

Guidelines for Trouble Shooting and Maintenance of ICP-MS Systems



Presented by Dr. Gareth Pearson
ICP-MS Supplies Product Manager



The Agilent Atomic Spectroscopy Lineup

An Instrument for Any Customer Application!



55B Flame AA



240FS/280FS Flame AA



240Z/280Z Furnace AA



4210 MP-AES



5110 ICP-OES



7800 ICP-MS



7900 ICP-MS



NEW! 8900 ICP-QQQ

Leading the way in atomic spectroscopy innovation

www.agilent.com/chem/atomic

Agilent ICP-MS at a Glance

Quadrupole ICP-MS (ICP-QMS) Agilent 7800 and Agilent 7900

High matrix tolerance with HMI/UHMI

He mode removes common polyatomics

Wide dynamic range

Applications from routine to research



Agilent 7800 and 7900 ICP-MS

Triple Quadrupole ICP-MS (ICP-QQQ) Agilent 8900

MS/MS for controlled reaction chemistry

Unmatched interference removal

High sensitivity and low backgrounds

Superior Abundance Sensitivity



Agilent 8900 ICP-QQQ

Top 5 Tips for Flawless ICP-MS Performance



Prevent nebulizer blockage



Pay attention to the interface cones



Keep it clean



Set high standards



Don't neglect the pump tubing





How to Prevent Nebulizer Blockage?

Micro-flow nebulizers

- Zero tolerance to undissolved solids
- Plugging of annulus and/or capillary

Rinse at least 10 minutes with a reagent blank before extinguishing plasma

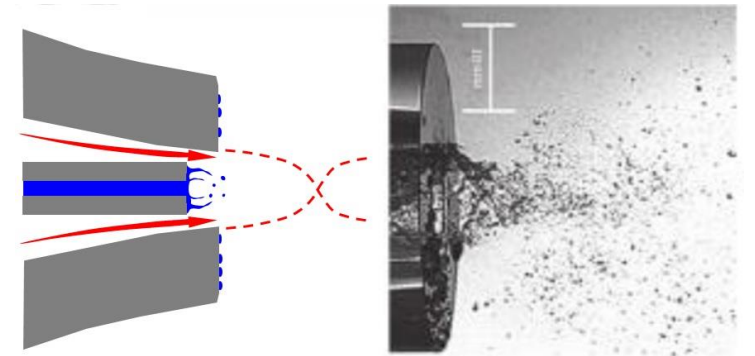
Filter/Centrifuge/gravitational settling

Use only lint less wipes

Autosampler enclosures

Autosampler probe height

Think “**PREVENTION**”



1. Image modified from “*Pneumatic Nebulizers and Spray Chambers for Inductively Coupled Plasma Spectrometry. A Review, Part 1. Nebulisers*” by Barry Sharp, *JAAS*, vol.2, p. 613-652, 1988
2. Image provided by Meinhard Glassblowing Products



Improve Efficiency of Sample Filtering

Most users should filter samples prior to analysis

- Reduces maintenance and downtime from blockages in the sample introduction system

Agilent Captiva syringe filters provide an efficient solution

- Captiva filters provide the industry's highest flow rates and loading capacities
- Available in a variety of membrane types and pore sizes, to suit your application
- Recommended for spectroscopy applications:
 - Captiva Premium, 100/pk
 - PTFE, 0.45um Pore, 15mm dia. (5190-5085) or 25mm dia. (5190-5087)
 - Captiva Econofilters, 1000/pk
 - PTFE, 0.45um Pore, 13mm dia. (5190-5266) or 25mm dia. (5190-5268)

http://www.agilent.com/cs/library/brochures/5991-1230EN_Filtration%20Bro.pdf



- Ideal for busy, high-volume labs
- Choose from a variety of membrane types and pore sizes
- Money-saving 1,000 packs



Instructions for use – Captiva Syringe Filters

Follow these steps to realize the full benefits of filtration

<https://www.agilent.com/en/products/sample-preparation/sample-preparation-methods/filtration/stepbystep>



Before filling with sample, draw approximately 1 mL of air into the syringe. This will minimize fluid retention.



Draw your sample into the syringe, then draw in about 1 mL of air. Invert the syringe and wipe residue off tip.



Connect the syringe to the syringe filter using a luer connection. Twist gently to ensure a secure seal.



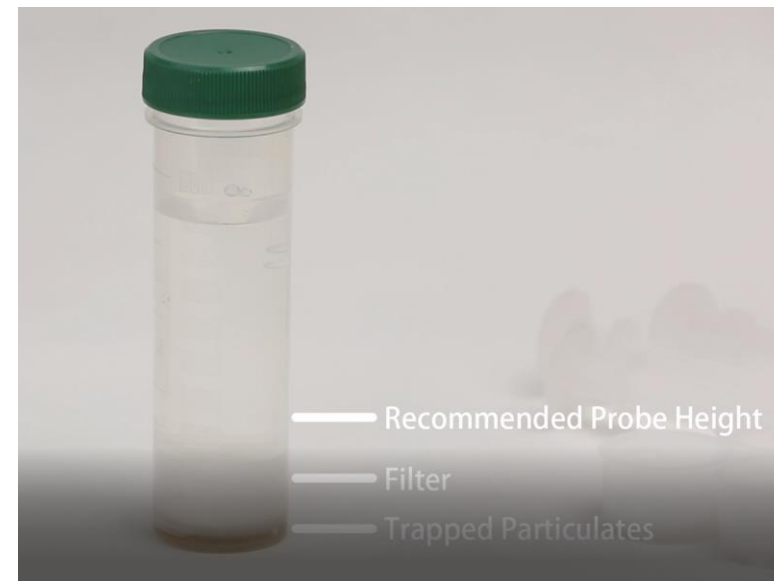
Filter syringe contents into a vial. Afterwards, remove the syringe filter, draw air into the syringe, re-attach the syringe filter, and press the plunger to filter the residual sample. This will maximize sample recovery.

Warning: Use caution with syringes smaller than 10 mL. They can easily generate enough pressure to burst the syringe filter. Agilent syringe filters are for laboratory use only. Pre-wetting the filter, while not mandatory, can be performed as an extra step.

Improve Efficiency from Digestion to Filtration to Analysis

The **FilterMate™ Filtration System** provides a convenient, economical method for digestion, filtration and analysis, improving sample preparation efficiency by eliminating transfers.

- Weigh sample directly into the vessel and use the graduations to accurately add reagents
- Digest in the Hot Block, Dilute to volume, Filter directly and place on the autosampler rack for analysis



190048000 FilterMate™ 2 micron PTFE, 100/pk
190048100 FilterMate™ 0.45 micron PTFE, 100/pk
190047900 Digestion Tubes, Polypropylene for 36- and 54- well hot block, 500/pk

<https://www.agilent.com/en/products/sample-preparation/sample-preparation-methods/filtration/filtermate>

Cleaning the Nebulizer

Never sonicate or attempt to clean with wire!

For normal cleaning:

- Soak in 5% nitric acid for ~10 mins.

To remove a nebulizer blockage:

- Use a dedicated nebulizer cleaning tool to force methanol solution through the tip; OR
- Reverse pump the nebulizer with the tip in solvent; OR
- Apply suction from the wide end of the capillary using a vacuum aspirator; OR
- Apply high pressure clean air via a tubing snugly fitted over the nebulizer tip (use with caution)

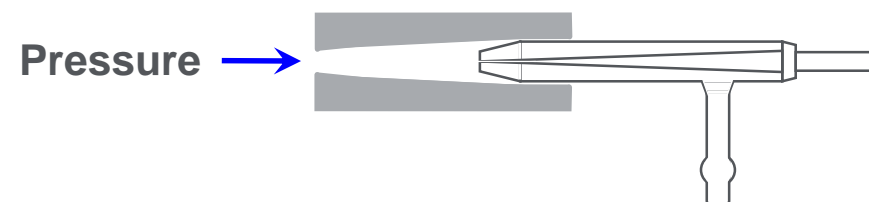
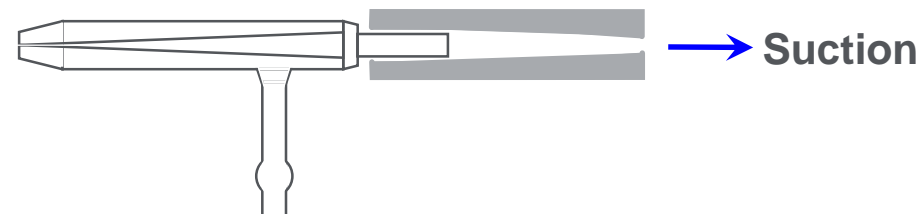
For salt deposits:

- Soak the nebulizer overnight in a beaker of 25% alkaline lab detergent. Rinse with pure water

For “stubborn” deposits:

- Soak the nebulizer overnight in conc. nitric acid. Use a pipette to ensure there are no air bubbles in capillary. Rinse with pure water

<https://www.agilent.com/en/products/lab-supplies/nebulizertips>



Nebulizer cleaning tool
Agilent pn G3266-80020

<http://www.agilent.com/en-us/promotions/icp-ms-resource>



Pay attention to the Interface Cones

- Visually inspect the cones
 - Agilent's LED measuring magnifier (pn 5190-9614) is a tool to help user's achieve optimum ICP-MS instrument performance and maximum cone life
- ICP-MS users can use the magnifier to:
 - Visually inspect a cone to evaluate its condition (e.g. check for excess matrix build-up at the tip)
 - Check if a cone has a damaged orifice and needs to be replaced (e.g. enlarged or damaged orifice)
 - Confirm if maintenance procedures and cleaning have been successful



Why and When to Clean Your Interface Cones?

