



GLASS EXPANSION

Quality By Design

APPLICATION SPOTLIGHT

ICP Nebulizer Selection and Maintenance

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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:

- 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.

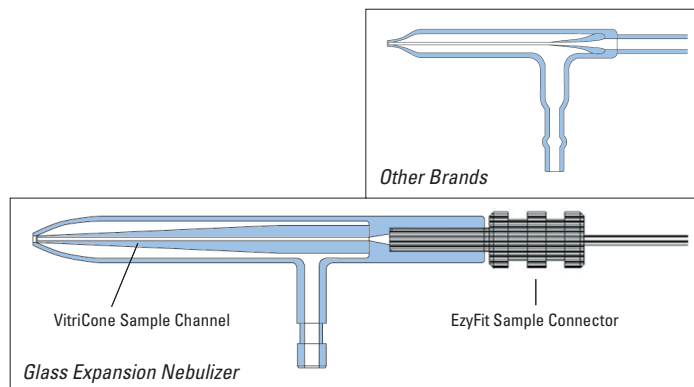


Figure 1. Rugged VitriCone design for Glass Expansion concentric nebulizers

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.

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

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

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) 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 ul/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:

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.



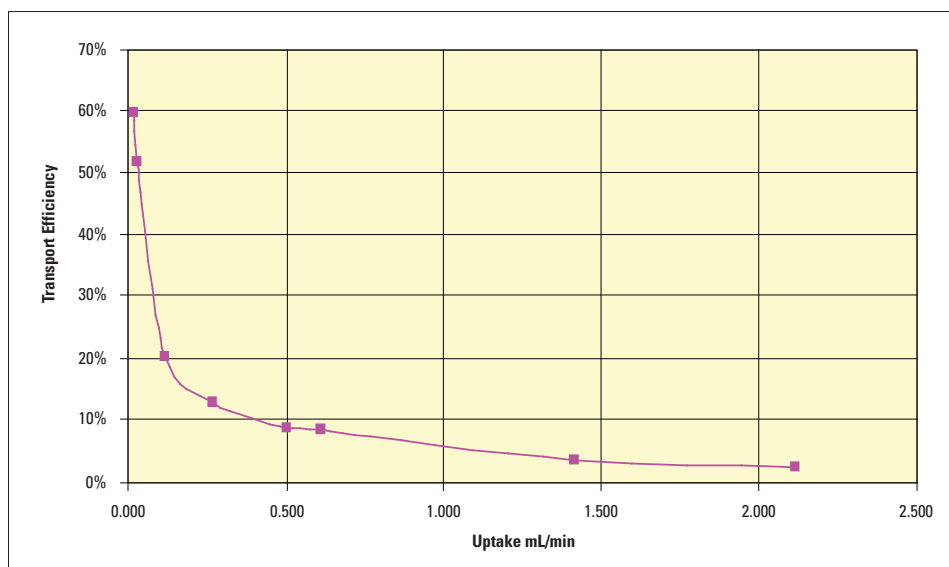
Figure 8. Eluo nebulizer cleaning tool for glass nebulizers

- 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

Figure 5. Nebulizer transport efficiency vs. uptake rate



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.

REFERENCES

(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.

INTERNATIONAL

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