



GLASS EXPANSION NEWSLETTER

Quality By Design

June 2008

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- Tolerance to total dissolved solids (TDS)
- Tolerance to particulates
- Tolerance to hydrofluoric acid (HF)
- Precision
- Purity
- Robustness
- Financial investment

In short, you should select the nebulizer with the lowest price tag that meets all of the requirements of the application. Table 1 below summarizes the characteristics of all of the Glass Expansion nebulizers.

Environmental Waters

Our Conikal concentric glass nebulizer is used in hundreds of environmental laboratories because it provides excellent precision and transport efficiency, is cost effective, and quite robust in comparison to other concentric glass nebulizers. The robustness results from the unique VitriCone™ design of all Glass Expansion concentric glass nebulizers as shown in Figure 1 (page 2).

The VitriCone channel is created by machining constant bore heavy stock glass tubing to create the desired aerodynamic exterior while maintaining a consistent internal diameter throughout its length. Because of this, the Conikal nebulizer can tolerate dissolved solids up to about 5%, depending upon the solubility of the salts.

APPLICATION SPOTLIGHT

ICP Nebulizer Selection and Maintenance

We are often asked which is the best nebulizer to use for a specific application. This article will attempt to answer that question for a variety of common applications as well as address the proper maintenance of nebulizers in order to achieve optimum performance and durability. To select the most appropriate nebulizer, you should consider a number of characteristics as follows:

Table 1. Comparison of Glass Expansion Nebulizers

Nebulizer	TDS (%)	Particulates (um)	HF	Precision	Purity	Robustness	Material
Conikal	5	75	No	High	Good	Good	Glass
SeaSpray	20	75	No	High	Good	Good	Glass
MicroMist	5	75	No	High	Good	Good	Glass
Slurry	5	150	No	High	Good	Good	Glass
OpalMist	30	75	Yes	High	Excellent	Good	PFA
PolyCon	5	75	Yes	High	Good	Very Good	Polyimide
Quartz VeeSpray	40	300	No	Moderate	Very Good	Very Good	Quartz
Ceramic VeeSpray	40	300	Yes	Moderate	Good	Excellent	Ceramic

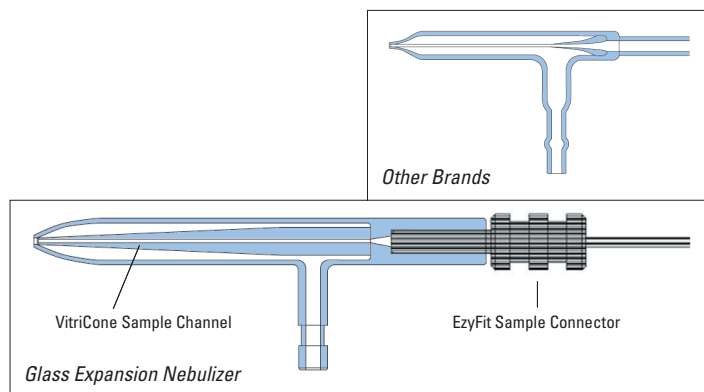


Figure 1. Rugged VitriCone design for Glass Expansion concentric nebulizers

Wastewater and soil extracts

These samples are very similar to the environmental waters with one exception; they are likely to contain much higher concentrations of dissolved solids. Because of this, we recommend going from the Conikal to the SeaSpray nebulizer. Although identical in appearance to the naked eye, the SeaSpray nebulizer has a recessed VitriCone channel that creates a self-washing action making it even more tolerant to dissolved solids. Again depending upon the specific salts, the SeaSpray nebulizer can tolerate up to 20% dissolved solids.

Soil analysis for agricultural applications

In this case, we are dealing with samples that are partially digested with acids and contain high concentrations of dissolved solids and, very likely, particulates. Typically, a soil lab runs hundreds of samples per day and is interested in achieving both speed and ruggedness. Precision and accuracy are usually less of a concern. A quartz VeeSpray nebulizer (Figure 2) is ideal for this application due to its tolerance of both high dissolved solids and particulates. Since the VeeSpray nebulizer is a modified Babington design, it is not self-aspirating and can be operated at very high uptake rates without degrading its performance. Faster uptake translates into faster filling and voiding of the sample lines leading to a shorter analysis cycle.



