

APPLICATION SPOTLIGHT

CHARACTERISTICS OF A SYRINGE-DRIVEN SAMPLE INTRODUCTION SYSTEM FOR ICP SPECTROMETRY

Introduction

The Assist™ automated sample introduction system has greatly enhanced the performance of ICP-OES and ICP-MS instruments. The Assist combines Flow Injection technology with purpose-built programmable syringe drives to reduce the typical analysis time by more than 50%. This combination provides the highest level of accuracy, stability and sample throughput by controlling the delivery of both sample and internal standard or diluent. It eliminates the inaccuracies and pulsations caused by peristaltic pumps, as shown in Figure 1. In this report we will present the benefits of the Assist and introduce three different configurations: the Basic Package, Premium Package, and Oils Package; to show how the Assist can tackle most ICP-OES and ICP-MS applications.

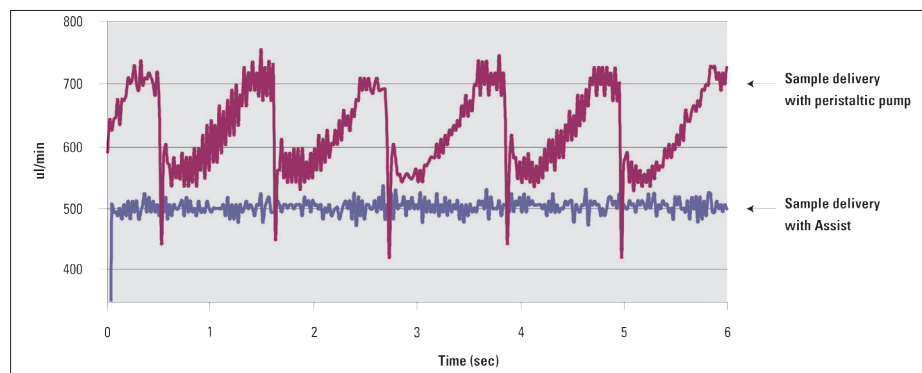


Figure 1: Chart comparing syringe delivery versus peristaltic pump delivery.

Assist Basic System

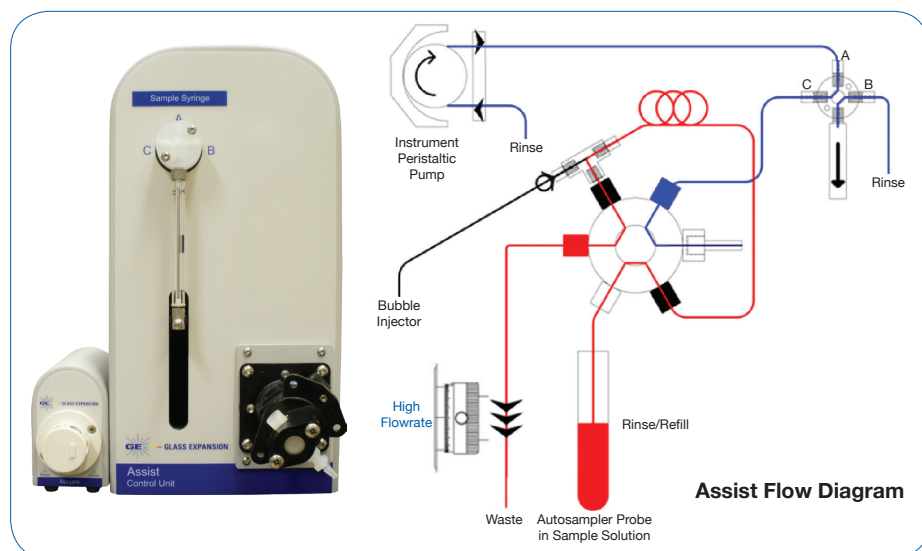


Figure 2: Assist Basic Package (single syringe) and flow diagram.

