GLASS EXPANSION
Quality By Design

ICP Sample Introduction System: Diagnostics and Care

Presenter:

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Organized By:

Instrument Solutions Benelux B.V.

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www.geicp.com

About Glass Expansion

Expertise:

- Over 40 years focused on ICP sample introduction
- Design focus: **Quality, Value, Analytical Performance, Usability**

Engineering & Innovation:

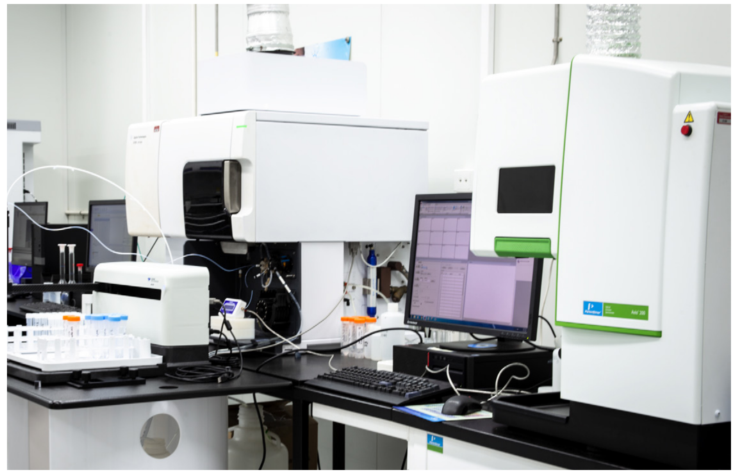
- ~10% annual investment in R&D
- **Advanced manufacturing:** CNC machining, electron beam welding, laser technology, particle analyzer, 3D printing

Quality & Testing:

- **Full control of manufacturing process** from raw material to finished product
- **6 in-house ICP instruments** for testing and quality assurance

GE Warranty: Components design to **meet or exceed OEM specificaitons**

Global Support: Offices in Australia, USA, and Germany

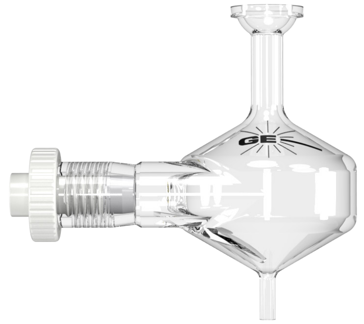


GE fully equipped ICP laboartory for QC and R&D

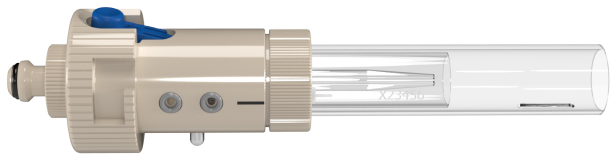
Glass Expansion Germany

Product Lineup

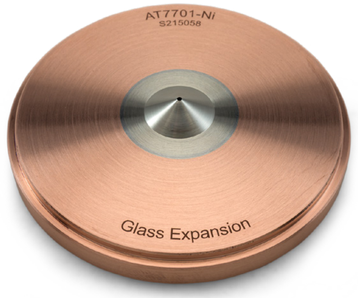
Nebulizers



Spray Chambers



Torches

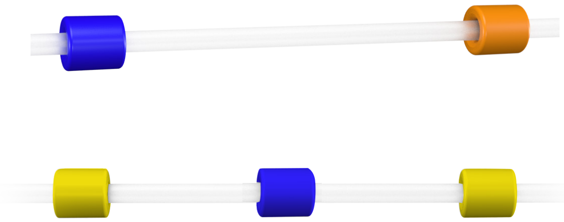


ICP-MS Cones

RF Coils

Accessories

Laser Ablation Adaptors



Peristaltic Pump Tubing

All Products Manufactured to Exacting Specifications

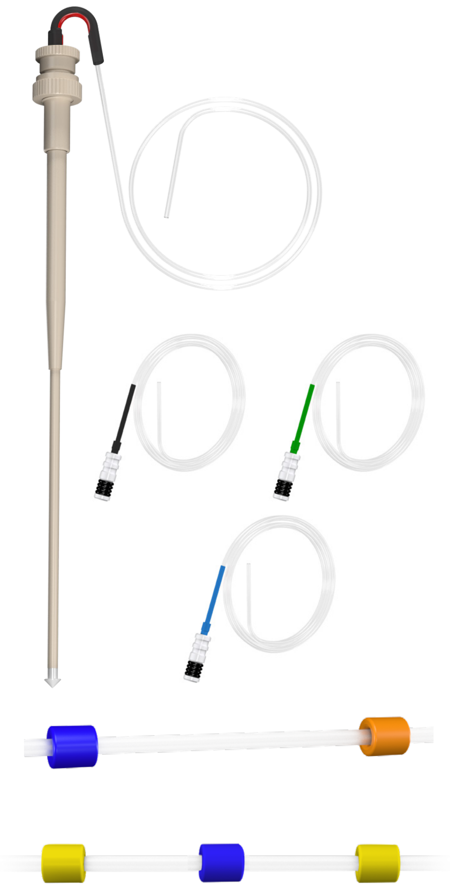
Trademarks: SeaSpray™ , MicroMist™ , IsoMist™ , E-Torch™, D-Torch™ , Elegra™ , Twister™ , and many others

Manufacturers Supported: Thermo® | Agilent® | PerkinElmer® | Shimadzu® | Spectro® | Analytik Jena® | Others

Sample Introduction: Critical Source of ICP Variability

Sample Delivery

Sample Probe & Tubing



Challenges:

Clogging

Tubing misfit/wear

Pump instability

Air bubbles

Impact:

Variations in flow lead to:

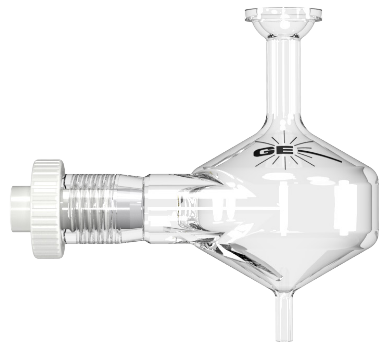
Signal drift/ High RSDs

Inconsistent sample transport

Reduced reproducibility

Aerosol Generation

Nebulizer & Spray Chamber



Challenges:

Blockages

Large/Dense Droplets

Sample Matrix Deposits

Dead volume

Impact:

Inefficient aerosol formation:

Reduces sensitivity and DLs

Results in unstable signals

Plasma Interface

Torch; Injector; RF Coil & Cones

Challenges:

Devitrification

Blockages

Cone deposits

RF coil degradation

Impact:

Signal loss

Reduced stability

Poor accuracy

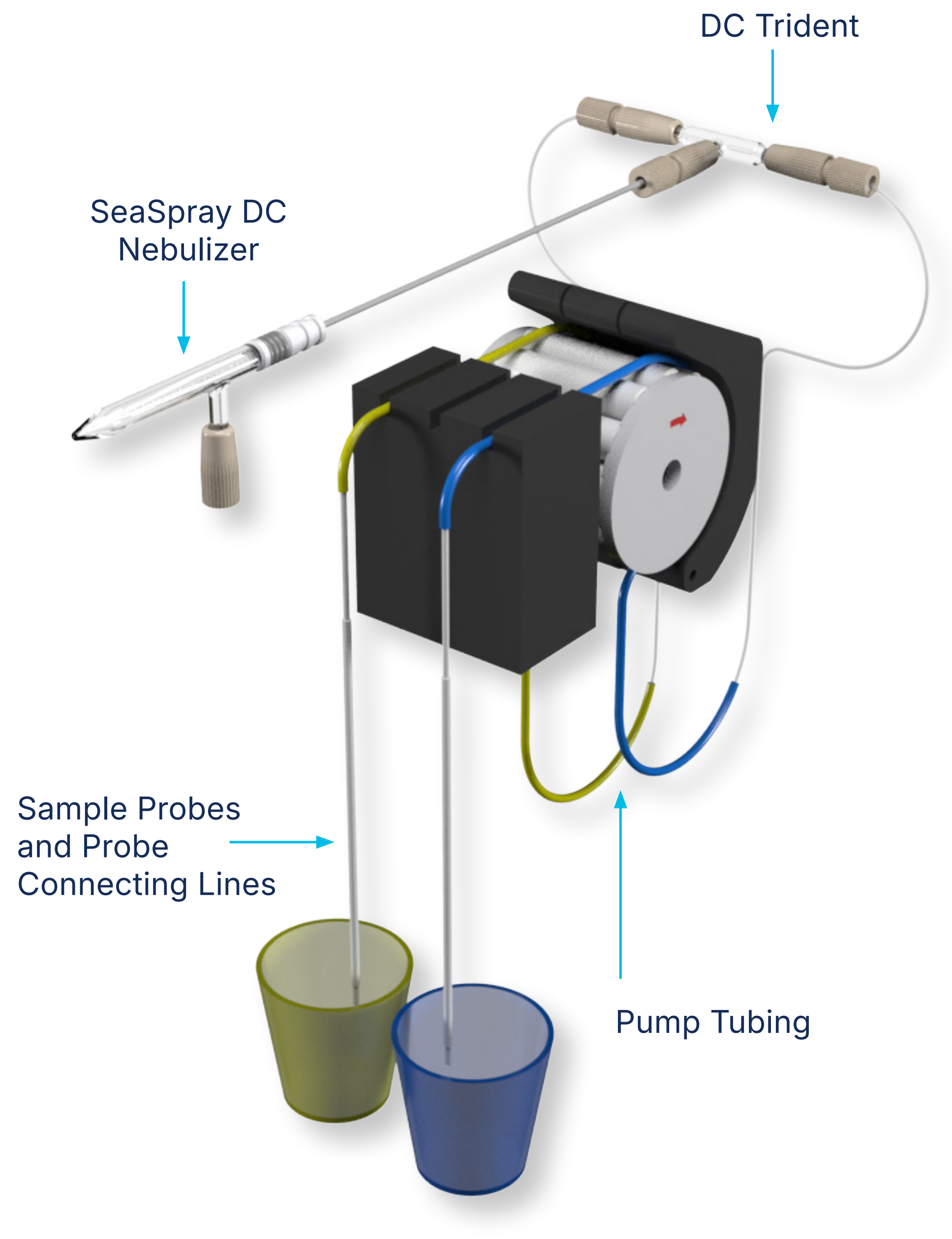
- Since only a small fraction of the sample reaches the plasma, the efficiency of each stage strongly influences analytical performance.

1. Sample Delivery System

The sample delivery system transports liquid samples from the sample tubes to the nebulizer at a controlled and stable flow rate.

Requirements for Optimal Sample Delivery

- Ensure optimal, stable and consistent sample uptake rate
- Unobstructed flow from sample container to nebulizer
- Proper mixing when internal standards are added
- Minimal carryover or contamination between samples



Trident CT™ Internal Standards Addition Kit

Sample Delivery: Component Considerations

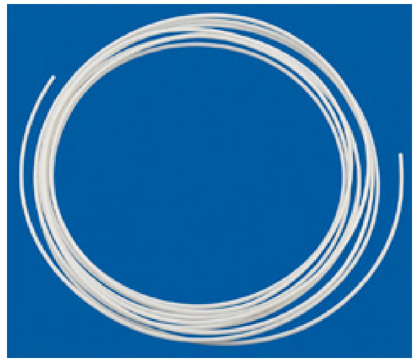
1. Sample Probe:

- Ensure optimal Internal Diameter (ID) Selection:
  - 1.0 mm ID: for high-TDS samples or fast valve systems
  - 0.75 mm ID: standard applications
  - 0.3 and 0.5 mm ID: limited sample volumes

