



Guidelines for Trouble Shooting and Maintenance of ICP-MS Systems

Today's Agilent: Atomic Spectroscopy

World's best, most complete atomic spectroscopy portfolio!



ICP-MS



Flame AAS



Graphite Furnace AAS



4200 MP-AES



New Agilent 7900 ICP-MS Performance Highlights

Rewriting the rules on ICP-MS

Better Analytical performance experience

- Ultra high matrix tolerance
- Superior sensitivity and lower background noise
- Wider dynamic range
- New Productivity Option (ISIS 3)
- Ultra fast scan speed for Single Nanoparticle analysis

Better Software experience

- ICP-MS MassHunter 4.1
- Method Wizard
- Mobile device support

Better Support experience

- Familiarization Tutorials/Videos
- Remote Advisor support



For more details, see: http://www.agilent.com/chem/icpms10xbetter



Next Generation 4200 MP-AES Performance Highlights Change is in the Air!

Better Analytical performance experience

- 2nd generation of proven MP-AES technology
- Robust torch design for superior analytical performance with complex matrices
- Mass flow control of nebulizer gas and robust sample introduction
- Enhanced accuracy and long term stability with tough samples
- Expands the application range of MP-AES

Better Software experience

- MP Expert V1.2
- Intuitive software interface
- Advanced features in the 'PRO' pack

Better after sales experience

- No flammable gases or ongoing gas costs
- Runs from Nitrogen extracted from air using Agilent's nitrogen generator



For more details, see: http://www.agilent.com/chem/runsonair



Agilent's Atomic Spectroscopy Portfolio - Features

Flame AA

MP-AES

Graphite Furnace AA

ICP-OES

ICP-MS



Lowest price

- Single element
- DLs typically ~100's ppb
- Fast (for 1 element)
- Good elemental coverage
- Low running cost



Lowest running cost

- Multi element
- DLs typically single to 10's ppb
- Faster
- Broader elemental coverage
- Lowest running cost



Trace levels at lowest price

- Single element
- DLs typically 10's to 100's ppt
- Very slow
- Limited elemental coverage
- Moderate running cost



Fastest measurement

- Multi element
- DLs typically single ppb
- Very fast
- Can measure most elements
- High running cost



Broadest coverage

- Multi element
- DLs typically single or sub-ppt
- Fast
- Can measure almost all elements
- Highest running cost

Lowest

Selling Price

Highest



Common ICP-MS Problems Reported by Customers

Sensitivity:

- Sensitivity is worse than it used to be
- I have a new application and I can't get the sensitivity I need
- How come I can't get the instrument to meet published detection limits?

Precision

Sensitivity is acceptable but precision is terrible

High noise

Can get the "right answers", but very noisy signal – this is also giving bad precision.

Accuracy

Instrument does not give the "right" results.

Poor Sample Throughput

- The instrument throughput needs to improve
- Nebulizer and/or interface cones blocks too quickly



Causes of Poor ICP-MS Sensitivity

Worn pump tubing Sample Blocked nebulizer introduction Blocked injector in torch system Worn or deformed torch Blocked interface cones Poor optimization – especially the neb. flow Method setting – tuning? **Optimization** Torch alignment Wrong tubing type High blank level **Standard** (& sample) Standards prepared correctly? preparation Samples prepared correctly? – internal standard selection Reaction gas purity



Causes of Poor ICP-MS Precision

Worn pump tubing Beading in spray chamber Sample introduction Nebulizer condition and performance system Air leaks in transfer tubing Sample line not grounded Torch alignment Interface cones not "conditioned" with your sample matrix **Optimization** Poor optimization – especially the neb. flow Method setting – appropriate delay times? Nebulizer choice for your samples **Standard** Wash-out (memory effects) (& sample) preparation



Causes of Poor Accuracy in ICP-MS

Worn pump tubing Sample Contamination from pump tubing introduction system Blockage in nebulizer and/or torch Blockage in interface cones Interferences Poor optimization – especially the neb. flow **Optimization** Choice of internal standard Insufficient stabilization time Standard preparation **Standard** Incomplete digestion – particles in solution (& sample) No matrix matching preparation Wash-out (memory effects)

ICP-MS Sample Introduction System Tips



Do:

Check performance report each analysis

Check/monitor the nebulizer uptake

Check/adjust the peri pump tubing

Check the blank reading

Rinse between samples & at the end of the run

- Rinse should match sample matrix

Clean the torch/nebulizer/cones regularly

- Inspect condition of the nebulizer tip
- Inspect condition of the interface cones

Condition the interface cones with your sample matrix before analysis





Assume system is still optimized

Assume nebulizer flow rate is the same

Overtighten the pressure adj. screw

Use a simple water blank

Wait until you have blockage before cleaning

Peri Pump Tubing Tips

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

- Tubing diameters
 - Want tubing used for waste to be larger ID than sample ID
- Chemical compatibility
 - · Ensure tubing is resistant to the solvent being used
- Replace frequently
 - Preclean 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



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.

Sample Introduction

Nebulizer Tips

Think "PREVENTION"

- Micro-flow nebulizers
- Zero tolerance to undissolved solids
 - Plugging of annulus and/or capillary
 - Filter/Centrifuge/gravitational settling sampler positioning
 - Use only lintless wipes
- Autosampler enclosures
- Rinse at least 10 minutes with a reagent blank before extinguishing plasma



Image modified from "Pneumatic Nebulisers 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

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:

- 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); OR
- Use a dedicated nebulizer cleaning tool to force methanol solution through the tip

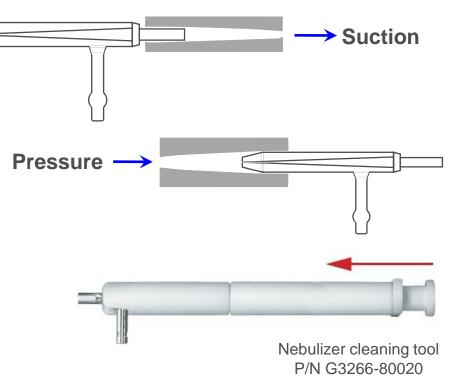
For salt deposits:

 Soak the nebulizer overnight in a beaker of 25% Fluka RBS-25 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







Cleaning the Spray Chamber

For routine cleaning, soak the end cap and spray chamber in 5% nitric acid for ~30 mins

Rinse, allow to dry and refit

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

- 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
- Rinse, allow to dry and refit





Cleaning the Torch

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

Replace if deformed or chipped

Do not sonicate!

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

- For more stubborn stains, soak in aqua regia (1:3 HNO₃: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

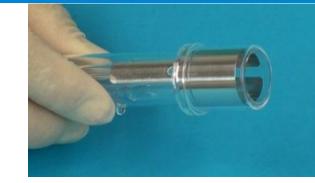
Ensure the torch projection fits into the slot on the torch holder

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

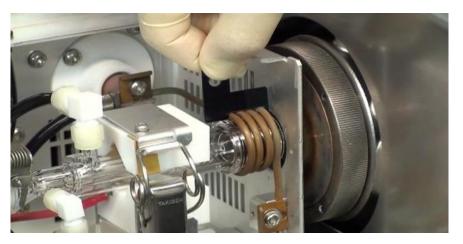
Reconnect gas fittings and transfer tube from spray chamber

Test plasma ignites and instrument switches to "analysis" mode

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



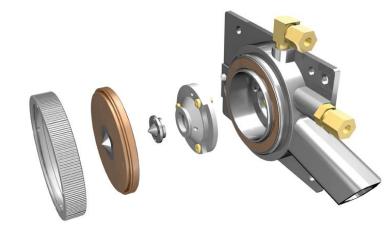




Interface Cone Choices

Sampler and skimmer cones extract a portion of the plasma ion population and transfer this into the higher vacuum mass spectrometer for detection

- Nickel cones are standard
 - Inexpensive
 - Good thermal and chemical resistance
 - Uses standard brass skimmer base
 - Typically use 3-5/year (based on ~350 samples/day)
- Ni plated sampling cone (optional)
 - Used when samples containing > 0.5% HCl are analyzed
 - Also used for routine operation with HMI aerosol dilution
 - Requires stainless steel skimmer base
- Pt tipped cones are optional
 - Essential for users wanting to analyze aggressive acids (esp. HF digests)
 - Also used when O₂/Ar option gas is used to analyze organic solvents
 - Pt skimmer with the Ni base used for organics analysis requires brass skimmer base
 - Pt skimmer with the Cu base used for best DLs and high matrix samples (incl. HF) - requires brass skimmer base
 - Typically use 1-2/year (based on ~350 samples/year)













Interface Cone Cleaning –Why?