GE NEWS

PITTCON
CONFERENCE & EXPO 2012

Pittcon 2012

A wide selection of Glass Expansion products will be on display at Pittcon 2012, Orlando, Florida, USA, March 12 – 15, 2012. The display will include nebulizers, spray chambers, torches, RF coils, ICP-MS cones and accessories. You will also be able to see a demonstration of the new Assist syringe-driven ICP sample introduction system. Glass Expansion specialists will be on hand to answer your questions and assist you to choose the optimum components for your ICP. Please visit us at Booth 2859.

We will also be presenting the following posters:

220-29P. Improved Plasma Robustness with a Ceramic Torch

220-30P. Advances in Enhanced Productivity Sample Introduction Accessories for ICP Spectrometry

Session 220 – New Developments in Analytical Instrumentation and Software

Sunday, March 11, 2012, Afternoon
Valencia Room, W415

IN THIS ISSUE:

- **Application Spotlight** 1 – 5
- **GE News** 1
- **New Products** 5
- **Instrument News** 6
 - From Agilent
 - From Bruker
 - From Spectro

The Assist Basic System (Figure 2) consists of a single syringe drive and a Niagara switching valve. This system is used when inline delivery of internal standard or diluent is not required. Currently, ICP-OES and ICP-MS instruments use a read/uptake delay, stabilization delay (incorporated in the read delay), read/analysis time, and rinse delay. With the Assist, only a read/uptake delay and read/analysis time are required. The rinse cycle of the nebulizer occurs during the read/uptake delay and the rinse cycle of the autosampler probe and uptake tubing is performed during the read/analysis time, thus eliminating the rinse delay. The constant flow of solution to the plasma and uniform internal diameter throughout the entire system allow the stabilization time to be reduced significantly and it is incorporated into the read/uptake delay to simplify the setup process. With the Assist, the stabilization time can be reduced further due to the steady solution flow rate from the syringe drives.

In Figure 3, the precisions for a number of different masses measured on an Agilent 7500 ICP-MS were averaged together. The precisions achieved with natural aspiration and with a peristaltic pump are compared to the Assist. The data in Figure 3a. shows that the precision syringe drive of the Assist is even more precise than natural aspiration and a factor of two better than the peristaltic pump. The graph in Figure 3b. shows that one can achieve the smallest variation between two replicate measurements with the precision syringe drive of the Assist in comparison to the other two methods.

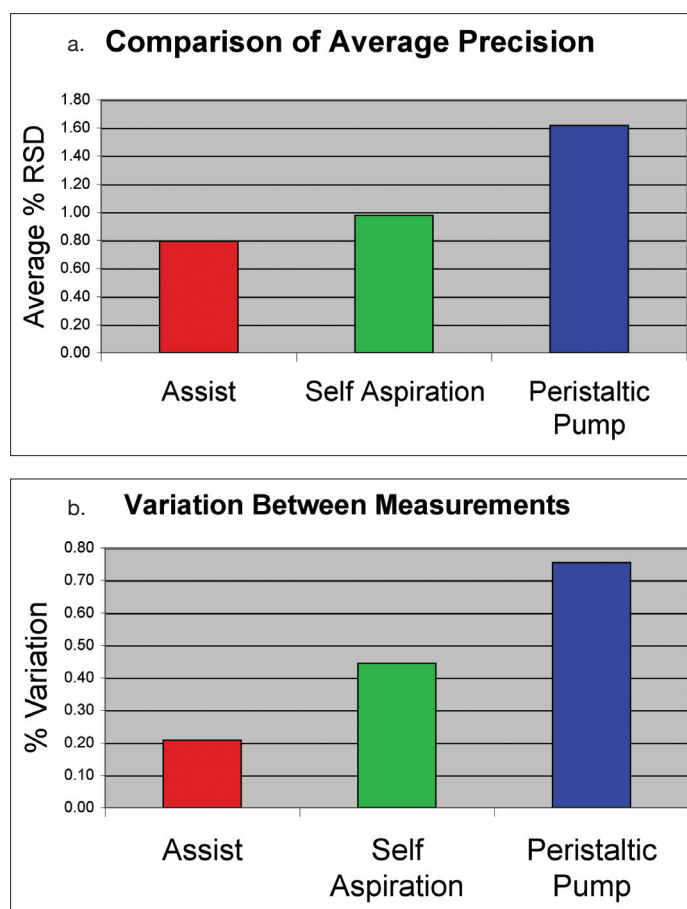


Figure 3: Comparison of average precision (%RSD) and measurement variation (%).

