

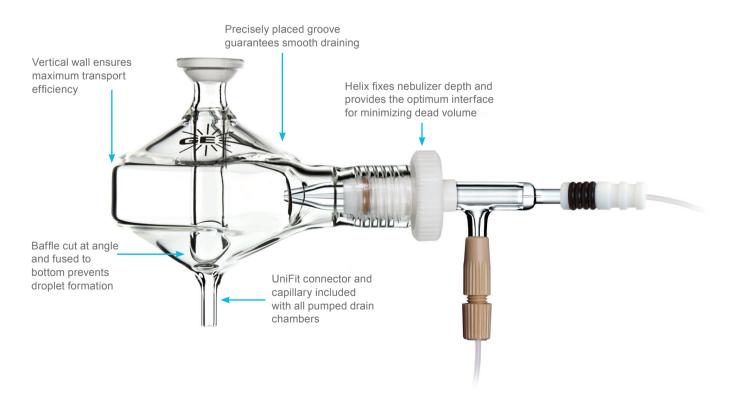
ICP Spray Chamber Update

Glass Expansion revolutionized spray chamber design for the ICP-OES and ICP-MS analyst by introducing cyclonic spray chambers. Prior to the Glass Expansion Tracey[™] cyclonic spray chamber, Scott-style spray chambers were the standard option provided with your ICP. The design of Glass Expansion's cyclonic spray chambers helps the ICP analyst achieve sensitivity gains, reduced washout times, and reduced matrix effects not possible with other designs.

This article will provide a summary of Glass Expansion's unique cyclonic spray chamber features and describe the advantages of each spray chamber type. We will also detail the evolution of the Glass Expansion spray chamber-nebulizer interface, covering the benefits of the Helix design. Finally we will address proper spray chamber maintenance and review an application-specific selection guide so that you can select the best spray chamber to suit your application.

Unique Design Features:

Figure 1: Characteristics of Glass Expansion's Cyclonic Glass Spray Chamber



Glass spray chamber:

Glass Expansion spray chambers are made from high-quality borosilicate glass with unique features to provide the ICP analyst with the utmost performance. The features of the Twister[™] cyclonic spray chamber are highlighted in Figure 1. Only a Glass Expansion cyclonic spray chamber will provide all these benefits.

Tracey cyclonic spray chamber:

The Tracey cyclonic spray chamber is Glass Expansion's standard glass cyclonic spray chamber (50 mL volume). It gives the best sensitivity and lowest memory effects for standard ICP analyses. The typical application uptake range is 0.2 to 3.0 mL/min.

Twister cyclonic spray chamber:

The Twister cyclonic spray chamber features a central transfer tube which acts as a secondary droplet filter to reduce the mean droplet size. This reduces solvent load in the plasma without compromising detection limits, while improving precision (% RSD). The typical application uptake range is 0.2 to 3.0 mL/min.

Low volume spray chamber:

Glass Expansion offers two low volume spray chamber designs, the CinnabarTM and TwinnabarTM. The advantage of a low volume spray chamber is to provide a very fast washout for sample uptake rates in the range of 20 to 400 µL/min. The Cinnabar is a mini-cyclonic based on the Tracey design while the Twinnabar is mini-cyclonic based on the Twister design. Like the Twister, the center baffle of the Twinnabar offers a reduced solvent load and excellent precision, in addition to a very fast washout.

PTFE and PFA spray chamber:

In 2006, Glass Expansion released a new line of PTFE and PFA spray chambers which have an internal surface that is specially treated using a proprietary process, named Stediflow[™], to improve the wettability of the surface and provide consistent drainage. Without the Glass Expansion Stediflow treatment, droplets tend to form on the surface and degrade both sensitivity and precision.

Glass Expansion offers two styles of HF-resistant spray chambers, the Tracey TFE and PFA44. The Tracey TFE is made from PTFE (Teflon) which is more chemically resistant than the old polypropylene HF-resistant spray chamber. The Tracey TFE spray chamber can be teamed with the DuraMist HF-resistant nebulizer to form the ideal chemically inert sample introduction system for ICP-OES. The Tracey PFA44 is made from ultra-pure PFA to provide exceptional purity and excellent sensitivity for ICP-MS techniques. The PFA44 spray chamber can be teamed with the OpalMist PFA nebulizer to form the highest purity, most inert sample introduction system available. A Tracey PFA44 spray chamber molded specifically for an airtight fit within the IsoMist module, is provided to those customers who require an HF-resistant temperature controlled option. Further details on the IsoMist are covered in the following section.

IsoMist:

The temperature of a sample introduction system has a profound effect on the performance of an ICP-OES and an ICP-MS. A cooled spray chamber is often used to reduce the volatility of certain solvents so that the plasma is sustained. Heating the spray chamber results in higher sample transport. Maintaining the spray chamber temperature can be critical when trying to achieve long-term signal stability.