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

- Sensitivity are orifices getting plugged?
- Long term precision are cones adequately conditioned?
- High background or distorted peak shapes
- Interface vacuum changing deposition?

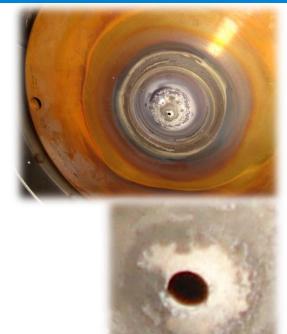
Other reasons to clean the cones?

- If there is a build up of deposits on the orifice (should be circular and free of deposits)
- If the orifice of the cone is discolored (due to excess heat)

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

If analyzing the same type of samples, clean to remove only superficial deposits. Step 1 may be all that is required



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

Interface Cone Cleaning – Step 1



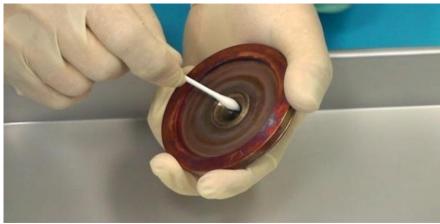
For normal contamination:

Just need a simple clean with pure water:

- Dip a cotton swab in pure water and clean both sides of the cone
- Rinse with pure water
- Ultrasonic clean the cones in pure water for more than 5 minutes



Agilent P/N 9300-2574





Interface Cone Cleaning – Step 2

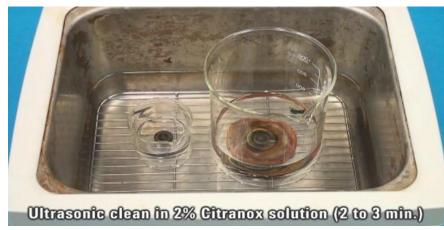


For normal contamination:

If performance is still not satisfactory, clean with a 2% Citranox solution

(NOT MORE THAN 2%!)

- Ultrasonic clean the cones in a 2% Citranox solution for 2 to 3 minutes
- Rinse with pure water
- Ultrasonic clean in pure water for an additional 2 to 3 minutes





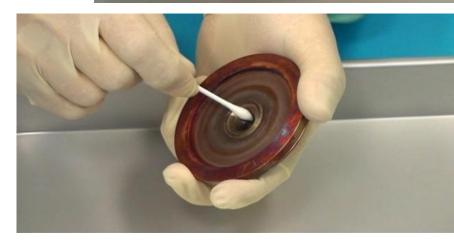
Interface Cone Cleaning – Step 3

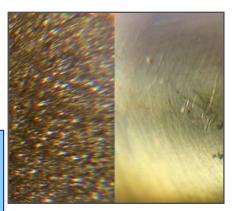
ONLY For more severe contamination:

Clean with a 2% nitric acid solution

- Dip a cotton swab in 2% nitric acid and clean both sides of the cone (DO NOT SOAK IN ACID!)
- Rinse with pure water
- Ultrasonic clean in pure water for 2 to 3 minutes
- Rinse with pure water
- Ultrasonic clean in pure water for an additional 2 to 3 minutes

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

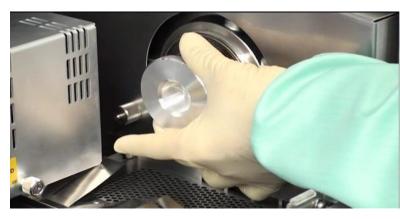
Refit the sample cone and tighten by hand

Check the vacuum levels to confirm correct installation

- Interface pressure: 500 Pa (~4torr, 0.005atm)
- Analyzer pressure: 0.002 Pa (~1.5 x10-5 torr, 2 x10-8 atm)

