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Quality By Design

A Simple, Low Cost Approach to Improving ICP Productivity

**The following report is the result of a collaboration between Glass Expansion and Inorganic Ventures*

In the October 2013 Glass Expansion Newsletter[1] we collaborated with Inorganic Ventures, the well-known Certified Reference Material (CRM) manufacturer (www.inorganicventures.com) to organize an application spotlight titled, "How to Achieve High Accuracy with Difficult Samples." The purpose of this article was to emphasize the importance of choosing a proper ICP sample introduction system based on your sample types. In this particular case we focused on the ICP-OES challenges associated with analyzing samples in a hydrofluoric acid (HF) matrix and samples high in total dissolved solids (TDS).

Once again we have teamed with Inorganic Ventures to introduce a simple, low cost approach to improving your ICP productivity. In this report we will highlight the advantages and versatility of Glass Expansion's Niagara CM Rapid Rinse Accessory, in addition to summarizing the latest updates to the operating software, which better facilitate ease of use and installation.

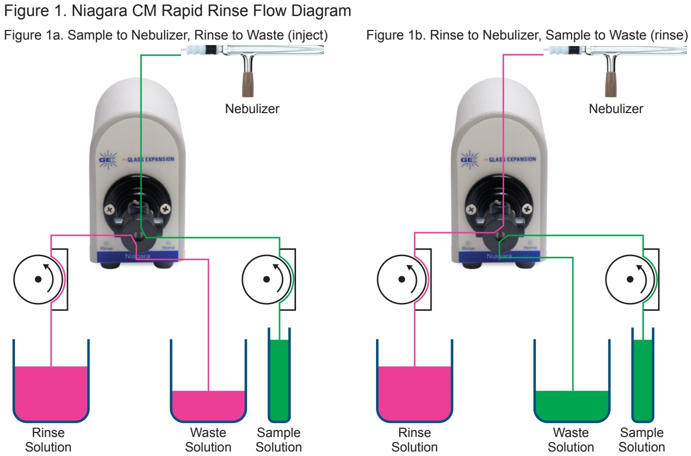
Introduction

Most ICP systems incorporate an autosampler. With these systems there is a significant delay between the time when the autosampler probe enters the sample and the time when the sample reaches the nebulizer. There is also similar delay between the time when the probe enters the rinse solution and the time when the rinse solution reaches the nebulizer. If these delays could be eliminated, the analysis time per sample could be reduced significantly and the sample throughput increased. This is exactly what the Niagara achieves.

The Niagara begins rinsing the nebulizer and spray chamber the instant the sample measurement is completed and continues to rinse until the next sample is ready. Thus the rinse is carried out in the time that is usually wasted waiting for the sample and the rinse solutions to flow from the autosampler probe to the nebulizer. In Figure 1, we show a flow diagram of the Niagara in both the "Home" and "Rinse" positions. The valve is in the "Home" position in Figure 1a., where the sample is being directed to the nebulizer while the rinse solution is directed to waste. At the completion of the sample measurement, the valve switches instantly to "Rinse" position and the rinse solution is directed to the nebulizer (Figure 1b), allowing for the nebulizer and spray chamber to begin rinsing immediately, with no uptake delay. The valve remains in the "Rinse" position while the autosampler probe moves to the next sample. Only when the next sample has made its way through the full length of the uptake tubing does the valve switch back to the "Home" position (Figure 1a).

The typical time saved with the Niagara is around 30% and since the valve switches immediately after the read is complete, you also minimize the amount of sample introduced into the plasma. This feature can drastically reduce your consumables costs, particularly for challenging sample matrices like high TDS, HF and organic solvents where the ICP torch and/or ICP-MS cones require frequent cleaning and can suffer from a shorter life.