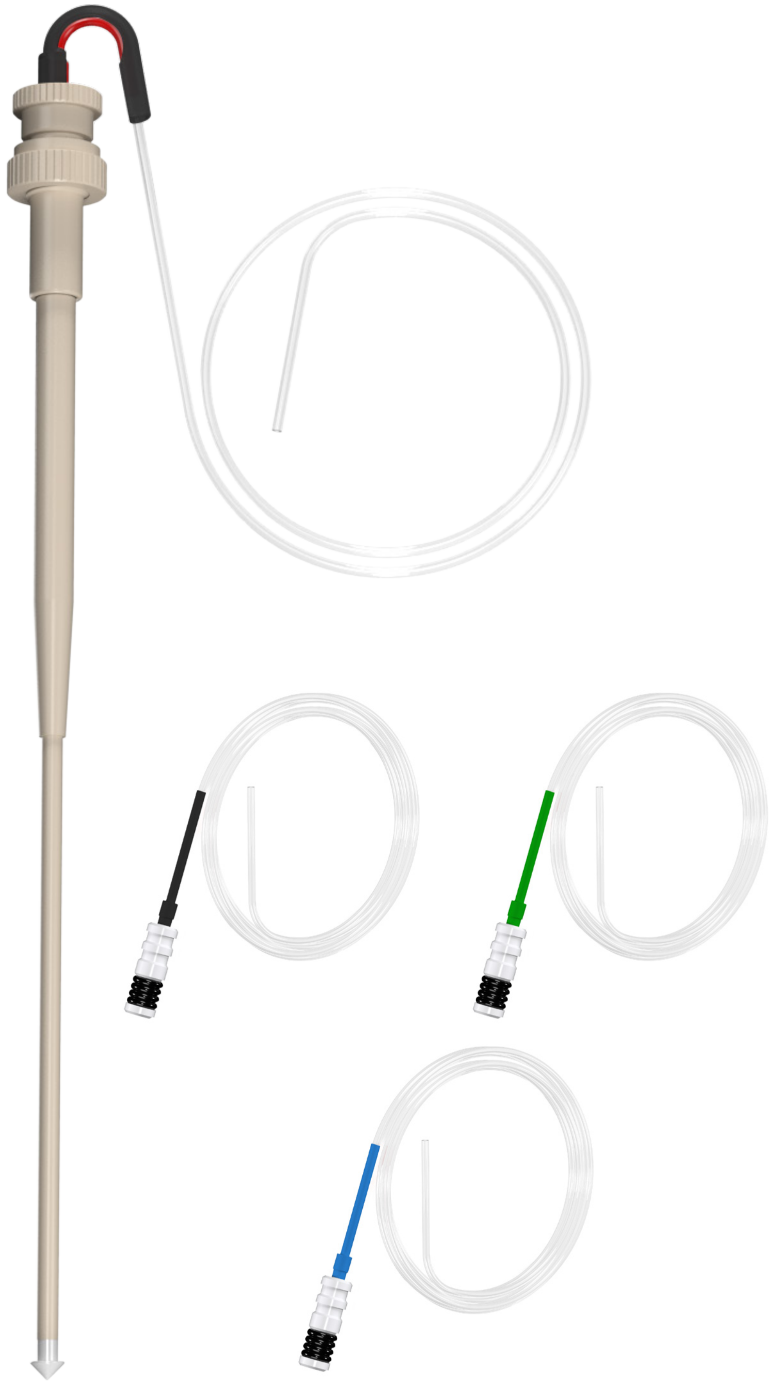
2. Teflon Sample Transfer Tubing:

- Probe → Pump Tubing → Nebulizer
- Typically made from PTFE/PFT/PFA because these materials are:
  - Chemically inert
  - Resistant to strong acids
  - Low adsorption (minimal contamination)

3. Pump Tubing & IS Kit



PFA Sample Tubing 1/16 OD x 0.13mm ID x 10m long



Guardian Probe for Agilent SPS3, SPS4, AIMS, 0.75mm

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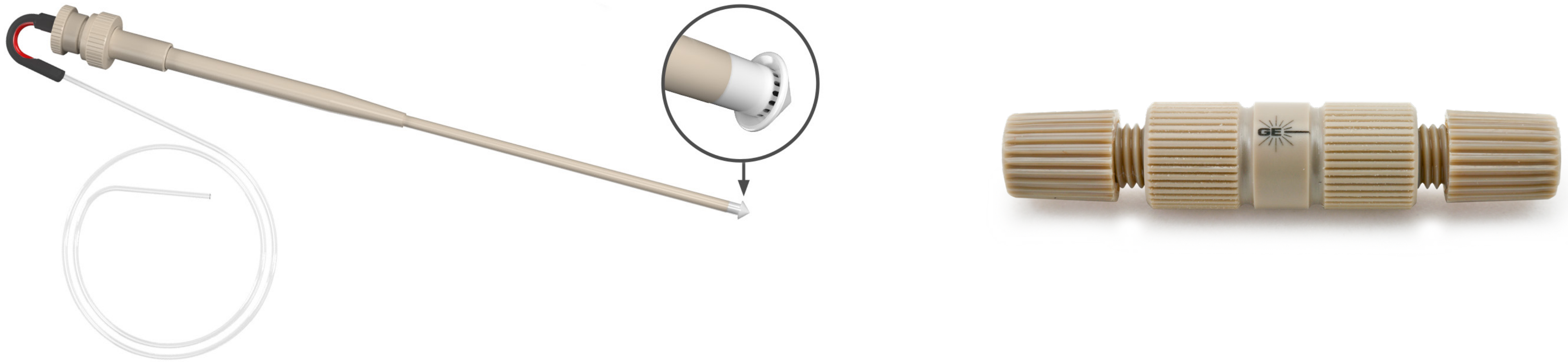
1a. Sample Delivery: Sample Probe

Common Issue	Possible Cause	Practical Recommendation
Unstable Signal (High RSDs) **Reduced or Inconsistent Uptake Rate** **Sudden Signal Loss**	Probe/ probe connecting line partially clogged by particulates or precipitates	• Inspect the probe and connecting tubing (**use a magnifier tool**) • Attempt **backflushing** using the pump in reverse • If unresolved, **remove/replace the affected tubing** (e.g., UniFit™ interchangeable sample lines) • **Apply Guardian accessories** (Guardian Probe with built-in filter or Guardian inline filter) to minimize particulate blockage
Delayed Washout **Memory Effect**	Carryover from previous samples	• **Adjust rinse and washout procedures**. • If available, use **Smart Rinse** in the software; otherwise run a blank before the next sample. • **Guardian Probe will help to minimize carryover.**

10x magnification with LED illumination



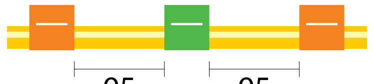
Guardian Accessories help prevent clogs in capillary tubing

1b. Sample Delivery: Pump Tubing Materials

3. Peristaltic Pump Tubing:

PVC Standard (Tygon ST): Economical and suitable for most routine aqueous samples using diluted acid solution.

Solva (Tygon HC or Solvaflex): Special tubing for hydrocarbons, petroleum products and distillates.



Tygon MH: Special PVC of high purity; No additives and no plasticizer; enviro-friendly; highly resistant to solvents (MIBK).