The necessity to clean the cones depends on your (in)tolerance limits for:

- Sensitivity
- Long term precision
- Elevated background (cps)
- Interface vacuum changing

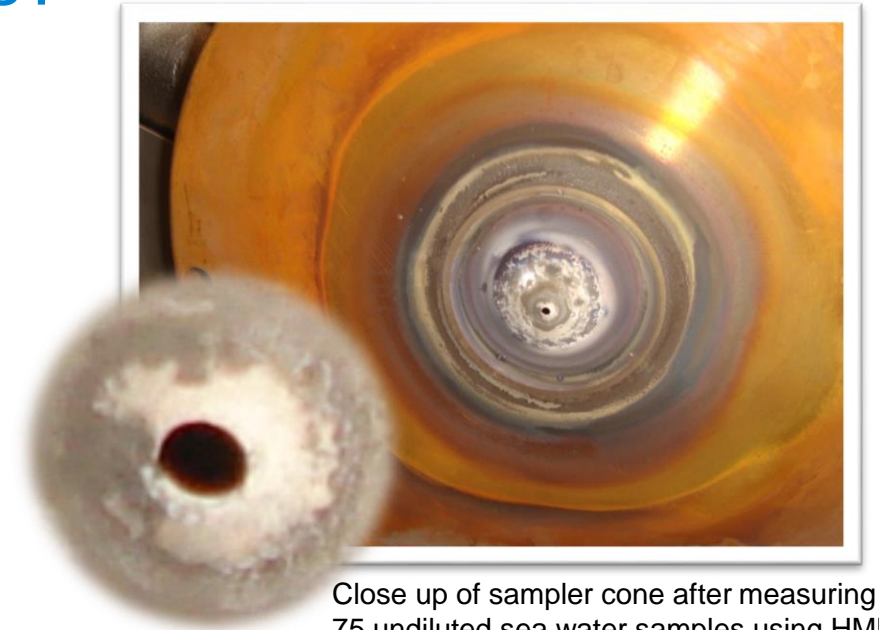
Other reasons to clean the cones?

- If there is an excessive build up of deposits on the orifice (should be circular and free of deposits)
- If the orifice of the cone is blocked / non-circular / unusually discolored

If analyzing the same type of samples, clean to remove only superficial deposits. Ultrapure water clean may be all that is required

A conditioned cone has a uniform coating that leads to long term stability

If analyzing different sample types where a major element in the first sample type is a trace element in the second, more than one cleaning step is required



Close up of sampler cone after measuring 75 undiluted sea water samples using HMI



What's the Right Way to Clean Interface Cones?

Routine Cleaning:

Simple clean with pure water

- Dip a cotton swab (pn 9300-2574) in pure water and clean both sides of the cone
- Rinse with pure water
- Ultrasonicate the cones in pure water for >5 mins (typ. 20 mins)
- Repeat as required (aim for water to stay clean)



Only if performance is still not satisfactory, clean with a 2% Citranox solution (pn 5188-5359) **(NOT MORE THAN 2%)**

- Ultrasonicate in a 2% Citranox solution for max. 2-3 mins
- Rinse with pure water
- Ultrasonicate in pure water for >5 mins

<http://www.agilent.com/en-us/promotions/icp-ms-resource>

What's the Right Way to Clean Interface Cones?

ONLY For more severe contamination:

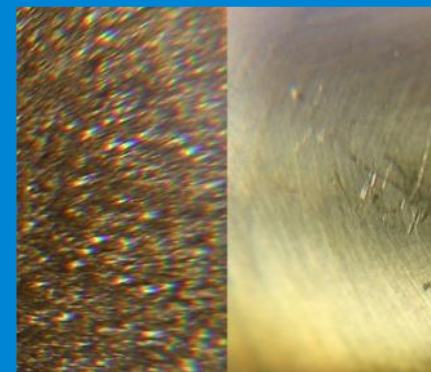
Clean with a 2% nitric acid solution

- Dip a cotton swab in 2% HNO₃ and clean both sides of the cone
(DO NOT SOAK IN ACID)
- Rinse with pure water
- Ultrasonicate in pure water for 2 - 3 mins
- Rinse with pure water
- Ultrasonicate again in pure water for an additional 2 - 3 mins

<http://www.agilent.com/en-us/promotions/icp-ms-resource>

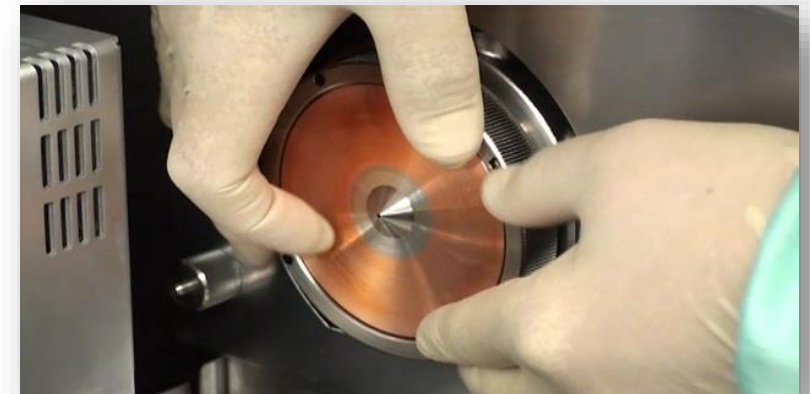
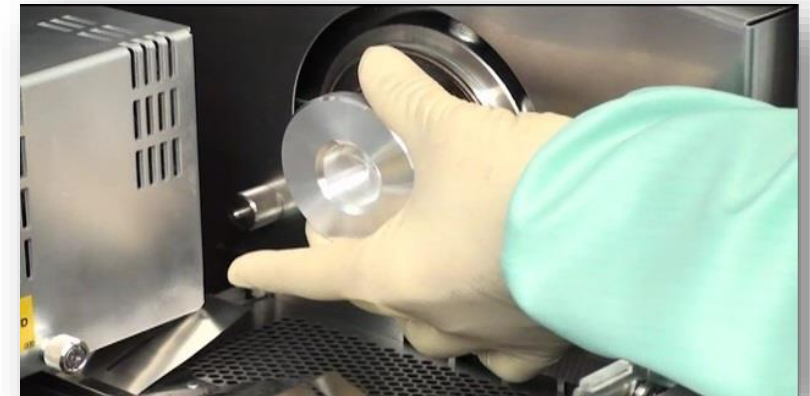
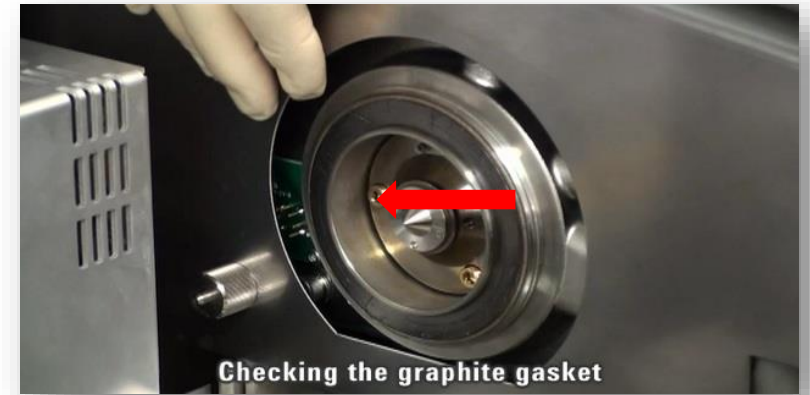


Pitted nickel cone from effect of HNO₃ soak (left side) and clean machined metal on right.



Re-installing the Cleaned Cones

- Check the condition of the graphite gasket and replace if necessary
- Refit the skimmer cone using the removal tool
- Refit the sample cone and tighten by hand
- Check the vacuum levels to confirm correct installation
 - Interface pressure: 500 Pa (~4 torr, 0.005 atm)
 - Analyzer pressure: 0.002 Pa ($\sim 1.5 \times 10^{-5}$ torr, 2×10^{-8} atm)



Common Interface Cone Issues

Cones are fragile (esp. skimmer cone) – Handle with care!

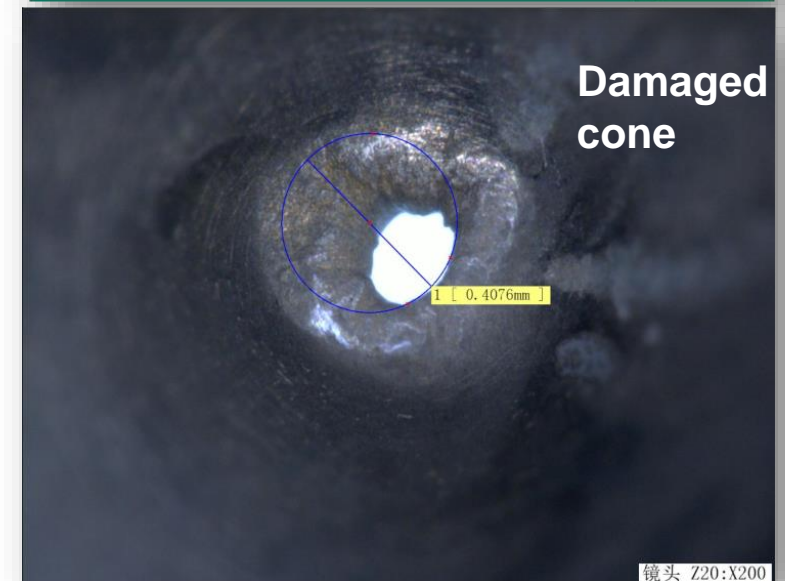
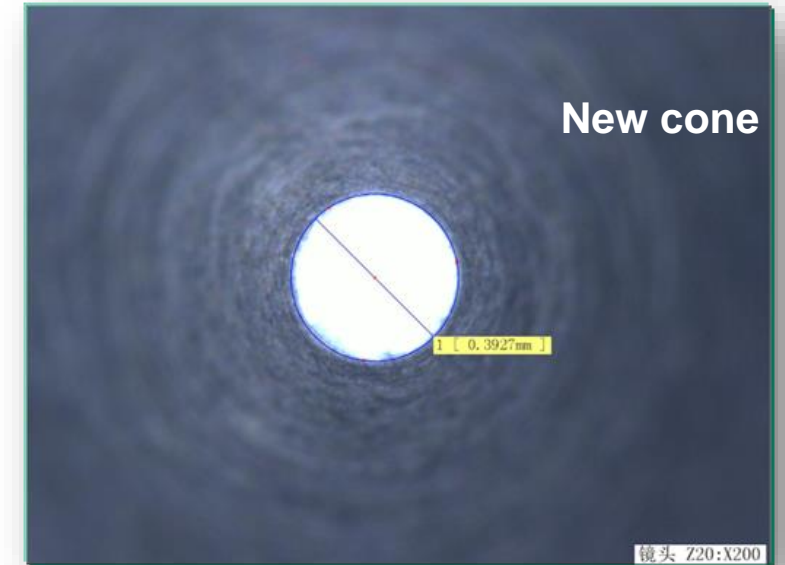
- Poor handling, harsh cleaning or physical abuse can irreversibly damage cones
- Never place tip down on any surface or during cleaning
- Don't try to clean back to original condition

Check that you're using the right skimmer base

- Increases deposition on the cone
- Using the wrong skimmer base can overheat and damage the cone
 - Ni cones – use the stainless steel base
 - Pt cones – use the brass skimmer base

Condition new or cleaned cones prior to use

- Reduces drift due to initial deposition of sample matrix on the clean cone surface
- Aspirate your highest matrix sample for >30mins and then your blank / rinse solution for 10 mins.
- Alternatively aspirate 6020 Interference Check solution A (pn 5188-6526) diluted 10 times in General Purpose mode for 30 mins.; follow with a 5% HNO₃ solution for 10 mins.