If necessary, retension the skimmer and sample cones



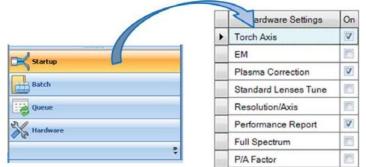


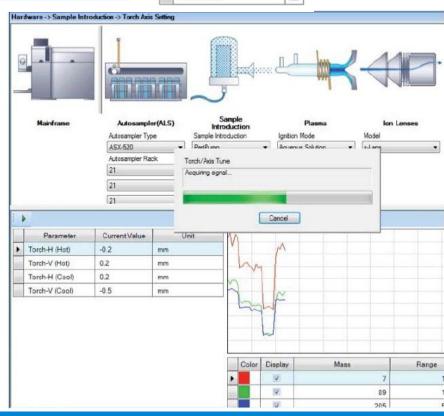




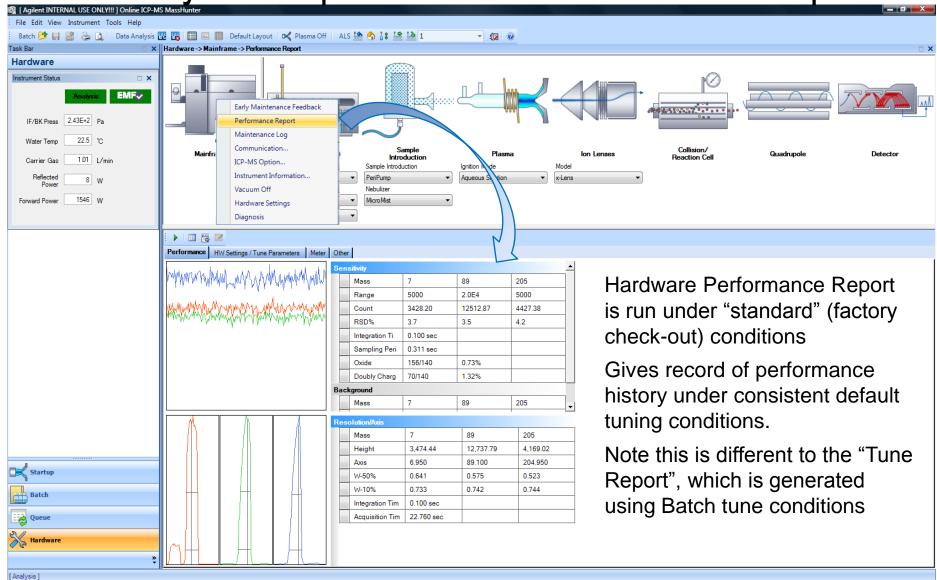
ICP-MS System Optimization – Automated "Expert" Tuning

- Startup provides a simple, user-configured schedule of system optimization and performance checks
- One-click expert autotuning for simple plasma optimization
- Ensures consistent performance from day to day
 - Independent of operator experience
- Automatically generate a Performance Report
 - Provides a continuing record of system performance





ICP-MS System Optimization – Performance Report

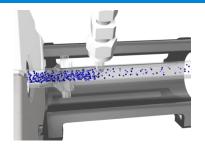


Recommended Procedures at End of the Day

- 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
- 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. a) Close the current worksheet leave Mass Hunter S/W running
 - b) Leave mains power and argon on
 - Keeps instrument in stand-by mode (ensures fastest start-up)

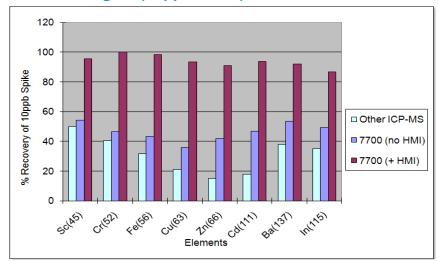
Agilent ICP-MS Performance - Benefits

Best matrix tolerance & less interferences

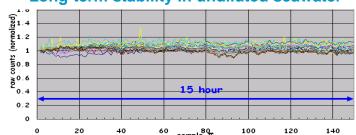


- Handles tough sample matrices better than any other ICP-MS
 - Highest plasma temperature (lowest CeO/Ce) as standard
 - HMI (high matrix introduction) allows direct analysis of difficult samples e.g. sea water
- ORS³ operates in He collision mode, to remove ALL polyatomic interferences
 - Enables reliable removal of all polyatomic interferences using a single set of conditions
 - Gives lower levels of interference and better long-term stability

