Guidelines for Troubleshooting and Maintenance of ICP-MS Systems

Presented by Eric Vanclay, Atomic Spectroscopy Consumables Product Manager

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.
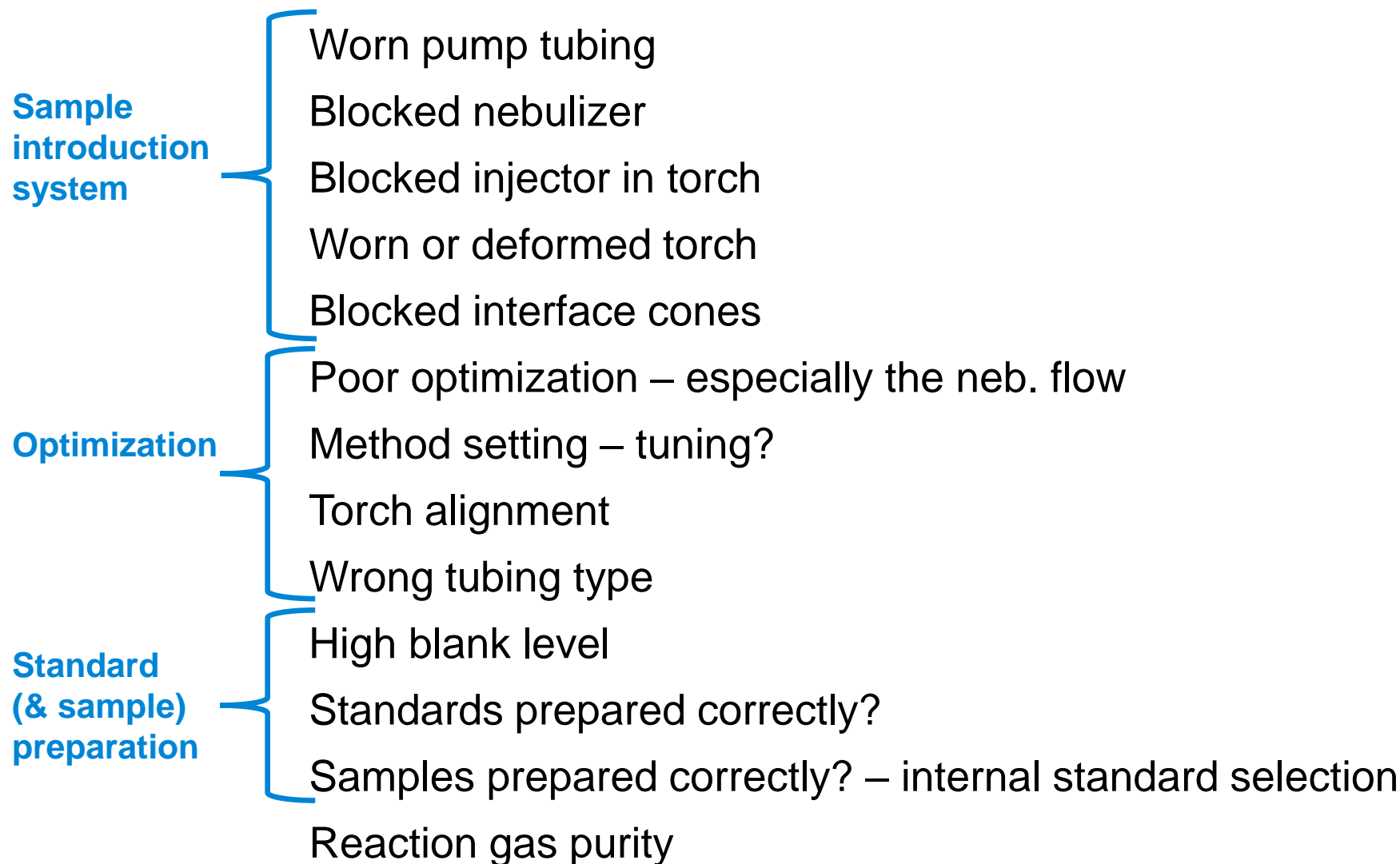
Accuracy