Choose from three Cone Care Kits—each includes our LED measuring magnifier

Nickel Cone Care Kit
(Part No. 5067-0294)



Order now

Nickel-plated Cone Care Kit
(Part No. 5067-0295)



Order now

Platinum Cone Care Kit
(Part No. 5067-0296)



Order now

Each kit contains:

- 2 sampler cones
- 1 LED measuring magnifier
- 1 package of sampling cone graphite gaskets (3/pk)
- 1 package of cotton swabs for cleaning (100/pk)

Remember...

You can save 25% on all skimmer cones ordered with any Cone Care Kit

<https://www.agilent.com/en/promotions/icpms-conecare-online>

Product flyer



Take a closer look at our exclusive Cone Care Kits.

https://www.agilent.com/cs/library/flyers/public/5991-8673_icpms_conecarekit_flyer.pdf



Are You Using the Right Interface Cone?

Type of Cone	For which Model ICP-MS?	Skimmer Base Required	Recommended Applications
Nickel sample / skimmer cones	Standard on 7500a/i/c/ce/cx, 7700x/e, 7800/7900 and 8800/8900 with x-lens	Stainless steel	Suitable for most common applications. Good thermal and chemical resistance Provides most economical operation Typically use 3-5/year (based on ~350 samples/day)
Nickel plated sampling cone	Optional for all 77/78/7900 and 88/8900 models	-	For samples containing > 0.5% HCl, or for routine operation with (U)HMI with max. aerosol dilution ratio
Platinum sample / skimmer cones	Standard on 7500s/cs, 7700s, 7900 with s-lens, and 8800/8900 semicon configuration. Optional for all other models	Brass	Required for analysis of aggressive acids (esp. HF) and when O ₂ /Ar option gas is used for analysis of organic solvents Use sample cone with larger 18mm insert for high viscosity & high boiling point acids e.g. H ₂ SO ₄ or H ₃ PO ₄
Platinum skimmer with Copper base	Standard on 7700s, 7900, 8800/8900 semicon configuration and 8900c	Brass	Recommended for the lowest LODs and for higher matrix samples Typically use 1-2/year (based on ~350 samples/day)
Platinum skimmer with Nickel base	Standard on 8900m	Brass	Recommended for organics analysis



Skimmer cone, Nickel



Skimmer base, Stainless Steel



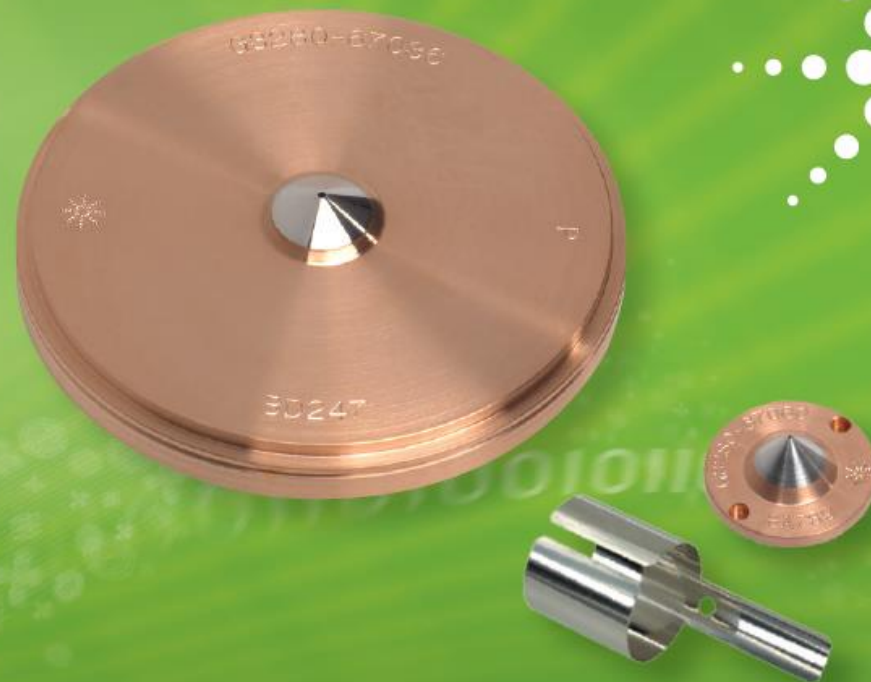
Skimmer cone, Platinum



Skimmer base, Brass

SAVE MONEY AND REDUCE YOUR ENVIRONMENTAL IMPACT WITH THE AGILENT PLATINUM CONE TRADE-IN PROGRAM

The Measure of Confidence



Agilent ICP-MS Platinum Cones – cut costs & go green by returning your used cones

- ICP-MS interface cones are expensive and need regular replacement
- User's purchasing new Agilent platinum cones can return their used cones
- You can receive a trade-in credit on your order
- The value of the credit is based on the reclaim value of the platinum
- This program lowers the net cost of purchasing a new cone, and enables recycling of the precious Pt metal in the cone

<http://www.agilent.com/chem/Ptcone>

3

Keep it clean Sample Introduction System

Spray chamber

Routine cleaning:

- Soak the end cap and spray chamber in 5% nitric acid or Citranox for >30 mins
- Rinse, dry and refit

If you see precision problems or droplet formation on the walls of the spray chamber (beading):

- Soak overnight in a 25% detergent solution
 - Best to leave it soaking for 24 hours
 - Use any laboratory detergent e.g. Fluka RBS25, Triton X-100, Decon 90 etc.



Cleaning the Torch

Visually check the torch, bonnet and shield when removing the torch

- Replace if deformed or chipped

Do not sonicate!

For routine cleaning:

- Soak in >5% nitric acid for ~30 mins

For more stubborn stains:

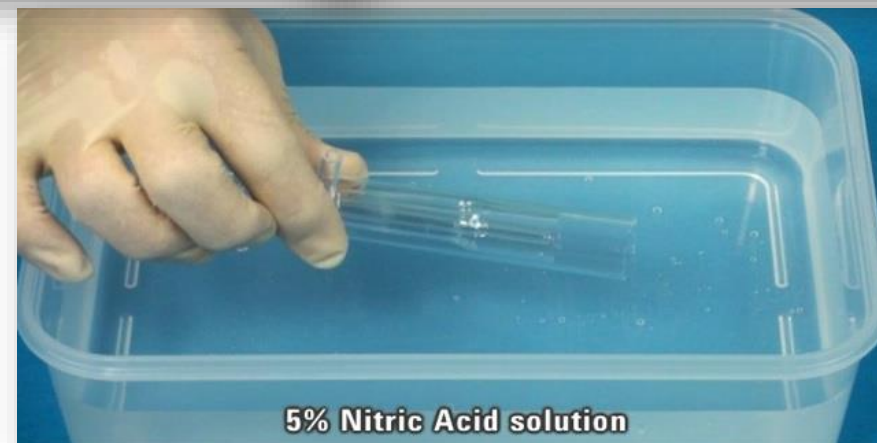
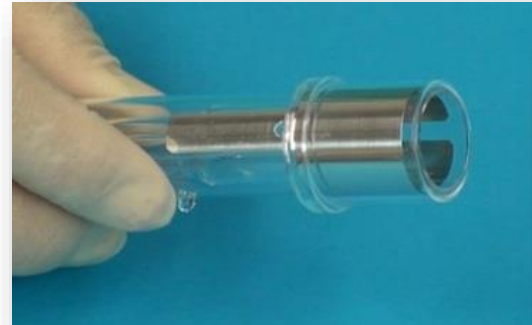
- Soak in bleach (e.g. Chlorox ©) overnight
- Soak in aqua regia (1:3 HNO_3 :HCl)

For salt deposits:

- Rinse with water to remove deposits
- Soak the torch overnight in a beaker of 25% Fluka RBS-25 detergent

