A Guide to Choosing Nebulizers and Spray Chambers for your ICP-OES.

Introduction

There are many different nebulizers and spray chamber combinations available for your ICP-OES and choosing the right one can be difficult, confusing and costly if you get it wrong. To help de-mystify nebulizer and spray chamber selection, we have produced a comparison using the most common figures of merit: sensitivity, short-term precision, signal to root background (SRBR), price and washout for our most popular products.

Spray Chambers

There are two main types of spray chambers used with ICP-OES – the cyclonic spray chamber and the Scott spray chamber. The cyclonic spray chamber was introduced to the ICP world by Glass Expansion in 1989. The Scott spray chamber dates from the origins of ICP in the early 1970’s. Both cyclonic and Scott spray chambers are made from various materials, which include borosilicate glass, quartz or a polymer such as PTFE, PFA or Ryton.

The material of the spray chamber is often chosen based on the tolerance to the sample matrix. For example, when analysing solutions containing hydrofluoric (HF) acid, a polymer spray chamber is recommended. If your laboratory analyse samples containing HF, please refer to our July 2018 Newsletter for a detailed application note focused on inert sample introduction systems for ICP-OES applications.

Cyclonic spray chambers are available in two configurations, a single-pass or double-pass. The cyclonic spray chamber is overwhelmingly the most popular design. It is most often the standard option found on virtually every new ICP sold. In this article we highlight the design advantages and performance of two Glass Expansion cyclonic spray chambers, the Tracey™ and Twister™, in addition to a non-Glass Expansion Scott-style design to help guide your ICP-OES spray chamber selection process.

A detailed evolution of Glass Expansion’s cyclonic spray chamber designs were summarized in a 2014 article, and most recently the release of the Helix CT (constant torque) spray chamber. These design innovations have provided sensitivity gains, reduced washout times and reduced matrix effects not possible with other spray chambers.

The Tracey cyclonic spray chamber is a single-pass design (50 mL volume), manufactured from high-quality borosilicate glass. It provides the best sensitivity and lowest memory effects for standard ICP analyses.
The Twister cyclonic spray chamber is a double-pass design (50 mL volume), also manufactured from high-quality borosilicate glass. The many unique design features of the Twister design are highlighted in Figure 1. The most important feature of the Twister is the central transfer tube or baffle. This acts as a secondary droplet filter to reduce the mean droplet size, which in turn reduces matrix effects and improves precision.

For ICP-OES instruments with a solid-state detector (as virtually all are now), the most reliable figure of merit for detection limits is the signal to root background ratio (SRBR). SRBR gives a quick, reliable guide to the detection limits of shot noise limited elements such as As, Pb and Se which are the hardest elements to measure by ICP. With SRBR, the higher the value, the lower the detection limits. Measuring detection limits with multiple solutions with large numbers of replicates is slow with significant statistical variation.

As shown in Figure 2, the Tracey spray chamber provides the best SRBR and hence lowest detection limits. Closely followed by the Twister spray chamber and finally the Scott spray chamber, providing the lowest SRBR and potentially poorest detection limit performance.

Application Spotlight

Figure 3 shows the Twister spray chamber reaches 4 order of magnitude (0.1 ppm) washout in 36 seconds whereas the Scott spray chamber is still above 0.1 ppm at 60 seconds. The cyclonic action of nebulizer gas in the Twister spray chamber effectively sweeps analyte away from the nebulizer tip ensuring good washout.

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Figure 2. Signal to Root Background ratio of 5 ppm Mn for the Tracey, Twister and Scott spray chamber with a SeaSpray nebulizer operated at 0.7 L/min nebulizer gas.

Figure 3. Washout comparison of a 1000 ppm Mn in a Twister and Scott type spray chamber.
Whether you choose the Tracey (single-pass) or Twister (double-pass) spray chamber depends upon the sample matrix being analysed and the efficiency of the RF generator in the ICP. Typically, the higher sensitivity Tracey (Figure 4) is preferred for the lowest detection limits, especially with "clean" samples, such as drinking water. The Twister design is preferred for the analysis of organics and samples with higher total dissolved solids (TDS). For ICP-OES analyses requiring the use of very volatile solvents a cooled cyclonic spray chamber is recommended, such as the IsoMist XR.5