Figure 2. Quartz VeeSpray nebulizer

Geological samples

Geological samples often contain refractory oxides which require HF as part of the digestion process. Depending upon the level of completion of the digestion, particulates may also be present as a sediment. So in this case, we need a nebulizer that can handle high dissolved solids, particulates and HF. The ceramic VeeSpray answers the

call (Figure 3). Made of rugged alumina ceramic, this nebulizer is otherwise identical to the quartz VeeSpray.



Figure 3. Ceramic VeeSpray nebulizer

Another nebulizer which is often used for this application is the OpalMist (Figure 4), but only when the digestion is taken to completion and total dissolution is achieved. It is made of inert PFA and can tolerate up to 30% dissolved solids. However, it is limited in its ability to tolerate large particulates.



Figure 4. OpalMist inert nebulizer

Wear metals in used engine oils

This sample matrix varies in viscosity but is usually diluted in kerosene by at least a factor of five and more commonly a factor of ten, resulting in a more consistent matrix. The presence of metals in the used oils provides an indicator of wear of the engine and the particular metals found provide clues as to the component that is wearing. The metals exist as particulates in the oil providing a dilute slurry of very small particles (typically less than 1 micron). Therefore, we recommend the Slurry nebulizer for this application. Some analysts also like to use a V-Groove nebulizer such as our quartz VeeSpray. The VeeSpray can indeed handle samples with particulates but we believe this is overkill for this application and yields inferior performance. The Slurry nebulizer is ideally suited to handle this type of sample and is less expensive than a VeeSpray nebulizer.

Radioactive samples

A major concern for the analysis of radioactive samples is the disposal of "hot" waste. At typical sample uptake rates of 1 to 2 ml/min, only 2 to 5% of the sample is transported into the torch while 95% goes to waste. For radioactive samples, this waste is particularly expensive to dispose of. A micro concentric nebulizer can be employed to minimize waste generation. As the ratio of gas flow to liquid uptake increases, so does the transport efficiency of the nebulizer. Figure 5 (page 3) shows the transport efficiency taken at varying uptake rates and a constant gas flow. As shown, when sample uptake dips below 0.2ml/min, the efficiency increases rapidly. For this reason, a MicroMist nebulizer (Figure 6 - page 3) designed to operate at 0.1ml/min provides

minimal waste and adequate intensity. Previous work has shown that heating the spray chamber allows even lower uptake rates for more efficient transport without sacrificing intensities¹. This approach is compatible with any application with limited sample volume, such as many forensic or clinical applications.



Figure 6. MicroMist low uptake concentric glass nebulizer

Determination of ultra trace impurities by ICP-MS

The low detection limits attainable by ICP-MS make it the instrument of choice for applications requiring the quantitation of ultra trace impurities. One such application involves the semi-conductor industry where impurities limit the effectiveness of a chip. We recommend the OpalMist line of nebulizers when this is the case. The OpalMist is composed of high purity perfluoroalkoxy (PFA) resin and is available in a range of sample uptake rates from 10 $\mu\text{L}/\text{min}$ to 2 mL/min . It is a self-aspirating nebulizer like other concentric types and can be used either with or without pumping. For high purity applications, it is often advisable to avoid the use of pump tubing as it may be a source of contamination.

Analysis of refractory metals and alloys

These difficult to digest samples usually require HF to achieve complete dissolution and are often digested with the aid of a microwave system using elevated pressures and temperatures. The accuracy of these determinations

is usually quite demanding since both impurities and constituent elements can have a crucial influence on the properties of the alloy. The PolyCon nebulizer (Figure 7) is often a good choice because of its HF resistance and durable construction, which provides consistently precise measurements. The PolyCon nebulizer body and internal capillary are both composed of rigid polyimide polymer enhancing the precision of aerosol production.



