

Optimizing Sample Introduction: A Practical Guide for Better ICP Performance



Presenter:

Dr. Maja Budanovic
ICP Application Specialist,
Glass Expansion GmbH
Contact: gegmbh@geicp.com

Organized By:

MS SPEKTRUM
Contact: biuro@msspektrum.pl
Web: www.msspektrum.pl



Introduction: About Glass Expansion

- GE have been specializing in sample introduction components for ICPs since 1985
- Global recognition for manufacturing precision and reliability
- OEM to every major ICP-OES and ICP-MS manufacturer
- Approximately 100 staff
- Sales Offices Worldwide: Australia, USA and Germany
- Official Distributor for Poland: MS SPEKTRUM



Glass Expansion - Europe Weilburg, Germany



Glass Expansion - Americas Cape Cod, Massachusetts



Glass Expansion – Asia-Pacific Melbourne, Australia



ul. Lubomira 4/4, 04-002 Warszawa, Poland

How is Glass Expansion Different?

Our products are designed by our experienced R&D team:

- Invest ~10% p.a. in R&D
- Specialized equipment for Manufacturing: Particle analyzers, Electron beam welders, Multiple CNC Machines, 3D Printing & Laser Technology, High precision elaborate machining of plastics, glass, metals, ceramics

We provide our customers with full technical support:

- Diverse industry/research partnerships
- Extensivley equipped laboratory with 6 in-house ICPs

Quality Control:

- Control the entire manufacturing process, from raw material to final product
- Manufactured to exacting specifications: Guaranteed to meet/exceed OEM specifications

Glass Expansion Warranty:

All products are supplied with a CUSTOMER SATISFACTION GUARANTEE

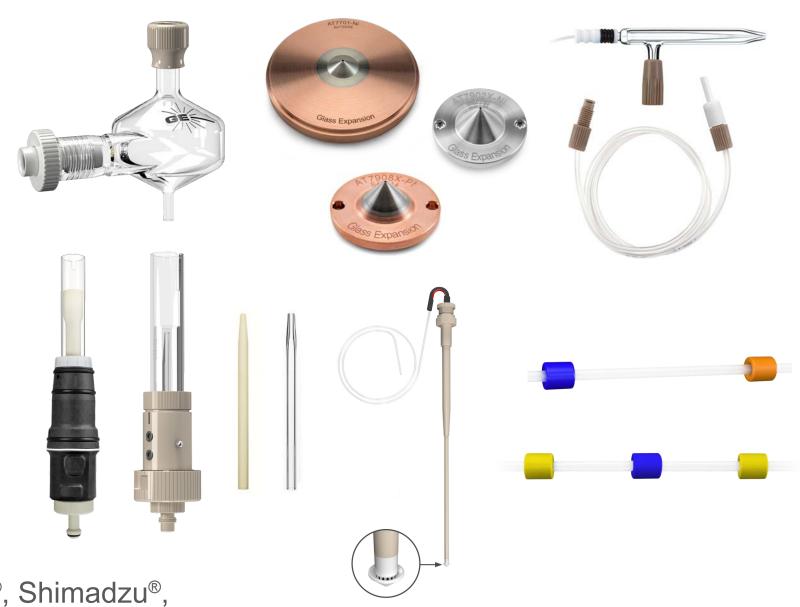




Product Lineup

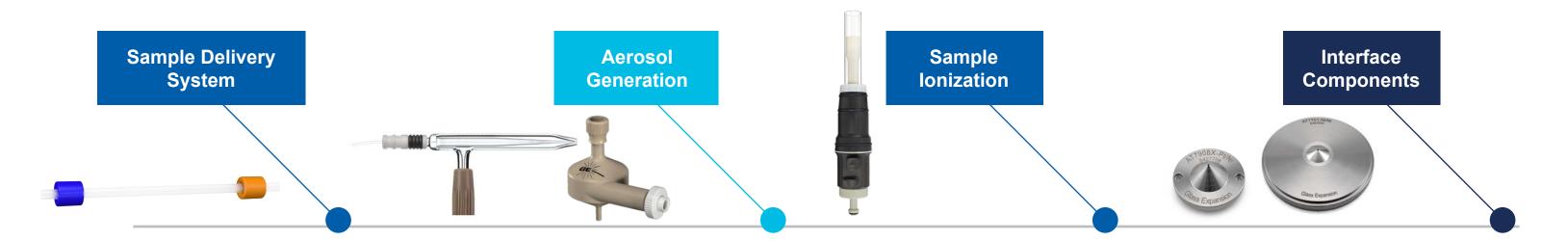
- Autosampler Probes
- Pump Tubing
- **Nebulizers:** Custom-manufactured for optimal performance with each ICP
- Spray Chambers: Pioneered cyclonic design
- Torches & Injectors: Introduced the FDT
- Cones, RF Coils
- Other Accessories

Manufacturers Supported: Thermo[®], Agilent[®], PerkinElmer[®], Shimadzu[®], Spectro[®], Analytik Jena[®], Horiba[®], Nu Instruments[®], Others





Agenda



- Sample Probe and Pump Tubing
- Practical Selection Considerations
- Internal Standard Addition Kit
- Care and Maintenance

- Nebulizer and Spray Chamber
- Optimal Design Considerations
- Application Suitability
- Care and Maintenance