Rinse and allow to dry

Caution! Reinstall only when dry



Torch damage due to incomplete drying

Re-installing the Torch

Refit the torch shield & torch bonnet

Replace the torch into the torch holder

Ensure the torch projection fits into the slot on the torch holder

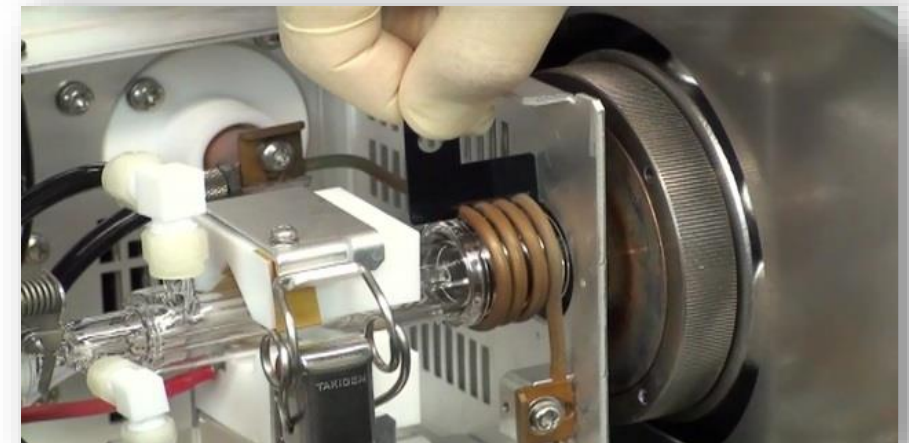
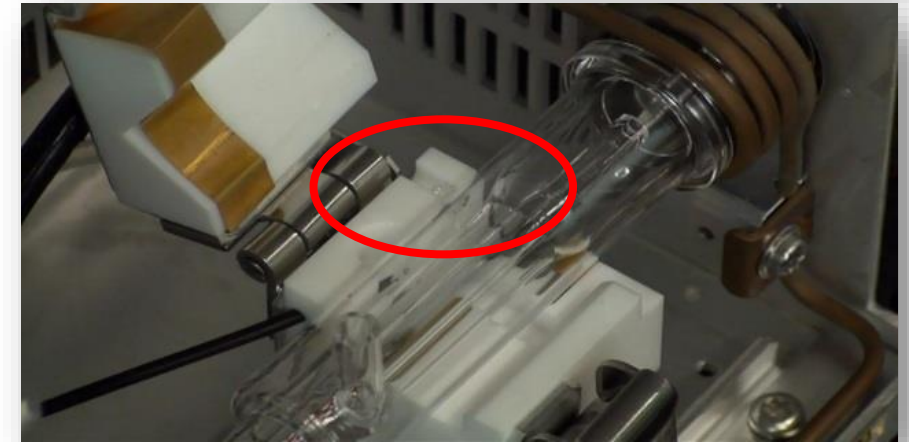
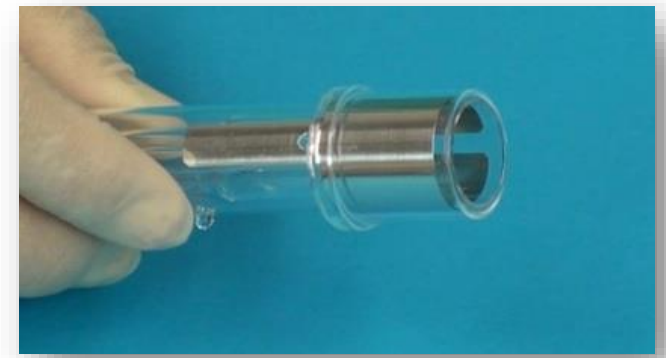
Can check the alignment of the RF coil when re-installing the torch

Reconnect gas fittings and transfer tube from spray chamber

Check torch alignment – esp. sampling depth (z position)

Test plasma ignites and instrument switches to “analysis” mode

- If plasma fails to ignite, check all connections for possible air leaks





Set high standards

Prepare accurate standards using Certified Reference Materials

Most atomic spectroscopy techniques need a standard of “known” composition to calibrate the instruments; then you can determine the “unknown” in your sample.

What’s in my sample?

The result of your analysis is largely dependent on the accuracy of your “known” standard.

How can I be sure these results are accurate?

I don’t have the extra time or money to redo this work...
I can’t risk my results by using inaccurate standards!

Errors during preparation or contamination of your “known” standard leads to:

- Inaccurate results
- Lost time through trouble shooting
- Instrument downtime
- Preparation of new standards and re-analysis of samples
- Premature replacement of instrument supplies
- Failed audits & potential loss of ISO accreditation

The Value of Agilent's Certified Reference Materials

Highest ISO Accreditation!

- Manufactured in an ISO 9001, ISO Guide 34 facility and certified in an ISO 17025 testing laboratory

High purity

- Manufactured from high purity raw materials and solvents which are tested for impurities

NIST traceable

- Certified using the NIST high performance ICP-OES test protocols
- Directly traceable to the NIST 3100 Series of SRMs

Contamination free

- Packaged in pre-cleaned, high purity HDPE bottles
- Shipped in poly sealed bags

Long shelf life

- Most offer a shelf life of 18 months
- Supported by short and long term stability studies

Thorough confirmation

- Trace impurities assayed using Agilent ICP-MS
- Actual concentration reported on CoA for up to 68 trace impurities



Agilent Certified Reference Materials

HIGH QUALITY INORGANIC AND METALLO-ORGANIC STANDARDS FOR ATOMIC SPECTROSCOPY

The Measure of Confidence



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Custom Inorganic Standards for ICP-MS, ICP-OES, AA, and MP-AES



Design your own standard for your unique sample and Agilent experts will guide you through stability and compatibility while maintaining the highest quality for precise, accurate calibration data.

All custom standards are of the highest quality - manufactured in an ISO Guide 34 facility and certified in an ISO/IEC 17025 testing laboratory. Each standard will be accompanied with Certificate of Analysis (CoA) highlighting ISO conformity, actual concentration, measurement uncertainty, and NIST traceability.

All Agilent's Certified Reference Materials can be used with Agilent, or any other brand (PerkinElmer, and Thermo) instruments.

Element Concentration (Click here for instructions)

Ag		Nd	
Al		Ni	
As		Os	
Au		P	
B		Pb	
Ba		Pd	
Be		Pr	
Bi		Pt	
Ca		Rb	
Cd		Re	
Ce		Rh	
Co		Ru	
Cr		S	
Cs		Sb	
Cu		Sc	
Dy		Se	
Er		Si	
Eu		Sm	
Fe		Sr	
Ga		Ta	
Gd		Tb	
Ge		Te	
Hf		Th	
Hg		Ti	
Ho		Tl	
In		Tm	
Ir		U	
K		V	
La		W	
Li		X	
Lr		Y	
Mg		Yb	
Mn		Zn	
Mo		Zr	
Na			

Name

Company

Address 1

Address 2

City, State Zip

Phone

Email

Preferred Matrix:

Units of Concentration

Bottle Size

Quantity

Intended Use (instrument/technique)

Previous Quote # (if applicable)

Your Summary:

Email this form >

Name:

Company:

Address 1:

Address 2:

City, State, ZIP:

Phone:

E-mail:

Element concentration:

Preferred Matrix:

Additional Notes (optional):

FOR OFFICE USE ONLY

Company PN

SPQ#

Comments

SUBMIT

To Submit a Custom request (For US/Canada Only):

1. Complete the [order form](#) for a quote. Please complete all fields in the form.
2. Submit form by using "E-mail this form" button in the [order form](#). This button compiles an e-mail message, which can be sent immediately

<https://www.agilent.com/en-us/custom-inorganic-standards>

Tips to Improve Standard Preparation

How are they prepared?

- Ensure purchased standards are still within “Use By” date
- Avoid all use of glassware for ICP-MS
- Perform dilutions by weight using a 4-decimal place balance
 - Calibrate and Periodically, check accuracy & reproducibility of your pipettes
- Use calibrated pipettes and class ‘A’ volumetric flasks for dilutions
- Use de-ionized water (Type I - conductivity $\geq 18\text{ M}\Omega/\text{cm}^3$)
 - Lower grades may have contamination
- Use serial dilutions for preparing low concentrations from 1,000 ppm stock
 - Please don’t do large dilutions ($> 1:10,000$) in 1 step



What concentration are they?

- Low concentration standards have a finite life
 - Prepare ppb and sub ppb (ug/L) concentration standards daily from high conc. stock
 - Prepare low ppm (mg/L) concentration standards weekly

How are they stored?

- Plastic vessels ensure better stability (PFA or FEP)
- Stabilize with acid – low pH ensures better stability

Intended use

Manufactured in an ISO 9001, ISO Guide 34 facility and certified in an ISO/IEC 17025 testing laboratory

Made from the highest purity raw materials and solvents

Agilent ICP-MS* used to assay the impurities

Traceable to NIST

Instructions for proper use and appropriate conditions of storage

Period of validity

Date of release

Date of expiration verified by short and long-term stability studies for all standards

Agilent Technologies

CERTIFICATE OF ANALYSIS

Agilent Product Name: Copper Standard 1000 ug/ml Cu in 5% HNO₃

Agilent Part No.: 5190-0308

Lot No.: Sample

Product Specifications				
Analyte	Starting Material	CAS #	Matrix	Certified Concentration
Cu	Cu	7440-50-8	5% HNO ₃	994 ± 2 ug/L (w/v) 994 ± 2 ug/g (w/w)

Intended Use: This solution is intended for use as a certified reference material or calibration standard for inductively coupled plasma optical emission spectrometry (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS), atomic absorption spectrometry (AAS) or (EPAAS), microwave plasma atomic emission spectrometry (MP-AES), or a very fluorescence spectrometry (VFS), and other techniques for elemental analysis.

Certification & Traceability: This CRM was manufactured under a quality management system that is accredited to ISO Guide 34, ISO/IEC 17025, and registered to ISO 9001. This CRM was prepared to a nominal concentration of 1000 ug/ml, for gravimetric methods using 50.0000 g of water dissolved in high purity nitric acid (2-9624) and diluted with ASTM Type I Water. The balances used in the preparation of this CRM are calibrated regularly with traceability to NIST. All volumetric dilutions are performed in Class A calibrated glassware. The certified concentration and uncertainty were determined using the "High Performance ICP-OES" method determined by NIST and both the certified concentration and uncertainty values are traceable to NIST SRM 3114, lot #611017. The uncertainty associated with the certified concentration represents the expanded uncertainty at the 95% confidence level using a coverage factor of k=2.

Uncertified Values: Agilent ICP-MS was used to determine trace metal concentrations for this product (but is not determined).