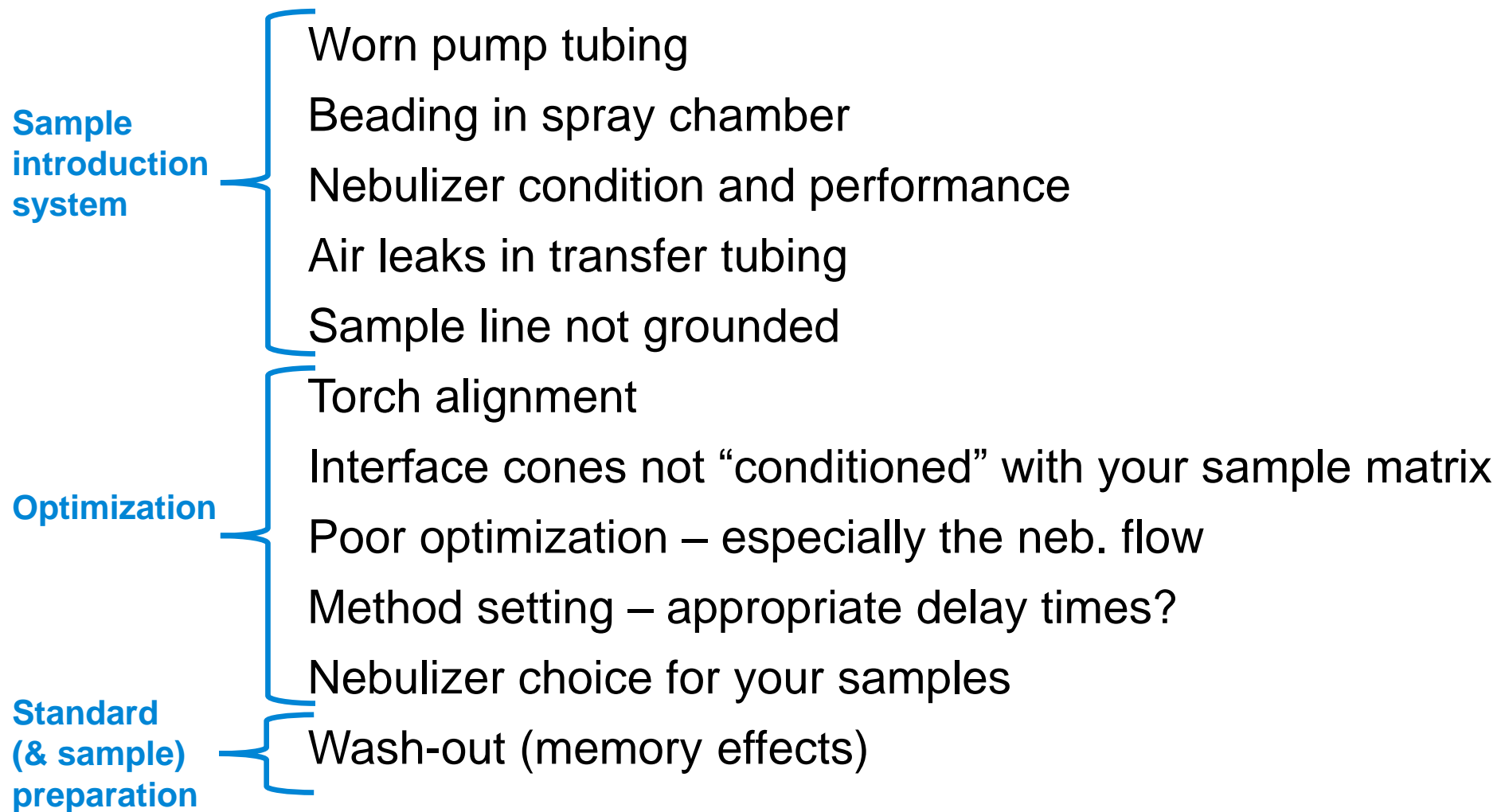- Instrument does not give the "right" results.