Figure 1. Niagara CM Rapid Rinse Flow Diagram

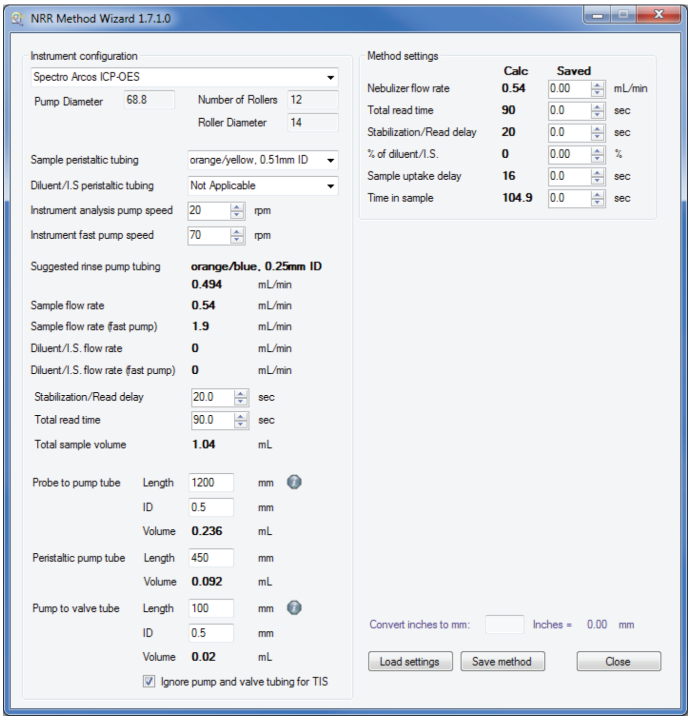


Niagara CM Rapid Rinse Updates

Since the original Niagara was introduced in January 2005,[2] Glass Expansion's line of enhanced productivity accessories has been continuously evolving to keep up with the demands of ICP analyses. Most recently, in 2013 we introduced the addition of a Control Module (CM), adding the suffix CM to denote this[3]. With the introduction of the CM, all Glass Expansion enhanced productivity accessories are now controlled by a dedicated central command unit. This provides faster and more reliable communication to the instrument PC and an easy avenue for upgrading. Further details on the benefits of the CM can be found in the February 2013 Newsletter.[3]

We are now introducing a suite of software upgrades that are specific to the Niagara CM Rapid Rinse. Software version NRRV2 includes a redesigned Method Wizard (Figure 2), which features an Uptake Delay Calculator, Rinse Tubing Calculator, and Time in Sample (TIS) Calculator. The addition of these calculators greatly simplifies method development so that you can quickly add the Niagara to your existing ICP method and optimize for maximum savings.

Figure 2. Niagara CM Rapid Rinse Method Wizard



The Uptake Delay Time is the time required for the sample to reach the valve and pass through to waste, ensuring that the first bit of sample diluted with the previous rinse is flushed out to waste. Once the Uptake Delay Time has finished, the Niagara valve will switch from Rinse to the Home position, directing the sample to the nebulizer (Figure 3a). The Uptake Delay Time is calculated based on the ID and length of the autosampler probe; peristaltic pump tubing ID and length; and RPM of the peristaltic pump (Fast Pump Speed). The Rinse Tubing Calculator calculates the optimum peristaltic pump tubing to be used in order to achieve the same flow rate that is used during analysis while using a fast pump during the uptake and post-acquisition rinsing steps of the analysis. Time in Sample (TIS) is the time required for the autosampler probe to remain in the sample tube during the acquisition (Figure 3b). The TIS is based on the total read time, RPM of the peristaltic pump (Analysis Pump Speed) and dimensions of the uptake tubing referenced above.

To improve washout, it is well known that adding a bubble or series of bubbles can help "scrub" the uptake line. However, air bubbles introduced into the plasma can lead to instability and require longer stabilization delay times. With the Niagara, we can easily add a series of air bubbles with our new post wash command without the worry of introducing any air or extra argon into the plasma. To achieve this, the NRRV2 software sends the autosampler probe to the rinse station after the TIS has completed, followed by a series of commands to slowly raise and lower the probe in the rinse station. This creates a series of air gaps between the rinse solution (Figure 3c) helping to rinse the probe, uptake tubing and sample peristaltic pump tubing. Once the ICP method rinse begins, the air bubbles and any excess sample are directed to waste while rinse solution is directed to the nebulizer (Figure 3d).