Firstly, let’s examine the differences in nebulizer design. All glass concentric nebulizers from Glass Expansion feature the unique VitriCone sample capillary (see Figure 6). Non-Glass Expansion designs typically use a hand-drawn glass sample capillary. With hand drawn sample capillaries, the internal diameter can vary preventing a laminar nebulizer flow and creating points where particulates may lodge. The Glass Expansion VitriCone sample capillary is entirely different. It is machined from a thick-walled glass capillary, providing a highly reproducible geometry and constant internal diameter – from sample inlet to tip. As it is a substantial capillary, it resists harmonic vibration caused by the high velocity Ar flows, giving the best short-term precision. The tightest manufacturing tolerances possible with the machined VitriCone sample capillary ensure consistent analytical performance from one nebulizer to the next, saving the ICP laboratory valuable time.

Nebulizers

The two most common types of nebulizer designs used with ICP-OES instruments today are – concentric or parallel path (or v-groove). With concentric nebulizers being the most popular as they generally provide the best analytical performance. Parallel path nebulizers are typically used when there are large undissolved particulates in the sample. Glass Expansion offers the largest range of concentric nebulizer designs by any manufacturer, each model having a variety of applications for which it is ideally suited. In this study we compare the performance of our most popular concentric nebulizer designs for ICP-OES applications; the SeaSpray, Conikal, DuraMist and Slurry nebulizer. A parallel path or V-groove nebulizer from another vendor is included as a comparison, providing a complete selection guide based on performance and cost of ownership.

Application Spotlight

Figure 4. Comparison of 5 ppm Mn Intensity for Tracey, Twister and Scott spray chambers with a SeaSpray nebulizer operated at 0.7 L/min nebulizer gas.

![Mn Intensity Graph](image1)

Figure 5. Short term precision of Mn for the Tracey, Twister and Scott spray chamber with a SeaSpray nebulizer operated at 0.7 L/min nebulizer gas.

![Short-Term Precision Graph](image2)

VitriCone capillary

A. Glass Expansion SeaSpray nebulizer

Self-washing tip

UniFit Sample Connector

VitriCone Sample Channel

Direct Connection (DC) Gas Line

B. Other brands

![Diagram of alternate nebulizer design](image3)
All Glass Expansion UniFit concentric nebulizers also feature a Direct Connection (DC) gas line (see Figure 7). Providing an inert metal-free, instrument specific gas line. The reliable ratchet fitting to the nebulizer ensures a leak-free gas connection, maintaining optimal backpressure for consistent day-to-day nebulizer performance.

The SeaSpray nebulizer is glass concentric nebulizer that features a uniquely engineered self-washing tip with smooth surfaces to avoid build-up of salt crystals. It provides outstanding nebulization efficiency and excellent tolerance up to 20% dissolved solids to give the best detection limit performance of the nebulizers here, even in difficult samples.

The Conikal glass concentric nebulizer is a great value, high precision nebulizer for routine analysis of aqueous and organic samples.

The HF resistant DuraMist concentric nebulizer is made of inert PEEK materials. It is highly sensitive, with excellent short-term precision and the highest tolerance to dissolved solids of any concentric nebulizer. It is a great all-rounder and the choice for analysis of diverse sample types.

The Slurry glass concentric nebulizer is designed for analysis of slurries, suspensions and wear metals. It provides the excellent sensitivity and short-term precision of a glass concentric nebulizer but is tolerant to undissolved particles of up to 150 um in diameter.

Parallel path nebulizers vary greatly in cost and performance. The large bore capillary is designed to resist blockages, but results in larger droplets and a wider drop size distribution, effecting both efficiency and precision. Parallel path designs also typically suffer from poorer reproducibility in performance from one nebulizer to the next.

Short-term analytical precision is one of the most critical figures of merit for an ICP-OES. With short-term precision (%RSD) the lower the figure, the better, as there is less variability in the analytical signal. In Figure 9 the SeaSpray nebulizer shows the best short-term precision of 0.26% RSD. Whereas, the parallel path V-groove nebulizer gives the worst precision at 1.3% RSD. Due to the design of the V-groove nebulizer, precision can be improved at higher sample flow rates (1.5 to 2 mL/min). However, higher sample flow rates are not desirable in many ICP applications due to plasma stability issues, limited sample volumes or autosampler capacity (higher sample flow rates require larger sample tubes, limiting the number of samples that can be loaded on an autosampler).