The Assist Basic System has recently been installed at American Assay Laboratory in Reno, NV on a Varian Vista Pro Axial ICP-OES fitted with an SPS-3 autosampler. The main purpose for installing the Assist was to eliminate lengthy rinse times from peristaltic pump tubing and improve sample throughput.

The method parameters with and without the Assist are listed in Table 1. The Assist has eliminated more than 70% of the time required to complete one measurement cycle. The time saved per sample has led to a factor of 3 improvement in sample throughput. The precise delivery of sample via the syringe drive allowed the replicate read time to be reduced to 8sec and still achieve a factor of 2 improvement in precision (%RSD). With the rapid uptake of the sample by the positive displacement pump, the fast pump option available on the Varian ICP can be turned off. This translates to a saving in sample consumption in addition to an improvement in the lifetime of consumables.

	Without Assist	With Assist
Probe to sample (sec)	4	4
Replicate read time (sec)	15	8
Replicates	2	2
Sample uptake delay (sec)	40	7
Stabilization time (sec)	10	5
Pump rate (rpm)	40	25
Rinse time (sec)	30 (up to 2 min)	0
Fast Pump	Yes	No
Total Time (sec)	114	32

Table 1: Method parameters with and without the Assist, provided courtesy of American Assay.

The Assist has two unique features, Time in Sample (TIS) and a Bubble Injector. The TIS feature allows the analyst to program the time the autosampler probe sits in the sample tube instead of waiting until the loop is filled. The Bubble Injector introduces a series of air bubbles at the end of the sample segment to reduce boundary diffusion and carryover. Yet no air is allowed to reach the plasma, maintaining maximum stability. The TIS and Bubble Injector permit a smaller volume of sample to be used and prevent any sample dilution/mixing with the carrier solution. These two features, combined with the inert sample loop and tubing, provided American Assay the capability of analyzing a 200ppm Au sample and achieving 5ppb Au in the next blank with no added rinse. One of the most noted performance improvements for American Assay is the elimination of lengthy rinse times, which could previously take up to 2 minutes to achieve blank level.

In summary, American Assay has achieved the following benefits since installing the Assist:

- Sample to sample time has been reduced from 114 seconds to 32 seconds, providing a three-fold increase in sample throughput.
- The signal precision has been improved by a factor of 2.
- Correlation coefficients of at least five nines have been obtained for the wavelengths examined, demonstrating excellent correlation.
- Carryover is greatly reduced, providing the capability of analyzing a 200ppm Au sample and achieving 5ppb Au in the next blank with no added rinse.
- Previously a lengthy rinse time of up to 2 minutes was sometimes required to achieve blank level; with use of the Assist the rinse time is eliminated.