Relative signal (suppression) in undiluted seawater



Long-term stability in undiluted seawater



HMI gives lower suppression and better long-term stability



Tips to Improve Standard Preparation

- How are they prepared?
 - Ensure purchased standards are still within "Use By" date
 - Use calibrated pipettes and class 'A' volumetric flasks for dilutions
 - Periodically, check accuracy & reproducibility of your pipettes
 - Use de-ionized water (Type I conductivity ≥ 18 M^{\text{\Omega}}/cm³)
 - Lower grades may have contamination
 - Use serial dilutions for preparing low concentrations from 10,000 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 (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
 - Stabilize with acid low pH ensures better stability



Tips to Reduce Contamination

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



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 (Cu, Fe, Zn, Cd)
- Powdered gloves (esp. for Zn)

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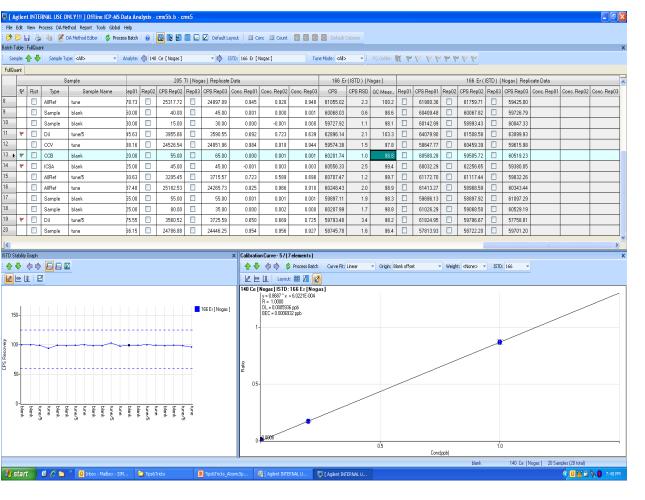


Tips to Improve Accuracy of Results

- 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 Improve Accuracy – Internal Std. Recovery



- Reprocess data post run using an alternate internal standard
- Ensure Internal Standard used is similar in mass
- Check whether the Internal Standard is present in the sample
 - If necessary, do a Semi-Quant measurement
- Dilute with calibration blank matrix

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



ICP-MS – Recommended Maintenance Schedule

Daily:

- Argon gas pressure
- 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)

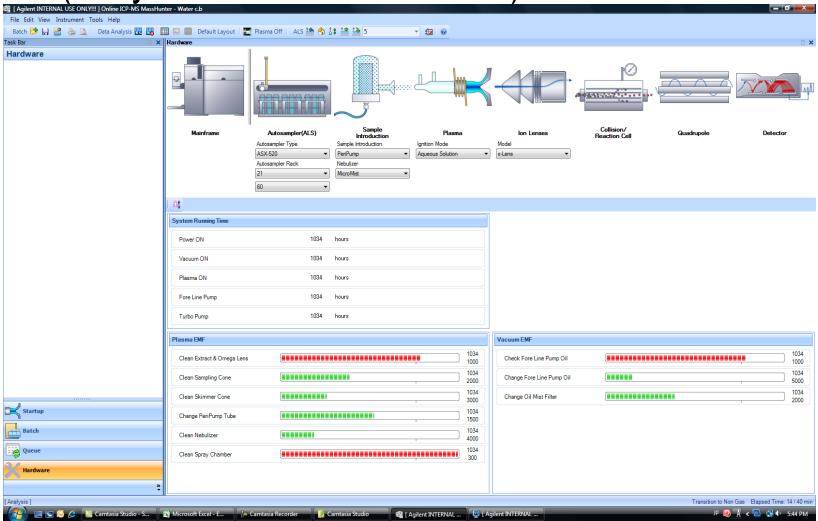
Frequently, as needed -- perform these operations:

- 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)

