

Glass Expansion Newsletter | February 2013 | Issue 30

APPLICATION SPOTLIGHT

LATEST DEVELOPMENTS IN ENHANCED PRODUCTIVITY PRODUCTS

The Glass Expansion line of enhanced productivity accessories has been specifically designed for ICP and ICP-MS applications and has been evolving since the original Niagara Rapid Rinse Accessory was introduced in January 2005. The Niagara Rapid Rinse Accessory provided the analyst with typical time savings of 30%, by starting the rinse cycle the instant the measurement was completed and rinsing the nebulizer and spray chamber during the sample flush time. In April 2009 the Niagara Plus was introduced, featuring a purpose-built, fully serviceable 12-port flow injection valve with a built-in internal standard mixing chamber. The new valve in combination with a programmable positive displacement pump dramatically increased sample throughput, (providing typical time savings of 50%) by rapidly filling the sample loop and rinsing out the sample lines. A simpler 7-port valve with removable internal standard tee was released in April 2011. The current 7-port valve reduces the number of components and swept volume, providing a faster uptake and easier maintenance.

In this report we will introduce the latest evolution in Glass Expansion's line of enhanced productivity accessories. One of the major improvements is the addition of a Control Module (CM) across the product line and the product names now have the suffix CM to denote this. The CM is a new addition to the Niagara II Rapid Rinse Accessory, Niagara Plus, and Assist packages, but this report will focus on the advantages specific to the Niagara Plus-CM (Figure 1).









Pittcon 2013

A wide selection of Glass Expansion products will be on display at Pittcon 2013, Philadelphia, Pennsylvania, USA, March 17 – 21, 2013. 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 and Niagara Plus enhanced productivity systems. 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 1149.

We will be presenting a seminar on advances in ICP sample introduction on Wednesday March 20 from 11:30 to 1:00 in Seminar Room SRA. The cost is \$10 per person and lunch is included. Please email **geusa@geicp.com** to register or to get an agenda.

IN THIS ISSUE:

 Application Spotlight 	1 – 4
• GE News	1
Product Design Focus	4 – 5
Instrument News	5 – 6
- From Agilent - From Bruker - From Spectro	





Save As

The Control Module

- With the introduction of the CM, the Niagara Plus is now controlled by a dedicated central command unit. By removing the task of sending commands to the Niagara Plus from the PC, the PC is now dedicated to running the Instrument, which in turn speeds communication.
- The timings for control of the Niagara Plus are no longer dependent on the USB host setting the processing priority, ensuring proper sequence of events. The processing priority setting isw becoming more of an issue with instruments using USB for accessories like an autosampler and valve system, as they can time out if the process priority is set to low.
- The Niagara Plus-CM has the option of running unattended without any connection to the instrument PC once the method has been loaded.
- The dedicated connections to the CM and new generic Assist syringe drives combine to simplify upgrading a Niagara Plus to an Assist.

New Software

The new features of the Niagara Plus-CM software include the following.

- An option to add a Wash Time for the autosampler probe and uptake tubing. Figure 2 shows the Method Settings, including "wash time after loop uptake".
- In combination with this wash time we have added the Post Wash command allowing the autosampler probe to be sent to any position once the wash time has concluded. This allows for the autosampler probe to be sent to a position ready for the next analysis, thereby eliminating most of the autosampler move time and further increasing throughput.
 Figure 3 shows the Autosampler Settings, including the "Post wash command".
- The Post Wash command can also be used to allow for up to three different rinse solutions. The method can be setup to flush the autosampler probe and uptake tubing with two different solutions while the ICP is analyzing a sample. The second of these solutions is then used to rinse the autosampler probe, uptake tubing and sample loop, while the third rinse is used to prepare the system for the next sample to ensure there is no added plasma stabilization time switching between different matrices.

Nethod settings Standby settin	igs Control	box		
Method:	NPlusII	~		New method
Loop uptake delay	7	sec		Save method
Uptake pump speed (Refill)	350	rpm		Save As
Uptake pump speed (Inject)	100	rpm		
Uptake pump speed (Move)	150	rpm		
Time in sample	6	sec		
Bubble inject time	6.8	sec		
Wash time after loop uptake	4	sec	🕖 Set to ze	ro to disable timeout
Turn off uptake pump after w	ash 🔽			

Figure 3: Niagara Plus-CM autosampler settings page.

