then moved on to carbon, gas chromatography, and finally liquid chromatography.

and develop better water-management policies. “Little is known about what effect these concentrations have on humans or ecosystems,” Furlong said.

\* \* \*

A 2-day short course on Report Training, Writing, and Colleague Review is slated January 12 and 13 at the NWQL. Instructor is **Keith Lucey**, reports specialist for the Central Region Office in Denver. The course includes instruction in report planning and organization, writing in Survey style, grammar, USGS policy issues, and an introduction to colleague review. Target audience is new authors and authors who need a refresher.

\* \* \*

**Robert Eganhouse**, research chemist, Reston, presented a seminar November 9 at the NWQL entitled, “Comprehensive Two-Dimensional GC Time-of-Flight Mass Spectrometry: A New Tool for Analysis of Highly Complex Mixtures of Organic Compounds.”

\* \* \*

“Profiling Fuels for Environmental Forensics” is the title of a seminar presented October 25 at the NWQL by **Colleen Rostad**, research chemist in the Branch of Regional Research.

\* \* \*

## Metro Wastewater inspects the Lab

Barbara Orr, Metro Wastewater Reclamation District, visited the NWQL October 20 to verify compliance with the building 95 Wastewater Contribution Permit. The Safety Office reports no violations of the permit requirements.

Orr toured the facility, including the sewer vault, the waste storage area, and several laboratories. She reviewed documents dealing with waste streams, hazardous waste manifests, and contingency plans.

The Safety Office was pleased with the results and lauded all employees in the building for the successful inspection.

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## Ethics, integrity of data subject of short course

NWQL chemists, analysts, technicians, and supervisors took part in data-integrity training November 30 and December 1 at the Denver Federal Center. The training is required by the National Environmental Laboratory Accreditation Conference (NELAC).

Training focused on data-integrity concerns, including examples of unethical procedures, with emphasis on the need for honesty and full disclosure in data reporting.

The instructor was Joe Solsky, a chemist with the U.S. Army Corps of Engineers. Solsky provides oversight of the Corps-wide Hazardous, Toxic and Radioactive Waste Chemical Data Quality Management activities. He also provides technical support for the Corps' Lab Validation Program and conducts laboratory audits.

## USEPA approves alternate test for total phosphorus and total nitrogen

The U.S. Environmental Protection Agency (USEPA) has recognized the U.S. Geological Survey alkaline persulfate digestion methods for total nitrogen and total phosphorus as alternate test procedures for nationwide surface- and ground-water monitoring. The methods are total nitrogen and total phosphorus in filtered samples (I-2650-03), and total nitrogen and total phosphorus in whole-water samples (I-4650-03).

William A. Telliard, director of analytical methods for USEPA, in a letter dated November 29, 2004, said the methods "would be a suitable alternative" for the analysis of total nitrogen and total phosphorus in filtered and whole-water samples.

The alternate test procedures were not extended to the USEPA compliance monitoring programs "because use in such programs would require proposal and promulgation of the methods in the Code of Federal Regulations." An example of a compliance monitoring program would be the National Pollution Discharge Elimination System (NPDES). The letter was addressed to Charles J. Patton, NWQL research chemist and principal investigator.

The USEPA's approval is based on a study by Patton and Kryskalla (2003).\* The NWQL authors obtained data for about 2,100 surface- and ground-water samples that were analyzed for Kjeldahl nitrogen and Kjeldahl phosphorus in the course of routine operations at the Laboratory. These samples then were analyzed independently for total nitrogen and total phosphorus using an alkaline persulfate digestion method developed by the NWQL Methods Research and Development Program.

Data from the study, as outlined in Telliard's letter, indicated that in the case of total phosphorus, "there is not a statistically significant difference" between the alternate test procedure and the Kjeldahl digestion method. The letter further states that in the case of total nitrogen, where analytical results differed significantly between the alternate test procedure and the reference method, Patton and Kryskalla (2003) present "a persuasive argument that the difference is due to nitrate interference...[in the] reference method."

Advantages of the alkaline persulfate digestion method compared to the Kjeldahl digestion method include the following:

- Hazards to analysts and toxic wastes are substantially less (reduced mercury waste);
- Alternate procedure more amenable to automation and less labor intensive;
- Method detection limits are substantially less than for existing methods; and
- The alternate method is more sensitive, accurate, and environmentally responsible for routine, simultaneous determinations of total nitrogen and phosphorus in surface and ground water.

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\*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.

## Frequently asked questions

### ***New analytical method for polycyclic aromatic hydrocarbons in sediment by gas chromatography/mass spectrometry***

Polycyclic aromatic hydrocarbons (PAHs) are hydrophobic compounds associated with fossil fuels and their combustion, and other complex carbon sources. They are widely distributed in the environment, and many are considered to be toxic and carcinogenic. This suggests that long-term exposure poses a risk to terrestrial and aquatic life.