- Torch and Injector
- RF Coil
- Selection Considerations
- Care and Maintenance

- Cone Material
- Care and Maintenance



1. Sample Delivery System

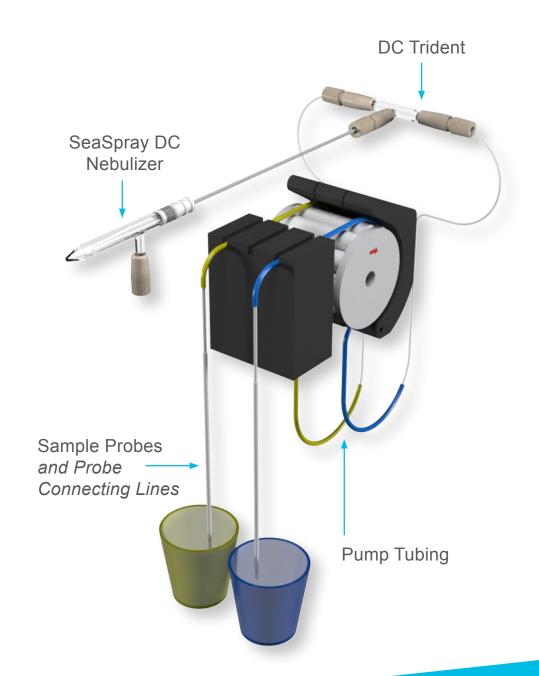
- Sample Probe
- Peristaltic Pump Tubing
- Teflon connecting tubing
- Internal Standard Addition Kit
- DC Trident

Key Functions:

- Consistent and accurate transport of the sample from its container to the nebulizer
- Allows for precise control over sample volume for reproducible measurements

Common Challenges:

- 1. Ineffective Sample Digestion: Precipitates and undigested particles
- 2. Clogging and Blockages
- 3. Cross-Contamination
- 4. Sample Flow Issues: Variation in sample flow rate results in an unstable signal (poor RSDs)





Sample Probe: Selection

1. Standard Option: Carbon Fibre Probe

- Encapsulated carbon fibre tube with continuous PFA tubing
- Available for most common Autosampler models, with IDs of 0.50, 0.75 and 1.00 mm

2. Advanced Option: Guardian Autosampler Probe Features:

- Robust tip design prevents crushed and damaged tips due to misalignment
- Combines drip-resistance and built in filter to minimize crosscontamination, while protecting the nebulizer and capillary tubing
- Completely inert design, Ceramic, PEEK and PTFE construction
- Interchangeable UniFit™ sample lines IDs: 0.3, 0.50, 0.75 and 1.0mm
- Autosamplers: Teledyne Cetac[®], Agilent[®], PerkinElmer[®], Shimadzu[®], Aim Lab and Thermo Fisher Scientific[™] Autosamplers.



Guardian™ Autosampler Probe

Drip-resistance prevents cross contamination of samples, especially with oils:

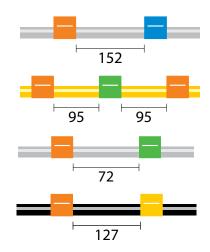
Customer Testimonials:

- Remarkable performance in environmental laboratories worldwide
- Unclogged operation even with particulate-containing samples
- Significantly reduces nebulizer blockage and downtime
- Enhances analytical efficiency





Peristaltic Pump Tubing: Selection



Tubing Material:

- PVC Tubing
- Solva Tubing
- Viton Tubing

2-Tag/Stop vs. 3-Tag/Stop:

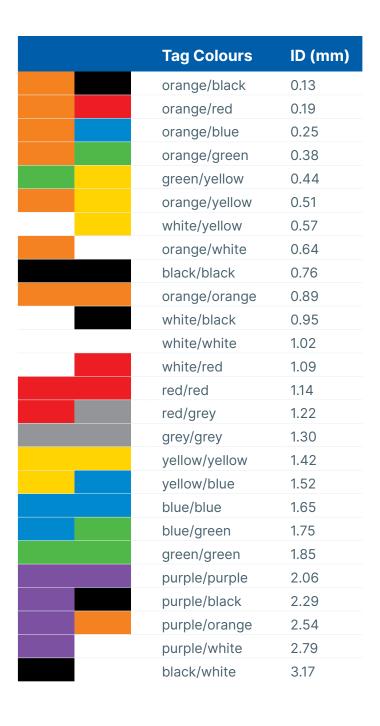
3-Tag/Stop: When one section wears out, a fresh section is ready to use, extending tubing life

Flared vs. Non-Flared Options:

Flared-end pump tubing makes it easier to insert larger sample capillary tubing

Internal Diameter (ID):

Smaller ID Tubing (0.2-0.4 mm): Ideal for precise, low-flow applications **Larger ID Tubing**: Suitable for higher flow rates and larger sample volumes



Customer Pain Points with traditional pump tubing:

1. Premature Tab Failure:

- **Description:** Tabs on the tubing can become loose or break off prematurely, especially if they are not properly bonded or are of poor quality.
- **Impact**: This can lead to detachment of the tubing from the connectors, causing interruptions in sample flow and requiring tubing replacement.

2. Tubing Slippage/Displacement:

- **Description:** Tubing can slip from the connectors or the pump rollers, particularly if the tubing is not properly fitted or if the connectors are worn.
- **Impact:** This can cause interruptions in the sample flow, inconsistent sample delivery and analysis interruptions.

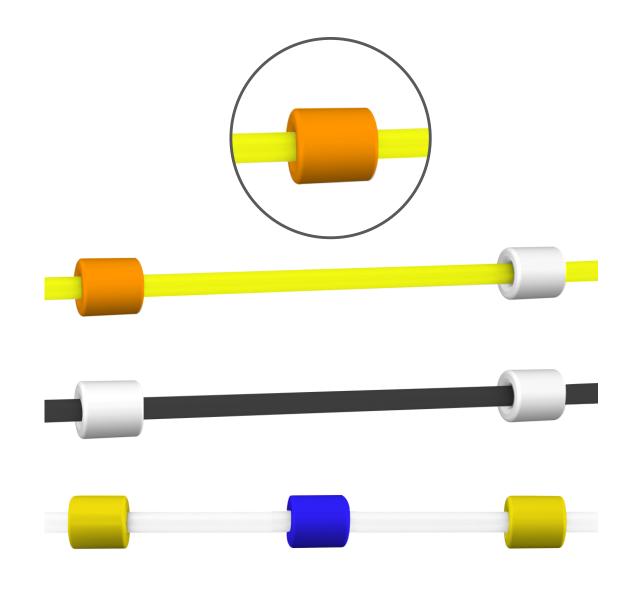


Traditional Pump Tubing

ProLok™ Peristaltic Pump Tubing

Features & Benefits:

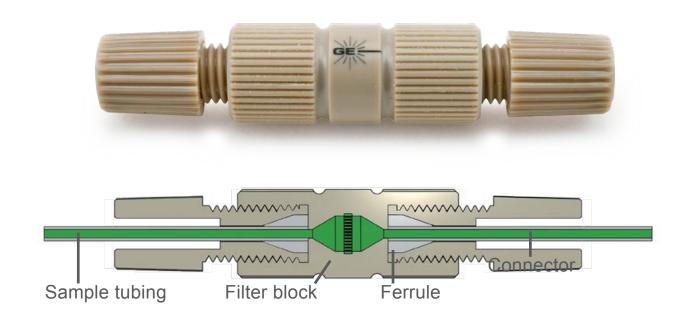
- **1. Enhanced Bonding Strength:** The color tab now features twice the surface area, ensuring a stronger and more secure attachment to the pump tubing.
- **2. Durability:** Designed to prevent premature failure, the reinforced tabs eliminate issues with loose tubing connections.
- **3. Superior Material Quality:** Crafted from high-quality Tygon[®] material, this product delivers premium performance and exceptional consistency.
- **4. Consistent Compatibility:** Maintains the same GE part numbers for seamless integration.
- **5. Precisely controlled tab spacing** designed to meet and exceed industry standards for ICP-OES and ICP-MS peristaltic pumps.



ProLok™ Peristaltic Pump Tubing

Suggestions for Common Challenges

Guardian In-Line Particle FIIter P/N 70-803-1108:





Benefits

- Prevent large particles from clogging your nebulizer
- Insert between probe and nebulizer
- Re-usable PEEK filter (120 μm)
- Easily backflush to remove build up

In-Line particle filter: "So far it has worked great, we have noticed significantly less clogged lines."

Fertilizer manufacturer - USA

In-Line particle filter: "By the way, the particle filters that we have purchased are working out very well with our soil sample analyses on our ICP-OES units, <u>have saved a lot of headaches with blocked nebulizers!"</u>

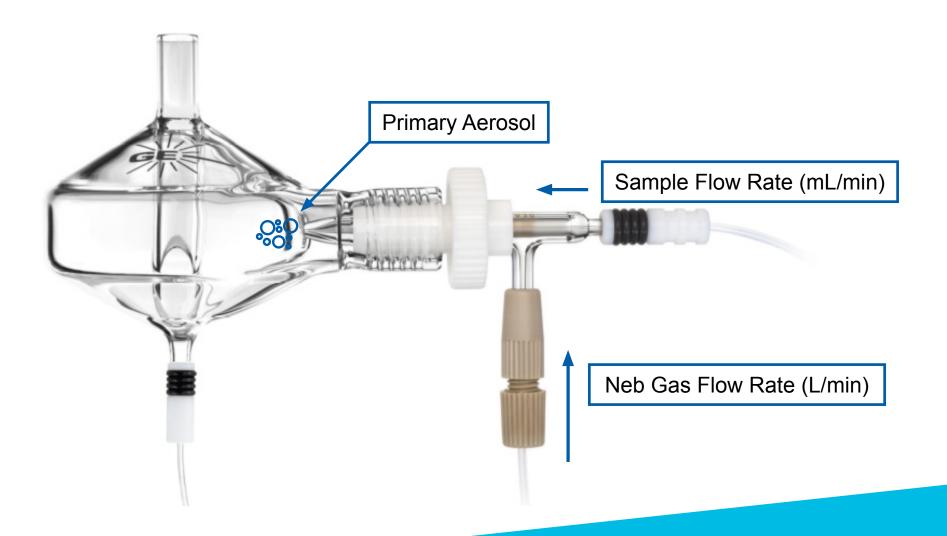
Soil & Plant Laboratory - Australia



2. Aerosol Generation: Nebulizer & Spray Chamber

- Only the smallest droplets (<10 µm) are transmitted to the plasma & 95-98% of nebulized sample is drained as waste.
- Primary aerosol is produced by the nebulizer
- Droplet size decreases as argon gas velocity increases and sample liquid flow rate decreases
- For optimal performance, aim for a higher concentration of droplets with a diameter of <10 µm

Quality of Aerosol \(\text{Quality of Results} \)





Concentric Nebulizers: Key Design Considerations (I)

- The most commonly used nebulizers due to their: efficiency, stability, ruggedness, natural aspiration
- Constant and reproducible nebulization efficiency is crucial for accurate analysis = nebulizers evaluated by stability

Potential Design Challenges:

- All ICP Nebulizers are not created equal
- Inconsistent taper which effects seal and depth within spray chamber

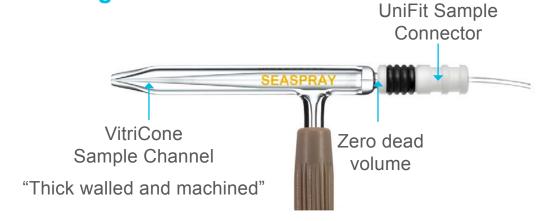
Sample channel constructed from drawn-out capillary tubing:

- The tube is tapered, encouraging salt deposition as the tube narrows.
- The tubing if very fragile and can vibrate under the influence of the high speed argon flow, leading to poor precision.
- It is very difficult to reproduce the same performance with different nebulizers.

