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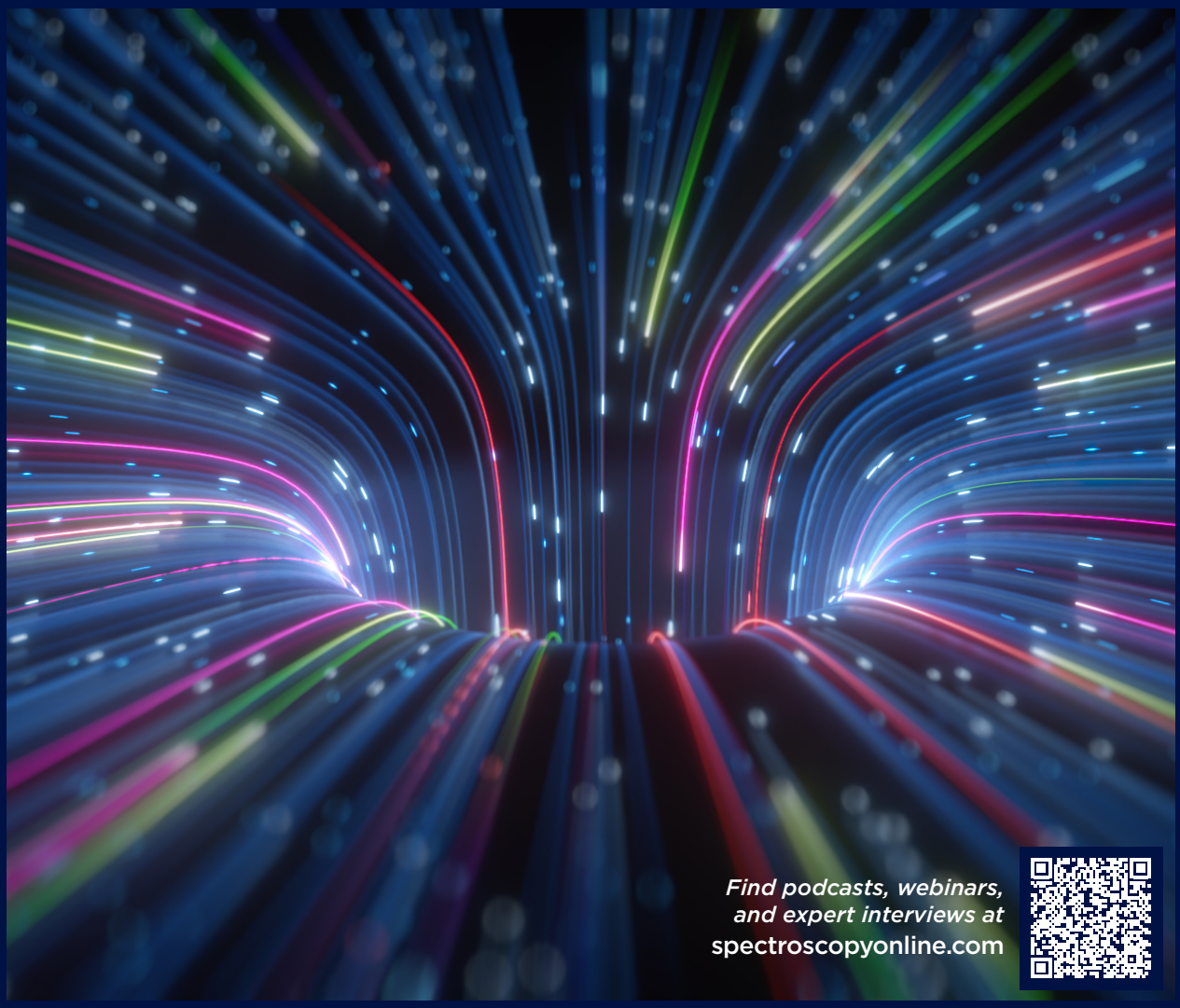

ADVANCES IN ICP-MS BEST PRACTICES

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The Role of the Nebulizer in ICP-MS: Design Considerations, Selection Criteria, and Optimization Guidelines

Justin Masone

Nebulizers play a pivotal role in the sample introduction systems of inductively coupled plasma mass spectrometry (ICP-MS), a technique that has revolutionized elemental analysis since its development in the early 1980s. Primarily designed for liquid samples, which constitute the majority of ICP-MS applications, nebulizers convert solutions into fine aerosols that can be transported to the plasma for desolvation, atomization, ionization and detection. However, the sample introduction system—encompassing the nebulizer and spray chamber—has long been regarded as the "Achilles' heel" of ICP-MS (1). This stems from its inherent inefficiency, where typically only 1-2% of the sample reaches the plasma, with the remainder lost to waste. Despite these limitations, advancements in nebulizer technology have significantly mitigated such issues, contributing to improved transport efficiencies and analytical outcomes.

