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Inorganic Program backlog at all-time low

Merle Shockey, chief of the NWQL Inorganic Program, reports that the sample backlog for the program is at an all-time low. The backlog--samples received at the NWQL but not analyzed within a 2-week period--is at the lowest level in Shockey's 14 years at the Laboratory. Moreover, this low level has been maintained longer than at any previous time.

"Reducing the 2-week backlog has always been an important goal of the Inorganic Program," said Shockey. "In the past, we approached a zero backlog towards the end of winter when the sample load was the lowest, but as the work load increased during spring and summer the backlog would also increase." As shown in figure 1, the backlog was reduced during the winter months of fiscal year 1994 and has remained low throughout the spring and summer.

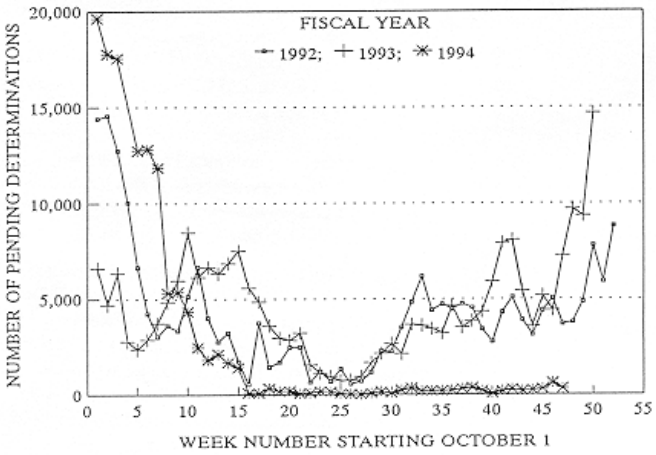

Figure 1.--Backlog history for Inorganic Chemistry Program.

Shockey says the improved performance is not the result of a decrease in samples. In fact, the number of determinations logged for fiscal year 1994 has increased about 10 percent compared to the same period in fiscal year 1993 (see fig. 2).

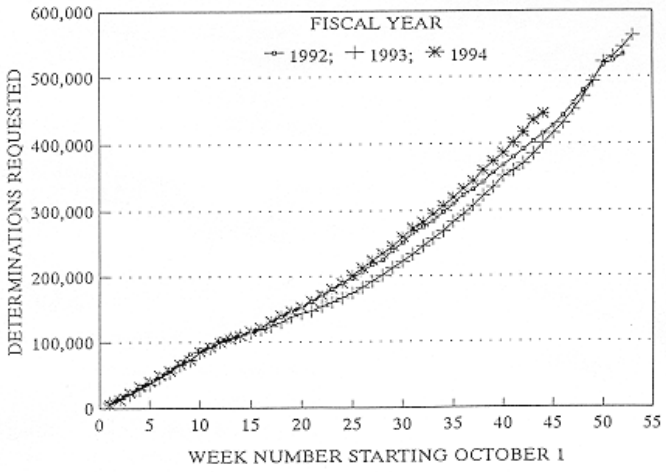

Figure 2. --Login history for Inorganic Chemistry Program.

The program chief offered the following reasons for the decrease in backlog during a period of increased work load:

- Reduced staff turnover,
- High-quality production staff,
- Increased cross-training resulting in more efficient use of analytical systems,
- Full-performance level raised from GS-7 to GS-9 for physical science technicians,
- Program reorganized from four to five units in August 1993, and
- Improved analytical capabilities with assistance of the Methods Research and Development Program.

by Jack DeGiacomo
and Tom Bushly

Family matters--Profile of a chemist

You arrive at Juan Vasquez' door and are greeted with glad welcome. This time you are here to conduct an interview for an article you hope to use in the *Newsletter*. And since this is a quiet activity, he closes the door so we will not be disturbed. Juan is a very considerate person.

You notice this thoughtfulness again as Juan begins to talk about his work as supervisor of the Nutrients Unit in the Inorganic Program. He patiently answers all questions, erudite or not so, spontaneously spelling technical terms, and bringing you gently to a better understanding of analytical chemistry and the processes we use at the Lab.