Figure 3a. Niagara CM Rapid Rinse – After Uptake Delay

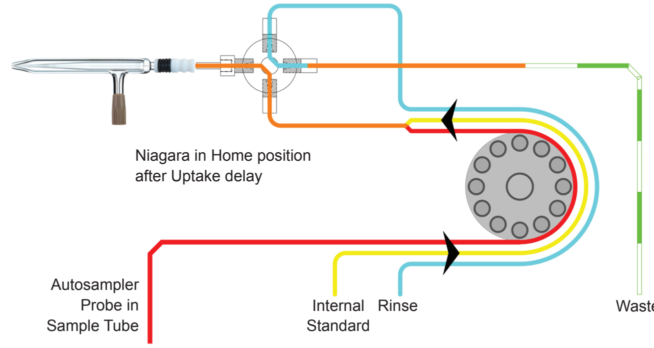


Figure 3b. Niagara CM Rapid Rinse – During Acquisition

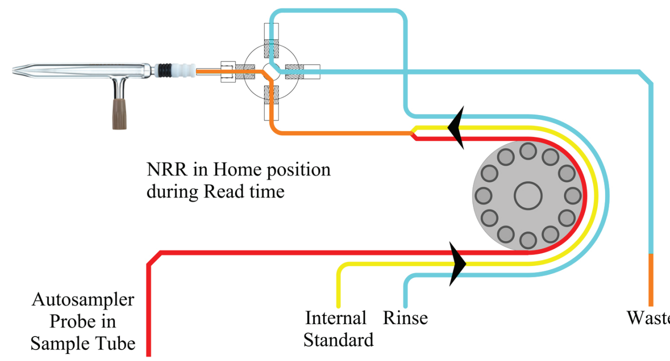


Figure 3c. Niagara CM Rapid Rinse – After TIS

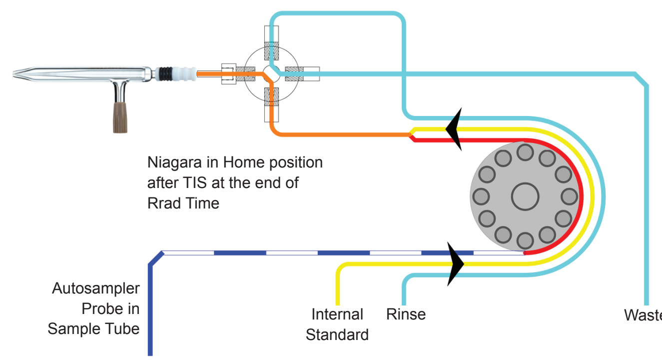
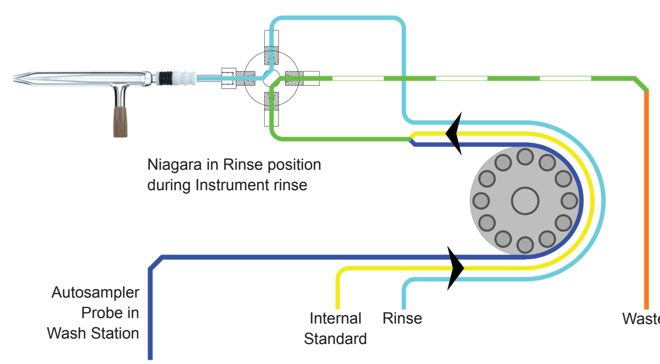


Figure 3d. Niagara CM Rapid Rinse – During ICP Method Rinse (post-acquisition)



Experimental

The speed and performance of Inorganic Venture's single element CRM ICP-OES certification analysis was compared with and without the use of the Niagara. For these certification experiments a Spectro Arcos EOP (Axial) ICP-OES instrument was used in combination with two sample introduction systems from Glass Expansion. For samples without HF, the SeaSpray DC nebulizer was used with the Twister spray chamber and a single piece quartz torch (Figure 4a). The HF-resistant package consisted of the DuraMist DC nebulizer, Tracey TFE spray chamber and fully ceramic D-Torch (Figure 4b). The instrument parameters for the Arcos are listed in Table 1. These particular sample introduction components and ICP operating conditions were previously optimized.[3]

Figure 4. Glass Expansion Sample Introduction systems for Arcos EOP ICP-OES

Figure 4a

Aqueous sample introduction system, SeaSpray DC Nebulizer, Twister spray chamber, glass elbow and single piece quartz torch for Spectro Arcos EOP ICP-OES. Also required is mounting bracket for Niagara Rapid Rinse.