The core function of a nebulizer is to deliver liquid samples into the plasma, where intense heat/energy—around 6000-10,000 K—atomizes and ionizes analytes for detection. Early designs focused on basic aerosol generation, but modern iterations prioritize precision, durability, and compatibility with challenging matrices. This evolution has been driven by the need to handle diverse samples, from trace-level environmental waters to high-matrix industrial effluents, while minimizing matrix interferences and maximizing signal stability. By understanding nebulizer mechanics, analysts can optimize parameters like gas flow rates and sample uptake, directly influencing aerosol quality and instrument performance.

The Importance of Nebulizer Droplet Size and Transport Efficiency

At the most fundamental level, effective nebulization is characterized by smaller droplet sizes and narrower distributions, as these factors determine how much of the sample reaches the plasma without overloading it—the plasma can only tolerate limited liquid loading before cooling or extinguishing it. Ideally, the sample introduction system should deliver 40 µL/min of water to the plasma or less, and exceeding 80 µL/min can extinguish the plasma (2). Additionally, if droplets are too large, they will not desolvate completely, significantly reducing plasma temperature and causing some elements to be released before others. This results in a higher signal for easily-ionizable elements (such as Na, K, and Al, for example) as compared to poorly-ionizable elements (such as Hg, As, and Se, for example). Thus, consistent droplet size is critical for accurate analysis. While all nebulizers generate a wide distribution of droplet sizes, nebulizers which generate a more consistent and narrower distribution will lead to better sensitivity and precision, as droplets over about 10 µm do not fully vaporize.

It is important to differentiate between what is called the primary aerosol and the tertiary aerosol. The primary aerosol is the aerosol that is produced from the nebulizer (as seen in Figure 1), whereas the tertiary aerosol is the aerosol that exits the spray chamber (and transfer tube, if used) and is injected into the plasma. As previously mentioned, all nebulizers produce a wide range of droplet sizes, and spray chambers are necessary to remove these larger droplets, with the smaller droplets going to the plasma and the larger droplets going to waste. This ratio of the amount of sample volume entering the plasma to the amount that is initially aspirated by the nebulizer is referred to as transport efficiency and is typically estimated by measuring the volume of waste draining from the spray chamber versus the total sample uptake volume.

Evolution of Nebulizer Designs

Nebulizers fall into two main categories: pneumatic and ultrasonic. Pneumatic nebulizers, the most common for ICP-MS, use the mechanical force of a gas flow to generate the sample aerosol, and will be the focus of this article. The early models of pneumatic nebulizers that were developed in the 1970s to support ICP-OES instrumentation achieved modest efficiencies but struggled with matrix effects and blockages. Over time, innovations in materials, fluid dynamics, and manufacturing have led to more robust and efficient designs.

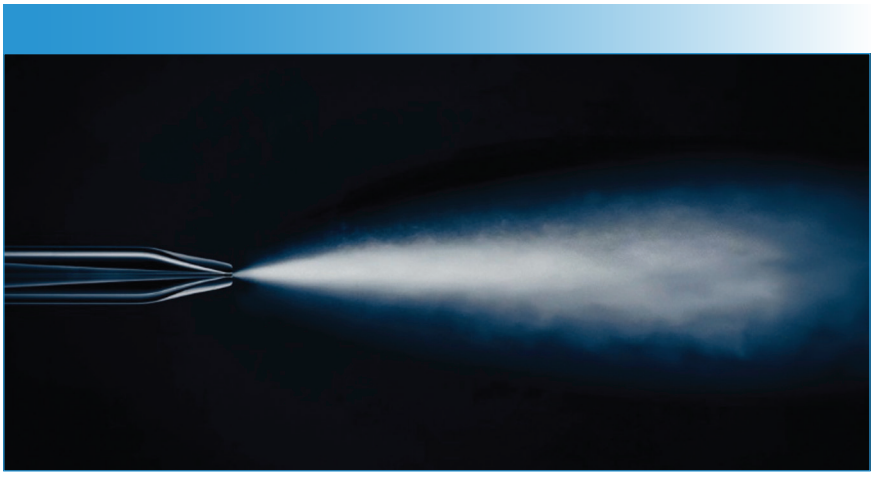

FIGURE 1: Close-up of the primary aerosol produced at the tip of a concentric nebulizer (image provided by Glass Expansion).