But the purpose of the article is not so much to describe Juan professionally as humanly, for you heard he had volunteered to conduct tours of the Aztec Exhibit at the Denver Museum of Natural History a year or so ago. As he discusses how he became involved with it and why, you swiftly become acquainted with a man who endeavors to share his educational experiences and Hispanic heritage with others. He regularly volunteers for Community Resources, Inc., giving chemistry and cultural presentations (he holds degrees in Chemistry and Spanish) throughout the Denver public school system. Then he tells you about his annual treks to Mexico, and fires your imagination with color.

Vasquez

Juan actively participates in a six-week Foreign Studies course taught by Francisco Padilla through Red Rocks Community College. Course work in Spanish (Padilla's forte), history and anthropology (Juan's contribution) is followed by a 20-day excursion to Mexico. Juan earned his credentials as a tour guide while an undergraduate student at the University of Colorado at Denver and has made over 15 tours to Mexico. He describes a typical itinerary through southern Mexico: Mexico City, Chiapas, Oaxaca, Veracruz, Puebla, Cuernavaca, Taxco; perhaps a long weekend at one of the beaches--Puerto Vallarta, Acapulco, or Cancun; day-tours of archaeological ruins or villages in which the whole community participates in an indigenous industry (weaving, for example); and, for recreation, horseback riding, rafting, boating, scuba-diving, parasailing. All the while he talks, he's spelling all the foreign terms because Juan is an educator. And unwittingly, he's inspiring you to save up for the next tour because Juan is an enthusiast.

Strongly committed to self-development physically as well as mentally and spiritually, Juan's hobbies include handball and bicycling. Throughout the interview, Juan acknowledges pride and respect for his family. He is the youngest of 12

children; his parents immigrated to the United States to escape the violence of the Mexican Revolution. Juan is married to Marta; their children are 21-year-old twins Faustina Abigail and Juan Gabriel.

Our interview becomes a conversation which encompasses the North American Free Trade Agreement (NAFTA) and its effect on the economy of Mexico. We also discuss the importance of dedication to our histories, to future generations, and to the enrichment of life around us. But the time grows short too soon and it's back to work--with a renewed hope and enthusiasm for the day.

by Karlin Allen

Tracking the elusive sample

Tom Bushly, computer engineer in the Laboratory Operations Program, has developed a software program that enables the NWQL to track samples when they are moved from the 30-day to 180-day storage area.

Upon removal from the 30-day area, bottles are scanned into the tracking system to verify availability. A comparison is made between the initial sample log-in and the relog from 30-day to 180-day storage. Any bottles not found are listed on a missing- bottle report and provided to Inorganic Program supervisors each Monday.

Bushly called this new software program an "improvement that will help the NWQL to track samples and reduce the number of misplaced bottles."

Sodium method automated

The Majors Unit in the Inorganic Program has added a new "analyst"--an Alpkem 301 autosampler. It is used with an atomic absorption spectrophotometer and a computer to analyze water samples for sodium concentration.

A human analyst no longer needs to manually aspirate the sample into the instrument and press a button to obtain results. This process saves time: the analyst attends to other tasks while the instrument analyzes samples. The majority of instruments in the Inorganic Program already are automated. Program managers plan to upgrade the few remaining systems that can be automated.

Still more insights into sample handling and analysis at the NWQL

What happens to the bottle after you put it in the mail?
Editor's note: This column contains questions that originally were part of the Water Resources Division (WRD) training class "Water-Quality Principles." Through this question and answer format, Paul Capel (Minnesota District) and Ed Furlong (Methods Research and Development Program) answer some of the more common questions that WRD personnel ask the NWQL. If you have a question about the Laboratory, please send it to EFURLONG on EDOC. Selected questions and answers will be printed in future columns.