Figure 4b

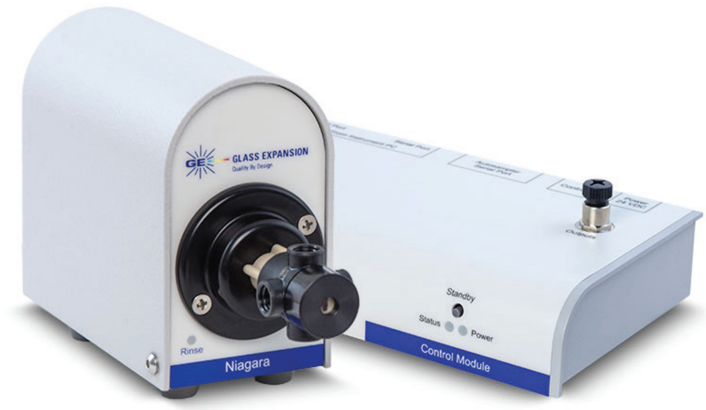
HF-resistant sample introduction system, DuraMist DC Nebulizer, Tracey TFE spray chamber, PuraFlex transfer tube, and fully ceramic D-Torch for Spectro Arcos EOP ICP-OES. Also required is mounting bracket for Niagara Rapid Rinse.

Table 1. Instrument parameters for Spectro Arcos EOP ICP-OES

	"Glass Setup"	"Inert Setup" (with HF)
RF Power (W)	1400	1400
Plasma gas flow (L/min)	15	15
Auxiliary gas flow (L/min)	1.0	1.0
Nebulizer gas flow (L/min)	0.75	0.75
Nebulizer flow rate (mL/min)	0.54	0.54
Torch	Single Piece Quartz Torch (P/N 30-808-0317)	Ceramic D-Torch (P/N 30-808-3371 & P/N 31-808-3425)
Injector i.d. (mm)	2.5	2.4 (P/N 31-808-3388)
Nebulizer	SeaSpray DC (P/N A21-07-USS2)	DuraMist DC (P/N A21-07-DM1)
Spray Chamber	Twister (P/N 20-809-9199HE)	Tracey TFE (P/N 20-809-2506)
Transfer Tube	31-808-3244	21-809-3298

The Niagara package (Figure 5), consists of an electronically controlled switching valve, dedicated control module and tubing kit. For maximum chemical resistance the 4-port valve is made entirely of Teflon and PEEK. An additional mounting bracket for the Arcos EOP ICP-OES was required to position the Niagara valve next to the nebulizer; this helps to reduce stabilization time. Please visit www.geicp.com/intro/niagara to find the recommended Niagara setup for your ICP.

Figure 5. Niagara CM Rapid Rinse



The method timings for the Niagara CM Rapid Rinse were calculated using the newly developed Method Wizard. These conditions are listed in Table 2, comparing the timings with and without the Niagara. Typically a very high "Fast Pump" would not be recommend due to the large amount of sample introduced into the nebulizer and plasma. However, with the Niagara the higher uptake rate of sample is directed to waste while the rinse solution is directed to the nebulizer at the analysis speed. This helps to maintain plasma stability while reducing the uptake delay time, stabilization time and sample load on the plasma. The Method Wizard also calculates the appropriate size "rinse" peristaltic pump tubing to select. This way when the Fast Pump option is used during the uptake, you are able to maintain a constant flow to the plasma. With the Arcos software, a minimum 1 second method rinse is required to properly trigger the Niagara actuator. A longer post rinse time can be used if necessary.

Table 2. Method settings with and without the Niagara Rapid Rinse

	Without Niagara	With Niagara
Autosampler Probe ID	0.5mm	0.5mm
Sample peristaltic pump tubing	Orange/Green, 0.38mm ID	Orange/Yellow, 0.51mm ID
Rinse peristaltic pump tubing	N/A	Orange/Blue, 0.25mm ID
Waste peristaltic pump tubing	White/White, 1.02mm ID	White/White, 1.02mm ID
Uptake Delay (sec)	50	23
Flush Pump Speed	45 rpm	70 rpm
Stabilization (sec)	30	15
Stabilization Pump Speed	45 rpm	20 rpm
Analysis Pump Speed	45 rpm	20 rpm
Read (sec)	90	90
Rinse (sec)	30	1