Figure 7. PolyCon nebulizer

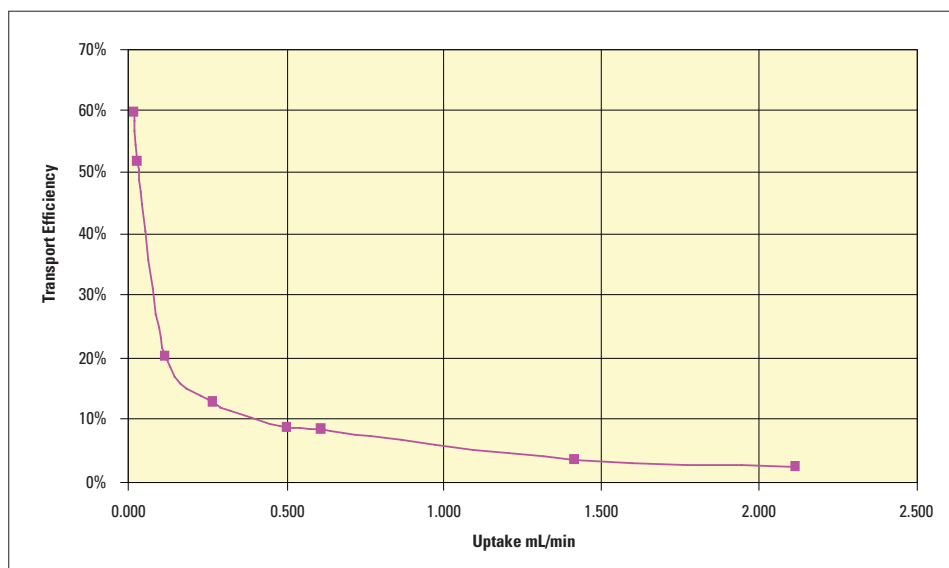
NEBULIZER MAINTENANCE

The lifetime of a nebulizer can be prolonged by proper maintenance. Maintenance recommendations vary slightly depending upon the type of nebulizer. First, here are some practices to be avoided.

- Do not insert anything through the orifice of the nebulizer. This is most likely to damage the nebulizer beyond repair.
- Do not use any concentration of HF to clean a glass or quartz nebulizer or a spray chamber when a glass nebulizer is installed. Even dilute HF can alter the orifice of the internal capillary and deteriorate the performance of the nebulizer.
- Do not place a glass nebulizer in an ultrasonic bath as it may dislodge the internal capillary and render the nebulizer useless.

The following procedures are recommended:

Figure 5. Nebulizer transport efficiency vs. uptake rate



Concentric glass nebulizers (Conikal, SeaSpray, Slurry, and MicroMist)

- At the end of each day's run, aspirate dilute acid or deionized water for 15 minutes prior to shutting down the instrument. Allowing a nebulizer to dry with a deposit at the tip can result in residue build up that can deteriorate performance and shorten its life.
- Periodically (weekly or monthly depending upon the complexity of the sample matrix) back flush the nebulizer. We recommend using the Eluo nebulizer cleaning tool (Figure 8) to lock the nebulizer in place while back flushing with methanol. Methanol is effective at removing both organic and inorganic deposits.



Figure 8. Eluo nebulizer cleaning tool for glass nebulizers

- For salt deposits, Fluka RBS-25 is very effective. Soak the nebulizer in a beaker of a 25% concentration of this solvent overnight. This procedure should be carried out whenever you observe a deterioration in precision.

Polymeric nebulizers (OpalMist and PolyCon)

These nebulizers can be treated the same as the glass nebulizers with the following distinctions.

- A separate Eluo tool is required to clean these nebulizers due to the difference in the gas arm. We call this tool an Eluo-OP as shown in Figure 9.



Figure 9. Eluo-OP for the OpalMist and PolyCon nebulizers

- These nebulizers can be repaired at our factory in ways that the glass nebulizers cannot. Often it is just a matter of removing a clog or in some cases replacing the internal capillary, both of which are very economical ways of repairing this type of nebulizer.