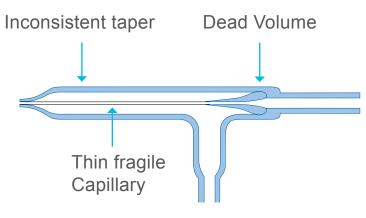


Concentric Nebulizers: Key Design Considerations (II)

GE Design:



Other Nebulizer Design:



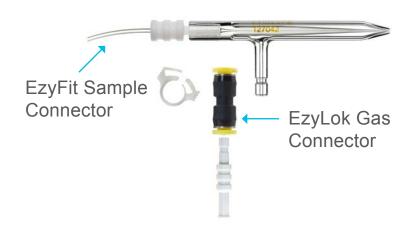
Practical Design Features of GE DC Nebulizers:

- VitriCone[™] is constructed from a heavy glass capillary which is machined to very high tolerances
- Resists blockage: A uniform sample channel prevents particulate trapping
- The rugged precision machined capillary resists vibration
- The industry's tightest tolerances ensure that each nebulizer will perform to the same high standards as the previous one

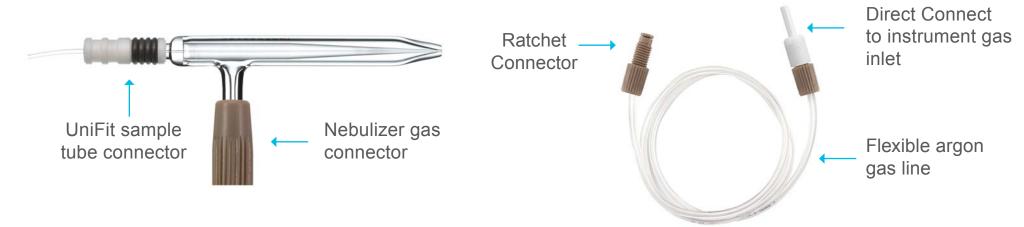
Design Considerations: Nebulizer Sample and Gas Connector

Optimal connection solutions support achieving accurate and reproducible results:

GE Old Design:



Current GE DC Nebulizer Developments



UniFit sample line connector:

- UniFit connector slides easily over the sample arm and creates an excellent seal
- Minimizes Dead Volume: Faster washout compared to EzyFit

Inert metal-free argon connector:

To prevent contamination and false positives (vs. EzyLok)

Instrument-specific Direct Connect flexible argon line:

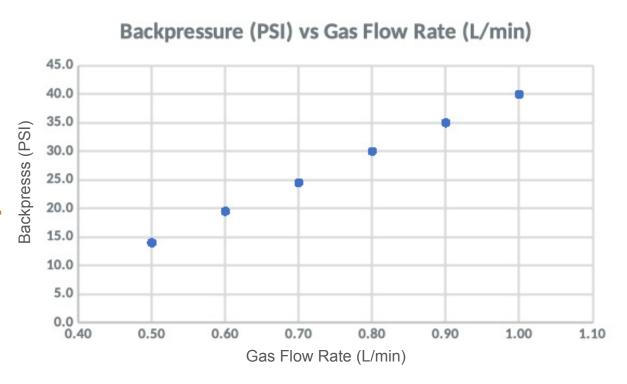
- Unlike hard tubing, which can restrict gas flow, the DC design ensures efficient, consistent gas flow
- Reliable ratchet fitting: Ensures leak-free gas connection

Nebulizer Selection

Selecting the right nebulizer requires careful consideration of various factors:

Nebulizer		TDS (%)	Particulates (µm)	HF	Precision	Purity	Material
SeaSpray™		20	75	No	High	Good	Glass
MicroMist™		15	40*	No	High	Good	Glass
Conikal™		5	75	No	High	Good	Glass
Slurry™		1	150	No	High	Good	Glass
Quartz SeaSpray	тм	20	75	No	High	Excellent	Quartz
OpalMist™		15	75*	Yes	High	Excellent	PFA
DuraMist™		30	75*	Yes	High	Good	PEEK
VeeSpray™	SHEET SHEET	30	300	Yes	Moderate	Good	Ceramic

^{*} Varies with nebulizer uptake



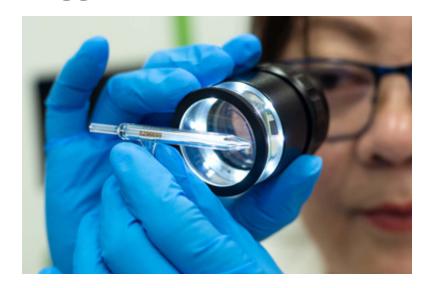
Important Nebulizer Operating Parameters

Example: GE P/N A13-<u>1</u>-UM<u>04</u>

- Optimum nebulizer gas flow = 1.0 L/min (40 psi)
- Sample uptake rate ≤ 0.4 mL/min

Nebulizer: Troubleshooting

Suggestions:



Verify the nebulizer back-pressure after instrument warm-up:

- 1. Low nebulizer back-pressure and a loss in sensitivity can indicate a leak on the supply line:
- Check Ar nebulizer gas connection at the instrument and at the nebulizer gas arm
- Nebulizer gas supply tubing can harden over time, losing their flexible, gas-tight seal
- Argon loss: Even a 1% loss can lead to significant changes in ICP analytical results



- 2. High nebulizer back-pressure can indicate a partially blocked or clogged nebulizer:
- Clean nebulizer or replace if necessary

Record your normal sample uptake rate

• A change in uptake rate can indicate a blockage, worn pump tubing or incorrect tension on the pump.

Nebulizer: Cleaning Procedure

Good Practice: To maintain your nebulizer, start and finish each run by nebulizing a mildly acidic blank solution, followed by DIW for 5-10 min.

Practices to avoid:

- Avoid inserting wires into the nebulizer orifice.
- Never touch the nebulizer tip.
- Avoid cleaning glass or quartz nebulizers with HF.
- Do not use ultrasonic baths for glass nebulizers.

For Blockages:

- 1. Initially flush with water using the Eluo.
- 2. Soak nebulizer tip in 25% Fluka for 24 hours. An initial flush of 25% Fluka may be required.
- 3. Flush 3x with water using the Eluo.
- 4. Stubborn deposits may require an additional soaking for 2 hours with 5% HNO₃.
- 5. Flush 3x with water using the Eluo.
- 6. For faster drying, flush with methanol.