Price

As we can see in Figure 8, the Conikal nebulizer provides the lowest cost, while the parallel path and “universal” DuraMist nebulizer are the most expensive.
Detection Limits

As shown in Figure 10, the SeaSpray nebulizer gives the highest SRBR value (indicating the lowest detection limits) with the universal DuraMist nebulizer next best. The V-groove nebulizer gives the lowest SRBR values and consequently, will have the highest detection limits.

Summary

If you want the best performance your ICP-OES can provide, it is essential that you choose the proper nebulizer and spray chamber combination to meet the demands of your sample types. In addition to following recommended maintenance procedures to ensure consistent performance and long-life. In this article we presented a selection guide based on several performance criteria and cost of ownership. If you require any further assistance selecting a sample introduction system please contact us at enquiries@geicp.com or visit www.geicp.com.

References

2. Glass Expansion October 2014 Newsletter, ICP Spray Chamber Update.
5. Glass Expansion June 2013 Newsletter, A Nebulizer Update.
HydraMist Simultaneous Cold Vapor Pneumatic Nebulization Spray Chamber

The Glass Expansion HydraMist is a sensitive, simple-to-use spray chamber for ICP that supports simultaneous operation of conventional pneumatic nebulization and cold-vapor/hydride generation. Cold vapor generation can provide more than 10-fold improvement in sensitivity on ICP for elements such as As, Sb, Se, Tl and Hg. The generation of volatile species of these elements results in increased analyte loading to the analytical plasma giving lower detection limits.

The design of the HydraMist spray chamber is based upon Glass Expansion’s industry-standard cyclonic spray chamber, giving excellent sensitivity and short-term analytical precision with fast washout. The HydraMist spray chamber features a secondary inlet port that mixes the aerosolized sample and liquid reductant in the aerosol phase inside the spray chamber for rapid conversion of the As, Sb, Se, TI and Hg analytes into volatile hydride species. The unique drain design ensures fast, complete removal of waste from the spray chamber, eliminating excess hydrogen build-up that causes sample reflux degrading analytical precision.

The HydraMist spray chamber features:

- The same outstanding short-term analytical precision and washout as other Glass Expansion cyclonic spray chambers
- Fast and complete generation formation of volatile As, Sb, Se, Tl and Hg species for the best detection limits in hydride generation mode
- A unique drain design and cyclonic aerosol action eliminates hydrogen build-up and sample reflux that degrades short-term precision
- Economic, just replace your current spray chamber and keep your existing nebulizer
- Improve productivity by analysing non-hydride forming elements and cold vapor elements simultaneously

Simple to setup with fast changeover, the HydraMist Spray chamber can be operated as a:

- Conventional cyclonic spray chamber with pneumatic nebulization
- Sensitive, simultaneous cold vapor and pneumatic nebulization mode with greater than 10 fold improvement in detection limits for the cold vapor elements without compromising performance of non-hydride forming elements
- Simple, simultaneous hydride and pneumatic nebulization mode improves hydride forming detection limits 5-fold, while maintaining detection limits for non-hydride elements

The HydraMist spray chamber uniquely mixes the aerosolized sample and liquid to ensure complete formation of the volatile hydride species. Other hydride generation systems rely on a slow liquid/liquid mixing of the sample and reductant which results in incomplete formation of the volatile hydride species and excess hydrogen build-up in the drain. These systems suffer from low sensitivity and poor analytical precision, significantly compromising detection limits.

<table>
<thead>
<tr>
<th>Element</th>
<th>Pneumatic Nebulization</th>
<th>Sensitive simultaneous hydride/pneumatic nebulization mode</th>
<th>Simple simultaneous hydride/pneumatic nebulization mode</th>
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<tbody>
<tr>
<td>Wavelength (λ)</td>
<td>(ug/L)</td>
<td>(ug/L)</td>
<td>(ug/L)</td>
</tr>
<tr>
<td>As 188</td>
<td>3.7</td>
<td>0.2</td>
<td>0.5</td>
</tr>
<tr>
<td>Hg 194</td>
<td>1.2</td>
<td>0.07</td>
<td>0.1</td>
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<tr>
<td>Sb 206</td>
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<td>0.2</td>
<td>0.4</td>
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<tr>
<td>Se 196</td>
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<td>0.2</td>
<td>0.5</td>
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<tr>
<td>Cd 214</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>Co 238</td>
<td>0.4</td>
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<td>0.7</td>
</tr>
<tr>
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<tr>
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<tr>
<td>Pb 220</td>
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<td>2.6</td>
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<tr>
<td>Tl 190</td>
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</tr>
<tr>
<td>Zn 213</td>
<td>0.2</td>
<td>0.2</td>
<td>0.2</td>
</tr>
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</table>