Poor Sample Throughput

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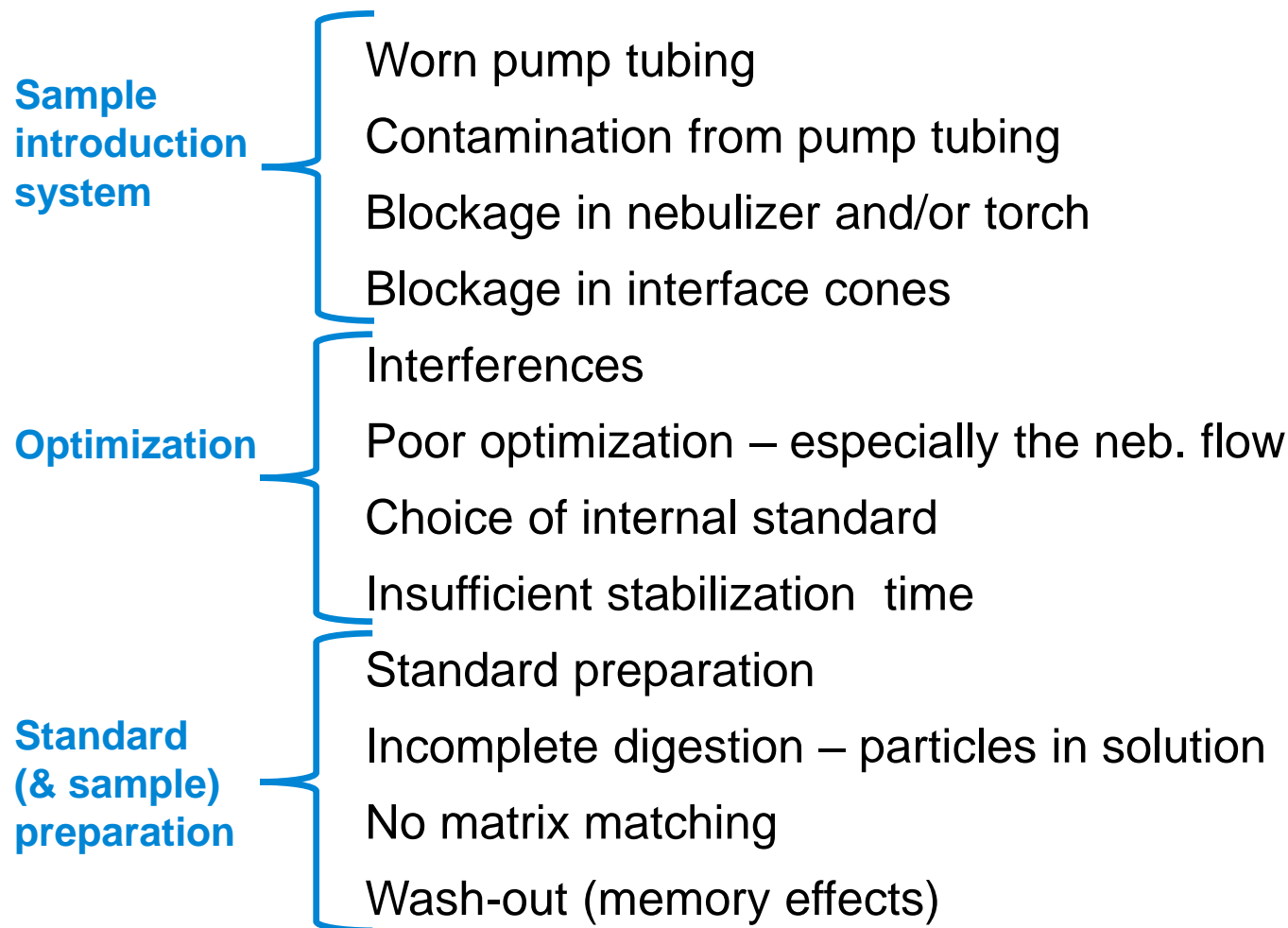- Nebulizer and/or interface cones blocks too quickly

Causes of Poor ICP-MS Sensitivity

Sample introduction system

- Worn pump tubing
- Blocked nebulizer
- Blocked injector in torch
- Worn or deformed torch
- Blocked interface cones

Optimization

- Poor optimization – especially the neb. flow
- Method setting – tuning?
- Torch alignment
- Wrong tubing type

Standard (& sample) preparation

- High blank level
- Standards prepared correctly?
- Samples prepared correctly? – internal standard selection
- Reaction gas purity

Causes of Poor ICP-MS Precision

Sample introduction system

Worn pump tubing

Beading in spray chamber

Nebulizer condition and performance

Air leaks in transfer tubing

Sample line not grounded

Optimization

Torch alignment

Interface cones not "conditioned" with your sample matrix

Poor optimization – especially the neb. flow

Method setting – appropriate delay times?