Assist Premium System

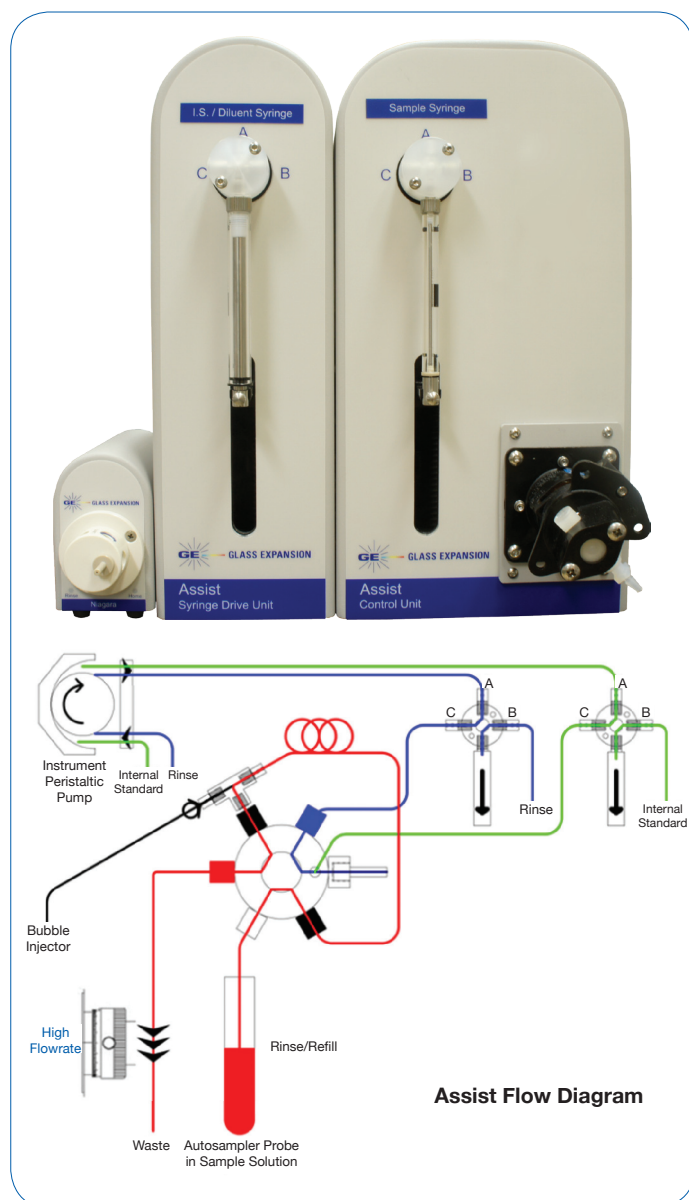


Figure 4: Assist Premium Package (two syringe drives) and flow diagram.

The Assist Premium System is the most commonly requested system. It incorporates two syringe drives and a Niagara switching valve (Figure 4). It provides all of the performance enhancements of the Basic Package described above with the added benefit of inline delivery and mixing of the sample and internal standard or diluent. The data obtained with the Assist Premium Package was obtained on the Agilent 7500 ICP-MS.

With the Assist Premium Package the second syringe drive is used to automatically deliver a diluent. The stabilization time required for a 20:1 dilution was less than 10 seconds (Figure 5) at a combined nebulizer solution flow rate of 0.4mL/min. The ability to complete accurate inline dilution with only a few seconds of added stabilization time can eliminate an entire day of sample preparation work. Figure 6 shows that the precision with inline dilution using the Assist is much better than that with the peristaltic pump.

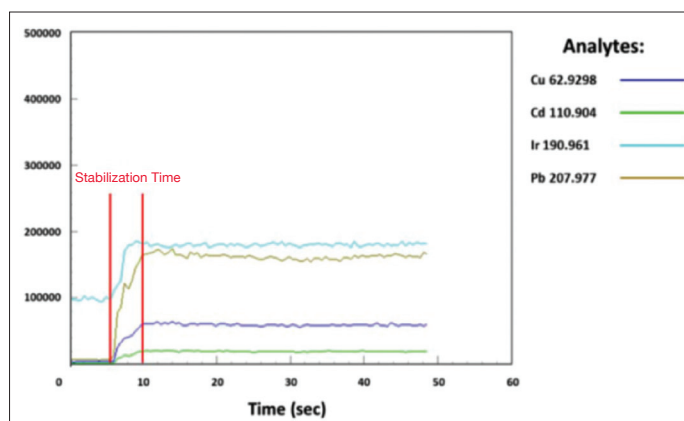


Figure 5: Stabilization time achieved with a 20:1 inline dilution.

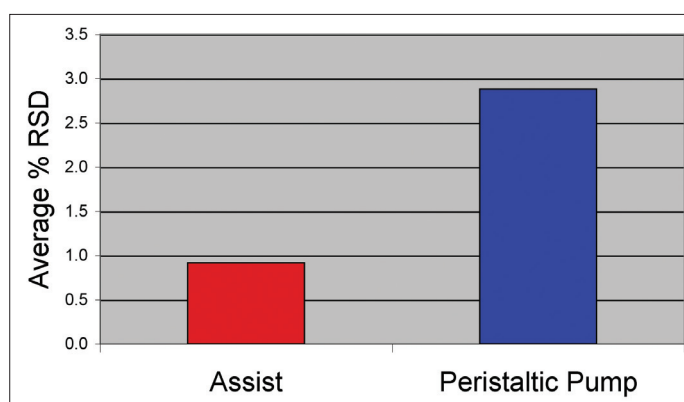


Figure 6: RSD for inline dilution using the Assist and peristaltic pump. Average of 21 elements, dilution factor 10:1.