Concentric Nebulizers

Concentric nebulizers represent a cornerstone of this evolution, featuring a central sample capillary encased in a glass sheath for coaxial flow

These nebulizers produce fine, uniform aerosols with small mean droplet diameters, ideal for high-sensitivity ICP-MS work. The earliest designs featured a heated and drawn inner glass capillary, which produced an excellent mist but resulted in a fragile, narrowing tip, prone to plugging by particles, as well as suboptimal relative standard deviations (RSDs) because of inconsistent concentricity and internal diameters (ID), increased glass porosity, and vibration issues caused by a high-velocity gas flow around the thin capillary. These issues are further exacerbated by similar inconsistencies in the forming of the nebulizer shell. Modern designs aim for a consistent ID along the capillary, as is seen with the VitriCone technology (Glass Expansion). This unique and revolutionary design involves machining precision-bore, thick-walled glass tubing, ensuring a uniform sample channel that resists clogging and provides high precision. Modern designs also use more advanced manufacturing methods to form the nebulizer jet nozzle, ensuring precise shape and structure.

Early designs of concentric nebulizers were prone to plugging and salting up with high-total dissolved solids (TDS) samples. This occurs as dissolved solids precipitate at the tip due to gas expansion and lower pressure. However, manufacturers have since developed improved designs for high-TDS samples, which feature recessed capillaries that move the salt precipitation zone away from the gas/liquid mixing zone, with some designs featuring a "self-washing nozzle," which uses the washing action of the dispersing droplets to further prevent crystal growth.

Benefits of Concentric Nebulizers

- Produce the finest aerosol at a variety of gas and sample flow rates, contributing to excellent reproducibility, transport efficiency, and stability.
- Can self-aspirate due to the natural Venturi effect, which can offset the pulsing effects of the peristaltic pump to maintain maximum precision.
- Are cost-effective, especially when made from glass.
- Easy to clean and maintain with convenient tools.

Potential Drawbacks of Concentric Nebulizers

- Can result in blockages from particulates or salts if the correct concentric nebulizer or accessories are not selected, as there are specific designs for different types of sample matrices.

Cross Flow Nebulizers

Cross Flow nebulizers, an early alternative to concentric nebulizers, intersect the sample and gas streams at right angles, offering robustness for high-salt samples. They can accommodate larger sample capillaries, but this results in a coarser aerosol with a wider droplet size distribution, leading to higher RSDs and limited suitability for ICP-MS (3).

Benefits of Cross Flow Nebulizers

- Offer larger liquid capillaries as compared to concentric designs, which allow larger particulates to pass through without plugging.

Potential Drawbacks of Cross Flow Nebulizers

- Provide a coarser mist than concentric nebulizers.
- Produce a wider range of droplet sizes and lower stability in the plasma
- Require higher liquid flow rates, typically limited to ICP-OES.

V-Groove Nebulizers

V-Groove nebulizers emerged to tackle high-particulate loads, as they have parallel gas and liquid paths, channeling liquid onto the gas orifice. They evolved based on non-plugging concepts and feature unrestricted sample paths that han-

dle unfiltered slurries, with particulate tolerance exceeding 150 µm in some cases. While they can be effective for complex matrices, they produce larger droplet sizes with greater variability, negatively impacting precision. With the advancements in concentric nebulizers designed for particulates, slurries, and dissolved solids, in additional to accessories such as humidifiers, in-line sample filters, and autosampler probes with integrated filters, the V-groove is often not the optimal choice if precision and sensitivity are of utmost concern (4).

Benefits of V-Groove Nebulizers

- Particles cannot plug the liquid capillary, as there is no liquid capillary restriction, and it can have a large ID.
- Can handle unfiltered samples.