Nebulizer choice for your samples

Standard (& sample) preparation

Wash-out (memory effects)

Causes of Poor Accuracy in ICP-MS

Sample introduction system
- Worn pump tubing
- Contamination from pump tubing
- Blockage in nebulizer and/or torch
- Blockage in interface cones

Optimization
- Interferences
- Poor optimization – especially the neb. flow
- Choice of internal standard
- Insufficient stabilization time

Standard (& sample) preparation
- Standard preparation
- Incomplete digestion – particles in solution
- No matrix matching
- 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
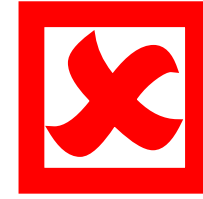
Don't:

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

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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"

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

- 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

Peri Pump Tubing Tips

Symptoms:

- Peri pump tubing that looks/feels worn or has a strange color
  - 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 Nebulizer or disconnecting tubing segments

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

Sample Introduction – Nebulizer Tips

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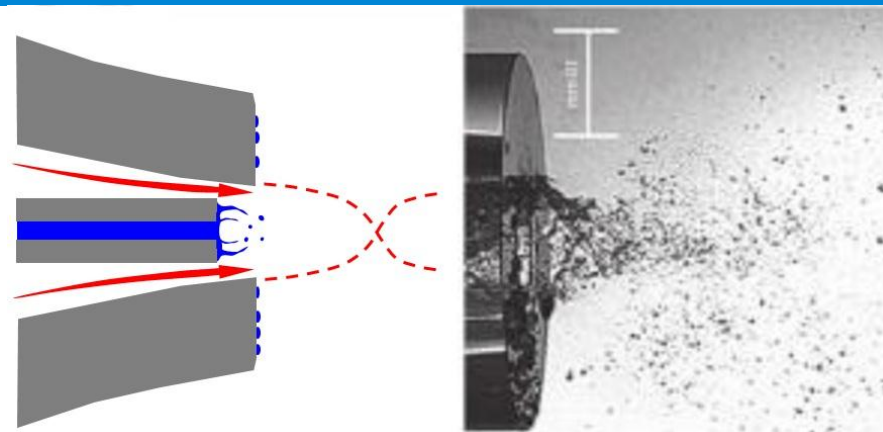
- Micro-flow nebulizers
- Zero tolerance to undissolved solids
  - Plugging of annulus and/or capillary
  - Filter/Centrifuge/gravitational settling – sampler positioning
  - Use only lintless wipes
- Autosampler enclosures
- Rinse at least 10 minutes with a reagent blank before extinguishing plasma

1. 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.

Do not use an ultrasonic bath.

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

Suction

Pressure
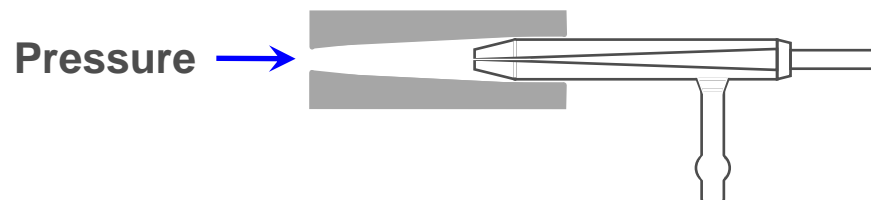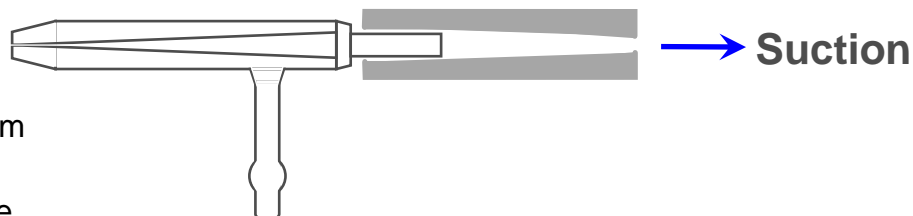
For salt deposits:

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

Nebulizer cleaning tool
P/N G3266-80020

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
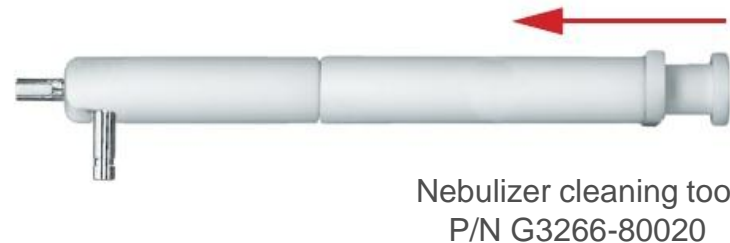
Cleaning the Spray Chamber

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 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

5% Nitric Acid solution
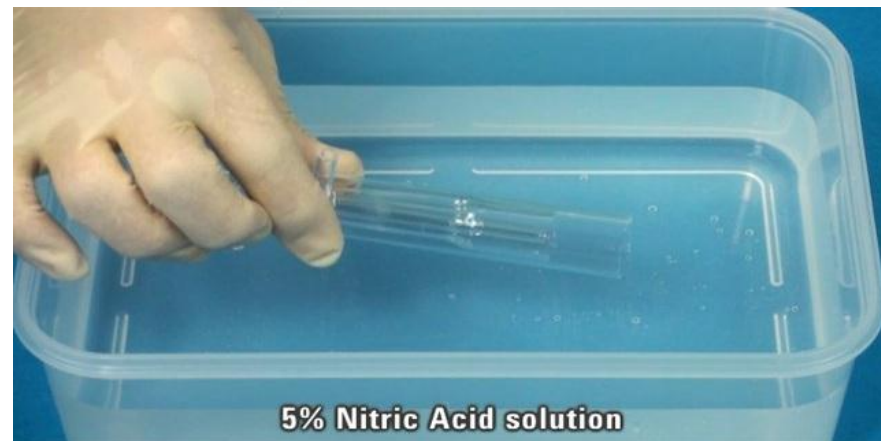
Torch damage due to incomplete drying

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
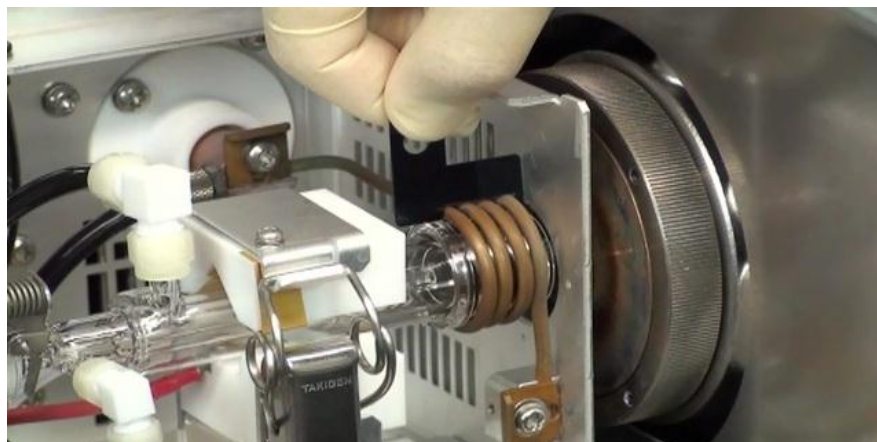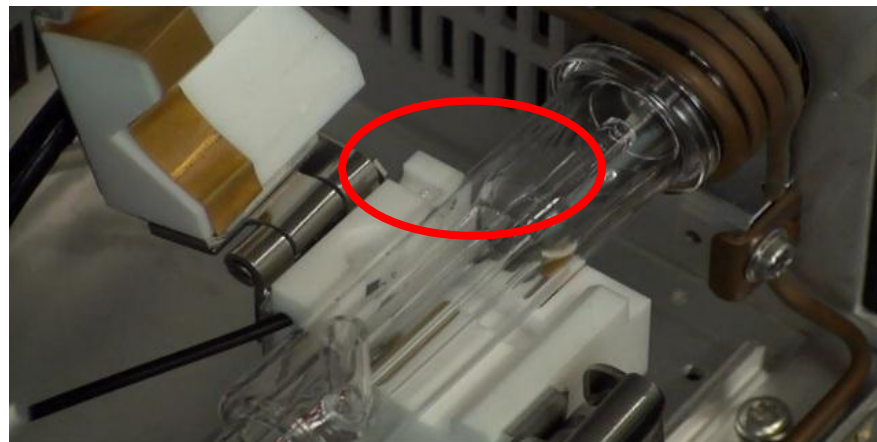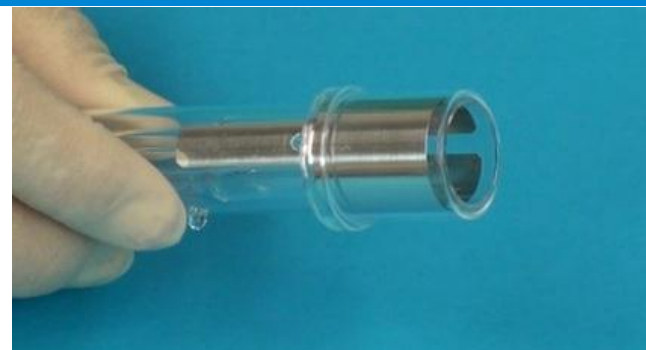
Interface Cone Choices