The second syringe drive also allows you to automatically add internal standard. When an internal standard is added to the sample in the ratio of 1:10, the error obtained with the Assist (Figure 7) is much smaller than that obtained with a peristaltic pump. This demonstrates that the ratio of internal standard to sample is accurately maintained with the Assist.

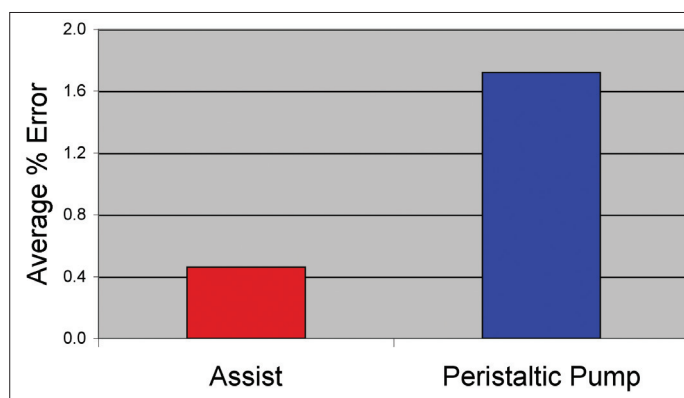


Figure 7: Concentration error with inline addition of internal standard. Average of 21 elements, ratio of internal standard to sample 1:10.

Assist Oils System

The Assist Oils Package (Figure 8) is configured specifically for the analysis of wear metals in raw (undiluted) lubricating oils. It incorporates three syringe drives, a Niagara switching valve, and a stainless steel diluter probe for the autosampler. It takes a sample of the oil, mixes it with a diluent such as kerosene and precisely delivers the diluted oil to the ICP spectrometer. It eliminates the need for manual dilutions and drastically increases the speed of analysis. A complete cycle time with the Assist oils package can be completed in as little as 28 seconds per sample, depending on the ICP method.

The measurement of metals in used engine oils provides valuable information about the engine and the state of the lubricant, information which can be used to increase the efficiency of the equipment in which the engine resides and lower the cost of maintenance. For example, copper is an indication of bearing wear while lead may come from bushings and chromium from piston rings and seals. Other elements such as calcium, magnesium, and zinc are added to the oil to produce a “soap” to protect engine components. So it is important to ascertain that these metals remain at an optimum concentration. Also, dirt may contaminate the oil which would be evidenced by the presence of elements like silicon and sodium.