P/N 70-ELUO



ICP Nebulizer Maintenance Made Easy Video





Elegra Argon Humidifier



Features:

- No heating or electric power required
- Membrane humidification technology
- Improved signal stability for samples with high TDS
- Inert metal free construction
- Dual-Channel version allows simultaneous humidification of nebulizer & aux. gas

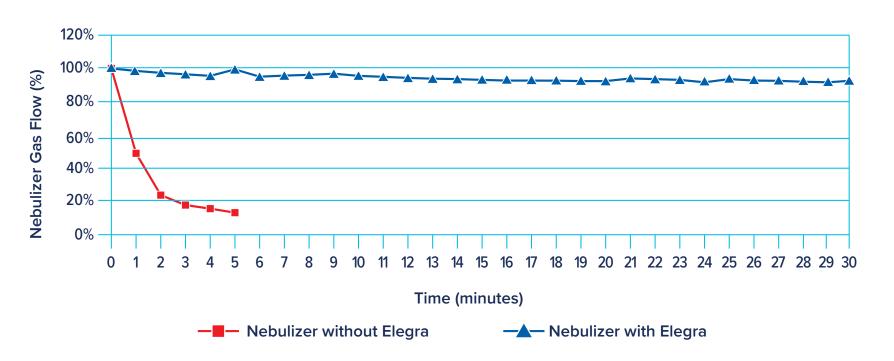
Other tips for high TDS:

- Increasing the auxiliary argon flow will lift the plasma higher off the injector, slowing salt buildup at the injector tip
- Extended rinses in between each sample are also recommended



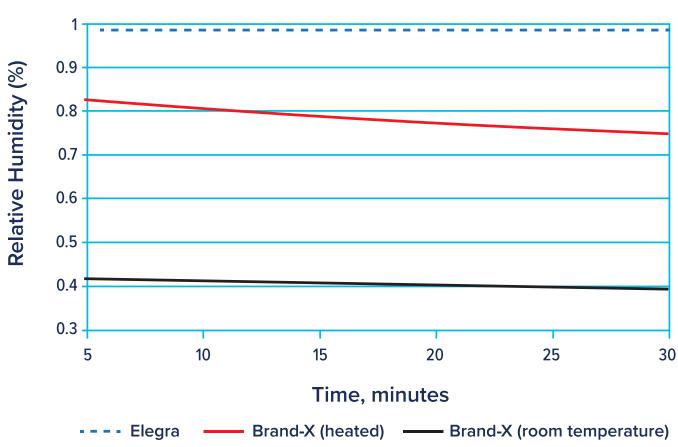
Elegra Argon Humidifier: Performance

Concentric Nebulizer with 25% TDS Tolerance



- Conikal nebulizer typically with up to 5% TDS tolerance
- Added moisture from Elegra prevented nebulizer clogging

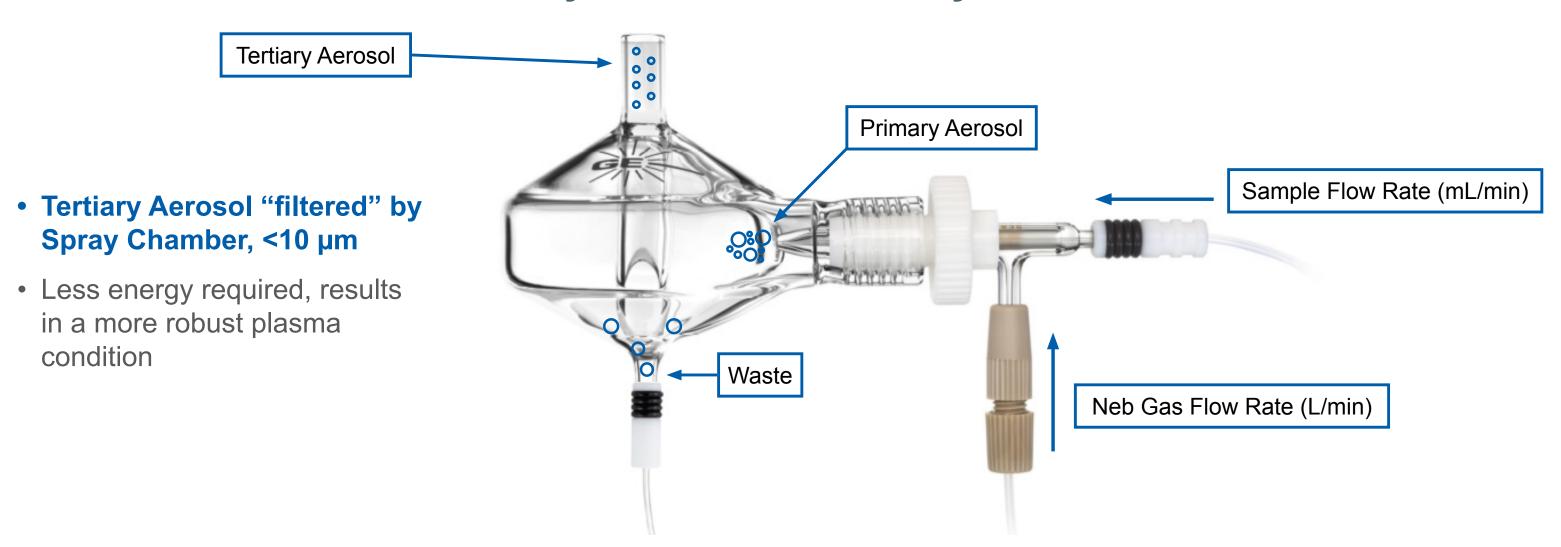
Percent Humidity Relative to Elegra (Elegra=1)