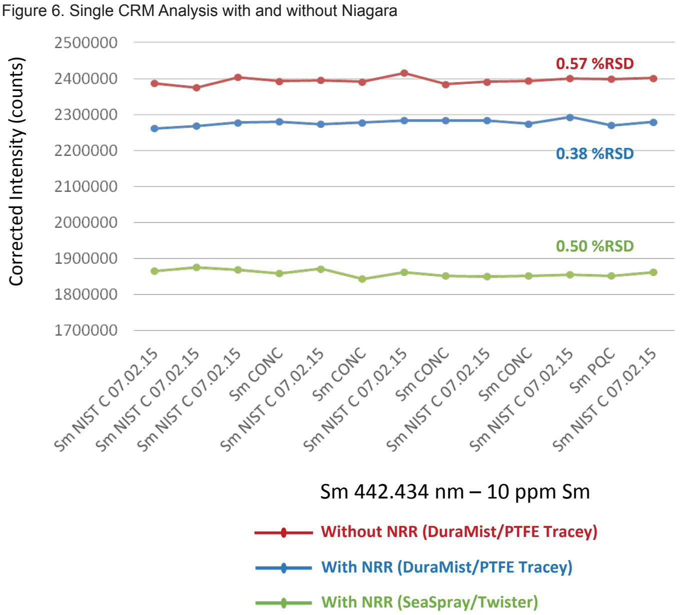
Results

A summary of the method time per sample with and without the Niagara-CM Rapid Rinse is shown in Table 3. The addition of the Niagara allowed Inorganic Ventures to reduce their certification analysis by 71sec per sample, roughly a 35% improvement. This reduction in time allows Inorganic Ventures to run an additional 10 samples per hour with the Niagara. The performance of the ICP certification analysis with the Niagara is also optimal. The stability and precision achieved certifying a 10ppm Sm CRM with the Niagara is summarized in Figure 6. With the Niagara, an average RSD of less than 0.5% is maintained with both Glass Expansion sample introduction systems.

Table 3. Method timing with and without the Niagara Rapid Rinse

	Without Niagara	With Niagara
Total Time (sec)	200	129
Improvement	N/A	35%
Samples per hour	18	28

Figure 6. Single CRM Analysis with and without Niagara



In addition to reducing sample to sample time, the Niagara also improves washout. To evaluate the performance of the new post wash command of the NRRV2 software, we compared the washout time achieved when adding a series of bubbles behind the sample to no bubbles. Thallium (Tl) is known to be a very "sticky" element that can result in the need for very long wash times. Thus a 1000ppm Tl solution was chosen for this test. These experiments were carried out using an Agilent MP-AES 4100. Results in Table 4 contain values in ppm Tl for three consecutive 2% HNO3 Blank solutions that were run after a 1000ppm Tl solution. The washout experiments were run three ways; without the Niagara, with a standard Niagara setup (no bubbles), and with the Niagara utilizing the new wash command adding a series of bubbles. With the new wash command, a dual rinse can be used in addition to adding a series of air bubbles. The dual rinse option provides the ICP analyst an option to use a more aggressive solution to rinse the uptake lines, but be directed to waste and not introduced to the ICP. With only a 10 sec rinse, the standard Niagara system achieved a better washout compared to a 30 sec rinse without the Niagara saving a considerable amount of time (31%). However, adding the bubbles and the possibility to have dual rinse stations with bubbles reduced the carryover even further, giving better than 4 orders of magnitude washout with a 46% savings in sample time compared to the standard instrument setup.

Conclusions

The Niagara CM Rapid Rinse provides a simple, low cost approach to improving ICP productivity. Typical time saved with the Niagara is approximately 30%, with little to no changes to the current ICP method settings and no degradation in performance. A new Method Wizard combined with the features of the NRRV2 software facilitate easy optimization and self-installation. A unique new post wash feature with the option to add a series of bubbles and dual rinse drastically improves washout and reduces rinse time.

References

1. Glass Expansion October 2013 Newsletter, *"How to Achieve High Accuracy with Difficult Samples."*
2. Glass Expansion October 2005 Newsletter, *"Increasing the Productivity of ICP Analyses."*
3. Glass Expansion February 2013 Newsletter, *"Latest Developments in Enhanced Productivity Products."*