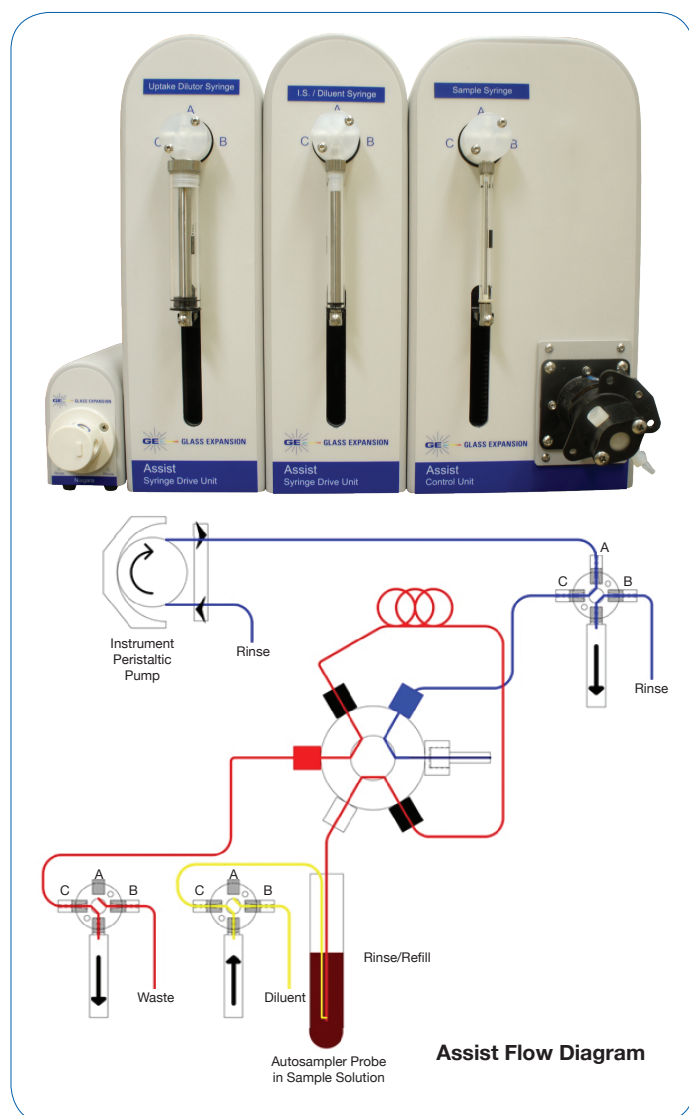


Figure 8: Assist Oils Package (3 syringe units) and flow diagram.

The data listed in Table 2 consists of wear metals, contaminants, and additives and was obtained with the Assist connected to a Varian Vista ICP-OES. The mean % recoveries are between 99 and 107%, showing the Assist is capable of providing accurate online dilution. Any carryover is reduced due to the totally inert sample path, as indicated by the mean wash concentration.

Element and λ	Mean % Recovery	Mean Wash Concentration (ppm)
Ag 328	103	(-) 0.21
Al 308	99	(-) 0.17
B 249	99	(-) 0.21
Ba 233	101	(-) 0.01
Ca 317	107	0.84
Cd 228	101	(-) 0.06
Cr 357	101	0.12
Cu 327	101	(-) 0.17
Fe 259	101	(-) 0.03
K 766	102	0.00
Mg 279	100	(-) 0.14
Mn 257	101	(-) 0.16
Mo 202	101	(-) 0.09
Na 588	100	(-) 0.09
Ni 231	101	(-) 0.12
P 214	106	0.03
Pb 220	101	(-) 0.02
Si 212	101	0.01
Sn 283	101	(-) 0.03
Ti 334	101	(-) 0.18
V 310	101	(-) 0.19
Zn 213	107	(-) 0.47

Table 2: Recovery and carryover with Assist Oils Package.

To investigate the accuracy of the Assist, a set of five 100 mg/L standards was prepared using the Assist and another set was prepared by manual dilution. With the Assist, the stock standards were loaded into the autosampler tray and a 10x dilution was completed inline while the other set of standards was diluted offline followed by sample delivery using the peristaltic pump. The absolute accuracy achieved with the Assist is better than manual dilution followed by delivery using a peristaltic pump and it was completed in half the amount of time (Figure 9).

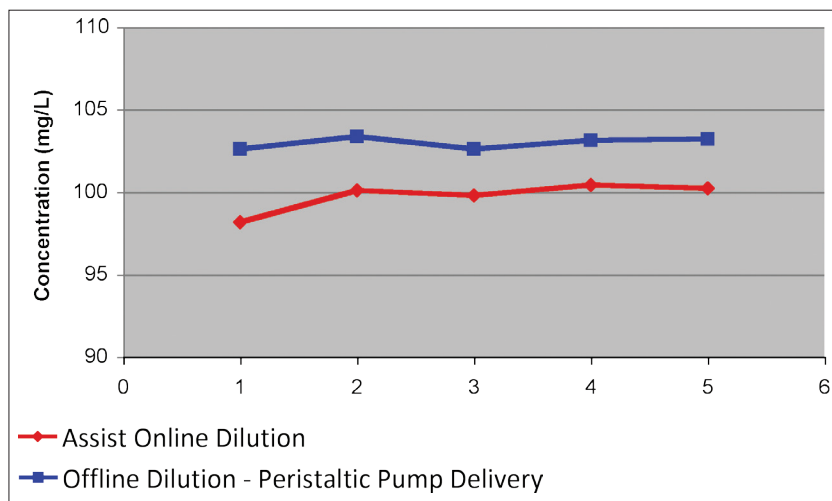


Figure 9: Accuracy of 100 mg/L standards prepared by inline dilution using the Assist compared to manual dilution followed by delivery using a peristaltic pump. Each point is the average for 22 elements.