Viton (Fluran): Fluoropolymer elastomer; special tubing for concentrated acids and corrosive solvents.

Chemical Resistance Chart

Important note on service life, temperature, compatibility and chemical resistance:
The data provided in the tables are advisory values and not guaranteed. In all cases customers should conduct tests to ensure compatability with their chemicals and processes.

We recommend:
Place the tubing in the medium to be used for a period of 48 hours. After this time, examine the tubing for signs of swelling, softening or hardening. A judgement can then be made as to the likely suitability of the tubing.

Legend
X **= Satisfactory**
O **= Use only after testing**
U **= Unsatisfactory**
- **= No data available**

Medium	PVC	Silicone	Viton	PVC Solva	Santo-prene
Acetaldehyde	U	X	U	X	X
Acetates (low mol. wt.)	U	O	U	X	X
Acetic acid (<5%)	X	X	X	X	X
Acetic acid (>5%)	X	U	O	X	X
Acetic anhydride	O	O	U	X	U
Acetone	U	X	U	U	U
Acetyl bromide	U	-	-	X	-
Acetyl chloride	U	-	-	X	-
Air	X	X	X	X	X
Alcohols	X	X	X	X	-
Aliphatic hydrocarbons	X	O	U	U	-
Aluminium chloride	X	O	X	X	-
Aluminium sulfate	X	X	X	X	X
Alums	X	-	X	X	-
Ammonia (gas-liquid)	O	X	U	X	X
Ammonium acetate	X	-	-	X	-
Ammonium carbonate	X	-	-	X	-
Ammonium chloride	X	-	X	X	-
Ammonium hydroxide	O	X	X	X	X
Ammonium nitrate	X	O	-	X	-
Ammonium phosphate	X	X	-	X	-
Ammonium sulfate	X	X	X	X	X
Amyl acetate	U	U	U	U	U
Amyl alcohol	X	U	X	U	X
Amyl chloride	O	U	X	U	-
Aniline	O	U	O	X	X
Aniline hydrochloride	O	U	X	X	-
Animal oils	U	O	X	X	X
Antimony salts	X	-	O	X	-
Aqua regia	U	-	O	X	U
Aromatic hydrocarbons	U	O	X	U	-
Arsenic salts	X	-	X	X	X
Barium salts	X	X	X	X	X

Medium	PVC	Silicone	Viton	PVC Solva	Santo-prene
Benzaldehyde	U	U	U	U	X
Benzene	O	U	X	U	U
Benzene sulfonic acid	O	-	X	X	-
Benzoic acid	X	O	X	U	U
Benzyl alcohol	X	-	X	U	X
Bleaching liquors	X	O	X	X	X
Boric acid	X	X	X	X	X
Bromine	X	U	X	X	U
Butane	O	U	X	U	U
Butanol	X	O	X	-	-
Butyl acetate	U	-	U	U	U
Butyric acid	U	-	O	X	X
Calcium salts	X	O	X	X	X
Carbon bisulfide	U	-	X	U	-
Carbon dioxide	X	O	X	X	X
Carbon tetrachloride	O	U	X	X	U
Chloracetic acid	U	-	U	X	U
Chlorbenzene	U	-	X	U	U
Chlorine (wet)	O	U	X	X	U
Chlorine (dry)	O	U	U	X	U
Chloroform	O	U	X	U	U
Chlorsulfonic acid	O	U	U	X	U
Chromatic acid	X	U	X	-	X
Chromium salts	X	-	-	X	X
Copper salts	X	X	X	X	X
Cresol	O	X	X	U	U
Cyclohexanone	U	U	X	U	U
Essential oils	O	-	-	X	X
Ethers	O	U	O	U	U
Ethyl acetate	U	O	U	U	U
Ethyl alcohol	O	O	X	X	X

Medium	PVC	Silicone	Viton	PVC Solva	Santo-prene
Ethyl bromide	U	-	X	X	-
Ethyl chloride	U	U	X	X	U
Ethylamine	U	-	U	X	-
Ethylene chlorohydrin	U	U	X	X	U
Ethylene di-chloride	U	U	X	X	U
Ethylene glycol	X	X	X	X	X
Fatty acids	O	O	X	X	-
Ferric chloride	X	O	X	X	-
Ferric sulfate	X	O	X	X	X
Ferrous chloride	X	O	X	X	-
Ferrous sulfate	X	O	X	X	-
Fluoborate salts	X	-	-	X	X
Fluoboric acid	X	-	-	X	X
Fluo-silicic acid	X	-	-	X	X
Formaldehyde	X	O	U	X	X
Formic acid	X	O	U	X	X
Freon	U	U	O	U	U
Gasoline (non-aromatic)	U	U	X	U	U
Gasoline (high aromatic)	U	U	X	U	-
Glucose	X	X	X	X	X
Glue	X	-	X	X	-
Glycerine	X	X	X	X	X
Hydriodic acid	X	-	X	X	-
Hydro-bromic-acid	X	U	X	X	X
Hydrochloric acid (dil.)	X	U	X	X	X
Hydrochl. acid (med. conc.)	X	U	X	X	X
Hydrochloric (conc.)	O	U	X	X	X
Hydrocyanic acid	X	U	X	X	X
Hydrofluoric acid	O	U	X	X	O
Hydrogen peroxide (dil.)	X	X	X	X	U

Example of a chemical compatibility chart for pump tubing

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Pump Tubing ID

3. Peristaltic Pump Tubing ID:

Ensure optimal ID selection: Directly determines the sample uptake rate to the nebulizer, which affects sensitivity, stability, and washout.