#### ***How did this method evolve?***

The method began through the proposal process as a custom method determining low concentrations of PAH compounds and their alkylated homolog groups in sediment by gas chromatography/mass spectrometry (GC/MS) using selected-ion monitoring. Because of improved instrumentation, it was updated to full-scan mode providing greater confidence at method detection limits (MDL) of 10 micrograms per kilogram ( $\mu\text{g}/\text{kg}$ ).

#### ***What does the new method offer?***

The method quantitates 28 PAHs and semiquantitates 25 alkylated PAH homolog groups for five parent PAHs. Alkylated PAHs are PAHs with attached alkyl constituents and are grouped by the number of carbon atoms present in their alkyl constituent. A particular homolog group includes all the isomers with the same number of carbon atoms in the constituents and the same parent PAH.

The method provides a base sample preparation, used to prepare samples for analysis by five different analytical schedules. The schedules include analysis for selected organochlorine pesticides, polychlorinated biphenyls, and organophosphate pesticides from the same sediment-sample extraction as the PAHs. A common extraction is efficient and reduces analytical costs.

#### ***What are the features of the new method?***

The method uses Soxhlet extraction, compound isolation by gel-permeation chromatography, and concentration through solvent reduction for sample preparation. The compounds are identified

and quantitated using capillary-column GC/MS in full-scan mode.

#### ***How are data reported?***

Compound concentrations for field samples are reported in micrograms per kilogram dry sediment to two significant figures. Surrogate data for each sample type are reported as percent recovered. Data for spike and quality-control reference material samples are reported as percent recovered. Compounds quantified in the set blank sample are reported in micrograms per kilogram.

For the individual PAHs and the alkylated PAH homolog groups, the MDL is set at 10  $\mu\text{g}/\text{kg}$ . The 25 alkylated PAH homolog groups are reported with an 'E' code because their calculated concentration integrates multiple individual compounds for which many have no available standards. Therefore, the accuracy of these groups cannot be assessed, and the uncertainty in the calculated concentration of each alkylated PAH homolog group requires qualification.

#### ***What is the new method number, and laboratory and parameter codes?***

The U.S. Geological Survey (USGS) method number is O-5505-03 and the NWQL schedule number is 2505 for the 28 PAHs and schedule number 2506 for the additional 25 alkylated PAH homolog groups. The other three schedules are: 5504 for organochlorine compounds, 1404 for organophosphate insecticides, and 2504 for selected PAHs plus total PCB.

The parameter and method codes, a list of the analytes, reporting limits, units, and sampling containers used for sediment samples are available on the NWQL USGS-visible web site at [http://www.nwql.cr.usgs.gov/USGS/USGS\\_gen.html](http://www.nwql.cr.usgs.gov/USGS/USGS_gen.html). Select LIMS Catalog (upper right corner).

Select a search category, such as schedule number, and enter the number to retrieve schedule information.

#### ***What procedures are required for shipping samples to the NWQL?***

Samples are collected into either 500- or 1,000-milliliter widemouth glass jars that have been precleaned by baking at 450°C for at least 2 hours. Lids must be lined with polytetrafluoroethylene (PTFE), a type of Teflon®. Sufficient water must be decanted to allow space in the jars for expansion from freezing.

Ship samples on ice by overnight carrier to the NWQL as soon as possible after collection.

#### ***May any District use the new methods?***

Yes. The Office of Water Quality approved the new water quality analytical method for the determination of PAHs in sediment on 25 November 2003 for all projects and programs.

#### ***How do I obtain a copy of the new methods?***

A copy of the report by Olson and others (2004) may be downloaded from the NWQL USGS-Visible web site (<http://www.nwql.cr.usgs.gov/USGS/pubs-only.html>), requested by E-mail to the NWQL Technical Editor ([jwraese@usgs.gov](mailto:jwraese@usgs.gov)) or [LabHelp@usgs.gov](mailto:LabHelp@usgs.gov), or calling 1-866-ASK-NWQL.

## Reference

Olson, M.C., Iverson, J.L., Furlong, E.T., and Schroeder, M.P., 2004, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of polycyclic aromatic hydrocarbon compounds in sediment by gas chromatography/mass spectrometry: U.S. Geological Survey Water-Resources Investigations Report 03-4318, 45 p.

- MARY OLSON, JANA IVERSON, EDWARD FURLONG, MICHAEL SCHROEDER, and ALLISON BRIGHAM

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## Students join IT section

Two new students recently joined the Information Technology section. Doug Smith, programmer, will be working on software development; and Darius Taylor will help out with technical support and documentation. Both are employed under the Student Career Experience Program (SCEP).