V-Groove nebulizers (quartz and ceramic VeeSprays)

These nebulizers are quite rugged and usually require only minimal maintenance. It is still advisable to run dilute acid or deionized water through them prior to shutting down the instrument. If you do get salt deposits, soak in Fluka RBS-25 as above.

(1) J. Dulude, V. Dolic, and R. Stux, *Spray Chamber Temperature: A Critical (yet often missing) Parameter in ICP Experiments*, Paper # TH16 at the Winter Conference on Plasma Spectrochemistry, Temecula, CA, January, 2008.

NEW PRODUCTS

D-Torch for Thermo Fisher iCAP 6000

The D-Torch is a new demountable torch design that provides the benefits of a fully demountable torch at a significantly lower cost. Interchangeable outer tubes made of quartz or ceramic are available. The ceramic outer tube is of particular benefit with oils analysis, where quartz outer tubes often suffer from short lifetime. The ceramic outer tube has a much longer lifetime, greatly reducing interruptions and downtime due to torch failure. Click on the following link for more information on the D-Torch: http://www.geicp.com/cgi-bin/site/wrapper.pl?c1=Products_torches_dtorch.

The D-Torch is now available for the Thermo Fisher iCAP 6000, in addition to the PerkinElmer Optima 4300/5300/7300V announced previously. We will soon be releasing the D-Torch for other ICP models. Please contact enquiries@geicp.com for information on the availability of the D-Torch for your ICP.



D-Torch for Thermo Fisher iCAP 6000

Products to suit the PerkinElmer Optima 7000 Series

Glass Expansion offers a wide range of sample introduction components for the new Perkin Elmer Optima 7000 Series. You can see the full range through the following links to our website:

For the Optima 7000 Series DV:

http://www.geicp.com/cgi-bin/site/wrapper.pl?c1=Products_byinstrument&inst=Perkin%20Elmer%20Optima%204300%20DV%20Series

For the Optima 7300 V:

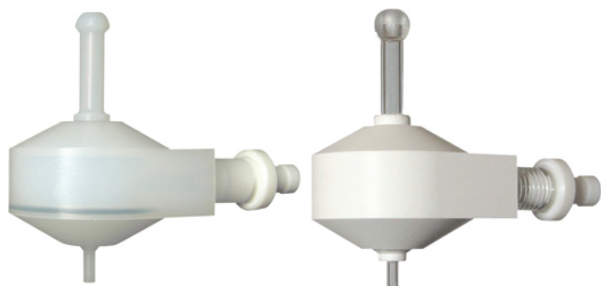
http://www.geicp.com/cgi-bin/site/wrapper.pl?c1=Products_byinstrument&inst=Perkin%20Elmer%20Optima%204300V

IsoMist Upgrade

Since its release last year, the IsoMist Programmable Temperature Spray Chamber has helped many ICP users to improve their analyses. However, we have had several users who want to be able to interchange PFA and glass spray chambers in the same IsoMist. It is not possible to do

this with the original IsoMist design but we have responded to this feedback by modifying the design.

With the new IsoMist (Version 2), you can swap between glass, quartz and PFA spray chambers in the same IsoMist. The new Version 2 is actually the same as the original design for the PFA spray chamber. We have changed the shape of the encapsulation for the glass and quartz spray chambers so that they fit in the same housing as the PFA spray chamber.



Interchangeable PFA and encapsulated glass spray chambers for the IsoMist

INSTRUMENT NEWS

From PerkinElmer:

NEW GENERATION OPTIMA 7000 SERIES ICP-OES

PerkinElmer Life and Analytical Sciences, a global leader in application-focused measurement and analysis solutions, has announced the Optima™ 7000 Series of ICP-OES. The Optima ICP-OES continues to evolve to meet changing customer needs. The Optima 7000 family is designed for best-in-class inorganic analysis, and is used in a variety of markets, including environmental, geochemical, product testing and biofuels. This newest generation in the Optima series includes a Universal Data Acquisition mode that records all of the spectral data for each sample. This enables customers to retrieve data that was not initially reported without needing to run the sample again, saving them time and increasing productivity.