Precision & Sensitivity: Design Considerations

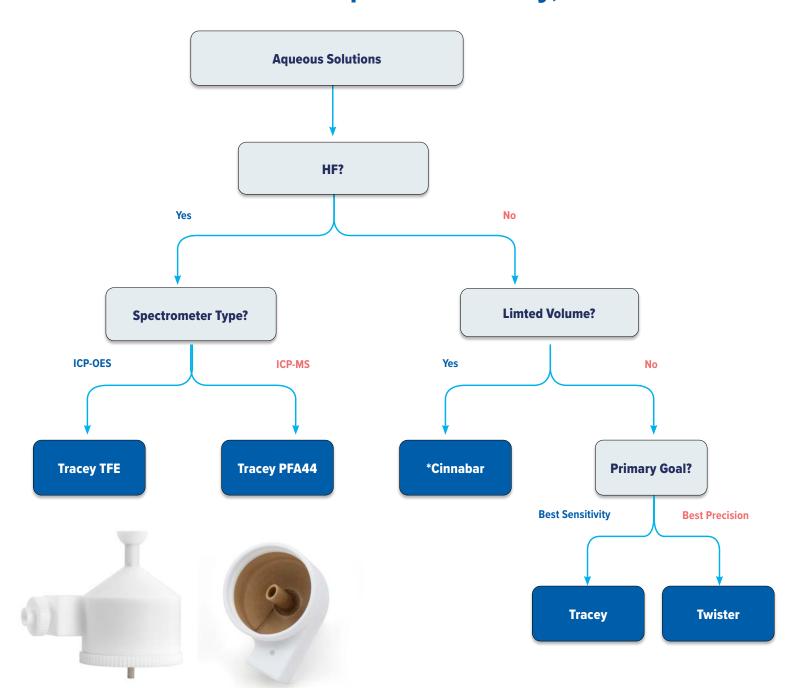
Quality of Aerosol \(\prec{Q}{\text{uality of Results}} \)



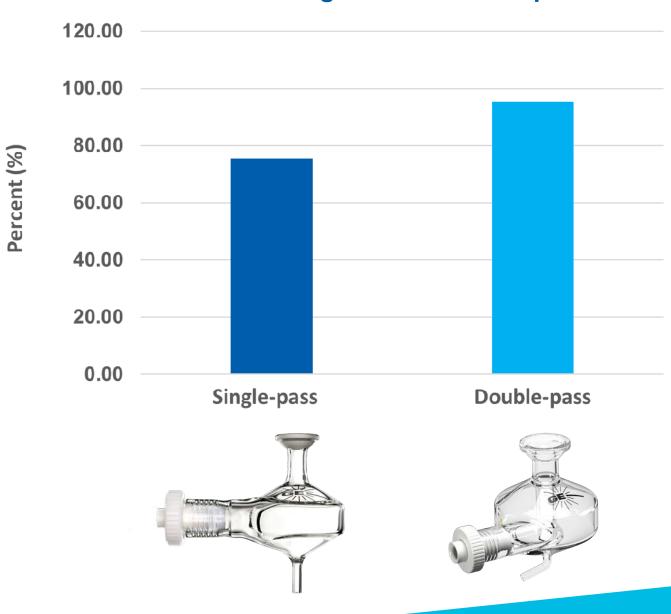
Smaller Droplets Require Less Energy = Efficient Ionization

Spray Chambers: Selection

Profound Effect on: Transport Efficiency, Precision and Washout



Percentage of Volume < 10µm

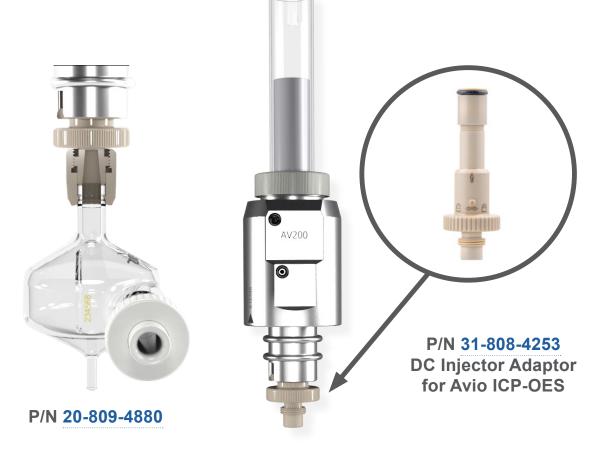


Latest Design: Direct Connect (DC) Spray Chambers

Features & Benefits:

- 1. Inert DC Connection: PEEK Construction ensures durability and chemical resistance. No ball joint clamps that corrode over time
- 2. Consistent Alignment: Provides precise alignment for enhanced accuracy and efficiency
- 3. Efficient Washout: 30mL low-volume cyclonic chamber with Helix CT technology
- 4. Cost-Effective: More affordable than traditional glass spray chambers
- **5. Wide Compatibility:** Fits most common ICP-OES models with E-Torch, D-Torch, and SDT/ FDT

DC Spray Chamber Video





DC PEEK Spray Chamber

Benefits of Tracey DC PEEK Spray Chamber:

- HF resistance up to 5%
- Excellent wetting characteristics of PEEK ensure the wetting properties are retained with general maintenance
- Spray chamber doesn't require internal surface treatment compared to TFE or PFA spray chambers
- Lower cost structure vs other HF spray chambers

