

APPLICATION SPOTLIGHT

Performance of a "Souped Up" ICP-OES System

Introduction

Typically nebulizer uptake rates of 1.0 to 2.0mL/min are utilized in ICP-OES in order to achieve acceptable sensitivity and precision. An even higher nebulizer rate, often referred to as "fast pumping" may also be required to washout a high standard or sample. Acceptable blank levels in some environmental applications require long rinse times at this fast pumping rate, which leads to long sample to sample time and reduces the lifetime of consumables. In this paper, we will examine the use of specific sample introduction accessories to dramatically improve both the performance and productivity of an ICP optical spectrometer with a focus on small sample volume. This is accomplished by optimizing the sample transport efficiency to the ICP and by maximizing the efficiency of sample delivery and washout.

The Niagara Plus CM[™] Enhanced Productivity Accessory was utilized in the "souped up" ICP configuration to reduce analysis time and sample consumption, while improving washout at a 300µL/min nebulizer flow rate. The Niagara Plus CM system is based on flow injection technology, combining a 7-port switching valve with a programmable positive displacement pump to fill a sample loop directly from the autosampler probe (bypassing the peristaltic pump) at a rate of up to 40 mL/min. The sample is rapidly loaded into the sample loop, drastically reducing uptake delays, without affecting plasma stability. Furthermore, the switching valve redirects rinse solution to the nebulizer as soon as the measurement is complete. The result is a dramatic increase in sample throughput, providing typical time savings of 50%.⁽¹⁻³⁾

In order to maximize the sensitivity of the ICP at a 300µL/min nebulizer flow rate, the IsoMist[™] Programmable Temperature Spray Chamber was incorporated. Replacing the ambient baffled cyclonic spray chamber with the IsoMist allows the ICP analyst to control the temperature of the spray chamber, whether it be for cooling or heating, from -10 °C to +60 °C. The benefit of this is two-fold. First, simply stabilizing the spray chamber temperature reduces the drift in transport efficiency that has been shown to occur in non-temperature stabilized chambers.⁽⁴⁾ Second, stabilizing the spray chamber at a temperature above ambient increases transport efficiency and thus intensity; a feature that is particularly important for samples with limited volume.⁽⁵⁻⁷⁾

Experimental

A Thermo iCAP 6500 Duo ICP-OES was used to compare the standard operating conditions with the "souped up" configuration. The parameters for both configurations are listed in Table 1. Note that the same plasma conditions are utilized for both sample introduction systems. The only difference is the incorporation of the IsoMist and Niagara Plus CM.



Glass Expansion News



New 2014 Catalogue

The new 2014 Glass Expansion catalogue is now available and finding the most suitable sample introduction system has never been easier. A full list of the available products for each of the most prevalent ICP-OES and ICP-MS models is included. There is also information on a wide range of performance and productivity-enhancing accessories and fittings. Please send your mailing address to enquiries@geicp. com and we will send you a catalogue immediately.

Upcoming Exhibitions and Conferences

A full range of Glass Expansion products will be on display and Glass Expansion specialists will be on hand to assist you at the following exhibitions and conferences:

NYAAEL/PaAAEL Environmental Conference and Exhibition, Corning, NY, USA, July 21-22 (www.nyaael.org) We will also be presenting "Optimizing ICP-OES and ICP-MS Sample Introduction Systems for Environmental Analysis" at the Technical Session.

JASIS 2014, Tokyo, Japan, September 3 to 5. Booth # 4A-609 (www.jasis.jp/2014/en/)

SCIX Conference and Exhibition, Reno, NV, USA, September 28-October 3, Booth #12 (www.scixconference.org)

Gulf Coast Conference, Galveston, TX, USA, October 14-15, Booth #1126 (www.gulfcoastconference.com)

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The measurement conditions and method timings of each ICP configuration are listed in Table 2. A maximum integration time of 30 seconds was used for both set-ups. In order to reduce the acquisition time only the axial view of the iCAP Duo ICP-OES was utilized. Working calibration standards were prepared in 2% HNO₃ by means of serial dilutions from single element solutions of 1,000 mg/L concentration or 10,000 mg/L (Elemental Standards, P.O. Box 649, Everton Park, QLD, 4053, Australia).