Trace Concentrations (ug/L)															
Ag	<0.5	As	<0.5	Cd	<0.5	Co	<0.5	Cr	<0.5	Fe	<0.5	Ge	<0.5	Se	<0.5
Al	<0.5	Cu	<1	Si	0.880	Ni	<0.5	Pb	<0.5	M	<100	Te	<0.5		
Ar	<0.5	Ca	<0.5	W	<0.5	Sr	<1	P	<0.5	Ba	<0.5	I	<0.5		
Au	<0.5	Cl	<0.5	Hg	<0.5	Mo	<0.5	Pt	<0.5	Sn	<0.5	V	<0.5		
B	<0.5	Cu	Mg	<0.5	Na	<0.5	Pb	<0.5	Si	<1	W	<0.5			
Be	<1	Se	<0.5	Ir	<1	Mo	<0.5	Pb	<0.5	Ta	<0.5	Ti	<0.5		
Ba	<0.5	Br	<0.5	V	<0.5	Ni	<0.5	Pb	<0.5	Ta	<0.5	Ti	<0.5		
Bi	<0.5	Br	<0.5	K	<0.5	Si	<0.5	Pb	<0.5	Ta	<1	Ti	<0.5		
Ca	<0.5	Fe	<10	La	<0.5	Co	<0.5	Sn	<0.5	Ta	<0.5	Ti	<0.5		
Cd	<0.5	Co	<0.5	Li	<1	P	<100	Sn	<0.5	Ta	<0.5	Ti	<0.5		

Instructions for Use: Agilent Technologies recommends that this solution be thoroughly mixed by repeated shaking or swirling of the bottle immediately prior to use. To achieve the highest accuracy the analyst should: (1) use only pre-cleaned containers and methodologies, (2) avoid pipetting directly from the CRM's original container, (3) use a minimum sub-sample size of 500 µL, (4) make dilutions using calibrated balances or certified volumetric class A flasks and pipettes, (5) allow to volume using the same matrix as the original CRM, and (6) never pour used product back into the original container. The solution should be kept tightly capped. Store at controlled room temperature per USP 36 (18.30.36). Do not freeze, heat, or expose to direct sunlight. Minimize exposure to moisture or high humidity.

Period of Validity: Agilent Technologies ensures the accuracy of this solution until the expiration date shown below, provided the instructions for use are followed. During the period of validity, the purchaser will be notified if this product is recalled due to any significant changes in the stability of the solution.

Sample for signature:

QA Manager

• Date of release: 5 February 2018
• Date of expiration: 31 August 2018

Certified concentration(s) reported (with uncertainty values in w/v and w/w)

Assayed by high performance ICP-OES, a method developed by NIST to assure direct traceability to the appropriate NIST 3100 Series of single-element SRMs

Method(s) used to determine certified concentrations

Actual concentration values reported for up to 68 impurity elements

*Impurities in wear metal, metallo-organic and bio-derived standards assayed using ICP-OES, XRF or other elemental analysis techniques

Tips to Improve Accuracy of Results

Sample preparation

- Is the most appropriate digestion being used?
- Are all of the analytes being quantitatively (and reproducibly) extracted and dissolved?
 - Many digestions are only partial extracts – efficiency will vary with the sample matrix
 - Some volatile analytes may be “lost” during digestion
 - Confirm by taking a solid certified reference material through your preparation and analysis procedure
- Is the digest stable – or are you seeing any precipitates or a suspension?
- Do you see any potential contamination from either reagents or the digestion equipment? e.g. especially with Si, B or Ca
 - Include a “Reagent Blank” with every sample batch to monitor



Tips to Reduce Contamination

Contamination can come from anything that comes into contact with your sample during storage, digestion (dilution) and analysis



Agilent Technologies

- **Check reagent purity**
 - Always buy the best reagents – use high purity or ICP-MS grade
 - Always check the certificate of analysis for elevated levels
 - Caution if buying in large quantities
 - Worst case – can use contaminated acid for cleaning
 - Ensure still within “use by” date
 - Reseal immediately after use
- **Other common contamination sources**
 - Reagent water
 - FEP containers preferred
 - Borosilicate glass can contribute Boron contamination
 - Airborne dust in the lab.
 - Pipette tips
 - Don't insert pipette tips into your acids
 - Use natural tips – colored tips may increase contamination (esp. with Cu, Fe, Zn, Cd)
 - Powdered gloves (esp. for Zn)

CERTIFICATE OF ANALYSIS

Agilent Product Name: Copper Standard: 1000µg/mL Cu in 5% HNO₃

Agilent Part No: 5190-8348

Lot No: Sample

Product Specifications

Analyte	Starting Material	CAS #	Matrix	Certified Concentration
Cu	Cu	7440-50-8	5% HNO ₃	994 ± 2 µg/mL (w/v) 984 ± 2 µg/g (w/w)

Intended Use: This solution is intended for use as a certified reference material or calibration standard for inductively coupled plasma optical emission spectroscopy (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS), atomic absorption spectroscopy (flame AAS or GFAAS), microwave plasma atomic emission spectroscopy (MP-AES), x-ray fluorescence spectroscopy (XRF), and other techniques for elemental analysis.

Certification & Traceability: This CRM was manufactured under a quality management system that is accredited to **ISO Guide 34, ISO/IEC 17025**, and registered to **ISO 9001**. This CRM was prepared to a nominal concentration of 1000 µg/mL by gravimetric methods using 99.999% pure copper (Cu) metal dissolved in high purity nitric acid (HNO₃) and diluted with ASTM Type I Water. The balances used in the preparation of this CRM are calibrated regularly with traceability to NIST. All volumetric dilutions are performed in Class A calibrated glassware. The certified concentration and uncertainty were determined using the “High Performance ICP-OES” protocol developed by NIST and both the certified concentration and uncertainty values are traceable to NIST SRM 3114. In-⁶³Cu and ⁶⁵Cu isotope ratios were determined using a secondary standard solution. The expanded uncertainty at the 95% confidence level using a coverage factor of k=2.

Uncertified Values: Agilent ICP-MS was used to determine trace metal concentrations for this product (nd = not determined).

Trace Concentrations (µg/L)

Ag	<0.5	Ce	<0.2	Gd	<0.2	Lu	<0.2	Pb	<1	Se	<2	Ti	<0.5
Al	<2	Co	<1	Ge	0.069	Mg	<5	Pd	<0.5	Si	<100	Tm	<0.2
As	<2	Cs	<0.5	Hf	<0.2	Mn	<1	Pr	<0.2	Sm	<0.2	U	<0.5
Au	<0.5	Cr	<0.5	Hg	<0.5	Mo	<0.5	Pt	<0.5	Sr	<0.5	V	<1
B	<5	Cu	Major	Ho	<0.2	Na	<25	Rb	<0.5	Sc	<1	W	<0.5
Ba	<1	Dy	<0.2	In	<0.2	Nb	<0.5	Re	<0.2	Ta	<0.5	Y	<0.5
Be	<0.5	Er	<0.2	Ir	<0.2	Nd	<0.2	Rh	<5	Tb	<0.5	Yb	<0.2
Bi	<0.2	Eu	<0.2	K	<25	Ni	9	Ru	<0.5	Te	<1	Zn	<2
Ca	<25	Fe	<10	La	<0.5	Os	<0.5	Sb	<0.5	Th	<0.5	Zr	<0.5
Cd	<0.5	Ga	<0.5	Li	<2	P	<100	So	<5	Tl	<2		



5

Don't neglect the pump tubing

Tubing diameters

- Waste to be larger ID than sample ID

Chemical compatibility

- Ensure tubing is resistant to the solvent being used

Replace frequently

- Pre-clean new tubing to remove potential contamination
- Using “old” tubing can lead to problems with precision and stability
 - Can also contribute to nebulizer blockage (if inside lining breaks down)
- Typical lifetime is ~5 days based on normal 8 hour working day
 - Detach from tube holder after use – allows tube to “relax”

Maintaining tubes – What to check?

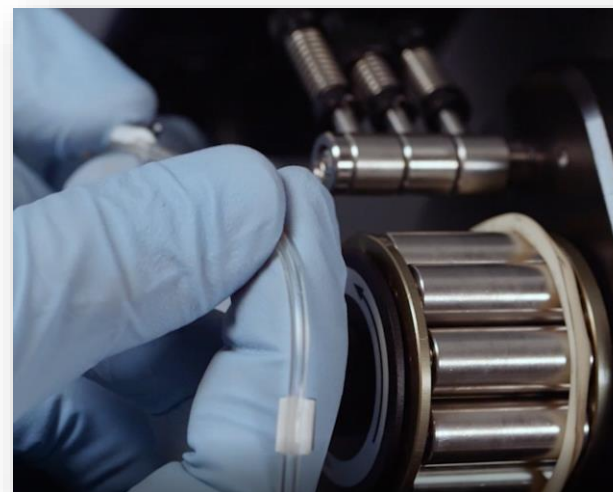
- Check 2 key things on pump tubing
 - Roundness of tube – should not be any “flat” spots
 - Tubing should still be elastic – replace if obviously stretched
- Don't over tighten – just need smooth and even sample flow

Remember to check other tubing for wear, leaks and crimps

Drain tubing 1.52 mm ID
Agilent P/N G1833-65570



Sample tubing 1.02 mm ID
Agilent P/N G1833-65569



Peri Pump Tubing Tips



Symptoms:

Peri pump tubing that looks/feel worn or has a strange colour

IF IN DOUBT, CHANGE IT

Erratic liquid flow

- Check tension from clamps

Bubbles in the liquid stream

- Check all gas fittings, tubing and connectors – deposits, burrs, damage

Spurting Nebuliser or disconnecting tubing segments

- Plugging in the transfer line. Requires cleaning or replacement.

Bad recovery or carryover on “indicator” elements that tend to become unstable first when the pump tubing has got an “active” coating.