New Actuator

Autosampler Settings

Sequence triggers

ExpertSPS3

Y

New

Save

Autosampler

The Niagara Plus actuator is the mechanism that triggers the switching valve to rotate to the programmed position. The new valve actuator provides extreme switching speed between ports, reducing the amount of back pressure on the system, thereby putting less stress on the connections to the valve and decreasing the chance of a leak.

Unique Features

The new CM product line maintains all of the unique features of the Niagara Plus as follows.

- **Bubble Injector:** The Niagara Plus-CM automatically injects several air bubbles at the end of the sample plug while filling the sample loop, as shown in Figure 4. These air bubbles prevent boundary diffusion, allowing for a smaller volume of sample to be used and prevent any sample dilution/mixing with the carrier solution. Because the bubbles are injected at a programmed time they are never introduced into the nebulizer which would cause plasma disturbances and require greater stabilization delays. When bubbles are placed between the sample solution and the carrier solution the surface tension restricts the mixing of the two solutions (see the bottom tube in Figure 4). Injecting the bubbles at the end of the sample allows virtually all the sample in the loop to be used for analysis. The bubble injector will put 8-10 bubbles at the end of the sample loop over approximately 25mm of tubing.
- **Time In Sample (TIS):** This feature allows the analyst to program the time the autosampler probe sits in the sample tube instead of waiting until the loop is filled. This prevents the sample in the uptake lines from being wasted, further reducing the amount of sample consumed and reducing the sample cycle time.

Figure 2: Niagara Plus-CM method settings page.

Ignore repeated triggers 🦳 Valve to rinse trigger move_z(0)\r A/S reply (optional) Valve to home trigger move_z(%1d%1[^6]%1d)\r OK\r A/S reply (optional) Autosampler emulation move_z(0)\rmove_sample(0,0)\rmove_z(160)\r Wash command move_z(155)\r Post wash command Error trigger Error string to send to host Automatic replies to instrumentation commands Command Reply position read\r **∩K**r Echo commands write_relay(d,0)\r OK\r Trigger checker One check the syntax of a trigger against the expected commands. Expected string/command Check Trigger to test Port settings COM Port BAUD rate 9600 COM Port stop bits 1 OK Cancel

NEWS



- Built-in Internal Standard Tee: This low volume removable mixing tee allows the operator to tee in diluent or internal standard without significantly lengthening either the stabilization time or washout time. If you select not to tee in an internal standard or diluent, the tee can simply be removed from the valve.
- Programmable Uptake Pump: With the Niagara Plus-CM software you have the option of having the uptake rate of the pump (and uptake syringe for HF model) automatically calculated using the method wizard. The wizard calculates the optimum pump speed for the length of the uptake tubing including probe and sample loop size. Using the optimum pump speed helps to reduce pump wear and to prevent degassing of the sample. Systems with a fixed rate vacuum pump do not allow for this type of adjustment. With the Niagara Plus-CM software you can also use the optimum speed (as low as possible) for the rinsing of the uptake lines during the "Inject" mode. The "move" mode can be used to either conserve rinse solution or to flush the uptake and sample loop with a large volume of rinse. This is user configurable.

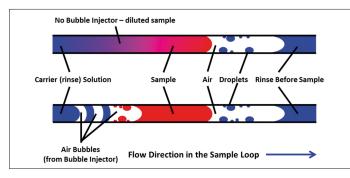


Figure 4: Comparison of sample loop with and without a bubble injector.

Bubble Injector and TIS Example

As an example, If an analyst has a an uptake tubing length of 1000 mm, a nebulizer flow rate of 1 mL/min, stabilization time of 10 sec and total read time of 50 sec (Total of 60 sec), the minimum amount of undiluted solution you would need to complete a read is 1.0 mL. With no bubble injector and no TIS option, 2.8 mL of sample is consumed in order to create enough undiluted sample to complete each read. The Bubble Injector reduces the amount of sample needed down to 1.7 mL and combining the Bubble Injector with the TIS option conserves an additional 0.4 mL of sample, bringing the total sample consumed down to 1.3 mL. These two features of the Niagara Plus-CM conserve sample and reduce sample-to-sample time.