Table 1. Thermo iCAP 6500 DUO ICP-OES Instrument Parameters			
	Standard ICP-OES	"Souped Up" ICP-OES	
RF Power	1350 W	1350 W	
Plasma gas flow	12.0 L/min	12.0 L/min	
Auxiliary gas flow	0.2 L/min	0.2 L/min	
Nebulizer gas flow	0.65 L/min	0.65 L/min	
Torch	Ceramic D-Torch (P/Ns 30-808-2844 and 31-808-3079)	Ceramic D-Torch (P/Ns 30-808-2844 and <u>31-808-3079)</u>	
Nebulizer	SeaSpray (P/N ARG-07-USS2)	SeaSpray (P/N ARG-07-USS2)	
Nebulizer flow rate	1.0 mL/min	300 µL/min	
Spray chamber	Twister (P/N 20-809-2951HE)	IsoMist (P/N KT-1015)	
Spray chamber temperature	N/A	29°C	
Autosampler	Cetac ASX-500	Cetac ASX-500	
Flow Injection	N/A	Niagara Plus with 1.2mL Sample Loop	

The sample loop size for the Niagara Plus is determined by the nebulizer flow rate, total acquisition time of the spectrometer, and the stabilization time. For the "souped up" ICP configuration a custom 1.2mL sample loop was used with the Niagara Plus. Approximately 2.4mL of sample was taken up by the Niagara Plus to efficiently rinse and load the sample loop. However, only 0.7mL of sample was introduced into the plasma with the Niagara Plus. The switching valve immediately directs rinse solution to the nebulizer once the ICP measurement is complete and any excess sample is pumped out to waste. Conversely, with the standard ICP any excess sample left in the uptake line will be flushed into the ICP during the rinse cycle. In these experiments the standard ICP set-up consumed over 5mL of sample all of which impacted on the sample introduction conponents (Table 2).

Table 2. ICP Measurement Parameters			
	Standard ICP-OES	"Souped Up" ICP-OES	
Max integration time	20 + 10 seconds (low and high)	20 + 5 seconds (low and high)	
Replicates	3 (Axial only)	3 (Axial only)	
Rinse time	40 seconds	0 seconds	
Flush time	60 seconds	17 seconds	
Stabilization time	20 seconds	20 seconds	
Total "sample to sample" time	270 seconds	150 seconds	
Sample volume	5.3 mL	2.4 mL	

Results

The sample transport efficiency of the "souped up" ICP was optimized at 29°C, utilizing the heating mode of the IsoMist. The sensitivity achieved with the IsoMist at a nebulizer flow rate of 300μ L/min was on average within 15% of that obtained at a nebulizer uptake rate of 1mL/min with the standard system (Figure 1). In some cases the sensitivity with the "souped up" ICP was greater than or equal to the standard ICP configuration.



A comparison of the detection limits obtained with the IsoMist at 29°C to the standard Twister spray chamber at ambient temperature is shown in Figure 2. Even though the nebulizer uptake rate was 70% lower, the detection limits obtained at 29°C, are on average within 30% of the standard ICP configuration. The sensitivity and detection limit results show the benefits of adding a temperature controlled spray chamber to the ICP by being able to increase the transport efficiency. Although not examined in this report, maintaining a constant spray chamber temperature also provides improved signal stability. Long-term signal stability helps to ensure that calibration checks pass throughout lengthy ICP methods while maintaining optimum ICP performance.



NEWS

Other than a loss of sensitivity, a main concern that an ICP analyst has with utilizing a low nebulizer uptake rate is the length of time it takes to deliver the sample to the plasma and the time required to effectively wash out the previous sample. As mentioned previously, a fast pumping rate can be employed during uptake and post-acquisition rinse, but the fast rate can lead to an increased stabilization time and decreased lifetime of the peristaltic pump tubing. By utilizing the flow injection technology of the Niagara Plus, the "souped up" ICP configuration has reduced the total sampleto-sample time to 150 seconds from 270 seconds (Table 2). Even at a nebulizer flow rate of 300µL/min, the Niagara Plus allows the ICP analyst to cut the cycle time by 45%. The Niagara Plus, in combination with a lower nebulizer uptake rate, reduces the amount of sample introduced into the plasma by 85%.. This would provide testing laboratories analysing harsh samples, such as high solids. with a significant improvement in the lifetime of their consumables.