- Ag and PGE (Platinum group elements. Ru, Rh, Pd, Os, Ir, Pt)

ICP-MS – Potential Autosampler Issues

More customers use autosamplers for automation

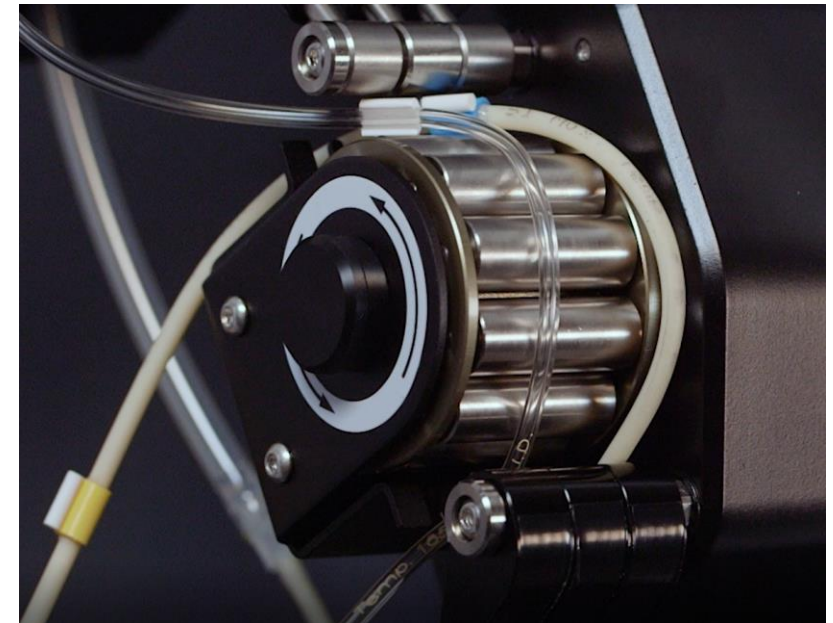
Issues to consider:

- Longer transfer tube between sampler and ICP-MS
 - May need to program a longer sample uptake delay
 - May exacerbate problems with memory effects
- Ensure probe diameter is appropriate for sample matrix
 - Use wider bore for high % TDS or viscous samples
- Sample stability - potential for sample changes while uncovered in racks – impacts accuracy
 - Dust ingress can introduce contamination
 - Sample evaporation may occur during long unattended runs
 - Sediment in the sample may settle out, esp. with wear metals or suspensions
- Ensure transfer line to ICP-MS is in good condition
 - Kinks in the line may cause poor uptake, or pulsing in the sample
 - Impacts on precision and accuracy



Recommended Procedures at End of the Day

1. Aspirate acid rinse solution for a few minutes before shutting off the plasma
 - Helps to prevent sample deposition inside the nebulizer after the run
2. Extinguish the plasma and switch off the chiller
3. Remove the sample capillary from the rinse, start the pump again and pump any remaining rinse solution from the spray chamber
4. Release the pressure bars on the pump tubing and remove the bridges from the securing slot
 - Ensure the tubes are no longer stretched over the pump rollers
5. Empty waste vessel
6. Close the current worksheet – leave Mass Hunter S/W running
7. Leave mains power and argon on
 - Keeps instrument in stand-by mode (ensures fastest start-up)



ICP-MS – Recommended Maintenance Schedule

Daily:

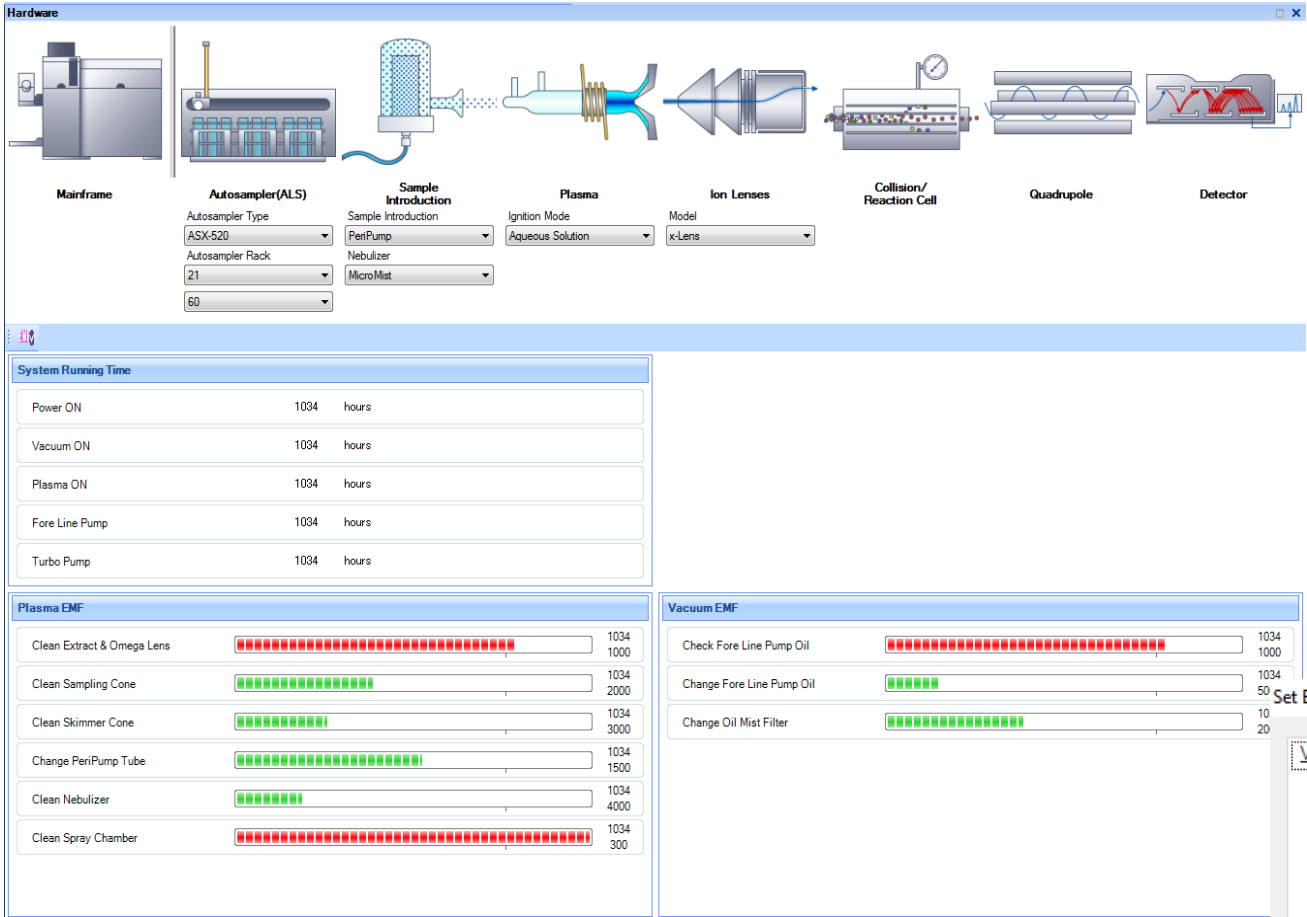
- Argon and cell gas (He, H₂, ...) pressures
- Check peristaltic pump tubing for damage/deterioration
- Visual check of glassware
(connections OK, no filling of spray chamber or connector)
- Visual inspection of sample cone exterior (orifice shape & deposition)

Frequently, as needed - perform these operations:

- Empty the drain reservoirs
- Thorough visual inspection of interface cones
- Check nebulization
- Replace peristaltic pump tubing
- Clean/replace torch
- Check recirculation water level

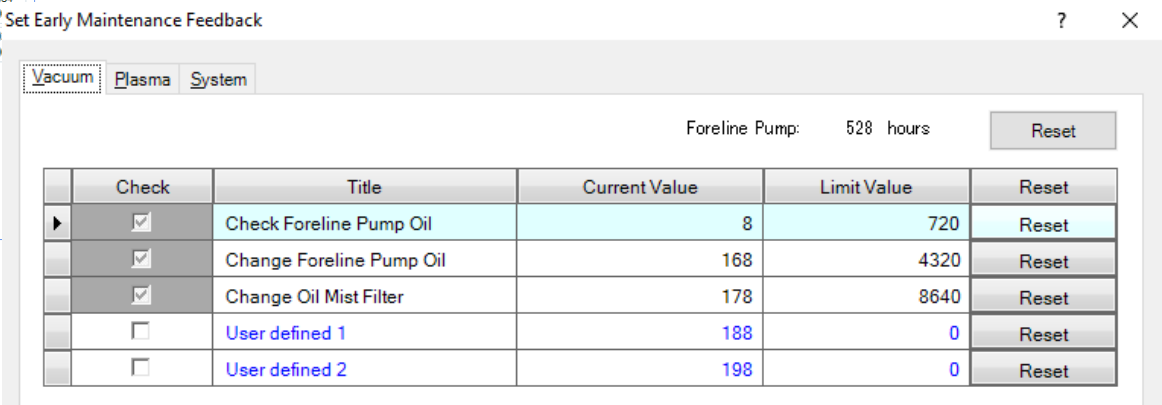
**Frequency and extent of maintenance depends on the usage of the instrument:
this overview assumes daily use, 8 hours/day.
For systems run 24/7, more frequent maintenance is required.**

EMF (Early Maintenance Feedback)



EMF window shows usage of various components and predicts when to perform maintenance

All gauges and limit values are user definable



ICP-MS System Tips – User Log

- Use the “Maintenance Log” to record routine and non routine maintenance activities
- Maintenance log can track:
 - When the maintenance activity was completed
 - Operator who completed the maintenance
 - Type of maintenance activity
 - Any operator comments

Add Maintenance Log

Operator: S.F.

Maintenance Log:

<input type="checkbox"/> Confirmed Drain Vessel	<input type="checkbox"/> Changed Peristaltic Tube	<input type="checkbox"/> Confirmed Foreline Pump Oil
<input checked="" type="checkbox"/> Cleaned Sampling Cone	<input type="checkbox"/> Changed Graphite Sheet	<input type="checkbox"/> Changed Foreline Pump Oil
<input type="checkbox"/> Changed Sampling Cone	<input type="checkbox"/> Confirmed Shield Torch	<input type="checkbox"/> Confirmed Foreline Pump Mist Filter
<input type="checkbox"/> Cleaned Skimmer Cone	<input type="checkbox"/> Changed Shield Torch	<input type="checkbox"/> Changed Foreline Pump Mist Filter
<input type="checkbox"/> Changed Skimmer Cone	<input type="checkbox"/> Confirmed Torch	<input type="checkbox"/> Cleaned Lenses
<input checked="" type="checkbox"/> Cleaned Nebulizer	<input type="checkbox"/> Changed Torch	<input type="checkbox"/> Changed EM
<input type="checkbox"/> Changed Nebulizer	<input type="checkbox"/> Confirmed Cooling Water Filter	<input type="checkbox"/> Miscellaneous
<input type="checkbox"/> Cleaned Spray Chamber	<input type="checkbox"/> Changed Cooling Water Filter	
<input type="checkbox"/> Changed Spray Chamber	<input type="checkbox"/> Changed Ion Gauge	

Comment: I changed sample tubing

Enter your Comment

File Edit View Instrument Hardware Startup Batch Queue Tools Help

Hardware Plasma Batch Tune Queue Data Analysis Report

Hardware -> Mainframe -> Maintenance Log

Mainframe Autosampler(ALS) Sample Introduction Plasma

	Date and Time	Operator	Maintenance Log
1	11/29/2013 11:19:23 AM	John	Confirmed Foreline Pump Oil Changed Foreline Pump Oil Confirmed Foreline Pump Mist Filter Changed Foreline Pump Mist Filter

Add Maintenance Log Delete Maintenance Log

I refer to the 7900 Maintenance Video.