Potential Drawbacks of V-Groove Nebulizers

- Produces a mist with much larger droplets than other designs.
- Greater variation in droplet sizes as compared to concentric nebulizers.
- Cannot self-aspirate via the Venturi effect to offset the pulsing effects of the peristaltic pump.

Parallel Path Nebulizers

Parallel Path nebulizers were developed in the 1990s and deliver liquid adjacent to the gas orifice, with separate streams for the liquid and the gas. The sample is drawn into the gas stream by induction caused by the low pressure around the gas stream. Enhanced Parallel Path nebulizers were developed in the early 2000s, and, instead of simply placing the liquid near the gas stream and relying on induction, they feature a "spout," which protrudes into the gas stream (5).

Benefits of Parallel Path Nebulizers

- Separate gas and liquid channels with a large bore sample path that eliminates particulate blockages

Potential Drawbacks of Parallel Path Nebulizers

- Produces a wide range of droplet sizes.
- Cannot self-aspirate via the Venturi effect to offset the pulsing effects of the peristaltic pump.

Flow-Blurring Nebulizers

Flow-Blurring nebulizers are a recent development, similar in function to modern concentric nebulizer designs, operating with more turbulent gas-liquid interaction. The nebulizer has a central liquid capillary with surrounding gas orifices, resulting in highly turbulent mixing of the gas and liquid and creating an aerosol, in a process called "flow blurring" (6).

Benefits of Flow-Blurring Nebulizers

- High tolerance to particulates and dissolved solids while still maintaining sufficient sensitivity and precision

Potential Drawbacks of Flow-Blurring Nebulizers

- Increased optimization often required, as performance depends on precise gas and liquid flow ratios.
- Operate with very high backpressure—may not be recommended for high-throughput valve systems, and can accentuate noise from the peristaltic pump.
- While often touted as a "universal" nebulizer, they may not perform as well when compared to dedicated concentric nebulizers optimized for specific sample matrices.
- Cannot self-aspirate via the Venturi effect to offset the pulsing effects of the peristaltic pump.

Impact of Nebulizer Design on Aerosol Characteristics and Analytical Performance

Nebulizer design and operating conditions critically affect performance, specifically sensitivity, precision, and overall analytical accuracy. In general, a higher concentration of smaller droplets leads to improved sensitivity, and a more consistent droplet size is paramount for accurate and precise results.

While the most important factor in maximizing nebulizer performance is simply choosing the optimal nebulizer for your analysis, the operating parameters (for example, gas and liquid flow rates) still have a significant effect on performance. How these two parameters affect nebulization can be explained by understanding the Sauter mean diameter ($D_{3,2}$) – the average measure of particle size—and the Nukiyama and Tanasawa equation used to calculate it (7).

$$D_{3,2} = \frac{585}{V}\left[\frac{\sigma}{\rho}\right]^{0.5} + 597\left[\frac{\eta}{(\sigma\rho)^{0.5}}\right]^{0.45}\left[\frac{10^3 Q_l}{Q_g}\right]^{1.5}$$

in which:

- $D_{3,2}$ = Sauter mean diameter (µm)
- V = velocity difference between gas and liquid (m/s)
- σ = surface tension (dyn/cm)
- ρ = liquid density (g/cm^3)
- η = liquid velocity (Poise or dyn.s/cm^2)
- Q_l = volume flow rate, liquid (cm^3/s)
- Q_g = volume flow rate, gas (cm^3/s)

For water or aqueous matrices, surface tension (**σ**), liquid density (**ρ**), and viscosity remain constant. The droplet size, then, is inversely proportional to the velocity difference of the gas and liquid (**V**); that is, droplet size ($\mathbf{D_{3,2}}$) decreases as the gas velocity increases and the liquid velocity (**η**) decreases. In practice, the smallest primary aerosol droplets are produced by using high argon gas flow and pressure, combined with low liquid flow and pressure.

The effect of lowering the sample flow rate can be seen in Figure 2 (it should be noted that while droplet size is an important factor for sensitivity, it is still dependent on the absolute amount of sample injected into the plasma). Three different nebulizers were compared using a constant gas flow rate and adjusting the sample uptake rate. Lowering the sample uptake rate for concentric designs increases the percent of droplet sizes less than 10 µm.