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 O_2/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)

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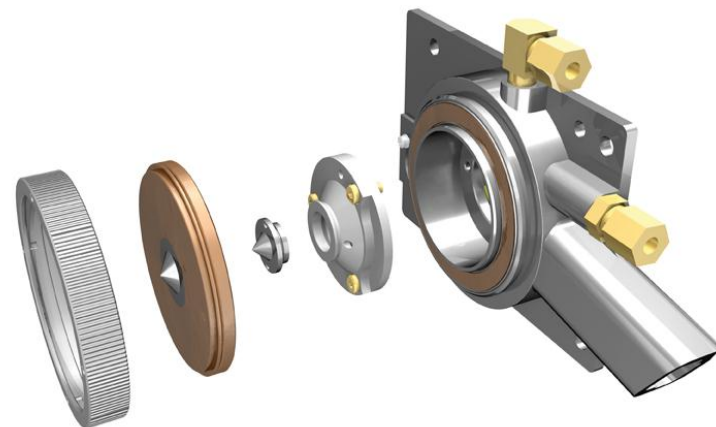
Interface Cone Cleaning –Why?

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?

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

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

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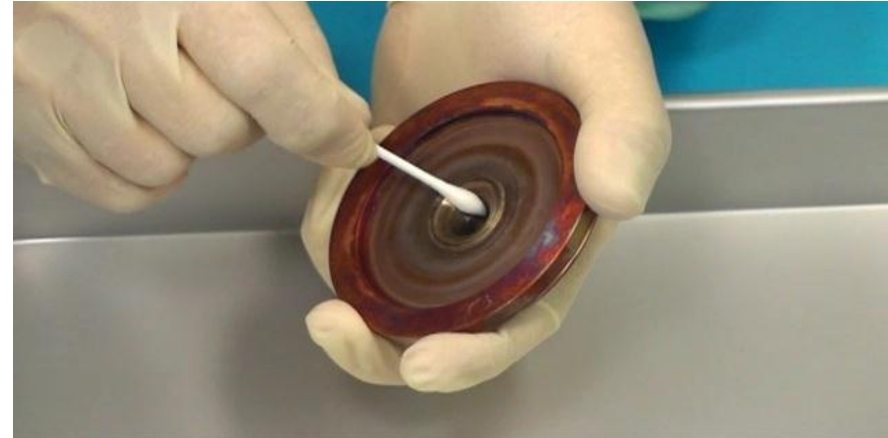
Interface Cone Cleaning – Step 1

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

Ultrasonic clean in pure water (more than 5 min.)

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

Ultrasonic clean in 2% Citranox solution (2 to 3 min.)

Ultrasonic clean in pure water (more than 5 min.)