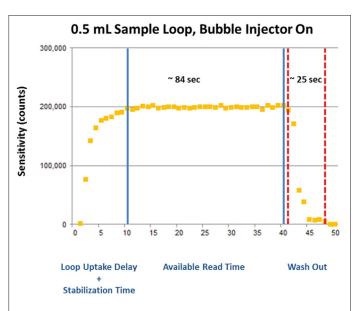
Bubble Injector and TIS Experiment

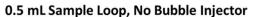
To further prove the advantages of the Bubble Injector, an experiment was setup up on an Agilent 7500 ICP-MS comparing the results with the bubble injector ON and the bubble injector OFF (Table 1). For this experiment a sample flow rate of 0.22 mL/min was utilized with a 0.5 mL sample loop. The ICP-MS delay time was purposely set to zero and 50 replicates were measured (2.78 sec/replicate) in order to show the available instrument read time with the bubble injector on versus no bubble injector. Figure 5 shows that, with the bubble injector on variately 84 seconds of available read time, compared to only 41 seconds with a system with no bubble injector. The graph with no bubble injector in use clearly illustrates the sample within the loop is starting to be diluted by the carrier flow around replicate 25. The use of the bubble injector also shows that rinse times would be reduced.

Comparison of analysis and washout time				
	Without Bubble Injector	With Bubble Injector		
Sample Loop (mL)	0.5	0.5		
Neb. Flow Rate (mL/min)	0.22	0.22		
Available Read Time (sec)	41	84		
Wash Out Time (sec)	> 70	25		

Table 1: Comparison of analysis and washout time, with and without a bubble injector.

Please note that the graphical data shown in Figure 5 does not include the rinsing of the uptake lines or a post-acquisition loop rinse that would typically occur at a much faster rate than 0.22 mL/min. However, with the bubble injector the blank level returns to baseline in as little as 25 seconds at only a flow rate of 0.22 mL/min, compared to greater than 70 seconds with no bubble injector. You will also have to consider that with no bubble injector, you will require twice the amount of sample, and even at the higher rinsing speeds and post-acquisition rinse, the washout will require more time compared to a sample loop of half the size.





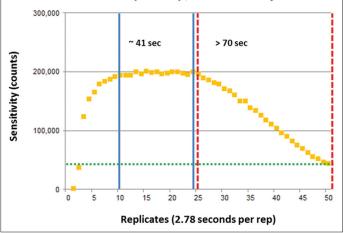


Figure 5: Comparison of sample loop with and without a bubble injector.

NEWS



Combining the Bubble injector and TIS, the Niagara Plus-CM can save up to 55% of the sample compared to a system without these features. A smaller sample loop also translates to faster sample to sample time. The Bubble Injector and TIS option can also help reduce carryover. One of the most noted performance improvements for some laboratories is the reduction in carryover. For example at one laboratory (ICP-OES), without the Niagara Plus the carryover for Mo from a 500 µg/L sample was >20 µg/L. With the Niagara Plus installed, the carryover is now undetectable at <4 µg/L. A Fe sample with a concentration of 10,000 µg/L previously had carryover >5 µg/L that is now undetectable at <2µg/L. At a mineral assay laboratory (ICP-OES), the Niagara Plus provided the ability to analyze a 200ppm Au sample and achieve 5ppb Au in the next blank with no added rinse. Previously a rinse time of up 2 minutes was used.

Summary

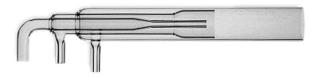
In summary, with the new control module, software, and components the Niagara Plus-CM combines versatility with ease of installation and operation, and an easy avenue to upgrade the system and features. The CM platform results in the fastest possible sample cycle time, minimizing carryover and wear on consumable components.