Q. My section chief told me to make sure you were doing a good job. How do I KNOW that you are doing a good job? Can I just take your word, or do I have to do something?
First, talk to NWQL personnel, request quality-control data, and find out whether a particular schedule is within control or not. Look at your data. Are the results "reasonable"? If not, at least in the case of inorganic samples, you can request reanalysis, but first discuss it with the Laboratory. For guidance, the NWQL has published a quality assurance/quality control (QA/QC) manual (Pritt and Raese, 1992) that indicates all quality-control steps the NWQL takes to ensure data quality. For organics in water, the NWQL has developed a field blank and spike program to provide onsite QA/QC. Onsite QA/QC allows both the client and the Laboratory objectively to assess whether your equipment or the samples you submit pose particular analytical problems.
The NWQL also has developed a course on sampling and analysis for trace organics (GO242). Instructors teach proper sampling techniques for organics in surface and ground water, discuss and demonstrate the uses of onsite and Laboratory QA/QC, and guide students through interpretation of QA/QC results. We envision developing a parallel course for inorganic constituents. Note that sample set blank and reagent spike recoveries are being transmitted for each sample analyzed by Schedule 2500 (subschedules 2501 and 2502; Organochlorine Pesticides and Semivolatile Compounds in Bottom Material) and Schedule 2101 (Organochlorine Pesticides in Tissue).

Q. I overheard some EPA people talking the other day about their low analytical detection limits. Being from the USGS, I felt bad. We don't even have detection limits! Why not? Is our reporting limit as good as EPA's detection limit?

I am not sure what is meant by an analytical detection limit. The terms used by the U.S. Environmental Protection Agency (USEPA) and NWQL are instrument and method detection limits. An instrument detection limit is the mass equivalent to a signal, due to the analytes, which is equal to three times the standard deviation of a series of 10 replicate measurements of a reagent blank signal (Currie, 1988). Note that this ratio is for a pure analyte and refers only to the sensitivity of an instrument. This is the best possible detection limit and the one that instrument manufacturers usually tout.
The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (U.S. Environmental Protection Agency, 1992). The 99 percent confidence is defined as three times the standard deviation calculated from seven replicate analyses of a matrix spiked at a concentration of 2.5 to 5 times the instrument signal-to-noise ratio, or 1 to 5 times the estimated detection limit. Note that in this MDL definition, the analyte has been through all steps (extraction, isolation, analysis) of the *method*. In addition, the MDL is matrix specific, so the MDL for DDT in wastewater will be higher than the MDL for DDT in ASTM Type I reagent-grade water (American Society for Testing and Materials, 1994). Given these matrix effects and the unavoidable analyte losses throughout the analytical procedure, the MDL can be 2 to 100X higher than the instrument detection limit.
The reporting level at the NWQL is the concentration that the Laboratory feels it can reliably report, regardless of varying sample matrixes in a given medium. Note that many NWQL reporting levels are lower than their corresponding USEPA MDLs for regulated analytes. The choice of a reporting level reflects a lot of variables, not all of them quantifiable. The NWQL is in the process of converting some schedule-reporting levels to MDLs. Also, for some new methods, the NWQL will report all values determined, whether or not they are less than the MDL (but still greater than an instrument detection limit). The NWQL also will provide guidance on how to use these uncensored data.

Q. How come some chemicals are listed more than once (at different prices, of course) in the Services Catalog (Timme, 1994)? From budget considerations, I'll always choose the cheapest analyses. What do you think?
Not always a good move. Typically, when one analyte is listed twice for one matrix, say dissolved potassium, the higher priced listing is for the analysis with a lower reporting level. This lower reporting level may be important if you are trying to calculate loads for a river and its tributaries, and the expected potassium concentrations are low or unknown. Basically, unless you have a lot of historical data, you cannot dismiss using the higher priced analysis with its lower reporting level. If you are not sure what to do, call the NWQL and we will provide you with a sense of what the analyst typically sees. You might get a better understanding of which analysis to use. Remember the study objectives when you are trying to determine your analytical needs.