The same conditions were used to compare the effect that lowering the sample uptake rate has on the mean droplet size, which is shown in Figure 3:

Similar effects can be seen when holding the sample uptake rate constant and adjusting the gas flow rate. Increasing the nebulizer gas flow rate increases the percent of droplet sizes less than 10 µm. This can be seen in Figure 4.

And likewise with the mean/average droplet size, shown in Figure 5.

Aerosol Filtration

In addition to modern advancements in nebulizer designs, new techniques have been developed to improve aerosol quality post nebulization, which helps in maximizing instrument performance.

One such design is the Jet Vortex Interface (JVI Glass Expansion), which works in conjunction with the existing "Make-up" or "Dilution/Auxiliary" gas option on an ICP. Aerosol filtration accessories like the JVI provide precise control of the aerosol droplet size transported to the plasma, which, for high matrix samples, results in extended lifetimes of the torch, injector, and cones by a reduction in sample deposition, which also serves to prevent signal drift, in addition to reduced matrix effects in general. Aerosol filtration accessories enable the analyst to optimize the analysis for a variety of sample matrices (such as brines or volatile organics), as well as performance criteria (such as controlling for transport efficiency and signal intensity). The impact of JVI gas flow rate on droplet size is shown in Figure 6.

Aerosol filtration can also be utilized to "fine tune" the plasma conditions to create a more robust plasma for high matrix samples by reducing the oxide ratio to well below 1%. As a consequence, a more robust plasma results in higher sensitivity and, because of reduced matrix deposition, improved analytical stability, as shown in Figure 7.

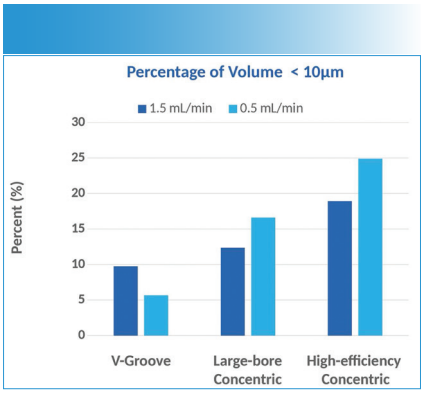

FIGURE 2: Effect of sample uptake rate on droplet size distribution.

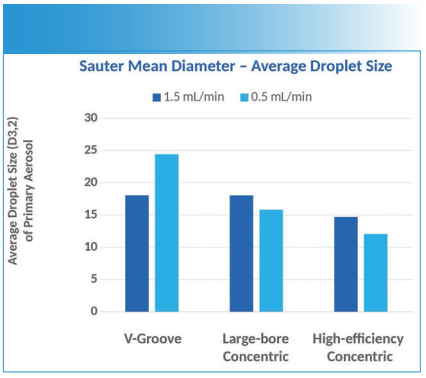

FIGURE 3: Effect of sample uptake rate on mean droplet size.

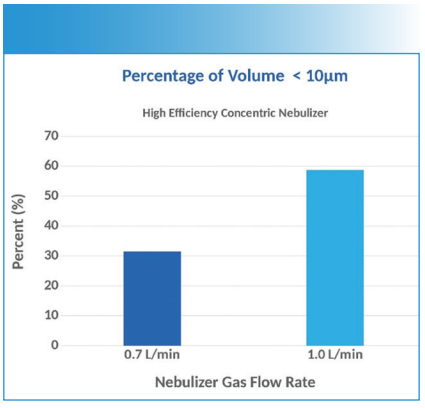

FIGURE 4: Effect of gas flow rate on droplet size distribution.

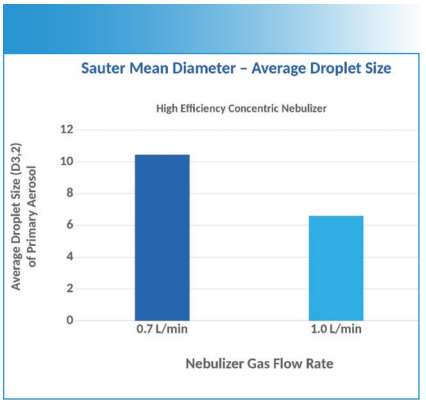

FIGURE 5: Effect of gas flow rate on mean/average droplet size.