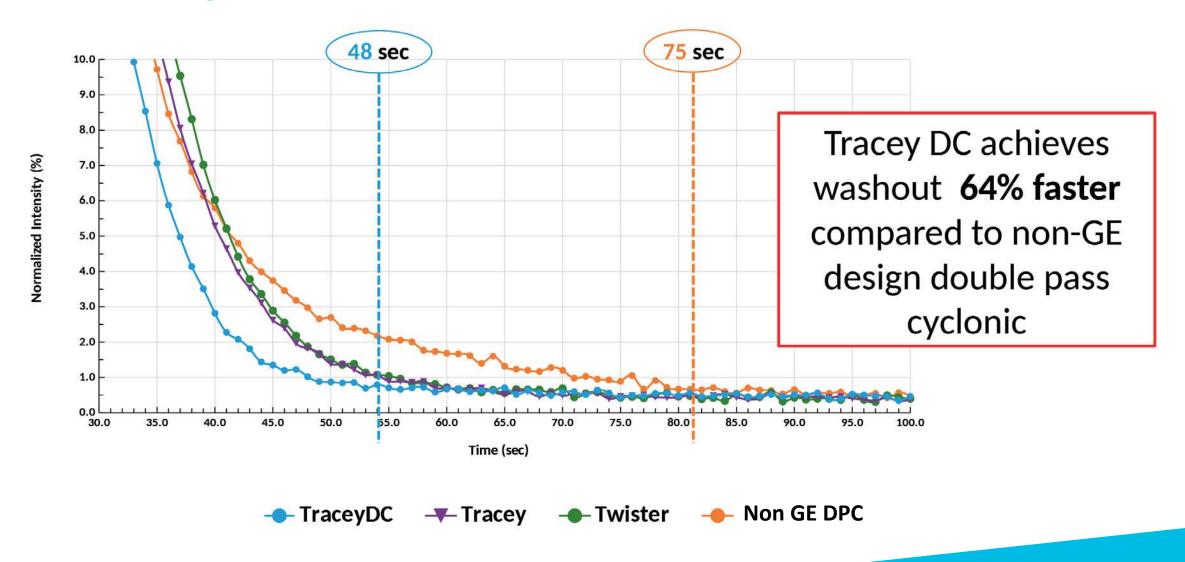
No metal clamp required





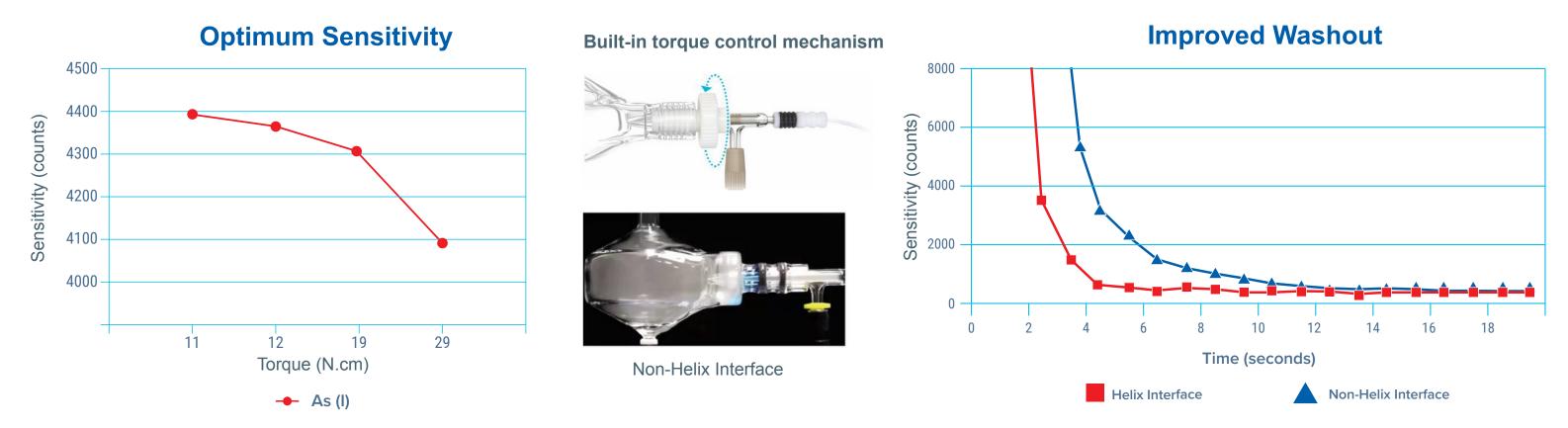
Direct Connect (DC) Spray Chambers

Washout Profiles for 1 ppm Hg:



Spray Chambers: Helix CT Interface

Helix CT: Constant Torque = Reproducible day-to-day ICP Performance



Download the Helix CT ICP Spray Chamber Application Note

Watch the Washout benefits the of Helix seal video



Spray Chambers: Maintenance

Suggestions for Glass Spray Chambers:

- **Do not:** use HF, sonicate, nor use metal or ceramic brushes.
- **Daily cleaning:** Start and end analysis by nebulizing mildly acidic blank followed by DI water.
- Initial cleaning: Nebulize 2.5% Fluka RBS-25 for 15 mins followed by DI water.
- Thorough cleaning: Overnight soak in 25% Fluka followed by DI water rinse.
- Check Helix CT seal and UniFit drain line, replace as needed.

Important note: Our glassware nebulizers, spray chambers, and torches are supplied clean and ready to use.

Care and Maintenance of Inert Spray Chambers



Replace Helix CT seal, e.g. **P/N 70-803-1456**



Replace UniFit drain line, e.g. **P/N UFT-16-75**

3.Torch (& Injector)

What are the common challenges encountered when using ICP Torch?



- HF
- High TDS

Organics

- Loss of Sensitivity: Blocked injector in torch
- Poor Plasma Stability
- RF coil alignment: to reduce devitrification

- Effects of Harsh Matrices: High TDS;
 Organics
- Worn or deformed torch
- Devitrification

Torch: Selection



ICP Torch Designs:

1. Single piece quartz torch:

General use torch: Lower initial cost structure with no removable parts

2. Semi-demountable torch:

Enables injector interchangeability without torch replacement:

- Narrow bore quartz, 1.0mm or less: volatile organics
- Large bore quartz, 2.0mm or greater: High TDS
- Ceramic (alumina): HF-containing samples

3. D-Torch:

Removable: injector, outer tube

4. Fully demountable torch (FDT):

Removable: injector, intermediate tube, outer tube

Torch: Demountable D-Torch

The D-Torch is a cost-effective alternative for any laboratory with a moderate workload.