The Optima 7000 has several time-saving enhancements for laboratory personnel. For rapid review of results, the Optima 7000 generates an error flag if a sample result falls above or below a user-specified value. To ensure a consistently high level of data and without requiring additional resources, the instrumentation can automatically generate Quality Control (QC) charts that show results for analyses run over a specified period of time.

The Optima 7000 Series replaces the Optima 2100, 5100, 5200 and 5300 models. For more information visit, www.perkinelmer.com/optima.

From Spectro:

NEW APPLICATION REPORT

The new application report “Analysis of Molybdenum oxide by ICP-OES with Axial Plasma Observation” documents the analysis of extremely pure molybdenum oxide, which is used for catalyst applications or in electronic components. Extremely pure transition elements – with a purity of up to 99.999 percent, for example – were, until now, considered to be extremely difficult or even impossible to analyze with ICP-OES. The ARCOS proves otherwise: It accurately determines the contents of all of the trace elements in such a sample in less than three minutes. An excellent recovery rate was determined for the analysis of reference materials. Olaf Schulz, Product Manager for ICP OES at SPECTRO, explains: “The optical system of the ARCOS achieves a very high resolution. It is 8.5 picometers in the wavelength range between 130 and 340 nanometers; 15 picometers above 340 nanometers. This represents the best resolution available, over the widest spectral range. The influence of spectral interferences is strongly reduced; accuracy and detection sensitivity effectively improved.” Important for SPECTRO customers: “The procedure described in the report can also be utilized for the examination of other subgroup elements,” reports the Product Manager. Obtain the application report, free-of-charge, per email spectro.info@ametec.com.

From Thermo Fisher:

DELIVERY OF 1000TH iCAP 6000 SERIES ICP EMISSION SPECTROMETER

Thermo Fisher Scientific Inc., the world leader in serving science, announces that the Plansee Group has made the 1000th purchase of the Thermo Scientific iCAP 6000 Series of ICP emission spectrometers. The Plansee Group, the world's leading manufacturer of refractory metal and composite materials products, has selected the iCAP 6500 Duo to perform R&D and quality control analyses of their specialized metal alloys at the company's production facility in Austria. Utilizing the latest design techniques and innovative new technologies, the compact iCAP 6500 Duo is a powerful system, achieving maximum levels of flexibility, performance and automation equal to the exacting demands of the busiest laboratories.

The Plansee Group required a system that was capable of distinguishing silica from tungsten background at low levels and achieving specific detection limits on silicon and potassium. The system had to have the inherent capability of performing accurate trace analyses in challenging alloy matrixes, including tungsten, molybdenum, titanium, niobium, tantalum, chromium, alumina and zirconium compounds as well as in environmental samples. Since its deployment at the Austrian plant, the iCAP 6500 Duo has

been able to detect the exact amount of impurities in even the most difficult alloy matrixes at low ppm levels. Analysis time has been considerably decreased for all elements.

For more information on the Thermo Scientific iCAP 6000 Series of ICP emission spectrometers, please email analyze@thermofisher.com or alternatively visit www.thermo.com/elemental.

HINTS FOR THE OPERATOR

Is Your Sample Uptake Rate Too High?

Many ICP operators are wasting sample and reagents by using high sample uptake rates which do not provide any analytical benefit. Traditionally, most ICP-OES analyses have been carried out with sample uptake rates in the range 2 – 3mL/min. There are several historical reasons for this:

- Early sample introduction systems were very inefficient and a relatively high sample uptake was required in order to obtain acceptable sensitivity.
- Reliable low-uptake nebulizers were not available.
- Detection limits were believed to be better with higher uptakes.
- Most operators used the ICP manufacturer's default conditions which almost invariably included a high sample uptake.

There are significant benefits to be obtained if lower uptake rates could be shown to be feasible:

- Smaller sample volumes could be used, leading to major

savings in reagent costs and the cost of standards.