Conclusions

This report shows that the Assist sample introduction accessory can greatly enhance the performance of your ICP-OES and ICP-MS instruments. The accessory uses Flow Injection technology to reduce analysis time by more than 50%, typically, resulting in reduced environmental impact and lower operating cost. In addition to the cost and environmental benefits, the Assist delivers an improvement in analytical performance with the precision syringe drives. With three different configurations, the Assist can tackle most applications.

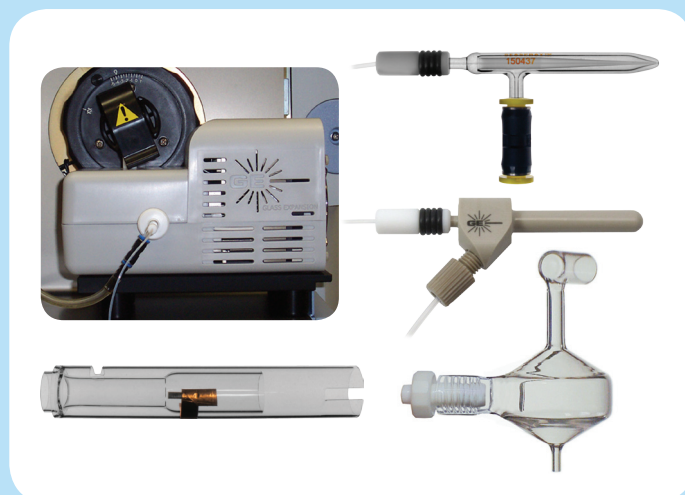
Reference

Based on a poster presented at the 2012 Winter Conference on Plasma Spectrochemistry, Tucson, Arizona, USA, January 9 – 14, 2012.

NEW PRODUCTS

Consumables for PerkinElmer Optima 8x00 ICP-OES

The PerkinElmer Optima 8000 series was released in 2011. We can now supply a full range of consumables for this model, including nebulizers, spray chambers and torches. You can see these products on our website. Click [here](#) to find products to suit your application or [here](#) to see the full range.



TruFlo for non-aqueous solutions

Since its release in 2009, the TruFlo Sample Monitor has proved to be an invaluable aid in solving sample introduction problems and minimizing instrument downtime. It measures the sample flow continuously in real time and alerts the operator if there is any change in flow. Typical customer comments are:

"The TruFlo from Glass Expansion is a great diagnostic tool for flow rate control."

"We like the TruFlo monitor. It has helped us on a number of occasions to track down some minor issues and we use it constantly while running our ICP."

"I have found the sample flow meter to be a very handy diagnostic tool."

The TruFlo is normally calibrated for aqueous solutions. With non-aqueous solutions, it can be reliably used to detect any change in the sample flow, but the actual flow reading will not be correct. For those who routinely run non-aqueous solutions (e.g. oil laboratories) we can now offer a TruFlo calibrated for your specific application. When you order the TruFlo, simply add: Part number **70-TFCAL**, TruFlo calibration service.

You will need to let us know what solution you are using and we will supply the TruFlo pre-calibrated for your solution.



INSTRUMENT NEWS

From Agilent Technologies – Launch of the World's First Triple Quadrupole ICP-MS

Agilent Technologies Inc. has announced the launch of the **Agilent 8800 triple quadrupole ICP-MS** (ICP-QQQ), the first and only instrument of its kind. The new ICP-QQQ system offers improved performance compared to single quadrupole ICP-MS, and provides MS/MS operation for controlled and consistent interference removal in reaction mode. ICP-QQQ also addresses high-end application requirements, with flexible analysis capabilities unavailable on single quadrupole machines.