Benefits:

- Replace just outer tube (fastest to degrade)
- Alumina intermediate tube, which resists wear and is tolerant to high temperatures, high TDS and acid attack
- In contrast, other demountable torch designs typically feature quartz intermediate tubes, which add to consumable costs

*The D-Torch is covered by US Patents



D-Torch Installation Video for Agilent® 5100/5110/5800/5900



D-Torch with Ceramic Outer Tube

Ceramic D-Torch:

- High Li conc. can degrade the torch's outer tube over time.
- The demountable option allows for replacing only the outer tube, avoiding the need to replace the entire torch.
- Injector: Alumina (~1.8 mm)
- Ceramic outer tubes outlast quartz, reducing maintenance, cleaning, and downtime, especially for high-TDS samples.
- Provides a higher average signal intensity



Analyte	Ceramic Outer Tube	Quartz Outer Tube	% Increase
As	173	148	17
Cd	4259	3367	26
Co	1050	855	23
Cr	5490	4435	24
Cu	5258	4558	15
Fe	3408	2767	23
Mn	49529	40237	23
Мо	954	778	23
Ni	721	584	24
Pb	285	226	26
Sb	326	278	17
Se	102	90	13
Ti	185	146	27
V	4677	3815	23

Six hours of running 10 % NaCl







Ceramic outer tube



The Role of the RF Coil

RF Coil Condition: Alignment, plating, and cleanliness improve energy transfer.

Key Factors for Energy Transfer:

1. Alignment

• Ensures a well-shaped and consistently positioned plasma.

2. Correct Dimensions

- Essential for circuit tuning and stability.
- Minor deviations can alter resistance and inductance.

3. Base Metal & Plating

- Copper: Cost-effective but oxidizes quickly.
- Silver: Best conductivity but tarnishes.
- Gold: Best corrosion resistance, slightly less conductive.





ICP-MS Cones

Cone Environment

- High temperature (6,000 8,000 K): Thermal degradation
- Chemical degradation (exacerbated by TDS, acid content, organic solvents, etc.)

Key Factors for High Quality Cones:

- 1. Purity of Raw Materials:
- Ensures performance and durability.

2. Advanced Machining:

- CNC, laser, and electron beam welders for precise manufacturing.
- Glass Expansion Warranty
- Refurbishment program for Pt Cones





General Guidelines on Cone Material

Copper:

- Often the lowest-cost option
- Most-susceptible to matrix effects, corrosion, and sample deposition
- Most-efficient heat transfer this means it "runs colder"
- Often need more frequent cleaning

SZT001-Cu S338141

Nickel:

- Often the "standard" option
- Good thermal and chemical resistance –more than Cu but less than Pt
- Moderate heat transfer: runs "hotter" than Cu but "colder" than Pt.





Platinum:

- Typically the most durable and longestlasting option
- Excellent chemical resistance: Suitable for aggressive acids or high-matrix samples
- Least-efficient heat transfer
 — this means
 it "runs hotter" than both Cu and Ni
- Can be refurbished



When to Clean Cones

Suggestions:

- Physical observation of cone condition using Magnifier Inspection Tool (P/N 70-803-1923) or indicated by the data and results
- Sampler cone is more exposed to the plasma: more frequent cleaning
- Always end the day by aspirating an acidified rinse solutions followed by UPW

Experimental indicators of cone cleaning:

- Increased background
- Memory effects
- Decreased sensitivity
- Change in vacuum

Observational indicators for cone cleaning:

- Visible deposits near or in the orifice
- Distorted Orifice





Magnifier
Inspection Tool
P/N 70-803-1923

How to Clean Cones

Suggestions:

3 recommended methods, from gentlest to most aggressive:

- Method A (Soak in Citranox);
- Method B (Sonicate in Citranox);
- Method C (Sonicate in Nitric Acid)

Order of severity (Don't use aggressive cleaners if it isn't needed):

- 1. Fluka RB-25
- 2. Citranox
- 3. HNO₃

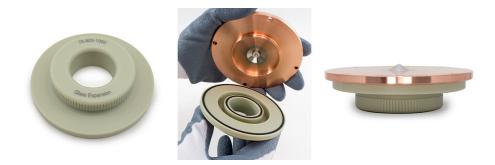
Use of a ConeGuard is highly recommended:

Damage to the threads, whether by corrosion or distortion, can lead to premature failure of the cone or worse - damage to the interface housing

Tips on Care & Maintenance



"Click here"
to view the Cone
Resouce Guide



ConeGuard with Agilent® Sampler Cone:

Protects the threads during the cleaning process

Helpful ICP Resources

Application dedicated SIS solutions: Suggestions for Common Issues

Agilent®: 5100, 5110, 5800, 5900

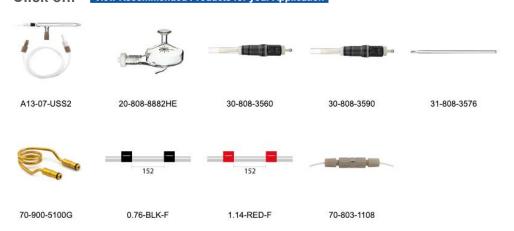
View All Products for this Model

Instrument Applications

- Animal feed
- Brines and salts
- · Chemicals and fertilizers
- Clinical and forensic materials
- Drinking, ground and surface water
- Food and drink
- Geological with HF
- Geological without HF
- Isotopic Analysis of Minerals
- Metals
- Petrochemicals
- Plants
- Soil and sediment with HF
- Soil and sediment without HF
- Waste water and sludge
- Wear Metals in oil

Example: https://www.geicp.com/cgi-bin/site/wrapper.pl?c1= Productsbyinstrument&inst=5100-5110-5800-5900&appno=4

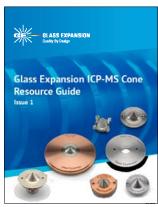




- Videos
- Webinars







Website





Flyers





Instructions



Application Notes



Thank You



Glass Expansion - Europe Weilburg, Germany

Europe

Friedenbachstrasse 9 35781 Weilburg Germany

Phone: +49 6471 3778517

Email: gegmbh@geicp.com

www.geicp.com