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
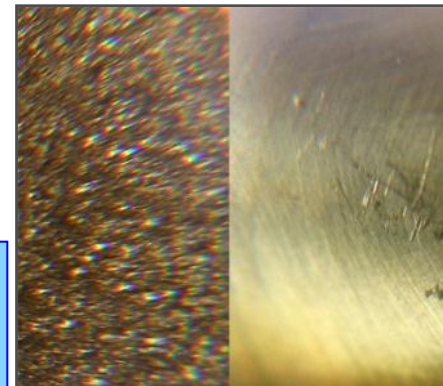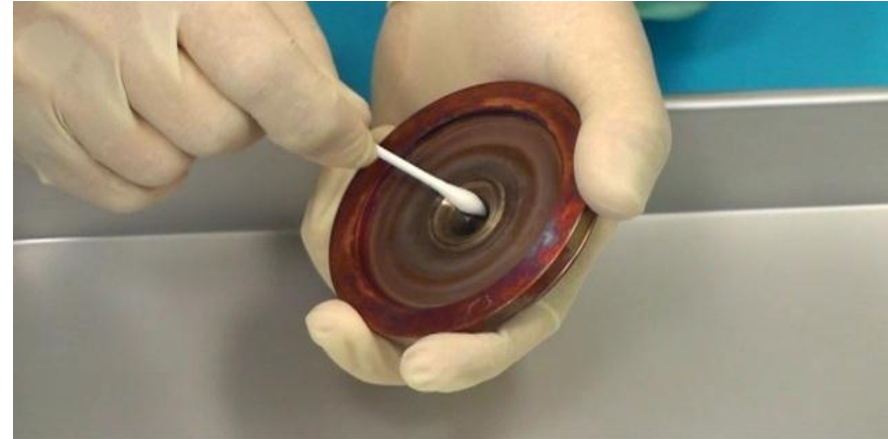
Pitted nickel cone from effect of HNO_3 soak (left side) and clean machined metal on right.

Re-installing the Cleaned Cones

Checking the graphite gasket

Check the condition of the graphite gasket and replace if necessary
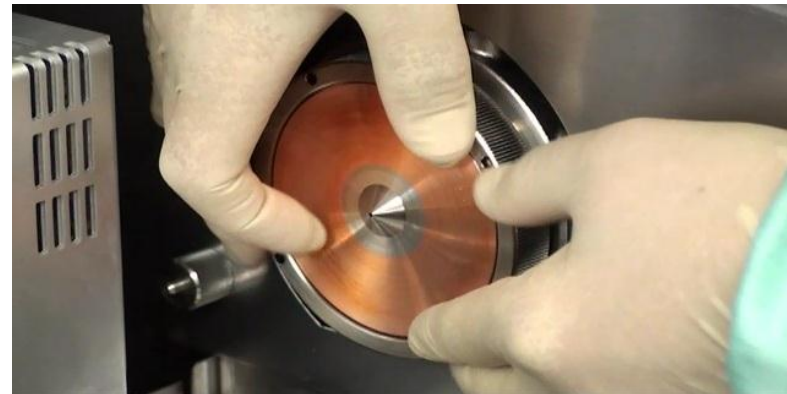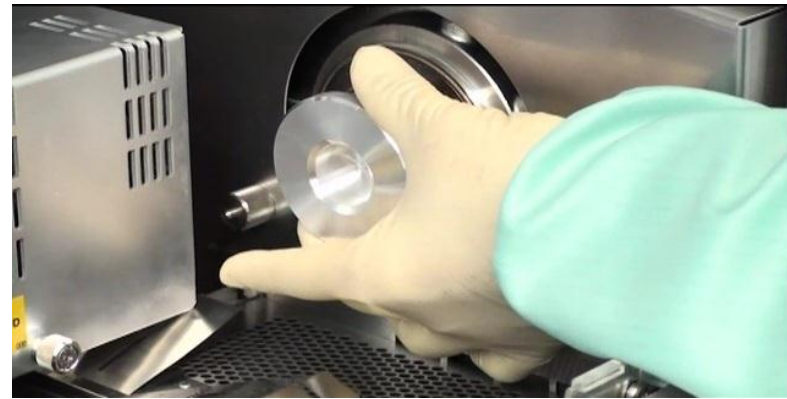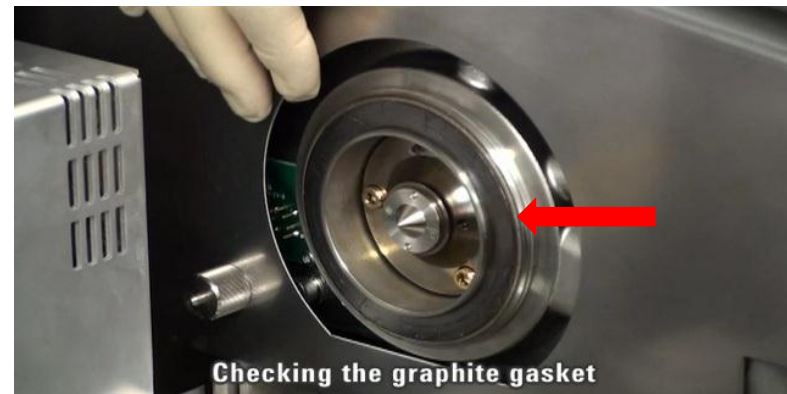
Refit the skimmer cone using the removal tool

