

CASE STUDY

CompuChem Pushing the Limits of ICP-MS Sample Productivity

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Traditionally the major appeal of ICP-MS is that its detection limits on average, are 1000-fold lower than ICP-OES and about 10-100x lower than graphite furnace AA. As a result, the majority of sample matrices analyzed involve the determination of very low analyte levels. A "typical" ICP-MS user would likely be satisfied with a 10-minute-per-sample analysis time for the determination of 22 elements in triplicate. In fact many analysts would probably be OK with much longer analysis times, especially if they had previously used two or three techniques to determine all twenty-two elements. However, if you ask an environmental laboratory that is carrying out CLP (Contract Laboratory Program)-type analysis under the Superfund Analytical Services, you might get an answer such as... "never fast enough."

The major reason for this kind of productivity demand is that the EPA needs a vast amount of data in support of the investigation and clean-up of contaminated hazardous waste sites under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA). The methodology that describes the determination of inorganic contaminants in these kinds of samples is outlined in the ILM05.3 (2004) and the updated ILM05.4 (2007) Statement of Work (SOW), which defines the analytical methods accepted by the CLP for the quantitation of 24 inorganic analytes (including mercury and cyanide) in water, soil and sediment samples using ICP-OES, ICP-MS, Cold Vapor (CV) AA, and colorimetric techniques.

The result is that the Superfund CLP program can generate extremely large numbers of samples, because the data is critical for determining the extent of contamination at hazardous waste sites, assessing the response based on risks to human health and/or the environment, deciding appropriate cleanup actions and making determinations as to when remedial actions are complete. In addition, the data may also be used in litigation against responsible parties in the enforcement of Superfund legislation, which means the contract lab that carries out these analyses, may be required to testify in court as to the integrity of their results. The bottom line is that the amount of data generated under this contract is exhaustive and must be of the highest quality, because it is used to make major decisions regarding public health and environmental safety issues.

One of the most experienced EPA contract labs is CompuChem, a Division of Liberty Analytical Corporation, based in Cary, North Carolina, which has been carrying out continuous and uninterrupted contract work with the EPA, since the CLP was initiated in 1981. It is well-recognized by EPA personnel that CompuChem has provided more support to the growth and maturation of the program than any other laboratory affiliated with the CLP. In the words of Laboratory Manager, Ken Grzybowski, "I would confidently say that in the 27 years CompuChem has been involved with the program, we have probably analyzed more CLP-type samples than any other lab in the U.S."

CORPORATE PHILOSOPHY

To meet the demands of industry and maintain a strong relationship with the U.S. EPA, CompuChem has always been firmly committed to a stringent program of quality assurance, which is described in detail in our Quality Manual. Each director, manager, supervisor, and staff member must comply with the quality program requirements set forth in our Quality Manual, and in its sister document, the complete Standard Operating Procedure (SOP) manual. CompuChem teams with our clients and strives to help solve problems that can be answered with valid data. CompuChem understands the significance of long-term business relationships and our highly trained and dedicated staff works with clients with the goal of fostering true partnerships.

MISSION STATEMENT

CompuChem's mission is to work towards a cleaner environment, providing quality analytical services economically and efficiently. Our highly trained and dedicated staff members work as partners to our clients to support environmental solutions.

So when CompuChem makes a decision to purchase a piece of equipment to analyze samples using SOW methodology, you know they have done their homework, to ensure they are investing in an instrument to keep up with the extreme demands of the application. This was exactly the scenario they were confronted with back in September 2008. CompuChem's existing 10-year-old ICP-MS had a catastrophic failure and was unable to run any samples until it was fixed or replaced. After a cost benefit analysis, it was determined that repairing the instrument was not an option. They contacted all the major vendors and based on a quick evaluation, talking with other users and estimated delivery times, they chose an ELAN® DRC-e. Ken went on to explain, *"All the vendors were telling us it would take 8-10 weeks to get an instrument, which was unacceptable and would have cost us numerous contracts and revenue. PerkinElmer, after initially quoting us 8 weeks, was able to readjust their time frame and have the instrument delivered to us in 3 weeks."*

Fortunately, PerkinElmer was able to supply a new ELAN® DRC-e to the lab, and install it in less than 4 weeks. Ron Buchanan, senior ICP-MS service engineer, installed the DRC-e instrument and provided CompuChem with basic training all in a few days. As Grzybowski said, *"Even though we were not familiar with the software, after the instrument was installed, it only took us one week for training and method development before we were running samples again. We were very impressed that we could get back on-line so quickly."*

Since then CompuChem has coupled an SC-FAST autosampler (Elemental Scientific Inc., Omaha, Nebraska) to its ELAN® and by optimizing sample delivery, measurement protocol and rinse out times (see SC-FAST sidebar for details), the lab is now analyzing a CLP sample for 30 elements in TRIPLICATE in 1 min 30 sec. As Grzybowski explained, *"We had no idea that we could have increased our sample throughput by this much. The same analysis on our previous instrument took us 9 min 53 sec, which translates into more than a 5-fold improvement in sample throughput. There is no doubt in my mind that the ELAN® DRC-e will be critical to our mission"* (see Corporate Philosophy/Mission Statement sidebar).

This kind of productivity combined with the recognized interference reduction capabilities of the ELAN®'s DRC technology for the more difficult environmental analytes like selenium and arsenic, will help the lab achieve its goal. CompuChem analysts are just beginning to realize that not only do they have an instrument that will keep up with the current demands of the EPA Contract Laboratory Program and other environmental test methods, but they also have an instrument that is going to be well-placed for future EPA trace element regulations as they inevitably fall to lower levels. We are pleased to welcome CompuChem into the ELAN® user community. They can be reassured that PerkinElmer will be there to help them achieve their corporate philosophy and mission.

SC-FAST

If you are not familiar with the SC-FAST, it is a rapid sampling approach integrated into an autosampler, which significantly reduces the pre and post measurement times involved with delivering a new sample to and removing the previous sample from the ICP mass spectrometer. Some of the areas of optimization include:

Autosampler response: This is the time it takes for the instrument to send a signal to the autosampler to move the sample probe to the next sample. By moving the autosampler probe over to the next sample, while the previous sample is being analyzed, a significant amount of time will be saved over the entire automated run.

Sample uptake: This is the time taken for a sample to be drawn into the autosampler probe and pass through the capillary and pump tubing into the nebulizer. By using a small vacuum pump to rapidly fill the sample loop, which is positioned in close proximity of the sample loop to the nebulizer, sample uptake time is minimized.

Signal stabilization: This is the time required to allow the plasma to stabilize after air has entered the line from the autosampler probe dipping in and out of the sample tubes (this can also be exaggerated if the pump speed is increased to help in sample delivery). However, if the pump delivering the sample to the plasma remains at a constant flow rate, and the injection valve ensures no air is introduced into the sample line, very little stabilization time is required.

Rinse-out: This is the time required to remove the previous sample from the sample tubing and sample introduction system. So, if the probe is being rinsed during the sample analysis, minimal rinse time is needed.

Overhead time: This is the time spent by the ICP performing calculations and printing results, so if this time is used to ensure the previous sample has reached baseline, minimal rinse time is required for the next sample.

By optimizing these steps, a significant improvement can be made in sample productivity, which for environmental contract laboratories like CompuChem, is extremely attractive.

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