Q. I don't really understand your "schedules." If there are 10 chemicals on a schedule and all I really want is the concentration for one chemical, why can't I just pay one-tenth the cost of the schedule?
Multiple-constituent schedules reflect the fact that analytes in the schedule are extracted, isolated, and determined identically. These constituents likely have similar environmental behaviors. It is also likely that if one analyte is present, others will be, too. Given these constraints and the high degree of computerized data acquisition and processing, it would cost 90 to 95 percent of the multiconstituent-schedule price to determine a single analyte in a multiple-constituent schedule.
Moreover, if you were to request and receive data on a single constituent, you might miss important information. For example, related herbicides often are applied in combination or in a year-to-year rotation to enhance effectiveness and avoid developing resistance. If you were to request an analysis of atrazine and not determine cyanazine (when cyanazine was used significantly), you would miss important environmental chemical information that is available when a multiple-constituent schedule is requested.

Call the NWQL's Method Research and Development Program (Mark Sandstrom, Program Chief, 303/467-8086) for answers to questions regarding schedules and compound determinations.

References -

American Society for Testing and Materials, 1994, Annual book of ASTM standards, Section 11, Water: Philadelphia, v. 11.01, p. 45-47.
Currie, L.A., 1988, Detection: Overview of historical, societal, and technical issues, *in* Currie, L.A., ed., Detection in analytical chemistry--Importance, theory, and practice: American Chemical Society Symposium Series 361, 191st meeting, New York, April 13-18, 1986, p. 1-62.
Pritt, J.W., and Raese, J.W., 1992, Quality assurance/quality control manual: U.S. Geological Survey Open File Report 92-495, 33 p.
Timme, P.J., 1994, National Water Quality Laboratory 1994 services catalog: U.S. Geological Survey Open-File Report 94-304, 104 p.
U.S. Environmental Protection Agency, 1992, Protection of environment (Appendix B to Part 136--Definition and procedure for determination of the method detection limit--Revision 1.11): U.S. Code of Federal Regulations, Title 40, parts 100-149, revised as of July 1, 1992, p. 565-567.

Bench-level review part of Nutrient program

Analysts in the Nutrient Unit at NWQL review data and determine whether or not to reanalyze specific samples at the bench level. This procedure helps to provide NWQL's customers with the best possible data quality as well as fast turnaround time.

An in-section data-quality program reviews and compares all data produced by the eight regular-level nitrogen and phosphorous analytical systems. This program, an integral part of the unit's data-review process for the past 4 years, enables analysts to use a personal computer to review their data and reanalyze questionable results within 24 hours of initial analysis. Up to three determinations can be performed before releasing the data for final review; and no data are released to the Laboratory Information Management System (LIMS) computer and the Quality Management Program (QMP) until they are processed through this program.

The bench-level quality-control program evaluates data produced by all eight analyses and requires a reanalysis for any compound failing any of several quality checks. The program also performs a computer-error analysis on designated Standard Reference Water Samples (SRWS). If a comparison or error analysis fails, the program identifies the analyses involved. The analyst then reanalyzes either part of or the whole batch of samples.

Using the bench-level data review has increased the data quality and productivity for these analyses. Analysts quickly spot and correct analytical or bottle problems that the program identifies. Same-day processing of analytical data yields turnaround times of 24 hours on most reanalyses instead of the minimum two days needed when all data were reviewed by the LIMS. With holding times of 8 days and disposal set at 30 days after log-in, the time saved in initial processing gives customers more time to review data before samples are discarded.

by Mary Cast and Juan Vasquez

End of FY94 water year

Once again the NWQL closed out the water year on the second Friday of September, as in the previous 3 years. Samples received after September 9, 1994, are charged to fiscal year (FY) 1995. This year, the Log-in Unit received 2,006 samples on the last day for a total of 3,762 during the last week of the FY94 water year. Close-out totals for FY93 were 1,670 samples received on the last day, 3,734 samples total for the last week.

New NWQL reports published

Brock, R.D., and Murtagh, L.K., 1994, Determination of chlorinated insecticides in bottom sediment using an electron-capture gas chromatography screening method, Austin, Texas, 1991 and 1992: U.S. Geological Survey Open-File Report 94-302, 18 p.

Fishman, M.J., Raese, J.W., Gerlitz, C.N., and Husband, R.A., 1994, U.S. Geological Survey approved inorganic and organic methods for the analysis of water and fluvial sediment, 1954-94: U.S. Geological Survey Open-File Report 94-351, 60 p.

Newsletter Staff

Jon Raese, Editor

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