The **IsoMist[™]** Programmable Temperature Spray Chamber (Figure 2A) is a convenient and universal device for both controlling and monitoring the spray chamber temperature. The temperature is electronically controlled using a powerful inbuilt Peltier device, allowing the ICP analyst to select any temperature between -10°C and +60°C in 1°C increments to provide the optimum conditions for any application.

Figure 2a: IsoMist Programmable Temperature Spray Chamber



Unique to the IsoMist is a Twister spray chamber encapsulated with a temperature conductive resin (Figure 2B). The encapsulated spray chamber provides a uniform temperature from top to bottom and an air tight fit within the IsoMist module to prevent condensation build-up and freezing (Figure 2C). These unique features of the IsoMist allow for a stable temperature to be held within +/- 0.1° C and a temperature of -5°C to be reached within 15 minutes. Other manufacturers rely on their standard spray chamber design to be fitted in a packing foam. This type of design can lead to unstable temperatures and an uneven temperature with respect to the top and bottom of the spray chamber.

Figure 2b: IsoMist Glass Twister Spray Chamber encapsulated in conductive resin

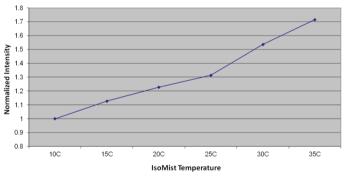


Figure 2c: Airtight seal of the IsoMist spray chamber in its case



The importance of a stable spray chamber temperature is demonstrated by Figure 3, which shows that a spray chamber temperature change of 1°C can result in a sensitivity change of 3%. In order to ensure an accurate and constant spray chamber temperature, the IsoMist Programmable Temperature Spray Chamber is preferable.

Figure 3: Average normalized intensity of 18 analyte lines vs. spray chamber temperature



31% increase from 25 to 35 °C or 3% per °C

Peltier Cooled Cyclonic (PCC) Spray Chamber for Agilent ICP-MS:

When the best temperature flexibility or temperature stability is required, we recommend the IsoMist. However, for many ICP-MS applications, a fixed temperature of 2 or 5°C is used with no required flexibility. For these applications, we recommend the **PCC Spray Chambers for the Agilent 7700/7900/8800** (Figure 4) and 7500 ICP-MS models. The PCC is based on the spray chamber and Peltier system of the IsoMist but is coupled to the electronics and water cooling of the Agilent ICP-MS and operated via Agilent software. Therefore Agilent 7700/7900/8800 and 7500 users can get the benefit of a cyclonic spray chamber, faster washout and increased sample throughput compared to the Scott style spray chamber.

Figure 4: PCC Spray Chamber mounted on an Agilent 7700 ICP-MS



Evolution of the Spray Chamber-Nebulizer Interface:

The interface between the nebulizer and the spray chamber in an ICP spectrometer needs to fulfill several criteria:

- 1. Seal the spray chamber from the external environment
- 2. Ensure that the nebulizer is always in the same position
- 3. Allow the nebulizer to be easily removed to be cleaned or replaced
- 4. Not contaminate the sample
- 5. Minimize carryover from one sample to the next

With early spray chambers, an attempt was made to satisfy these criteria by constructing the spray chamber with a glass arm containing grooves for o'rings as shown in Figure 5A.

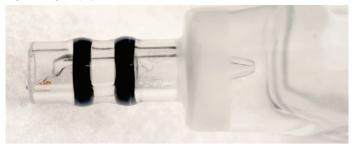
Evolution of the Spray Chamber-Nebulizer Interface:

Figure 5: Progression of spray chamber-nebulizer interfaces; all glass with o'rings (A), plastic adaptor with o'rings (B), and Helix o'ring-free adaptor (C).



While this design was generally satisfactory, there was a tendency for the o'ring to bond to the glass nebulizer. If this happened, the nebulizer could be difficult to remove, and it was not uncommon for the ICP analyst to break either the spray chamber arm or the nebulizer, see Figure 6.

Figure 6: All glass spray chamber with broken nebulizer



The introduction of the plastic nebulizer adaptor, as shown in Figure 5B, made the spray chamber much more robust and alleviated the problem of the spray chamber arm being broken. However, this design still relied on o'rings, and the problem of the o'rings bonding to the nebulizer remained.

Also, there is no o'ring material that is impervious to all of the solvents used with an ICP. In particular, some organic solvents cause the o'rings to degrade rapidly, leading to potential contamination and necessitating frequent o'ring replacement.

The new Helix, shown in Figure 5C, eliminates all of the problems with the previous designs. A smooth lock and release mechanism enables the nebulizer to be simply and easily inserted or removed (visit our <u>video page</u> for an instructional video). The Helix seal is made from Teflon, which is totally inert to all of the organic solvents and strong acids normally used in ICP analyses. This minimizes any possibility of contamination. The collar of the Helix provides a positive stop for optimal and reproducible nebulizer positioning.