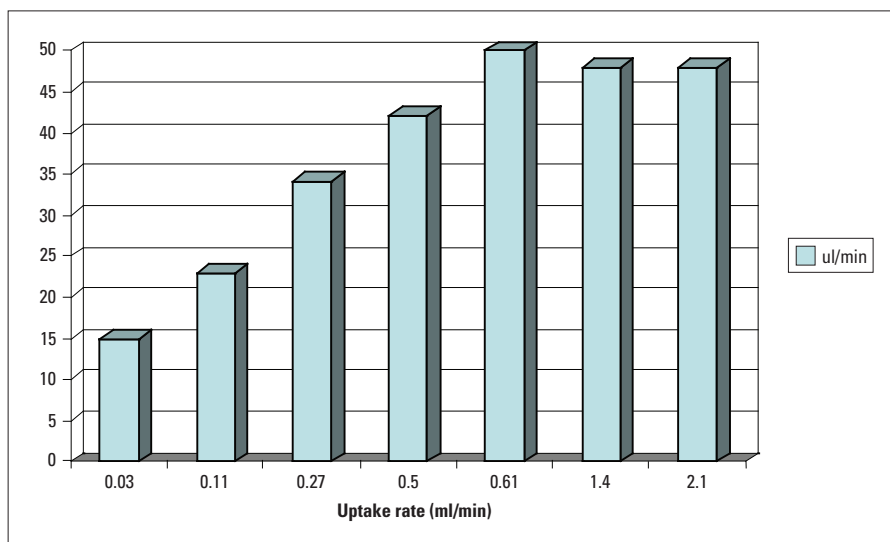
- The volume of waste could be reduced, leading to reduced waste disposal costs and obvious environmental benefits.
- The plasma is usually more stable with lower sample uptake.

With Glass Expansion's state-of-the-art sample introduction systems, the historical reasons for high sample uptakes have largely been eliminated, enabling the benefits of lower uptakes to be realized. The combination of concentric nebulizer and cyclonic spray chamber is much more efficient than the old cross-flow nebulizer with Scott-style spray chamber. Also Glass Expansion now offers a full range of low-uptake nebulizers which provide the same reliability and precision as the higher-uptake nebulizers. This range includes the MicroMist, SeaSpray, OpalMist and PolyCon. See the Application Note section of this newsletter for a detailed nebulizer comparison.

Furthermore, experiments carried out in our laboratory have shown that the sample uptake can be reduced by around a factor of 4 with minimal or no deterioration in performance. There is a large improvement in sample transport efficiency as the sample uptake is reduced. This means that there is very little reduction in the amount of sample that actually reaches the plasma. Figure 1 shows that there is only a minimal drop in the amount of sample reaching the plasma when the uptake rate is reduced from 2.1 to 0.5mL/min. This means that, for most applications, the same analytical performance can be obtained with a 0.5mL/min uptake as a 2mL/min uptake.

Some ICP users are reluctant to reduce the sample uptake rate because it extends the time between sample measurements, thereby reducing the sample throughput.

Figure 1. Amount of sample reaching the plasma as a function of sample uptake rate



However this problem is easily overcome by using smaller diameter sample and pump tubing. Glass Expansion supplies a range of sample tubing and pump tubing to cover all requirements.

GLASS EXPANSION NEWS

NEW REGIONAL MARKETING MANAGER

Brian Boyd has joined our Australian office as Regional Marketing Manager. He started his career as an applications chemist at Varian and since then has been working in mining, metals and environmental laboratories in Australia, UK and Canada. He has a wealth of experience and will enhance our ability to solve problems for ICP users. Brian can be contacted at bboyd@geicp.com.

JAIMA SHOW 2008

A full range of Glass Expansion products will be on display at the JAIMA Show, Tokyo, Japan, September 3 to 5, 2008, Booth 7A-206 and Glass Expansion specialists will be on hand to assist you.

FACSS 2008

Our products will also be on display at the 35th FACSS Conference, Reno Nevada, USA, September 28 to October 1, 2008, Booth 3. You are invited to visit our booth and discuss your ICP application with our specialists.