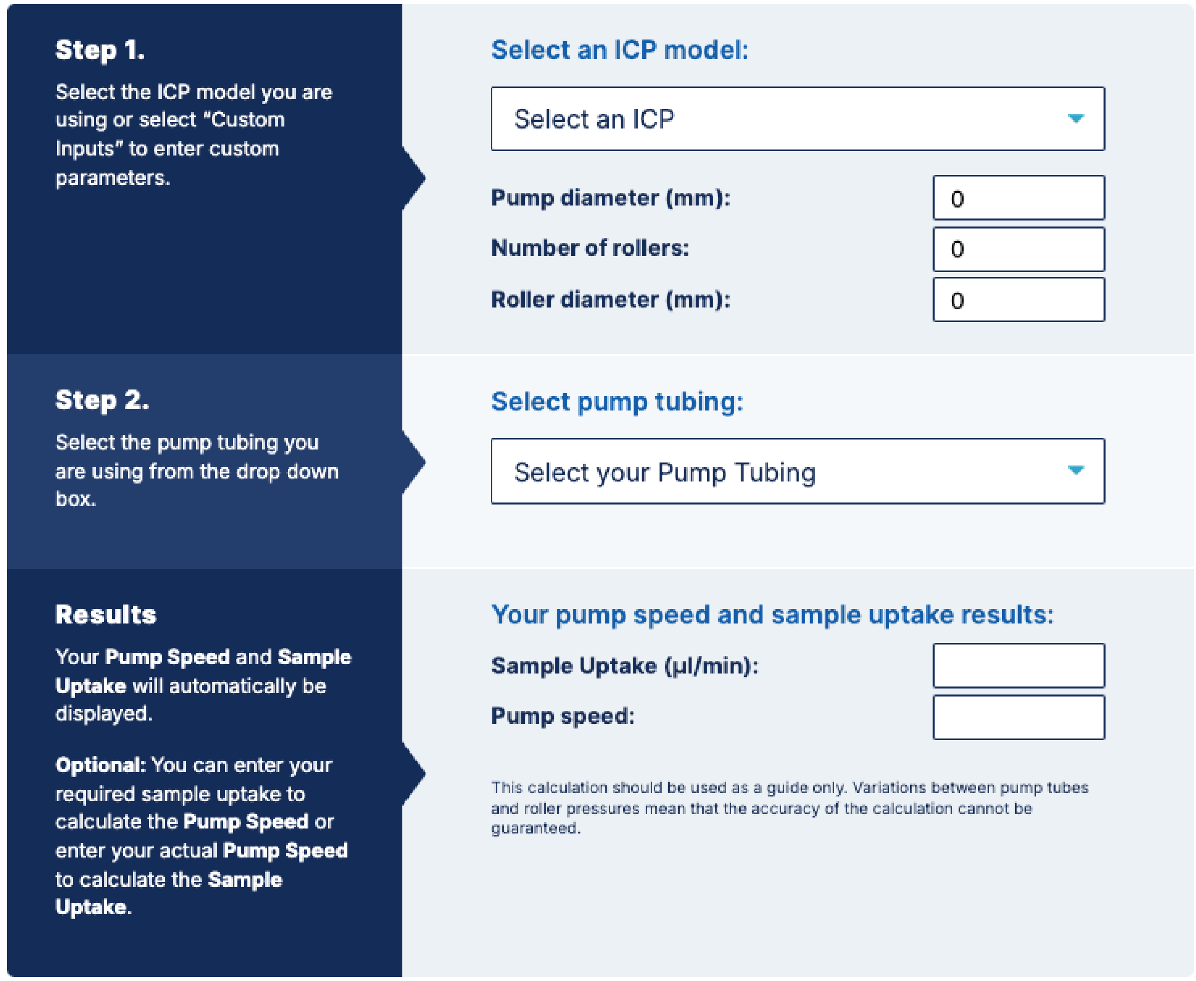
Smaller ID Tubing (0.2 – 0.4 mm): Ideal for precise, low-flow applications.

Larger ID Tubing (0.76 – 1.02 mm): Suitable for higher flow rates and sample volumes.

Internal standard tubing (0.25 – 0.38 mm): Controls IS flow and dilution.

Waste tubing (1.30 – 1.65 mm): Ensure efficient drainage.

Pump Speed and Sample Uptake Calculator



∗Use Sample Uptake Calculator (GE website or software) to estimate flow rate, then verify using clean/new SIS.

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Pump Tubing: Practical Recommendations

Performance Considerations

- Tubing elasticity determines consistent sample flow and signal stability.
- Worn or flattened tubing can cause **flow rate variations and high RSD values.**
- Incorrect tubing size may result in improper nebulizer uptake rates.

Maintenance Guidance

- **Inspect pump tubing regularly** for flattening, cracks, or discoloration.
- Replace tubing periodically depending on system usage and sample matrix.
- **Maintain correct pump tension** and roller alignment.

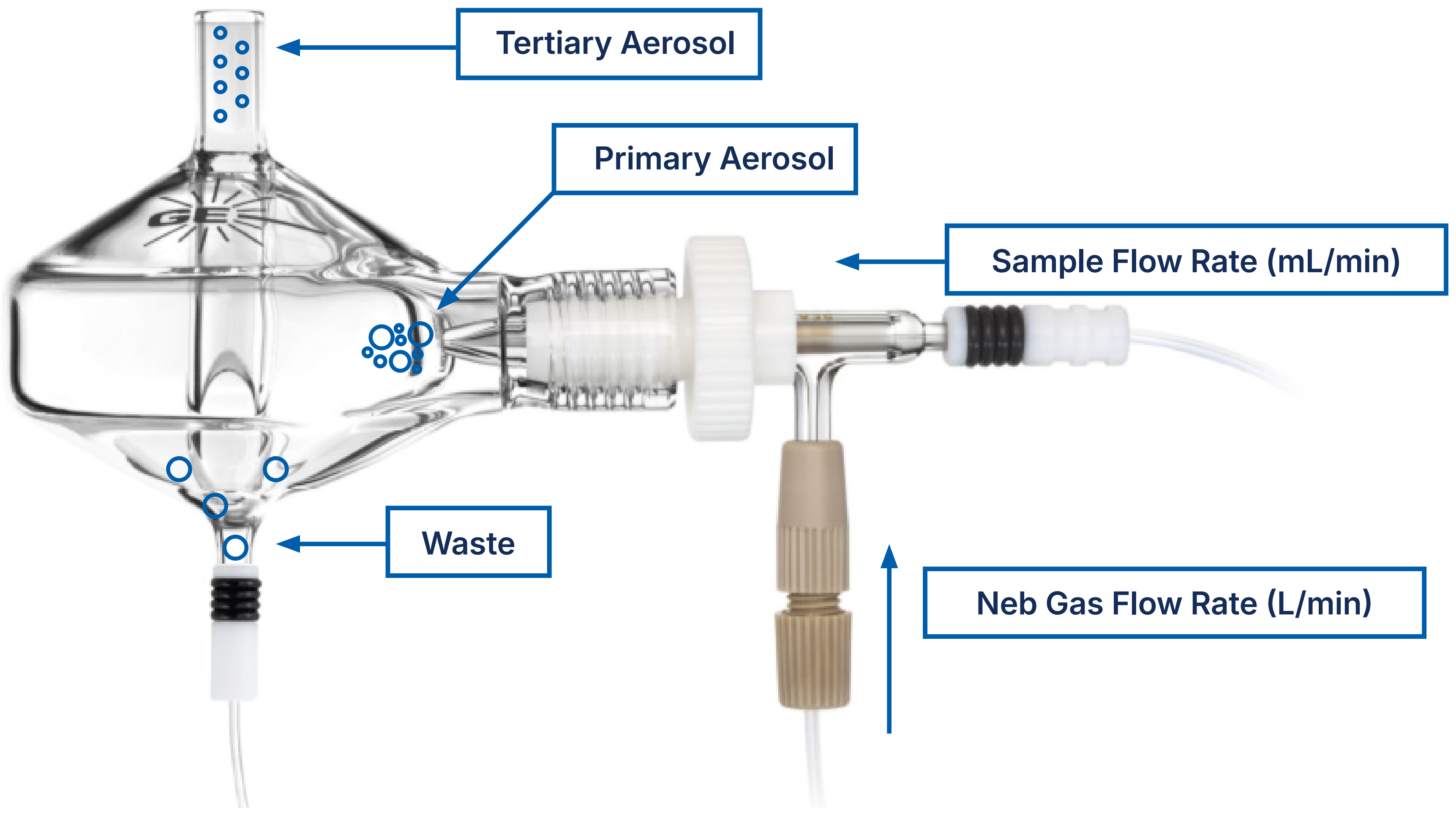
*****The EzyGlide Cloth** can be used to lubricate pump rollers. This lubrication reduces pump tubing wear to increase the service life of pump tubing.*