The Niagara Plus also provides superior washout at 300μ L/min with no post-acquisition rinse compared to the standard ICP set-up at a nebulizer uptake of 1mL/min and a 40 second post-acquisition rinse. The carryover data shown in Figure 3, displays the blank reading immediately flowing a 10ppm multi-element standard. An average carryover of 0.05% was achieved with the "souped up" ICP configuration. In order to achieve the same level of carryover obtained with the Niagara Plus, the standard ICP set-up required 50 seconds of post-acquisition rinse at a flow rate of 1mL/min. If the ICP was run at a nebulizer flow rate of 300μ L/min without a switching valve and no "fast pumping" it would require 350 seconds of post-acquisition rinse in order to achieve a carryover of 0.05% following a 10ppm standard.



Lastly, we wanted to compare the % recovery obtained at 300µL/ min with the "souped up" ICP conditions to the standard ICP at 1.0mL/min for a 50ppb and 1ppm standard. The results show that the "souped up" ICP achieves a % recovery between 98 and 109% (Figure 4a) for the 50ppb standard and between 98 and 102% for the 1ppm standard (Figure 4b). The recovery results for the "souped up" ICP are very comparable to the results achieved at 1mL/min, proving there is no degradation in ICP performance with the addition of the IsoMist and Niagara Plus CM.



Conclusion

This paper demonstrates that significant improvement in both performance and speed can be achieved on a commercially available ICP optical emission spectrometer simply by choosing the optimum sample introduction components and accessories. Adding the IsoMist and Niagara Plus CM to an ICP provides trace measurements using small volumes without sacrificing performance and significantly shortens the analytical cycle. The Niagara Plus provides improved washout and drastically reduces the amount of sample consumed and introduced into the ICP. This is beneficial to those with limited sample volumes and for those that want to reduce the exposure of their sample introduction components to harsh samples, such as those with high TDS. The IsoMist allows the ICP analyst to precisely control the transport efficiency and maintain the highest signal stability.

11670.7 279.

Standard ICP-OES

Element Wavelength

SoupedUp" ICP-OES

NEWS



References

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- 5. Glass Expansion Newsletter, Issue 15, February 2008, Spray Chamber Temperature: A Critical Parameter in ICP Experiments.
- 6. Glass Expansion Newsletter, Issue 20, October 2009, The Basics of Sample Transport into an ICP Spectrometer.
- 7. Glass Expansion Newsletter, Issue 23, October 2010, Improved Accuracy in the Analysis of Precious Metals by ICP-OES.





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:

"If sample flow rate is suspect for some reason, one way to monitor it would be with Glass Expansion's TruFlo Sample Monitor."

"It works exceptionally well. I have two units connected to ICPs and have saved a great deal of troubleshooting time because of them."

"A nice piece of kit to help with sample intro troubleshooting."

"I consider this an essential tool for every lab that uses ICP-OES, ICP-MS." "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. With the release of the new V2.0 software, users can now calibrate the TruFlo with their own samples. You now have the capability to create, edit, delete, import and export calibration profiles for specific solutions. This is particularly useful for those who routinely run non-aqueous solutions (eg. oil laboratories).

INSTRUMENT NEWS

From Agilent - On July 1 we correct the vision of Dual View

Deciding on an ICP-OES before then would be very short-sighted.

Agilent is the long-established innovation leader in atomic spectroscopy and in just a few short weeks we will continue that tradition. On July 1, 2014, we will introduce a revolutionary new dual-view ICP-OES instrument so advanced, so unlike anything else available, that you will never see "dual-view" the same way again.

Save time and money.

Run your samples faster, using less gas, without compromising performance - even on your toughest samples.

Achieve a new level of performance.

New innovative technologies will enable uncompromised axial and radial performance at the same time.

