

Glass Expansion Newsletter | October 2011 | Issue 26

# **APPLICATION SPOTLIGHT**

### EVALUATION OF A NEW HIGH PERFORMANCE INERT NEBULIZER

### Introduction

Glass Expansion has been developing innovations for the ICP community since the early 1980's. Our dedication solely to the field of ICP spectrometry has focused all of our resources toward improving the quality and productivity of elemental analysis. In this article, a new concentric nebulizer constructed of PEEK (PolyEtherEtherKetone) will be evaluated.

Until recently, Glass Expansion offered three inert nebulizers as follows:

- OpalMist: Concentric nebulizer with high purity PFA construction; ideal for ICP-MS
- PolyCon: Concentric nebulizer with high rigidity polyimide construction, ideal for high precision ICP-OES with relatively "clean" samples
- VeeSpray: Modified Babington type nebulizer with all ceramic construction, recommended for "dirty" samples (high salts and particulates)

The goal of the DuraMist development project was to offer HF users a high precision yet rugged nebulizer approaching the performance of the SeaSpray concentric glass nebulizer. The rigidness of the PEEK material allowed for the DuraMist to be constructed with high physical reproducibility, which should provide good analytical precision and nebulizer to nebulizer reproducibility.

The DuraMist, shown in Figure 1, consists of a PEEK body and insert both of which can withstand up to 5% HF. The nebulizer has a standard 6 mm outside diameter, which fits the standard cyclonic spray chambers (an adaptor is available for Scott type spray chambers). The DuraMist is manufactured in four different configurations as shown in Table 1. The DuraMist is fitted with a UniFit sample connector line that slides easily over the sample arm of the nebulizer creating an excellent seal and zero dead volume connection and a quick-connect EzyLok connector for safe, easy, and effective connection to the Argon supply line.

Part number	Argon flow rate (L/min)	Natural uptake rate (mL/min)
ARG-07-DM04	0.7	0.4
ARG-07-DM1	0.7	1.0
ARG-1-DM04	1.0	0.4
ARG-1-DM1	1.0	1.0

Table 1: Configurations of DuraMist nebulizer

This report highlights experiments conducted on two different ICP-OES systems to compare the performance of the DuraMist to the SeaSpray, OpalMist, PolyCon, and a non-concentric polymer nebulizer, referred to as NCPN throughout the remainder of the discussion. Characteristics studied included sensitivity, precision, stability, robustness, and tolerance to high TDS (total dissolved solids).

## **GE NEWS**



### 2011 Catalogue

Our new 2011 catalogue has now been sent to everyone who requested it. However, please contact us if you would like an extra copy or if you know someone else who would like a copy.

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### **NEWS**



### **Experimental**

The first experiments were conducted on a Thermo ICAP 6500 Duo, operated under the conditions listed in Table 2. To avoid any fluctuations in measurements due to temperature variations, the IsoMist Programmable Temperature Spray Chamber was utilized with a Twister baffled glass spray chamber held constant at 21°C. A ceramic D-Torch was also used to avoid the deleterious effects of salt during the high dissolved solids experiments. Under these conditions the performance of the DuraMist was compared to the SeaSpray and a NCPN, examining precision and sensitivity.

The second portion of this report compared the performance of the DuraMist to the OpalMist and PolyCon. For these experiments a PerkinElmer Optima 2100DV was utilized, operating under the conditions listed in Table 3. As in the initial experiments, an IsoMist and ceramic D-Torch were employed. The common ICP figures of merit established by Poussel and Mermet were used to compare plasma robustness, atomization/ionization, excitation, efficiency, and stability for each of the three inert nebulizers. The tolerance to a 20% TDS solution was also examined. As a final test to complete the DuraMist evaluation, long-term stability was examined in the presence of a 2.5% NaCl solution.



Figure 1: DuraMist nebulizer, UniFit sample line, ractchet EzyLok connector, and EzyLok kit.

Thermo iCAP 6500 Operating Conditions				
RF power (W)	1350			
Plasma gas (L/min)	15			
Auxiliary gas (L/min)	0.2			
Nebulizer gas(L/min)	0.65			
Replicates	3			
Sample flush time (sec)	65			
Plasma view	Auto			
Max integration time (sec)	15			
Peristaltic pump rate (rpm)	37			
Pump tubing	Orange/white (0.64 mm i.d.)			