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

2. Aerosol Generation: Nebulizer & Spray Chamber

- Only the smallest droplets (<10 µm) are transmitted to the plasma and around 95-98% of nebulized sample is drained as waste.
- **Tertiary Aerosol "Filtered" by Spray Chamber, <10 µm.**
- Less energy required, results in a more robust plasma condition.

******Because only ~1–2% of the sample reaches the plasma, even small disturbances can significantly affect signal stability and sensitivity.***



Smaller Droplets Require Less Energy = Efficient Ionization

2a. Nebulizer Assessment

Order Summary

1. Check aerosol production
2. Check nebulizer pressure
3. Check gas connections
4. Inspect for blockages
5. Replace component if damaged

Common Symptom	Possible Cause	Practical Recommendation
Poor sensitivity / Signal Loss	Partial nebulizer blockage, gas flow issue, improper installation, or spray chamber seal problem	**Verify nebulizer aerosol production.** If aerosol is weak, check nebulizer backpressure, gas connectors, and inspect the nebulizer tip for blockage.
High nebulizer backpressure	Salt deposits, particulates, or dried residues from high-TDS samples	**Clean the nebulizer and inspect the tip using a magnifier.** If deposits cannot be removed or damage is observed, replace the nebulizer.
Low nebulizer backpressure (often with sensitivity loss)	Gas leak, damaged nebulizer, or kinked tubing	**Inspect argon connectors and tubing for leaks.** Replace damaged tubing or the nebulizer if necessary.
Poor precision (high RSD)	Blocked or damaged nebulizer, unstable gas flow, worn pump tubing, or poor spray chamber draining	Inspect nebulizer and gas connectors; verify pump tubing condition and spray chamber draining.
No aerosol production	Complete blockage in nebulizer, sample tubing valve, or autosampler probe; damaged nebulizer tip	**Identify the blockage location and clean or replace the affected component.** If the nebulizer tip is damaged, performance cannot be restored and the nebulizer must be replaced.

Care of Nebulizers: Other Practical Tips

Check Argon Connections

- Increasing RSDs may indicate a small argon leak at the nebulizer connection.
- Polymer gas tubing can harden over time and lose a gas-tight seal.
- Even **~1% argon loss can significantly affect ICP signals.**

Quick Sample Uptake Check

- Observe the speed of an air bubble in a 10 cm section of uptake tubing.
- Example estimates:
  - 1.0 mm ID tubing: 10 cm in 10 s ≈ 470 µL/min uptake
  - 0.5 mm ID tubing: 10 cm in 10 s ≈ 118 µL/min uptake

Storage Recommendations

- Always **store nebulizers in their protective storage case.**
- Prevent dust or particles from entering the capillary.

Inspect gas connections

All Glass Expansion nebulizers are supplied in specially designed storage boxes

Nebulizer Cleaning Procedure

To maintain your nebulizer, start and finish each run by nebulizing a mildly acidic blank solution, followed by DIW for 5-10 min. This prevents sample deposits from forming inside the nebulizer when the solvent dries out.

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% HNO_3.
5. Flush 3x with water using the Eluo.
6. For faster drying, flush with methanol.

ICP Nebulizer Maintenance Made Easy

GE Nebulizer Resource Guide

2b. Spray Chamber Assessment

Common Symptom	Possible Cause	Practical Recommendation
Poor sensitivity / signal drift	Inefficient aerosol transport due to deposits contamination inside the spray chamber	Clean spray chamber and verify proper nebulizer installation
Poor precision / high RSD	Droplet accumulation on internal chamber walls causing unstable aerosol transport	Clean spray chamber to remove deposits and ensure proper draining
Delayed washout / memory effects	Residual sample trapped in spray chamber or drain line	Inspect drain tubing and increase rinse time if necessary
Excess liquid entering plasma	Blocked or poorly draining spray chamber outlet	Inspect and clear drain tubing to ensure proper drainage
Sensitivity varies with time or temperature	Spray chamber temperature fluctuations affecting aerosol transport efficiency	Use temperature-controlled spray chamber (e.g. IsoMist™) to stabilize conditions

Inspect UniFit Drain Connector (Left) and Helix CT Seal (Right)