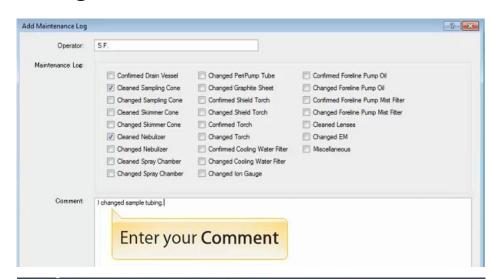


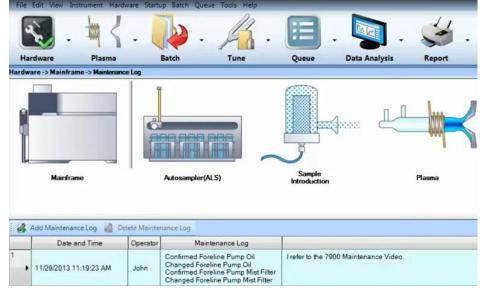
EMF window shows usage of various components and predicts when to perform maintenance (user definable)



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





Summary – 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

Part Number	Description	Content					
G3280-67003	Basic Consumables Kit for 7700x	Peristaltic pump tubing for sample intro Sample tubing (ID 0.5mm) Torch (2.5mm)	Graphite gasket for sampling cone Rotary pump oil (I L)				
G3280-67004	Comprehensive Spares Kit for 7700x/e	Peristaltic pump tubing Sample tubing (ID 0.5mm) Online ISTD addition kit Drain tube assembly Spray Chamber (G3280-80008) Ball joint connector Gas connector for dilution gas port Carrier gas connector for Micro Mist Plug for dilution gas port Torch (G3280-80001) Clamp	Long life shield plate Bonnet plasma gas tubing and gas line connector Graphite gasket for sampling cone 2 Ni Sampling and Skimmer cones Screw and spacer kit for x-lens O-ring for cell Octopole assembly Polishing paper for ion lens Oil mist filter element Rotary pump oil				
G3280-67007	Comprehensive Spares kit for 7700s	Peristaltic Pump tubing for sample intro Spray chamber (G3280-80008) Ball joint connector Gas connector for dilution gas port Clamp Torch (G3280-80001) Long life shield plate Bonnet Plasma / aux gas line tubing Work coil	Graphite gasket for sampling cone PT sampling and sklimmer cone (one each) Screw and spacer kit for x-lens O-ring for cell Octopole assembly Polishing paper for ion lens Oil mist filter element Rotary pump oil				
G4911-67001	Comprehensive Spares kit for ISIS	PFA sample tubing (0.3 mm and 0,5 mm ID) PTFE sample tubing (0.8 mm and 2.0 mm ID) Peristaltic Pump tubing for sample intro Tubing identification tags	Tee, Cross and Union joints Teflon nuts for 1.6 mm and 3.0 mm OD tubing Front and back ferrules for 1.6 mm and 3.0 mm OD tubing Tubing clamp Spiral tubing				

There's also Speciation kits – basic LC connection kit (G1820-65541) & comp. LC connection kit (G1833-65200)



ICP-MS Pt Cone Trade-in Program

- ICP-MS interface cones are expensive and need regular replacement
- Now user's purchasing new 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





For more details, see: http://www.agilent.com/chem/Ptcone



Where to Find the Right Consumable?

Analytical Consumables: Consumables & Supplies 1-800-227-9770 (Option 1,1) www.agilent.com/chem/contactus

Agilent Assist:
Instrument Sales &Services
1-800-227-9770 (Option 1,3)
www.agilent.com/chem/contactus

On-Line resources:

ICP-MS Parts & Supplies

2nd Edition Speciation Handbook

ICP-MS Journal Archives

ICP-OES Parts & Supplies

Atomic Absorption Supplies

AA FAQs

Instrument Parts & Supplies

Atomic Spectroscopy Application Notes

Recorded Agilent e-Seminars

Agilent Quick Reference Guide for ICP-MS (pub. # 5990-8182EN)

Agilent Spectroscopy Supplies Catalogue (pub # 5991-1060EN)

Instrument User Manuals







Other Support Resources for Agilent ICP-MS Users

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.chem.agilent.com/en-US/newsletters/icpmsjournal/Pages/default.aspx

Are you a subscriber to the Access Agilent newsletter?

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Summary – To Achieve Quality Data

- Most "instrument" failures occur in the sample introduction area
 - Includes
- Interface cones
- Peristaltic pump tubing
- Drain Assembly
- Torch
- Spray chamber
- Nebulizer
- Improper maintenance of this area can result in poor data quality
- Frequently less experienced analysts can fail to recognize problems resulting in productivity losses
- Establishing maintenance procedures can prevent problems

Questions?



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