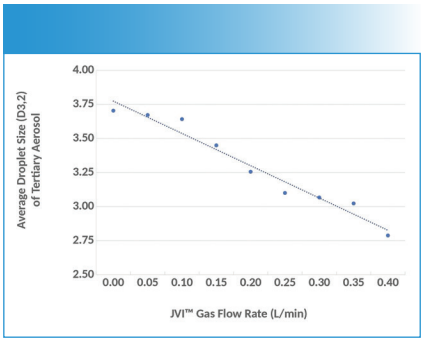

FIGURE 6: Effect of JVI gas flow rate on mean droplet size.

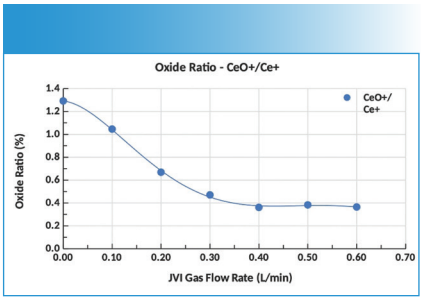

FIGURE 7: Effect of JVI gas flow rate on oxide ratio.

Nebulizer Selection Criteria

The choice of a nebulizer depends on several criteria, including:

- Reproducibility
- Sensitivity
- Precision
- Material (glass, PEEK (polymer), PFA (polymer), ceramic, etc.)
- TDS tolerance
- Particulates tolerance
- Hydrofluoric acid (HF) tolerance
- Purity
- Robustness
- Sample delivery method (self-aspirating vs. pumped)
- Cost

While the best advice is to consult with your instrument manufacturer or sample introduction supplier, some general guidance is offered in Table I

TABLE I: General performance guidance of nebulizers discussed in this article

Nebulizer Type	Material	Precision	TDS Tolerance	Particulates Tolerance	HF Tolerance	Purity	Self-Aspirating	Cost
Standard concentric	Glass	High	Good	Moderate	No	Good	Yes	Low
High-solids concentric	Glass	High	High	Moderate	No	Good	Yes	Moderate
High efficiency concentric	Glass	High	Moderate	Moderate	No	Good	Yes	Moderate
Large bore concentric	Glass	High	Moderate	High	No	Good	Yes	Moderate
PFA concentric	PFA	High	Moderate	Moderate	Yes	Excellent	Yes	High
PEEK concentric	PEEK	High	High	Moderate	Yes	Good	Yes	Moderate
V-Groove	Ceramic	Moderate	High	High	Yes	Good	No	High

(8). Similar charts, with more specific and detailed information, are often available from the manufacturer or supplier.

Nebulizer Maintenance

Regular cleaning is crucial for maintaining nebulizer performance and extending nebulizer lifespan. A good daily protocol is to always start and finish the use of a nebulizer by nebulizing a mildly acidic blank solution followed by deionized water for a couple of minutes. This ensures that sample deposits or crystals do not form inside a nebulizer when the solvent inside the nebulizer dries out.

If the sample capillary becomes blocked, tools like the Eluo (Glass Expansion) can be used for safe backflushing. For stubborn salt deposits, soaking the nebulizer in a 25% Fluka RBS-25 (Sigma Aldrich) solution for 24 h, and then flushing with warm water, followed by ethanol, is effective (9).

Note: *Never attempt to eliminate a blockage by inserting a wire or probe into the capillary—this may damage the nebulizer.*

Always handle the nebulizer tip with gloves to prevent detrimental effects from body oils. Be sure to store nebulizers in their specially-designed containers to prevent physical damage or contamination. Lastly, never put a nebulizer in an ultrasonic bath, as this can dislodge the sample capillary and damage the nebulizer.

Conclusion

Nebulizers remain a fundamental sample introduction component for ICP-MS. The diverse range of available nebulizer designs, coupled with an understanding of each design's impact on aerosol characteristics and analytical performance, enables analysts to select an optimal nebulizer for the wide variety of sample types and analytical challenges often encountered in today's laboratories. Ongoing advancements in nebulizer design and performance, and the integration of supplementary techniques such as aerosol filtration, continue to enhance the sensitivity, precision, accuracy, and robustness of ICP-MS. Lastly, proper nebulizer selection and dedicated maintenance are the key to maximizing instrument performance and minimizing instrument downtime.

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(9) ICP Nebulizer Maintenance Application Note from Glass Expansion Website https://www.geicp.com/site/images/application_notes/ICPNebulizer_Maintenance_Prolong_Performance_and_Lifetime.pdf

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