The unique QQQ configuration of the Agilent 8800 controls the ions that enter the collision/reaction cell, so the reaction conditions remain consistent and predictable even if the sample composition changes. The new instrument provides turnkey operation in a range of novel reaction modes, delivering effective and consistent interference removal for problematic elements in difficult samples. The Agilent 8800 can also be set up to operate like a single-quad ICP-MS, offering the security of replicating existing and familiar methodologies.

The 8800 ICP-QQQ analyzes problematic elements in samples across a range of applications, including:

- Ultra-trace analysis of elements that suffer interferences in high-purity samples (Ge and As in HCl; V and Ti in H₂SO₄, and more).
- Reliable low-level measurement of selenium and arsenic in soil, rock and plant materials, where both polyatomic and doubly charged interferences may occur.
- Quantitative analysis of sulfur and phosphorus in DNA and protein/peptides for life-science applications.

To learn more about the 8800 triple quadrupole ICP-MS (ICP-QQQ), visit www.agilent.com/chem/icpqqq.

From Bruker – Advantages of Automated Aerosol Dilution on the Bruker aurora M90 ICP-MS

A key limitation of ICP-MS is the level of total dissolved solids that can be introduced. If a sample with a very high TDS level is analysed by ICP-MS, the narrow orifices of the interface cones may be partially (or even fully) blocked and result in sensitivity loss and poor detection capability. One technique that has become popular in recent times is Aerosol Dilution. With Aerosol Dilution the normal flow rates of nebuliser gas (typically 0.8-1.1 L/min) and sheath gas (typically 0.1-0.3 L/min) are reversed. The result is a decrease in the efficiency of nebulization, meaning less sample aerosol is being generated and less sample is reaching the plasma.

A higher sheath gas flow keeps the total carrier gas flow to the plasma consistent and maintains optimum flow conditions. The higher flow of the clean and dry sheath gas, relative to the nebulizer gas flow, means the density of the aerosol is further reduced upon mixing. This combined dilution effect is known as aerosol dilution and can provide the equivalent of a 10-fold dilution without the need for additional sample preparation.

This aerosol dilution capability is available on the Bruker aurora M90 and former Bruker 800-MS series as standard. No additional hardware is required. The inclusion of a new auto-optimisation sequence for aerosol dilution within the Bruker Quantum ICP-MS software makes method setup, fast and easy. Press a button and you are ready in a few seconds to start analysing your toughest samples.

For further information on this new feature please visit the following web link for a free download. (see Tech Note CA-275622):

www.bdal.de/products/icp-ms/aurora-m90/learn-more.html

From Spectro – Conclusive Waste Oil Analysis: Start Your Condition Monitoring Now with the SPECTRO GENESIS

Doctors use a patient's blood count to get an idea about their general health. Similarly, chemists use the analysis of used oil to draw conclusions about the state of installations or motors. The wear particles and trace elements in oil reveal which motor components are under greater strain, making it possible to proactively repair or replace these.

It's worth it all and that's exactly why more industrial businesses and logistics companies are using "condition monitoring" to optimize their maintenance cycles and to increase the lifetimes of installations, equipment and fleets. SPECTRO is there to help: With our **SPECTRO GENESIS** ICP Optical Emission Spectrometer, it is possible to precisely analyze the components of waste oil down to a few ppb.

Find out which condition monitoring advantages SPECTRO GENESIS holds for you in our free **White Paper "Cost-Effective Condition Monitoring with the SPECTRO GENESIS ICP-OES"**. Here you'll read:

- Why ICP-OES is the ideal analytical method for condition monitoring
- Why the user-friendly SPECTRO GENESIS is the ideal instrument for waste oil analysis and for your start into condition monitoring
- Which elements the SPECTRO GENESIS records and what detection limits it achieves during elemental analysis

Sound exciting? Then request your copy of the new White Paper now! You'll find all kinds of information about condition monitoring and many conclusive measurement series that illustrate the impressive performance of the **SPECTRO GENESIS**.

More about the SPECTRO GENESIS ICP-OES instrument and its applications can be found in our new online magazine, www.spectrolive.com.