Table 2: Thermo iCAP 6500 Operating Conditions

PE Optima 2100 Operating Conditions			
RF power (W)	1400		
Plasma gas (L/min)	15		
Auxiliary gas (L/min)	0.2		
Nebulizer gas(L/min)	0.65		
Replicates	3		
Read delay (sec)	15		
Min integration time (sec)	1		
Max integration time (sec)	10		
Source equilibration delay (sec)	15		
Plasma view	Axial (Na 589 – Radial)		

 Table 3: PE Optima 2100 Operating Conditions

#### **Results**

The SeaSpray is the most popular nebulizer due to its excellent precision and sensitivity and ability to handle high TDS. In the first set of experiments the goal was to compare the performance of the DuraMist to the SeaSpray. We also compared its performance to that of a non-concentric polymer nebulizer. When comparing precision (Figure 2), the DuraMist has a slightly smaller range of relative standard deviation (RSD) values compared to the SeaSpray. The range in precision values of the NCPN is larger compared to both the DuraMist and SeaSpray, which is not unexpected due to its increased sensitivity to peristaltic pump pulsations.

The next step was to compare the sensitivity of the SeaSpray, DuraMist, and NCPN. In Figure 3 sensitivity measurements of the DuraMist and NCPN are normalized to the sensitivity measurements taken with the SeaSpray. On average the DuraMist is within 13% of the sensitivity of the SeaSpray and is slightly higher than the NCPN for almost all wavelengths examined.

In the second portion of this study the performance of the DuraMist is compared to that of the other two inert concentric nebulizers offered by Glass Expansion, the OpalMist and the PolyCon. In Figure 4 sensitivity measurements of the OpalMist and PolyCon are normalized to the sensitivity measurements taken with the DuraMist. On average the DuraMist shows a 25% higher sensitivity compared to both the OpalMist and PolyCon for a wide range of wavelengths.

The experiments for ICP figures of merit established by Poussel and Mermet are most commonly used to examine plasma robustness, atomization/ionization, excitation, efficiency, and stability (Table 4), all of which can be affected by nebulizer performance. The ICP figures of merit for the DuraMist, PolyCon, and OpalMist are compared in Figure 5 (a) and 5 (b). The Mg and Cr ratios are monitored to measure the robustness and atomization/ionization efficiency of the plasma. On the PE Optima 2100DV ICP the DuraMist shows a slightly higher robustness and atomization/ionization efficiency compared to the other two GE inert nebulizers.

The Zn 206/Ba 455 ratio is used to measure excitation efficiency of the ICP, which is virtually the same with all three inert nebulizers. As a measure of nebulizer efficiency, the RSD for Mg 285 is monitored; with the DuraMist showing the second highest efficiency and the OpalMist offering the highest efficiency. **NEWS** 

GLASS EXPANSION Quality By Design

The RSD for Ar 404, Zn 206, and Ba 455 lines are monitored to measure signal stability, with the lowest RSD being the most stable. The stability data varies from wavelength to wavelength. However, when averaging the stability for all three wavelengths together, the DuraMist offers the best stability.

In order to examine the TDS capability for each inert nebulizer a stress test was conducted. The conditions described below are not utilized during normal operation, where a rinse would typically be used after each sample was measured and the sample is typically pumped rather than naturally aspirated. So this is a worst case experiment designed to show the effects of high TDS quickly and to compare one nebulizer with another.







Figure 3: Comparison of measurement sensitivity to the SeaSpray.



OpalMist 
 PolyCon

Figure 4: Comparison of measurement sensitivity to the DuraMist.

Abbreviation	Test	Diagnostic
Mg-Mg	Mg280(II)/Mg285(I)	Robustness
Cr-Cr	Cr267(II)/Cr357(I)	Atomization/ionization
Zn-Ba	Zn206(II)/Ba455(II)	Excitation
Mg	Mg285 (I) RSD	Nebulizer efficiency
Ar	Ar404 (I) RSD	Stability
Zn	Zn206 (II) RSD	Stability
Ва	Ba455 (II) RSD	Stability

Table 4: ICP figures of merit<sup>1</sup>

b.











Figure 6: High TDS stress test (20% NaCl)



Figure 7: DuraMist precision and long term stability in the presence of 2.5% NaCl

To conduct the TDS stress test a 20% NaCl solution was aspirated for 20 minutes, during this test an ICP was not used. Throughout the 20 minutes the nebulizer gas flow for each nebulizer was monitored at constant pressure. The results depicted in Figure 6, show no change in the nebulizer gas flow for the DuraMist even with a 20% NaCl solution. Whereas the PolyCon begins to drop off after only a few minutes. The OpalMist shows only a small decrease in nebulizer gas flow for the first 15 minutes, but after 15 minutes the flow begins to drop off faster. These results show that of the three inert nebulizers tested, the DuraMist was the most tolerant of high TDS.