Spray Chamber: Cleaning Procedure

Standard Cleaning

1. Aspirate 2.5% Fluka RBS-25 solution (40× dilution) for ~15 minutes.
2. Rinse thoroughly with deionized water.

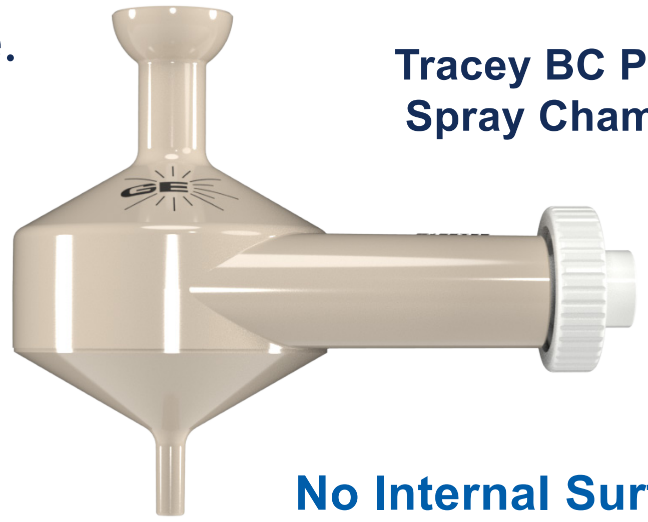
Deep Cleaning (if performance is not restored)

1. Soak overnight in 25% Fluka RBS-25 solution (4× dilution).
2. Rinse thoroughly with deionized water before reinstalling.

PTFE / PFA Spray Chambers

- Internal surface is specially treated to promote uniform wetting and efficient drainage.
- If the treated surface no longer recovers after cleaning, the chamber may need **Surface Re-treatment*.**

PTFE and PFA spray chmabers



Tracey BC PEEK Spray Chamber

No Internal Surface Treatment

3a. Interface: Torch and Injector Assessment

Torch failure can result from gas leaks, damaged/misalign RF coil, or suboptimal torch selection.

Common Symptom	Possible Cause	Practical Recommendation
Plasma instability **Difficulty igniting plasma**	Gas leaks from torch connection, worn O-rings, or injector ferrule deformation	**Inspect torch connections,** O-rings, and injector ferrule; replace if worn or damaged.
Poor sensitivity **Signal drift**	Injector partially blocked by salt deposits from high-TDS samples	Inspect injector tip for salt buildup or blockage; clean or replace if necessary. **Use Elegra™ Argon Humidifier to reduce salt deposition.**
Frequent torch cracking **Short torch lifetime**	RF coil damage, deformation, or misalignment causing uneven plasma heating	**Inspect RF coil for damage/** discolouration; clean or replace if needed.
Outer tube devitrification	Chemical attack or prolonged exposure to high plasma temperatures	**Inspect torch outer tube for clouding or crystallization;** clean or replace torch if necessary

Devitrification of quartz due to salt deposits (Left), and RF Coil corrosion (Right)

*******Even if gas flows are set correctly in the software, poor mechanical connections can cause flow instability and plasma issues.***

Torch and Injector: Cleaning Procedures

Contamination of quartz torches can reduce torch lifetime due to high-temperature interaction with sample deposits.

Carbon Deposits (Organic Samples):

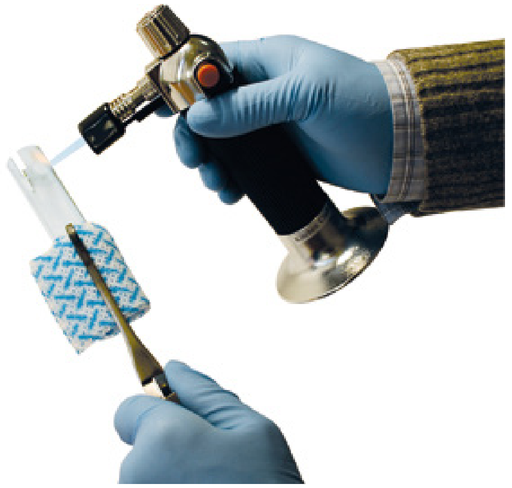
- Bake the torch in a muffle furnace (~500 °C) to remove carbon deposits.
- Alternatively, use a hand-held propane torch to burn off carbon deposits.

Salt Deposits:

- Soak the torch or outer tube in 25% Fluka RBS-25 solution (4× dilution).
- Rinse thoroughly with deionized water.

Metal Deposits:

- Soak the affected section of the outer tube in acid (typically the same acid used for sample preparation, except HF).
- Keep the acid level limited to the contaminated region to avoid damaging polymer components.



Removing carbon from outer tube

Elegra™ Argon Humidifier helps reduce salt deposition

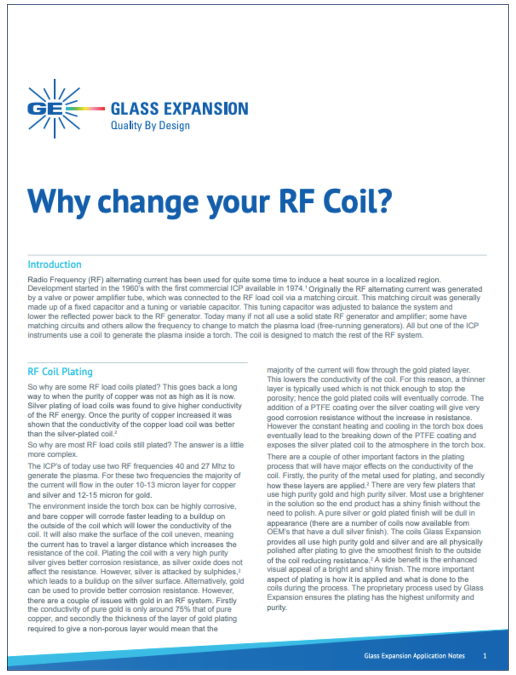
RF Coil: Assessment

Common Issue: RF Coil Corrosion

Can occur due to heat or chemical oxidation:

- Reduced energy transfer efficiency.
- Increased load on the RF generator / RF power tube.
- Less robust plasma and lower analytical signal intensity.

******Corroded coils may also create hotspots, leading to premature torch failure or melting.***



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Why change your RF Coil?

Introduction

Radio Frequency (RF) alternating current has been used for quite some time to induce a heat source in a localized region. Development started in the 1960's with the first commercial ICP available in 1974.[1] Originally the RF alternating current was generated by a valve or power amplifier tube, which was connected to the RF load coil via a matching circuit. This matching circuit was generally made up of a fixed capacitor and a tuning or variable capacitor. This tuning capacitor was adjusted to balance the system and lower the reflected power back to the RF generator. Today many if not all use a solid state RF generator and amplifier; some have matching circuits and others allow the frequency to change to match the plasma load (free-running generators). All but one of the ICP instruments use a coil to generate the plasma inside a torch. The coil is designed to match the rest of the RF system.

