

GLASS EXPANSION Quality By Design

A Practical Guide to ICP Sample Introduction System and Maintenance



Dr. Maja Budanovic

ICP Product Specialist Glass Expansion GmbH Contact: mbudanovic@geicp.com

www.geicp.com

Introduction: About Glass Expansion

- GE have been specializing in sample introduction components for ICPs since 1983
- Global recognition for manufacturing precision and reliability
- We are dedicated to:
 - Quality By Design
 - Value
 - Analytical Performance
 - Better usability

Industry Standard Trademark Designs:

• Conikal[™], SeaSpray[™], Tracey[™], Twister[™], IsoMist XR[™], Helix CT[™], others

We provide our customers with full technical support:

- Diverse industry/research partnerships
- Extensivley equipped laboratory with 6 in-house ICPs
- Satisfaction Guarantee: Guaranteed to meet or exceed OEM specifications.



SeaSpray[™] & MicroMist[™] Nebulizers



Tracey[™] & Twister[™] Cyclonic Spray Chambers with Helix CT[™]



D-Torch[™] Demountable Super Torch

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



Sample Delivery System

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

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 eliminates crushed and damaged tips due to misalignment
- **Combines drip-resistance and built-in filter** to minimize crosscontamination, while protecting the nebulizer and capillary tubing
- Interchangeable UniFit sample lines IDs: 0.3, 0.50, 0.75 and 1.0mm
- Autosamplers: Teledyne Cetac, PE S20, AimLab, and Agilent SPS3/SPS4





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

Tag Colours	ID (mm)
orange/black	0.13
orange/red	0.19
orange/blue	0.25
orange/green	0.38
green/yellow	0.44
orange/yellow	0.51
white/yellow	0.57
orange/white	0.64
black/black	0.76
orange/orange	0.89
white/black	0.95
 white/white	1.02
white/red	1.09
red/red	1.14
red/grey	1.22
grey/grey	1.30
yellow/yellow	1.42
yellow/blue	1.52
blue/blue	1.65
blue/green	1.75
green/green	1.85
purple/purple	2.06
purple/black	2.29
purple/orange	2.54
purple/white	2.79
black/white	3.17

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

Trident CT[™] Internal Standard Addition Kit

- Efficient mixing chamber ensures complete mixing of sample and reagent.
- ConstantTorque[™] (CT) ratchet fittings for a durable, leak-free seal on all connections.
- Consistency in torque application helps maintain the reliability and performance of the mixing chamber.
- Click here for P/N 60-703-1179 Trident CT Internal Standard Kit inclusions and options.
- Trident CT for HF Solutions P/N 60-808-1150



Trident CT P/N 60-703-1179

Sample Delivery System: Maintenance

Suggestions for Maintaining Pump Tubing:

- Pre-Stretch Tubing and Maintain the proper tension on tubing
- Frequent Replacements: Pump and capillary tubing can be a source of contamination
- Lubricate pump rollers (EzyGlide Cloth):
 - \circ Reduce wear and increase lifespan by minimizing friction
 - \circ Stabilize sample delivery by reducing pulsations

Regular cleaning protocols:

- Implement a rinsing protocol between sample types using dedicated solutions to remove residual materials
- Start and finish each run by aspirating a mildly acidic blank solution or the sample matrix, followed by DIW for 5-10 min

Check out the Pump Speed and Sample Uptake Calculator.



ove residual materials trix, followed by DIW for 5-10 min



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 ~ **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)



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





Quality By Design

Design Considerations: Nebulizer Sample and Gas Connector

Optimal connection solutions support achieving accurate and reproducible results:

GE Old Design:





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



Current GE DC Nebulizer Developments

Direct Connect to instrument gas inlet

Flexible argon gas line

Nebulizer Selection

Selecting the right nebulizer requires careful consideration of various factors:



* Varies with nebulizer uptake

Precision & Sensitivity: Design Considerations

Quality of Aerosol ~ **Quality of Results**



Smaller Droplets Require Less Energy = Efficient Ionization

Sample Flow Rate (mL/min)

Neb Gas Flow Rate (L/min)

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**



P/N 20-809-4880





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:



www.geicp.com

Tracey DC achieves washout **64% faster** compared to non-GE design double pass cyclonic



Design Considerations: Spray Chamber Interface

Drawbacks of an O-ring Seal:

- **Contamination:** Dead volume around the O-ring can trap contaminants.
- Chemical Resistance Limitations: O-rings may degrade with strong acids or organic solvents.
- **Difficult Maintenance:** Replacing O-rings often requires tools and can be challenging.
- Fragility: Bonding of the O-ring to the nebulizer.