Simplify your workflow.

Intelligent hardware and software designed to take the guess work out of method development, so you get the right results the first time, every time.

Interested in learning more? <u>Complete the registration form and you'll be the first to know during the official introduction</u>. This is dual-view analysis as it is meant to be. You've watched and waited long enough.

From Analytik Jena - PlasmaQuant® PQ 9000 - Peak Performance

Analytik Jena proudly presents PlasmaQuant® PQ 9000 – the future generation of high-performance ICP-OES – designed to master the most difficult analytical challenges in atomic spectroscopy with superiority in terms of precision, flexibility and convenience.

PlasmaQuant® PQ 9000 is:

- smart bench-top instrument design
- innovative high-end technology
- impressive analytical performance
- competitive sampling efficiency
- a new generation in operator comfort
- premium quality made in Germany

Combining uncompromised plasma stability, vertical plasma geometry, advanced dual view plasma observation with unique resolving power and superior sensitivity.

PlasmaQuant® PQ 9000 permits robust routine analysis for a working range stretching from the low ppb to the high percentage. Its detection limits reaching into the parts per trillion are an innovation in ICP-OES.

PlasmaQuant® PQ 9000 is specialized in the analysis of matrix-rich samples (e.g. brine or petrochemicals) and line-rich spectra (e.g. metals and rare earth materials)

PlasmaQuant® PQ 9000 analyzes major, minor and trace elements in one measurement with minimal sample dilution

PlasmaQuant® PQ 9000 guarantees ultratrace detectability, ultimate precision and supreme long-term stability with high ease of use

PlasmaQuant® PQ 9000 simplifies the handling of complex samples, plasma torch and analytical data

More information at: www.analytik-jena.com; info@analytik-jena.com





From Horiba Jobin Yvon - High Resolution, High Sensitivity and Stability for the Most Challenging Applications with ICP-OES

HORIBA Scientific introduces the ULTIMA Expert ICP-OES Spectrometer, specially designed to combine ease of use, a unique plasma torch design, comprehensive tools, highest resolution and low detection limits for the most challenging applications.

ULTIMA Expert includes a unique plasma torch that offers radial viewing mode far superior to competitive systems, thanks to the measurement of the entire normal analytical zone. The vertical torch, the original sheath gas device and the wide injector make the ULTIMA Expert more tolerant to difficult matrices, and offer the benefits of robust operation with minimal maintenance.

High resolution, with less than 5 picometers for the UV range and less than 10 picometers for the visible range, and excellent sensitivity allow elements with high, low and trace concentrations to be measured accurately.

Powerful software, with unique comprehensive tools offers advanced user guidance from sample to results. Image Navigator software provides qualitative and semi-quantitative analysis, S3-base and MASTER for facilitated method development.

The robustness of ULTIMA Expert makes it ideal for demanding applications such as mining, salt production, wear metals in oil analysis, petrochemical, metallurgical and chemical manufacture.

For more information, go to: http://www.horiba.com/scientific/ products/atomic-emission-spectroscopy/ icp-oes-spectrometer/



CATALOGUE OF ICP-OES/ICP-MS SUPPLIES AND ACCESSORIES





Essential reference for all ICP-OES and ICP-MS users

The 2014 Glass Expansion catalogue of ICP-OES/ICP-MS Supplies and Accessories is now available. It is an essential reference for all users of ICP-OES and ICP-MS equipment. It includes hundreds of components covering the most prevalent ICP-OES and ICP-MS models from the major manufacturers.

Its 58 full-colour pages show nebulizers, spray chambers, torches, RF coils and ICP-MS cones by instrument model, plus general consumables and accessories.

The catalogue also includes information on unique Glass Expansion accessories, including the Assist CM syringe-driven sample introduction system, Niagara Plus CM productivity enhancement system, TruFlo sample monitor, IsoMist programmable temperature spray chamber, Eluo nebulizer cleaning tool, Capricorn argon humidifier and Trident internal standard kit.

To order your copy, click here or send an email to enquiries@geicp.com



A wide range of useful accessories is included The most suitable sample introduction components are displayed for each ICP model