RF Coil Plating

So why are some RF load coils plated? This goes back a long way to when the purity of copper was not as high as it is now. Silver plating of load coils was found to give higher conductivity of the RF energy. Once the purity of copper increased it was shown that the conductivity of the copper load coil was better than the silver-plated coil.[2]

So why are most RF load coils still plated? The answer is a little more complex.

The ICP's of today use two RF frequencies 40 and 27 Mhz to generate the plasma. For these two frequencies the majority of the current will flow in the outer 10-13 micron layer for copper and silver and 12-15 micron for gold.

The environment inside the torch box can be highly corrosive, and bare copper will corrode faster leading to a buildup on the outside of the coil which will lower the conductivity of the coil. It will also make the surface of the coil uneven, meaning the current has to travel a larger distance which increases the resistance of the coil. Plating the coil with a very high purity silver gives better corrosion resistance, as silver oxide does not affect the resistance. However, silver is attacked by sulphides,[2] which leads to a buildup on the silver surface. Alternatively, gold can be used to provide better corrosion resistance. However, there are a couple of issues with gold in an RF system. Firstly the conductivity of pure gold is only around 75% that of pure copper, and secondly the thickness of the layer of gold plating required to give a non-porous layer would mean that the majority of the current will flow through the gold plated layer. This lowers the conductivity of the coil. For this reason, a thinner layer is typically used which is not thick enough to stop the porosity; hence the gold plated coils will eventually corrode. The addition of a PTFE coating over the silver coating will give very good corrosion resistance without the increase in resistance. However the constant heating and cooling in the torch box does eventually lead to the breaking down of the PTFE coating and exposes the silver plated coil to the atmosphere in the torch box.

There are a couple of other important factors in the plating process that will have major effects on the conductivity of the coil. Firstly, the purity of the metal used for plating, and secondly how these layers are applied.[2] There are very few platers that use high purity gold and high purity silver. Most use a brightener in the solution so the end product has a shiny finish without the need to polish. A pure silver or gold plated finish will be dull in appearance (there are a number of coils now available from OEM's that have a dull silver finish). The coils Glass Expansion provides all use high purity gold and silver and are all physically polished after plating to give the smoothest finish to the outside of the coil reducing resistance.[2] A side benefit is the enhanced visual appeal of a bright and shiny finish. The more important aspect of plating is how it is applied and what is done to the coils during the process. The proprietary process used by Glass Expansion ensures the plating has the highest uniformity and purity.

Glass Expansion Application Notes 1

RF Coil Application note

Alignment: Ensures a well-shaped and consistently positioned plasma.

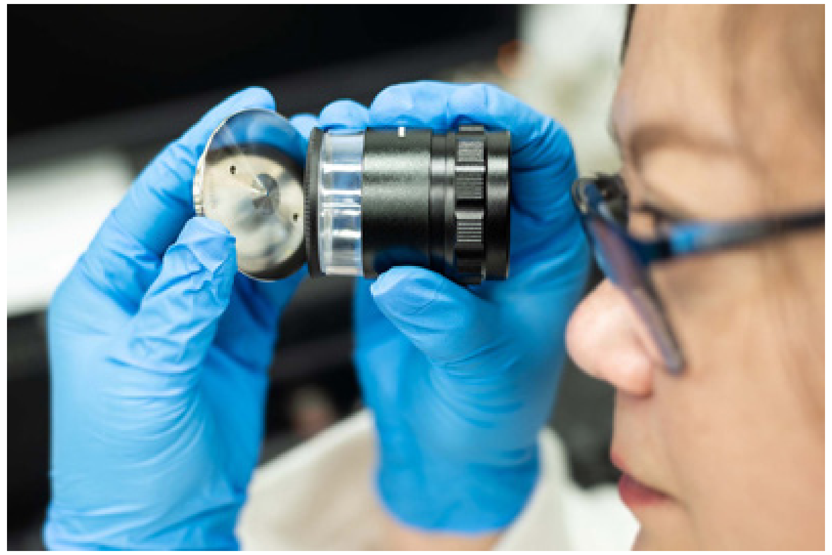
Material & Plating:

- **Copper:** cost-effective but oxidizes quickly
- **Silver-plated:** highest conductivity but prone to tarnish
- **Gold-plated:** best corrosion resistance for harsh environment

Maintenance Recommendations

- Clean periodically to remove deposits and oxidation.
- Replace coils showing copper migration, severe tarnish, or damage.

3b. Plasma Interface: Cones Assessment



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 solution 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

******Cleaning frequency depends on workload and sample matrix, ranging from daily (high-matrix samples) to monthly (clean samples).***

Cone Cleaning: Progressive Approach

1. Gentle Cleaning (Routine)

- Soak cones in **detergent solution (RBS-25)**
- Follow with **Citranox solution**
- Rinse thoroughly with **deionized water**

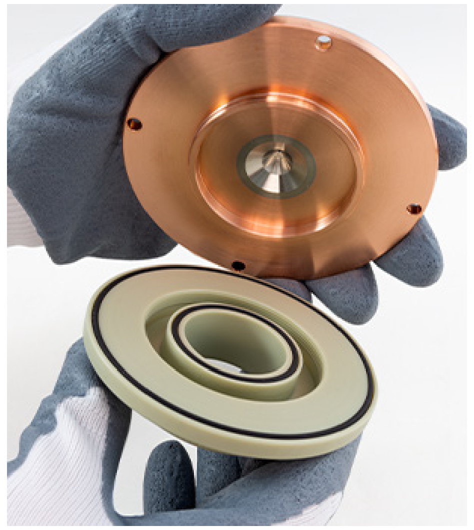
2. Enhanced Cleaning (Deposits Present)

- Sonicate cones in **Citranox solution**
- Rinse multiple times with **deionized water**
- Dry completely with clean gas or warm oven (~60 °C)

3. Deep/Aggressive Cleaning (Heavy Deposits)

- Sonicate in **dilute nitric acid** if detergent cleaning is insufficient
- Limit nitric acid exposure to prevent cone damage or orifice enlargement

Cleaning Expectations: Removing sample deposits is key; discoloration is normal and can improve signal stability.



ConeGuard to protect threaded cones from corrosion during cleaning

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