## Update on the Ocala transition—National Field Supply Service

Several improvements have been made to the National Field Supply Service (NFSS) catalog in 1-Stop Shopping. The catalog is now available for use by part number or by using alphabetical sequence. The item nomenclatures also are being standardized to better reflect the item description. Additional improvements are as follows:

- Several months ago, the commercial source for the capsule filter changed from Gelman to Whatman. The Whatman filter basically is the same as the Gelman filter, but it has a pressure relief valve, which should enhance field work. The paper label on the filter is being eliminated, and the information that includes lot number and flow direction will be printed on the end of the filter to make it easier to identify. Contract specifications are being developed to provide long-term vendor support for the filters.
- The Office of Water Quality and the NWQL have developed a list of commercial items that previously were available from Ocala but are no longer stocked by the NFSS. The list will include the item description, part number, and available source. A link to the list will be added soon to the 1-Stop catalog.
- The NWQL also is determining if off-the-shelf commercial items meet USGS performance standards rather than assembling or processing kits and spike mixtures, for example, in-house.
- To reduce the cost of field supplies to our customers, the NWQL now provides some items in large lots or in bulk quantities. A few examples include pH buffer, gloves, conductivity standards, and inorganic blank water. The NWQL also will try to purchase some items in large quantities to reduce quality-control costs.
- Finally, the NWQL is working with the Hydrologic Instrumentation Facility (HIF) to transmit field-supply orders using file transfer protocol (FTP) rather than Lotus Notes to improve the transfer of orders from the 1-Stop program. Testing of the new system will begin early this month. The NWQL will continue to work with the HIF to provide additional enhancements.

• WILL LANIER

## Workload estimates vital to NWQL prices, planning

Income/Workload Estimates from the Water Resources Discipline (WRD) Cost Centers were submitted to the NWQL at the end of calendar year 2004. These estimates from WRD are important to the Laboratory and to its customers because the estimated number of samples can affect prices.

With good information from the Cost Centers, the NWQL can properly staff analytical processes to minimize costs while maintaining quality and turn-around time. Conversely, incomplete or faulty information can cause delays in results and can drive up costs by causing the NWQL to work in an emergency mode for a part of the year.

The NWQL will be using initial estimates to pull reports after January 1, May 1, and August 26, 2005. This information is used to determine staffing levels and training needs for each analytical process. The NWQL understands that initial estimates may need to be changed and that is why it pulls an initial and an interim estimate (May 1, 2005).

For instructions regarding estimates and other processes, customers can open a help file at [http://nwql.cr.usgs.gov/usgs/budget/nwql\\_estimate\\_help.pdf](http://nwql.cr.usgs.gov/usgs/budget/nwql_estimate_help.pdf) or click on the word "Help" at the top of the Income/Workload Estimates webpage.

For other assistance, please contact as follows:

### Administrative/Data Entry problems

Merilee Bennett ([mbennett@usgs.gov](mailto:mbennett@usgs.gov))  
303-236-3531

Nancy Wydoski ([njwydosk@usgs.gov](mailto:njwydosk@usgs.gov))  
303-236-3545

### System problems

Jim Steverson ([jrstever@usgs.gov](mailto:jrstever@usgs.gov))  
303-236-3704

If customers would like information on how their estimates from last year compared to the actual logins, please contact Gary Cottrell ([cottrell@usgs.gov](mailto:cottrell@usgs.gov), 303-236-3490).

• GARY COTTRELL

# Letters, faxes, and e-mail

October 18

To: Greg Mohrman

Building 95 does an excellent job of quick evacuation and accounting for all visitors and employees once they are outside. This is a team effort that has been practiced many times, and it shows when an annual fire drill is conducted or when the alarm sounds and the building has to be evacuated. Thanks Greg [Mohrman], Helen [Wharry], and Carlos [Arozorena] for your constant efforts in keeping things running smoothly during building evacuations.

Dee Lucas  
Safety Specialist, USGS

Dear Labby,

**Question:** Why do I get results of “M” when I table my data in NWIS? I know the lab sent me a result.

**Answer:** This question arises frequently. If you choose default rounding within QWDATA, which is the default setting, the rounding array from the NWIS (National Water Information System) parameter-method table (PMT) is used. The selected rounding array matches the parameter-method code combination of your data. If the value reported by the lab is very small (generally less than the method-reporting level) and rounds to zero after applying the rounding array from the PMT, then the output for your result is reported as “M”—constituent identified in sample, but not quantified. This is an indication in the output that the value would have been displayed as zero, if a value had been displayed. If zero is an acceptable value for your result, the output will include a zero. Additional information about result rounding and censoring may be found at the following URLs:

<http://phoenix.cr.usgs.gov/www/roundzero.html>

<http://phoenix.cr.usgs.gov/www/ucv.html>

<http://phoenix.cr.usgs.gov/www/rounding.html>

- GLENDA BROWN and STEVE GLODT  
BUSINESS DEVELOPMENT TEAM

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*Water Logs*, the National Water Quality Laboratory Newsletter, is published quarterly by the National Water Quality Laboratory, U.S. Geological Survey, Box 25046, MS-407, Federal Center, Denver, CO 80225-0046. For copies, call Jon Raese 303-236-3464 or send e-mail request to [jwraese@usgs.gov](mailto:jwraese@usgs.gov).

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