Helix Washout:

Another important design feature of the Helix is the elimination of dead volume around the nebulizer seal. Eliminating dead volume leads to faster washout times and higher sample throughput. Figure 7 compares the time required to washout a 10ppm Molybdenum standard with a Glass Expansion spray chamber (Helix interface) and a "Brand-X" spray chamber with an o'ring interface. The results show that with the Helix nebulizer interface a 10ppm standard can be washed out in as little as 4 seconds, whereas "Brand-X" takes 16 seconds. One can expect this time to significantly increase for more troublesome or "sticky" elements that are more prone to carryover issues.

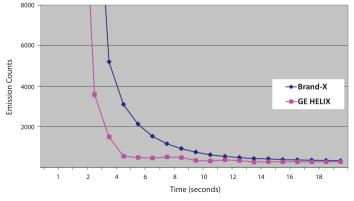


Figure 7: 10 ppm Mo Washout comparison between Helix and non-Helix spray chambers

Spray Chamber Maintenance

Glass and Quartz Spray Chambers:

It is good practice to always start and finish use of a glass spray chamber by nebulizing a mildly-acidic blank solution for several minutes. This ensures that sample deposits or crystals don't form inside a spray chamber when the solvent inside the chamber dries out. To avoid the risk of breakage, washing glass spray chambers in an ultrasonic bath is not recommended.

If you notice a degradation in performance (such as poorer precision or detection limits), then clean the spray chamber with Fluka 'RBS-25' (<u>P/N FLUKA25</u>). In the first instance, aspirating a 2.5% Fluka solution for 15 minutes will probably be sufficient to recover the performance. However, if this is not effective, the spray chamber should be soaked overnight in a 25% Fluka solution.

If you see droplets collecting on the internal surfaces of your spray chamber, this is a sure sign that stability is suffering - such 'resident' droplets in the spray chamber are the most common and visible indication of spray chamber instability, and they should be removed. A long soak in 25%-strength RBS-25 solution is recommended.

PTFE and PFA Spray Chambers:

The PTFE and PFA spray chambers have an internal surface that is specially treated to ensure that it wets evenly and provides consistent drainage. The treatment turns the surface a characteristic brown color. It should be noted that the treatment actually changes the molecular structure of the PTFE and PFA. It is not a coating and it does not introduce any potential contaminants.

While the surface treatment is long lasting, it may degrade after prolonged use. The lifetime of the treated surface depends on the type of samples used and could range from several months to several years. To ensure that you get the best performance from your PTFE and PFA spray chambers, we recommend the following:

Do not use H_2O_2 for cleaning the spray chambers as this will accelerate the degradation of the surface.

Do not make physical contact with the chamber interior surface with any instrument, including your hands or a brush.

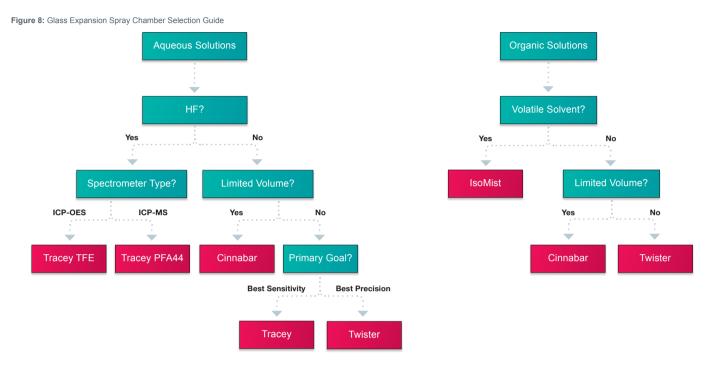
Do not be concerned if the brown color fades over time. This is normal and does not necessarily lead to a degradation in performance.

If you notice a degradation in performance (such as poorer precision or detection limits), then clean the spray chamber with Fluka 'RBS-25'. In the first instance, aspirating a 2.5% Fluka solution for 15 minutes will probably be sufficient to recover the performance. However, if this is not effective, the spray chamber should be soaked overnight in a 25% Fluka solution.

Eventually the surface may degrade to the point where it does not recover after soaking in Fluka. At this point the spray chamber needs to be returned to Glass Expansion where the surface can be re-treated for a nominal cost.

Spray Chamber Selection Guide:

Glass Expansion offers several types of cyclonic spray chamber designs to suit your application. Whether your ICP laboratory is analyzing clean aqueous samples, samples containing HF and/or high dissolved salts, or volatile organic solvents; Glass Expansion has a spray chamber to suit your needs. To find the most suitable spray chamber for your specific application, download our FREE smartphone app 'GE Selection' from the App Store or Google Play, or you can utilise our easy to follow Flow Chart, shown in Figure 8.



Summary

We hope that this spray chamber update has provided you with some helpful details regarding the unique design features and performance an ICP analyst can expect to achieve with a Glass Expansion cyclonic spray chamber. We believe that the best design, production, and service are all necessary to achieve the highest product quality. Our design and manufacturing processes ensure reliability in the performance of our products, which helps you minimize variables that can affect your analyses.

Following the maintenance guidelines described in this article will allow you to protect your investment and maintain optimum spray chamber performance. If you have any questions about spray chamber selection please contact us, we are happy to help.