Table 1. Measured 3σ detection limits (in ug/L) with an Agilent Technologies 5100 SV DV in axial mode at 1.4 kW RF power and 20 second integration time.
Enhanced Single Cell Sample Introduction System for ICP-MS

Single-cell analysis by ICP-MS (SC-ICP-MS) is enhancing the basic understandings in cellular biology, oncology and drug discovery. SC-ICP-MS provides quantification of metals in individual biological cells at ultra-low detection levels not previously seen in other techniques. SC-ICP-MS can be used to study disease aetiology, provide a better understanding of diseased cell states and develop new drug treatments for better patient outcomes.

The Glass Expansion Single-Cell Sample Introduction System (SC-SIS) for ICP-MS consists of a:

- High efficiency, low uptake rate, concentric glass nebulizer designed to efficiently nebulize single-cell suspensions without compromising cell integrity
- Low volume, on-axis spray chamber directly coupled to the ICP-MS for the highest transport efficiency of the nebulized cell suspension
- Patent pending MicroJet gas adapter shapes the nebulizer aerosol plume to reduce cell deposition on the spray chamber walls, enhancing the transport efficiency

In single-cell analysis, the aim of the sample introduction system is to convert a continuous stream of cell suspension efficiently into an aerosol made entirely of single cells into the analytical plasma. Critically, during the nebulization process the cells must remain intact and unruptured, to ensure each individual cell provides a single burst of ions in the plasma which can be measured by the mass analyser of the ICP-MS.

High Efficiency Single Cell Nebulizer

The novel design of the high efficiency nebulizer used in the Glass Expansion single-cell sample introduction system uses a low argon gas flow to provide high nebulization efficiency without rupturing the cell walls.

Glass Expansion's single cell nebulizer with proprietary VitriCone sample channel features:

- Superb transport efficiency at low sample uptake rates (15 to 45 uL/min) to ensure individual intact cells are transported into the plasma for measurement
- Constant diameter, large bore sample channel minimizes blockages from sample build-up commonly experienced with biological samples
- Rigid, precision machined thick-walled glass sample capillary gives the best analytical precision by resisting harmonic vibrations from the high linear velocity of the surrounding argon gas flow
- Inert, metal-free DC gas fittings with ratchet mechanism give reliable, reproducible leak-free Ar connections

Spray Chamber with MicroJet Adaptor

The challenge for SC-ICP-MS is to efficiently transport the intact cells one cell at a time, through the spray chamber and into the plasma. Conventional ICP-MS spray chambers have low transport efficiencies (< 5%) and filter out larger droplets (>5um) preventing a high percentage of cells from entering the plasma.

The low volume on-axis spray chamber with the patent pending MicroJet Adaptor to simultaneously provide a:

- On-axis, laminar flow sheath gas around the nebulizer aerosol plume preventing cell deposition on the spray chamber walls, ensuring high cell transmission efficiency
- Optimization of the velocity of the sample aerosol through the central channel of the analytical plasma, independent of the nebulizer gas flow
- Excellent washout between samples
Sapphire Dual Path MC-ICP-MS

The molecular species generated by argon ICP source will cause significant interferences on the mass spectrum of non-traditional stable isotope systems such as Mg, Si, S, K, Ca, Ti, V, Cr, Fe, Ni and Se, making precise and accurate isotope determination challenging on the MC-ICP-MS. A typical solution is to use high-resolution to fully or partially resolve the isotopes of interest from their respective interferences, but at the cost of significantly reduced ion transmission.

Sapphire from Nu Instruments is a next generation MC-ICP-MS. The collision cell capable instrument builds on the field proven Nu Instruments MC-ICP-MS platform (with over 180 installed instruments worldwide) to add unrivalled capabilities. The Sapphire features low and high energy switchable ion beam paths, when the low energy mode is selected, the ion beam is directed into a hexapole collision cell for interference removal and measurement of the isotope systems mentioned above. The instrument can also be switched to the high energy mode and used as a traditional MC-ICP-MS for measurements of conventional isotope systems with no compromise in analytical performance. Sapphire brings additional capability to the existing MC-ICP-MS market allowing the measurement of even more isotope systems by this well-established technique.