The TDS stress test demonstrated that the DuraMist has the capability to handle up to a 20% NaCl solution without clogging. In order to examine the performance of the DuraMist with a high TDS matrix, a multi-element solution was prepared in a 2.5% NaCl/2.0% HNO3 matrix and monitored over a period of 8 hours (Figure 7). The average RSD for a number of wavelengths remained < 0.5% for 8 hours, demonstrating that the DuraMist has the capability to run a high TDS sample without sacrificing performance.

### Conclusions

In summary the DuraMist is a concentric, self-aspirating inert nebulizer that provides excellent sensitivity and precision. Results show its ability to handle up to 20% TDS without clogging and excellent precision and long term stability in the presence of 2.5% NaCl. Data showed that the DuraMist had only slightly lower sensitivity than the SeaSpray, and out performed an NCPN in both precision and sensitivity. Although the OpalMist is still the preferred nebulizer for ICP-MS applications, we believe the DuraMist will gradually replace the PolyCon for ICP-OES application involving HF. Final specifications of the DuraMist are listed below.

#### **Specifications**

- HF resistant PEEK body and insert
- High TDS Tolerance: up to 20%
- Up to 75um particles (for larger uptake)
- 0.4 and 1.0mL/min uptake models
- Either 1.0 or 0.7L/min argon flow

#### Reference

1. E. Poussel, J.M. Mermet, and O. Samuel, Simple experiments for the control, the evaluation and the diagnosis of Inductively coupled plasma sequential systems, Spectrochimica Acta Part B, 48, 1993, 743-755.



## **NEBULIZER SELECTION GUIDE**





## **PRODUCT DESIGN FOCUS**

### **Inert Spray Chambers**

Early ICP spectrometers mostly used large volume (>100mL) "Scott style" spray chambers. These generally provided good stability and detection limits. However they had some limitations, including low efficiency and long wash-out times. The availability of the cyclonic spray chamber in the 1990's helped overcome these limitations.



With the cyclonic spray chamber, the aerosol is introduced tangentially. The aerosol flows in a vortex about the axis of the spray chamber and the larger droplets are eliminated by centrifugal action. These larger droplets strike the wall of the spray chamber and flow to the drain while the smaller droplets are swept into the torch by the argon stream.

Compared with the Scott style spray chambers, the newer cyclonic spray chambers are of lower volume (usually about 50mL) and provide improved efficiencies as well as faster washout times and reduced carryover from one sample to the next. At the same time, they are able to match the excellent precision of the Scott style spray chambers.

Both the Scott style and cyclonic spray chamber are usually made from borosilicate glass. Glass provides sufficient purity and chemical resistance to make it a cost-effective material for the majority of ICP applications. It also exhibits excellent drainage characteristics, leading to very good precision and low carryover. However, it does have some limitations. It is attacked by hydrofluoric acid (HF) and is therefore not suitable for the analysis of samples containing even low levels of HF. It is also not suitable for the ultratrace determination of some elements (eg. silicon, boron) by ICP-MS.

For applications where a glass spray chamber is not suitable, it has been common to use spray chambers made from various plastics. Again, the early plastic spray chambers were of the Scott style but more recent models are of the cyclonic design. A big problem with plastic is that the surface does not wet easily. This results in the formation of large drops, erratic drainage, poor precision and poor sensitivity. To reduce these problems, some form of surface treatment is necessary. One option is to sandblast the surface and this treatment was used by Glass Expansion for several years. It improves the performance significantly but the performance of sandblasted spray chambers is still markedly inferior to that of glass spray chambers.

A major breakthrough in the performance of polymer based spray chambers came with the introduction of the proprietary Stediflow surface treatment by Glass Expansion in 2006. The Stediflow treatment improves the wettability of the surface, ensuring efficient drainage, and delivering sensitivity and precision almost as good

as those achieved with a glass cyclonic spray chamber. The graph below shows the sensitivity obtained using a Teflon spray chamber with Stediflow surface treatment and a polypropylene spray chamber with sandblasted surface compared with a Tracey glass cyclonic spray chamber (Intensity = 1). Whereas the sensitivity with the polypropylene spray chamber is only about 50% of the sensitivity with the glass spray chamber, the sensitivity with the Teflon spray chamber is almost as good as that of the glass spray chamber.

Sensitivity of inert spray chambers relative to glass spray chamber (relative sensitivity = 1)



It is important to note that the Stediflow treatment actually changes the molecular structure of the polymer material. It is not a coating and it does not introduce any potential contaminants. We have standardized on PTFE (Teflon) for our inert ICP-OES spray chambers (Tracey TFE model) and, due to its higher purity, we use PFA for our inert ICP-MS spray chambers (Tracey PFA44 models). There is a Tracey TFE or Tracey PFA44 spray chamber with Stediflow treatment available for every ICP-OES and ICP-MS model.