PRODUCT DESIGN FOCUS

THE D-TORCH

First there was the one-piece torch

The ICP torch comprises three circular concentric tubes – an outer tube, an inner or intermediate tube and an injector tube which delivers the sample to the plasma. The torch is usually made from quartz and the three tubes have traditionally been fused to form a single piece.



...then the semi demountable torch

While this approach has worked well, it does not offer the analyst any flexibility. Some samples require torch components with different dimensions or made of different materials. For example, quartz is attacked by HF, and samples containing HF require an injector tube made from a ceramic material such as alumina. Also, organic solvents usually give best results when the sample flow is reduced and a narrow-bore injector is used. In order to accommodate the need for different injectors, the semidemountable torch was introduced. With this design, the inner and outer tubes are fused to form a single-piece torch body while the injector is separate. The two parts are held in position in an adaptor or base with o-ring seals. The semi-demountable torch allows the injector to be conveniently changed in order to provide optimum performance with different sample types.



...then the fully demountable torch

While the semi-demountable torch allows the injector to be changed, it does not address the common problem of outer tube damage. This tube is very close to the plasma and small changes in argon flow can cause it to overheat and melt. Also, salt particles from the sample can react with the quartz, causing devitrification. In either of these situations, the outer tube must be replaced. With a one-piece torch, this means that the whole torch must be replaced, even though the inner tube and injector may be fine. With a semi-demountable torch, the torch body must be replaced, even though the inner tube may be fine.

The fully demountable torch (FDT) was introduced as a solution to these problems. With this design, the outer tube, inner tube and injector are all separate components able to be replaced independently. With early FDT designs, the components were held in place by o-ring seals. However, it was very difficult to position the inner and outer tubes to the accuracy required using this method. It was only when Glass Expansion introduced the glass torch base that the tolerances required for routine replacement of all torch components could be met. With this design, the inner and outer tubes slid over a ground glass body that provided both the positioning and the gas seal. A drawback of this design was that the glass body and the quartz tubes had to be manufactured to micron-level tolerances so that any quartz tube could be fitted to any torch and still be perfectly positioned. This requirement made the FDT quite expensive.





...and now there is the D-Torch

The D-Torch is a revolutionary new demountable torch design. It provides the benefits of a fully demountable torch at a significantly lower cost. The benefits of the D-Torch include:

- Demountable outer tube instead of replacing the entire torch when just the outer tube wears, you need only replace the outer tube. And most operators will be saving money after only a few replacements.
- Interchangeable quartz and ceramic outer tubes.
- Much lower cost than other demountable torches.
- Interchangeable injectors choose the optimum injector for your application. Select from quartz for aqueous solutions, narrow bore for organics, wide bore for high dissolved solids or ceramic for HF.



...with a ceramic option

Glass Expansion pioneered the design of ceramic torches more than 20 years ago with the PT2000 FDT for the ARL Maxim ICP-OES. Since then we have provided ceramic versions of our PT2000 and ABC FDTs for several ICP models.

Ceramic outer tubes are also available for the D-Torch. In general the ceramic outer tube has a much longer lifetime, greatly reducing interruptions and downtime due to torch failure. The ceramic outer tube is of particular benefit for:

- the analysis of wear metals in engine oils, where quartz outer tubes often suffer from short lifetime.
- Si determinations, where quartz outer tubes often produce high background signals.
- fusion samples or samples with high levels of dissolved solids which cause quartz tubes to devitrefy.

INSTRUMENT NEWS

From Agilent Technologies – Latest Revision of ICP-MS MassHunter Software

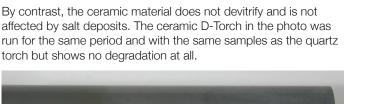
ICP-MS MassHunter software has been updated for the 7700 Series and 8800 ICP-QQQ, providing many new features and improved operation in key application areas. Agilent's ICP-MS MassHunter software has been supplied with the 7700 Series ICP-MS since 2009, with major updates in 2010 and 2011. The latest B.01.02 revision (released December 2012) adds more new functions, performance improvements and a range of other enhancements.