UniFit connector helps prevent the pulsating effect often associated with ineffective draining caused by pushfit connectors found in other spray chamber designs.

Latest Developments:



Helix CT fixes nebulizer depth and provides the optimum interface for minimizing dead volume.



Spray Chambers: Helix CT Interface

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



Built-in torque control mechanism



Download the Helix CT ICP Spray Chamber Application Note

Improved Washout



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. ۲
- 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

Conikal[™], SeapSpray[™], MicroMist[™], Slurry[™]

ICP Nebulizer Maintenance Made Easy Video



Quality By Desigr

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**



GLASS EXPANSION

26

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



www.geicp.com



Quality By Design

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**



Standard quartz torch body



Ceramic outer tube



As

Cd Со

Cr

Cu Fe

Mn Мо Ni

Pb

Sb Se

Ti

V

Six hours of running 10 % NaCl

www.geicp.com

te	Ceramic Outer Tube	Quartz Outer Tube	% Increase
	173	148	17
	4259	3367	26
	1050	855	23
	5490	4435	24
	5258	4558	15
	3408	2767	23
	49529	40237	23
	954	778	23
	721	584	24
	285	226	26
	326	278	17
	102	90	13
	185	146	27
	4677	3815	23



Quality By Design

Latest Design: E-Torch

Benefits

- Made from PEEK, PTFE & Quartz
- Shipped with P/N 31-808-2836, Capillary Quartz Injector 2.0mm (EMT)
- Interchangeable injectors, (Quartz, Ceramic, Sapphire)
- Compatible with Tracey[™] DC spray chamber which eliminates ball joint clamps
- Performance equivalent to the D-Torch
- Ceramic tube set available on request for HF and high TDS applications.
- Low cost Inner and Outer tube assembly which simplifies deposit removal maintenance and allows for oven cleaning of Carbon deposits.
- Compatible with the Jet Vortex Interface (JVI)





P/N 30-808-4388 E-Torch for Thermo® PRO

PEEK Retainer P/N 31-808-4454 and Ferrule P/N 31-808-4451





D-Torch: Maintenance

Suggestions:

Always wear safety gloves

Carbon Deposits Removal:

- Use a portable hand-held propane torch to burn off deposits.
- Avoid heating the outer tube in a muffle furnace (due to the polymer ferrule).

Salt Deposits Cleaning:

- Soak in a 25% Fluka RBS-25 solution or dilute acid. •
- Stand the tube upright in a beaker, covering the deposits with cleaning solution.

Metallic Films Removal:

- Soak in acid, preferably the same one used for sample preparation.
- Ensure the acid covers the metallic deposits while the tube stands upright.



We recommend that the polymer ferrule is not soaked in acid.



<u>Quality By Design</u>

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.





Benefits of Maintaining your RF Coil:

Key Challenge: Corrosion

- Caused by heat or chemical oxidation.
- Reduces energy transfer efficiency, increasing stress on RF components.

Steps for Prolonging Coil Life:

- Periodic Cleaning: Removes tarnish and buildup.
- Avoid Acid-Based Polishes: Use abrasive metal polish to clean without damage.
- **Replace When Needed:** Replace coils with copper migration or significant tarnish.

Advantages of GE RF Coils:

- High-quality, consistent plating for longer life.
- Supplied on a plastic former for correct dimensions and easier installation.
- Proper alignment minimizes devitrification of the outer tube.





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

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







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





Copper Cones



GLASS EXPANSION Quality By Design

35

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





ConeGuard with Agilent[®] Sampler Cone: Protects the threads during the cleaning process

"Click here" to view the Cone Resouce Guide





Catalogs



Helpful ICP Resources

www.geicp.com

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

Click on:	View Recommended Products for you	
0		
A13-07-USS2	20-808-8882HE	30-808-35
	152	152
70-900-5100G	0.76-BLK-F	1.14-RED



D-F 70-803-1108

38

Thank You



Glass Expansion - Europe Weilburg, Germany

Europe

Friedenbachstrasse 9 35781 Weilburg Germany

Phone: +49 6471 3778517 Email: gegmbh@geicp.com www.geicp.com

www.geicp.com