# **NEW PRODUCTS**

#### Niagara Plus upgrade

The Niagara Plus reduces the time for a typical analysis by half, thereby doubling your sample throughput, halving your operating costs and significantly reducing your environmental footprint. A positive displacement pump rapidly fills a sample loop and the sample is delivered to the nebulizer via a proprietary switching valve. The internal standard is added automatically and the sample does not contact any peristaltic pump tubing. Since its release two years ago, the Niagara Plus has allowed numerous laboratories to save many thousands of dollars in costs. We have recently implemented several improvements to make the Niagara Plus even more effective.

**Seven-port valve.** The original 12-port valve has been replaced by a 7-port valve, simplifying the installation. If inline addition of internal standard is not required, the mixer is simply removed from the front of the valve, turning the valve into a 6-port system. (The original valve needed to be configured with or without internal standard in advance.)



**Netbook computer.** The Niagara Plus is now supplied with a Netbook computer which has the operating software pre-installed. This streamlines the installation by eliminating the need to install new software on a host computer. Of course, installation of the software on the host computer is still an option, if preferred.

**Pneumatic actuator.** In order to get the maximum time saving, it is important to have the valve close to the nebulizer. Some ICP models have the nebulizer inside the torch compartment. It is not feasible to have an electrically-operated valve inside the torch compartment due to the high levels of RF radiation. For these models, an optional pneumatically operated valve is used.

For more information on the Niagara Plus, check our website at **www.geicp.com** or send an email to **enquiries@geicp.com**.

# **INSTRUMENT NEWS**

### From Bruker - New Quantum software package

Bruker has released the new 'Quantum' software package for its ICP-MS product range. With Quantum comes a range of new features, making your Bruker ICP-MS even easier to use. Now offered as standard on Bruker's recently released aurora M90 model, some of the new software capabilities include:

- Full support of existing ex-Varian and Bruker ICP-MS systems
- Complete automation of the 'aerosol dilution' technique allowing direct analysis of complex samples
- Multiple language support including English, Chinese, Japanese, German and French
- Nitrox-500 gas accessory control for online addition of  $\rm N_{2}$  and  $\rm O_{2}$  to plasma gases
- Operation under Microsoft's Windows 7 operating system
- Touchscreen compatible for greater ease-of-use

As a bonus, existing Varian/Bruker ICP-MS users who upgrade to Quantum will receive a new 'aurora M90 style' sample introduction tray (in dark grey).

To order your copy of Quantum software, contact your local Bruker representative. http://www.bdal.com/offices.html

Bruker brings in a new era in ICP-MS with the recently released aurora M90. To learn more about the aurora M90, check out our YouTube video explaining the innovative technologies that make the aurora M90 a powerful, yet easy-to-use system, no matter what your application.

Available in English and Chinese.

http://www.youtube.com/watch?v=mCgCqqNxa80 (English) http://www.youtube.com/watch?v=58wtFPJYxR8 (Chinese)

### From Spectro – High-Performance SPECTROBLUE ICP-OES Spectrometer Offers Simplified Operation, Greater Stability and Lower Cost

SPECTRO Analytical Instruments, a leading supplier of analytical instruments for optical emission and X-ray fluorescence spectrometry, has introduced the SPECTROBLUE, an ICP-OES that sets new benchmarks among compact, mid-range spectrometers for simplified operation, low maintenance and affordability.

"SPECTROBLUE ICP-OES is the ideal system for environmental laboratories that require a low-cost, high-throughput spectrometry solution. SPECTROBLUE was designed to establish new levels of performance for common laboratory analysis where stability, uninterrupted operation and throughput are as important as sensitivity and resolution. It couples an ultra-reliable design with no-compromise technical innovation to achieve the lowest cost of ownership in its class," comments Olaf Schulz, Product Manager ICP-OES.

#### **Robust Generator Handles Extreme Plasma Loads**

A robust generator design packs an ample power reserve that can handle the most extreme plasma loads. The heat from the generator's high-power ceramic tube is eliminated through the use of innovative air-cooled technology that alleviates the need for an expensive external water-based cooling system.

### Innovative Optics Eliminate Consumable Purge Gases, Ensure Long-Term Stability

SPECTROBLUE's optic system and unique full-transparency UV-PLUS approach eliminate consumable purge gases and ensure excellent long-term stability, adds Olaf Schulz. The instrument's sealed optic system abolishes gas purging for a lifetime savings totaling up to one-third of the instrument's purchase price. Additionally, SPECTRO's innovative Paschen-Runge optic technology permits a direct, high-luminance path for maximized light throughput, allowing the system to more easily process linerich spectra, improve measurement accuracy and reduce expensive rework.