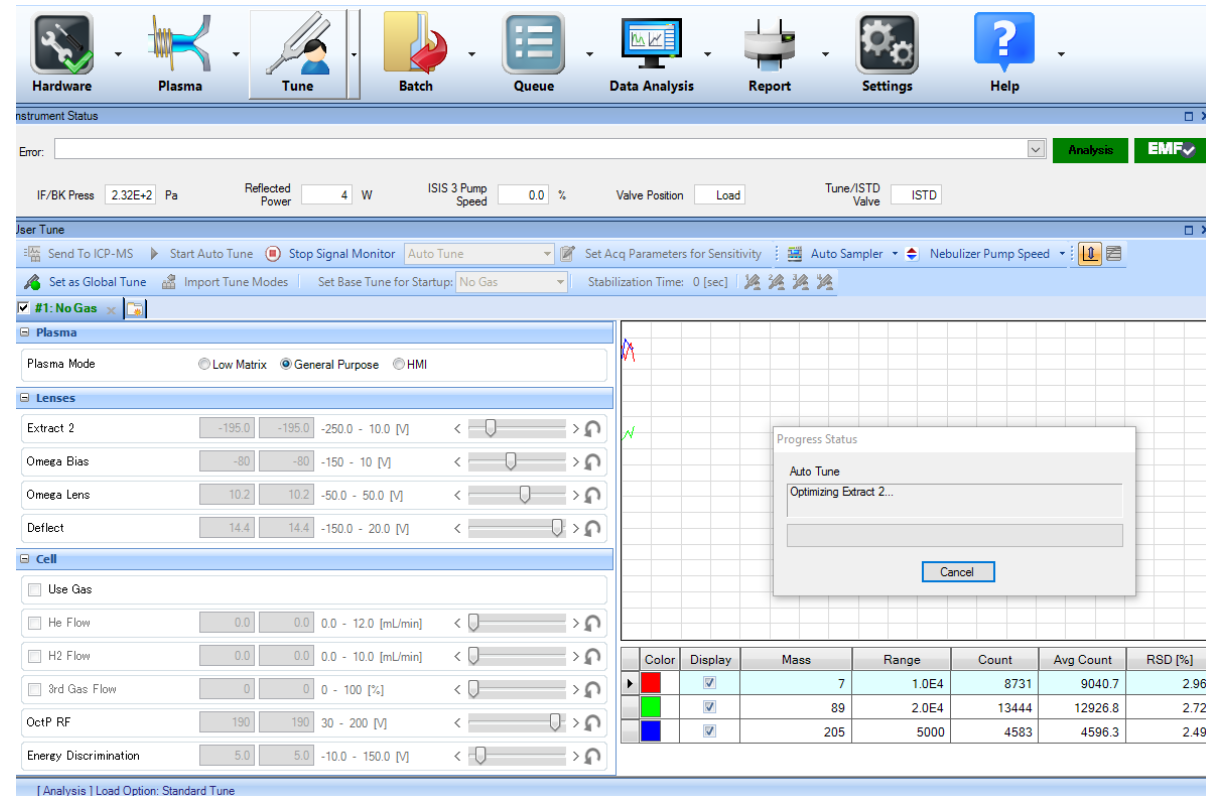
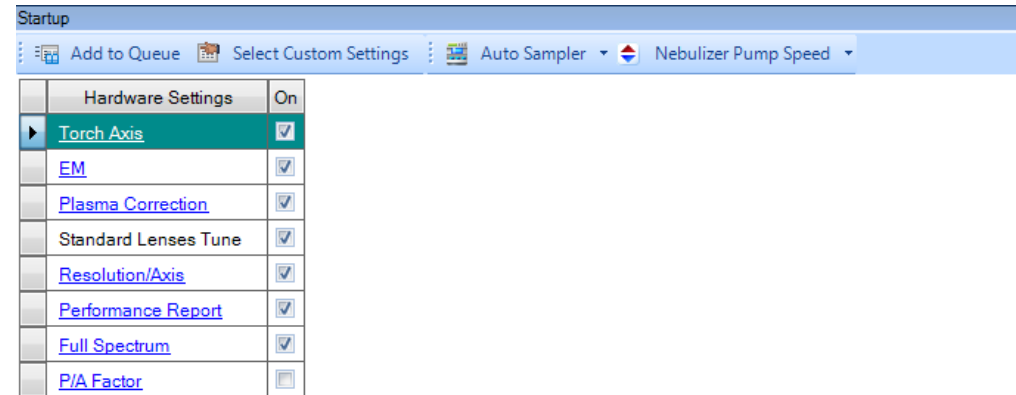
ICP-MS System Optimization

Startup provides a simple, user-configured schedule of system optimization and performance checks

- Automatically generate a Performance Report
- Provides a continuing record of system performance

One-click expert AutoTune for simple optimization

- Ensures consistent performance from day to day
- Independent of operator experience



Key Consumables for ICP-MS

Sample preparation/presentation:

- Peristaltic pump tubing
- Transfer and drain tubing
- ICP-MS standard solutions
- Internal Standard solutions
- Torches
- Spray chambers
- Nebulizers

Ion Extraction:

- Sampler and skimmer cones

Autosampling:

- Sample tubes, racks, probes and transfer tubing

ISIS:

- Peristaltic pump tubing, ferrules & fittings



Agilent ICP-MS Consumable Kits

MINIMIZE WORKFLOW INTERRUPTIONS



Operating Supplies Kits for Agilent ICP-MS

Reduce costs while ensuring long-term confidence in your results

Your Agilent ICP-MS instrument was engineered and manufactured to deliver superb performance. Don't risk the quality and reliability of your results, as well as possible instrument downtime, by using non-Agilent supplies. You'll find that using authentic **Agilent ICP-MS operating supplies kits** will enable you to:

- Maintain accurate analysis
- Save as much as 15% over the cost of purchasing supplies individually
- Keep your instruments running for up to 12 months (depending on use) without worrying about reordering supplies

Increase flexibility with Agilent configurable operating supplies kits

In addition to time and cost savings, you can configure the contents of these kits for your specific instrument configuration and application. That means you can select the type and quantity of sampling cones, skimmer cones, and other supplies you need.

These flexible supplies kits are available for:

- **Agilent 7700/7800/7900 Single-Quadrupole ICP-MS systems** fitted with x-lens
- **Agilent 8800/8900 Triple-Quadrupole ICP-QQQ systems**
- **Agilent 7700s/7900s and 8800/8900 semiconductor ICP-MS systems** fitted with s-lens

These are not just parts and supplies, they are essential components which guarantee the performance, reliability, and longevity of your instrument.

On the following pages you'll find a full portfolio of genuine Agilent ICP-MS operating supplies, listed by instrument. Identify the model you are using and make your selections.



Agilent offers configurable kits for all current Agilent ICP-MS systems, that allow you to select which key components are supplied in the kit e.g. type and quantity of interface cones

- G1131A for 7900 ICP-MS fitted with x- or s- lens
- M5141A for 7800 ICP-MS fitted with x-lens
- G1091A for 8900 ICP-MS fitted with x- or s- lens
- G3690A for 7700x/e and 8800 ICP-MS fitted with x-lens

Ask your Agilent representative for more information



Agilent Technologies

ICP-MS Maintenance & Trouble Shooting

If you need more help – count on the experts



Agilent University



Preventive Maintenance



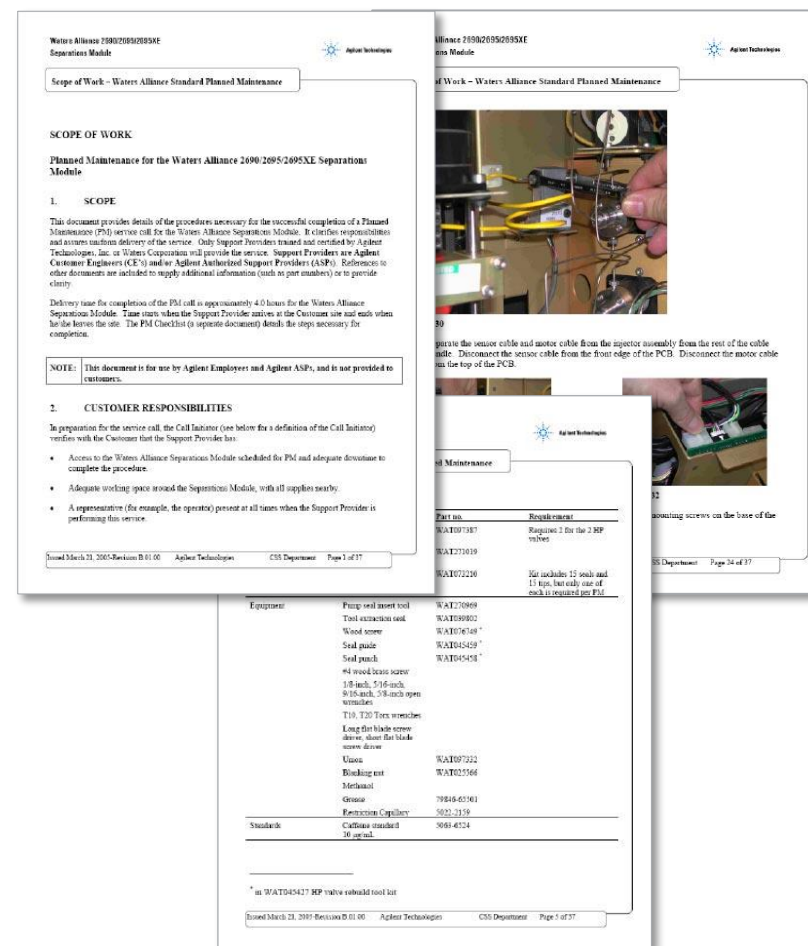
Method and Application Consulting

Agilent Preventative Maintenance Services

Studies show that 60% of instrument failures can be traced to a single cause – lack of preventive maintenance*. These studies also show that failure rates decrease by up to 25% for all mechanical systems when a laboratory implements a preventive maintenance program.