The major developments in the new revision are:

- Improved functionality for fast TRA and laser ablation
- More flexible tuning options for non-standard applications
- Additional data processing functions for chromatography
- Compatible with Agilent Spectroscopy Database Administrator (SDA) for Compliance in regulated pharma laboratories

The new B.01.02 revision of ICP-MS MassHunter ships as standard with all new 7700 Series ICP-MS and 8800 ICP-QQQ instruments from December 2012.







A combination of high temperature and salt deposit causes

and is already badly degraded.

a quartz torch to devitrify. Higher concentrations of salt in the

samples lead to more rapid devitrification. The quartz torch in the

photo was run for only 6 hours with samples containing 10% NaCl

Glass Expansion manufactures D-Torches for Agilent ICP-OES and ICP-MS, PerkinElmer ICP-OES and ICP-MS, Thermo Fisher ICP-OES and Spectro ICP-OES models. Please send an email to **enquiries@geicp.com** for more information.



INSTRUMENT NEWS

From Bruker – The Science of Sensitivity Powers the Business of Quantitation

Bruker is proud to announce the release of its new ICP-MS, the aurora Elite at the 2013 Plasma Winter Conference held this year in Krakow, Poland. In addition to the highly successful aurora M90, Bruker now offers an additional model, the aurora Elite, as its new high-end performance offering, setting new and exceptional standards for sensitivity and matrix robustness in ICP-MS.

The following features are now available with the aurora Elite:

- Industry leading Sensitivity: 1.5 billion cps/ppm sensitivity in 'High Sensitivity' mode and 400 million cps/ppm in 'Normal Sensitivity' mode for middle masses
- Faster scan speeds and shorter integration times: offers an unmatched performance combination for faster transient signal analysis such as LA-ICP-MS and LC-ICP-MS.
- Lowest cost of ownership: rugged design, durable cone system, no expensive spare parts, and a unique all-digital detector with a typical lifetime of more than 5 years.
- Full 21 CFR Part 11 compliance support for Quantum ICP-MS software: to meet the requirements of the pharmaceutical industry aurora's Quantum software with CompassCDS offers new features such as audit trails, electronic signatures and user management.
- External Device Control: bidirectional triggering for seamless connectivity to laser ablation and chromatographic devices.
- Improved air flow management inside the instrument: with a significantly reduced exhaust flow requirement which reduces installation cost and is of particular interest in clean room environments.

For more information go to: http://www.bdal.de/products/icp-ms/aurora-m90/learn-more.html.





From SPECTRO – Increase sensitivity analyzing trace elements by using ElectroThermal Vaporization and the SPECTRO ARCOS ICP-OES spectrometer

By using electrothermal vaporization (ETV) for sample introduction, SPECTRO has greatly improved the sensitivity of the SPECTRO ARCOS ICP-OES spectrometer for material analysis applications. Trials verify that detection limits can be improved by an order of magnitude. At a much lower cost, ICP-OES penetrates the sensitivity range of glow discharge sector field mass spectrometers, opening new application areas in solid material analysis for ICP-OES.

Typically, ICP-OES samples are first dissolved and then introduced to the instrument using a nebulizer. When using electrothermal vaporization (ETV), however, the solid samples are vaporized in an oven with a temperature range up to 3,000 degrees Celsius. The graphite vaporization chamber uses argon as the inert gas. After vaporization, the analyte is transported as a dry aerosol to the ICP instrument with an argon/reaction gas stream. Sample vaporization takes just two minutes.

ETV analysis opens up whole new possibilities especially for samples that are difficult to dissolve. No longer is it necessary to create solutions of ceramic materials, such as the extremely resistant silicon carbide, using time-consuming chemical digestion.

Electrothermal vaporization as a method for sample preparation has been available on the market for some time. "However, first generation ETV systems were often difficult to operate, not especially suited to routine use and prone to errors. There has been major success in making ETV more compact and user friendly," reports ICP-OES Product Manager, Olaf Schulz.

SPECTRO offers the ICP-OES ARCOS together with the ETV system as a complete system with an autosampler for up to 50 solid samples in its range of products.