This service includes:

- **Inspection:** Perform general inspection of the complete system.
- **System Cleaning:** Remove covers and clean dust from fans and vent covers.
- **Pump Maintenance:** Replace oil mist filter, drain and replace mechanical pump oil. Verify proper pump operation.
- **Lens Cleaning:** Remove and clean surfaces of the ion lens. Sonicate ion lens parts.
- **Vacuum System Maintenance:** Inspect vacuum hoses and exhaust tubes for possible problems. Check pump for evidence of leakage.
- **Verification:** Check quadropole matching. Replace octopole and perform octopole matching. Perform system auto-tune.
- **Documentation:** Record maintenance in instrument service logs.





Featured technique:
Agilent ICP-MS Online Resource Library

We're committed to bringing you the world's best ICP-MS systems... plus the critical information you need to keep them running at peak performance

To minimize costly downtime—and achieve the best results from your ICP-MS system—you must stay up to date with the best practices for instrument maintenance and operation.

The Agilent ICP-MS Online Resource Library makes it easy by giving you instant access to the latest how-to videos, maintenance procedures, training opportunities, and much more. So you can maximize your daily productivity—and positively impact your lab's success over time.

Top 5 tips for flawless ICP-MS performance

1

2

3

5

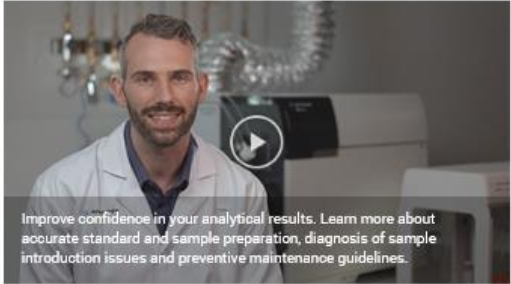
Set high standards

Ensure precise, accurate calibration data by preparing standards fresh from certified reference materials with known uncertainty. Only use high-purity reagents and de-ionized water to reduce contamination. [Learn more](#)

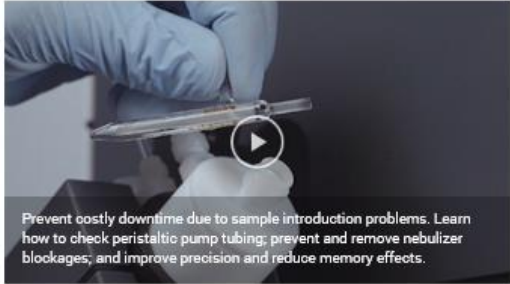
<http://www.agilent.com/en-us/promotions/icp-ms-resource>

ICP-MS maintenance and troubleshooting videos

Part 1: Overview



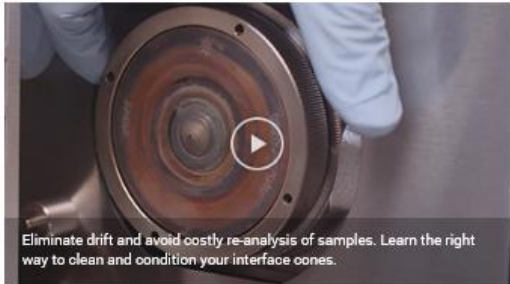
Part 2: Sample Introduction



Part 3: Torch Box



Part 4: Interface Region



ICP-MS workflow

Maximize data quality and productivity with the complete workflow and genuine Agilent supplies for your ICP-MS analysis.

[ICP-MS Supplies](#) | [Captiva Syringe Filters](#) | [ICP-MS Standards](#) | [Interface Cones](#) | [Agilent ICP-MS](#) | [Agilent ICP-QQQ](#)

Interface Cones

Trade-in your used Agilent, Spectron or Glass Expansion platinum interface cones when you purchase replacements from Agilent—save money and help the environment!*

Recycle now

Learn more

* The trade-in program may not be available in all geographies. Contact your local Agilent representative for more details.



Agilent supplies for Agilent instruments

Agilent Technologies is committed to optimizing your laboratory's productivity, so we have produced this list of the most commonly ordered supplies and parts for the 7800/7900 ICP-MS and 8900 Triple Quadrupole ICP-MS. Keep this list handy so you can quickly find the supplies you need and minimize instrument downtime.



Sampler Cones and Supplies (suits all 7800/7900 and 8900 ICP-MS)	Part No.
Graphite gasket for sampling cone, 3/pk	G3280-87009
Nickel sampling cone	G3280-87040
Platinum sampling cone (standard for semicon configuration)	G3280-87036
Ni plated sampling cone (for samples containing > 0.5% HCl and when using max. HMI aerosol dilution)	G3280-87061
Platinum sampling cone with 18 mm insert (for high viscosity and high boiling point acids e.g. H ₂ SO ₄ or H ₃ PO ₄)	G3280-87056
Skimmer cones and bases for 7800 ICP-MS	
Nickel skimmer (standard for 7800)	G3280-87041
Stainless steel skimmer base for nickel skimmer	G3280-80608
Platinum skimmer, Ni base (recommended for organics analysis - requires brass skimmer base)	G3280-87063
Platinum skimmer, Cu base (recommended for best LODs and with higher matrix samples [requires brass skimmer base])	G3280-87060
Brass skimmer base (required for use with Pt skimmer)	G3280-80621
Skimmer cones and bases for 7900 ICP-MS	
Nickel skimmer (standard for 7900 with x lens)	G8400-87200
Stainless steel skimmer base for 7900 with x lens (required for use with Ni skimmer)	G8400-80624
Platinum skimmer, Cu base for 7900 with x lens, (recommended for best LODs and with higher matrix samples [requires brass skimmer base])	G8400-87201
Platinum skimmer, Ni base for 7900 with x lens (recommended for organic analysis [requires brass skimmer base])	G8400-87202
Brass skimmer base for 7900 with x lens (required for use with Pt skimmer)	G8400-80625
Platinum skimmer, Cu base (standard for 7900 with s lens [requires brass skimmer base])	G3280-87064
Brass skimmer base for 7900 with s lens (required for use with Pt skimmer)	G8400-80626

Operating Supplies Kits	Part No.
Basic supplies kits #	
For Agilent 7900 ICP-MS fitted with x-lens and Edwards E2M18 roughing pump using Inland 45 oil	G3280-87003
For Agilent 78/7900 and 8900 ICP-MS fitted with x-lens and roughing pump using AVF 60 SHCE oil	G3280-87221
Configurable parts and supplies kits #*	
For Agilent 7800 ICP-MS system fitted with x- lens	M5141A
For Agilent 7900 ICP-MS systems fitted with x- or s-lens	G1131A
For Agilent 8900 ICP-MS systems fitted with x- or s-lens	G1081A

for kit contents, additional options and more information see Agilent publication "5991-5006EN: Spare Parts and Operating Supplies Kits for Agilent ICP-MS"
*configurable kits must be ordered via your Agilent Representative or Agilent Authorized Distributor. Configurable kits cannot be ordered through the online store.

Links to Other Useful ICP-MS Resources

- ICP-MS parts and supplies (On-line Store):
http://www.chem.agilent.com/store/en_US/Cat-SubCat1ECS_30319/ICP-MS
- Agilent atomic spectroscopy application notes:
<https://www.agilent.com/en-us/library/applications?N=129+900007284>
- Agilent ICP-MS Quick Reference Guide (lists most common consumables items):
http://www.agilent.com/cs/library/flyers/public/5991-7990EN_ICP-MS_Supplies_QRG.pdf
- Agilent Spectroscopy consumables catalog:
http://www.agilent.com/cs/library/catalogs/public/5991-5455EN_Spectroscopy_Catalog_LR.pdf
- Agilent high quality Inorganic and Metallo-Organic standards for Atomic Spectroscopy:
http://www.chem.agilent.com/Library/catalogs/Public/5991-5678EN_Chemical_Stnds_Catalog_LR.pdf
- Agilent supplies for PerkinElmer ICP-OES & ICP-MS systems catalog:
http://www.chem.agilent.com/Library/catalogs/Public/5991-6789EN_ICP_MiniCatalog_Offset_LR.pdf
- Agilent "Make Productivity Happen" workflow webpage:
<http://www.agilent.com/en-us/promotions/make-productivity-happen-spectro#home>
- Agilent recorded webinars for atomic spectroscopy:
<http://www.agilent.com/en-us/training-events/eseminars>

Agilent ICP-MS Journal

Are you a subscriber to the Agilent ICP-MS journal?

- An ICP-MS specific journal produced 4 times/year
- Includes applications, techniques, “real” user stories, news updates and other product information

To register, use this link to the registration form on the Agilent website (or ask your Agilent representative):

<http://www.agilent.com/en-us/newsletters/icpmsjournal>



Summary – To Achieve Quality Data

Most “instrument” failures occur in the sample introduction area:


- Interface cones
- Peristaltic pump tubing
- Drain Assembly
- Torch
- Spray chamber
- Nebulizer



Improper maintenance of this area can result in poor data quality

Frequently even experienced analysts can fail to recognize problems resulting in productivity losses

Establishing good routine maintenance procedures can prevent problems








Agilent Technologies

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Atomic Spectroscopy
Agilent Technologies • 26 videos • 1,299 views • Last updated on Jun 28, 2017

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- 1  **Troubleshooting and Maintenance of ICP-MS Systems PART 1: Overview**
by Agilent Technologies
- 2  **Troubleshooting and Maintenance of ICP-MS Systems PART 2: Sample Introduction**
by Agilent Technologies
- 3  **Troubleshooting and Maintenance of ICP-MS Systems PART 3: Torch Box**
by Agilent Technologies
- 4  **Troubleshooting and Maintenance of ICP-MS Systems PART 4: Interface Region**
by Agilent Technologies
- 5  **Agilent OneNeb Series 2 Nebulizer**
by Agilent Technologies

<https://www.youtube.com/user/agilent>

Thank you for your attention. Question & Answer session.



Prevent nebulizer blockage



Pay attention to the interface cones



Keep it clean



Set high standards



Don't neglect the pump tubing



<https://www.agilent.com/en/promotions/icp-ms-resource>