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 (~1.5 x10-5 torr, 2 x10-8 atm)

If necessary, retension the skimmer and sample cones

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. a) Close the current worksheet – leave Mass Hunter S/W running

    b) Leave main power and argon on
   -   Keeps instrument in stand-by mode (ensures fastest start-up)

Agilent ICP-MS Performance - Benefits

## Best matrix tolerance & less interferences
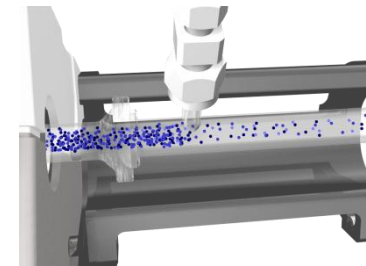
- Handles tough sample matrices better than any other ICP-MS

  - Highest plasma temperature (lowest CeO/Ce) as standard

  - HMI (high matrix introduction) allows direct analysis of difficult samples e.g. sea water

- ORS[3] 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
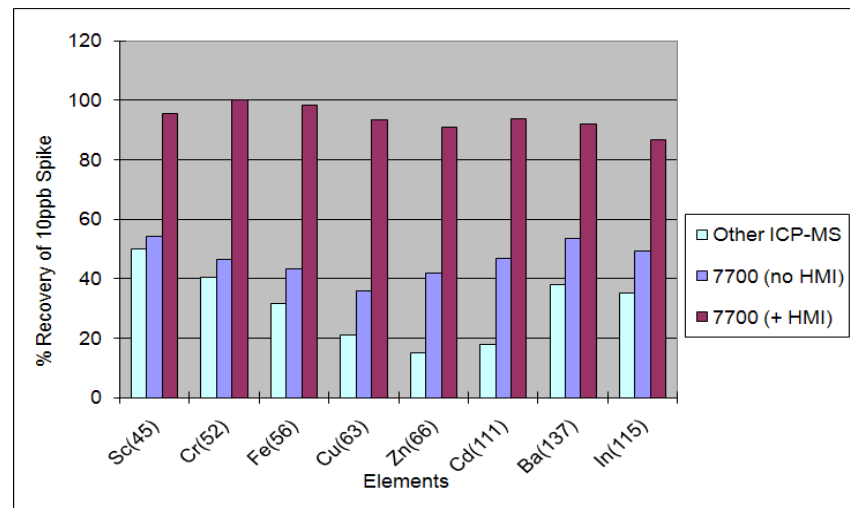
Long-term stability in undiluted seawater
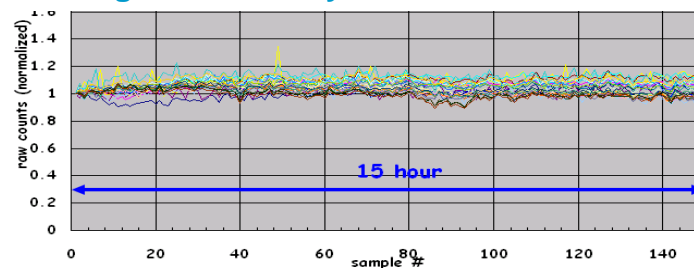
HMI gives lower suppression and better long-term stability

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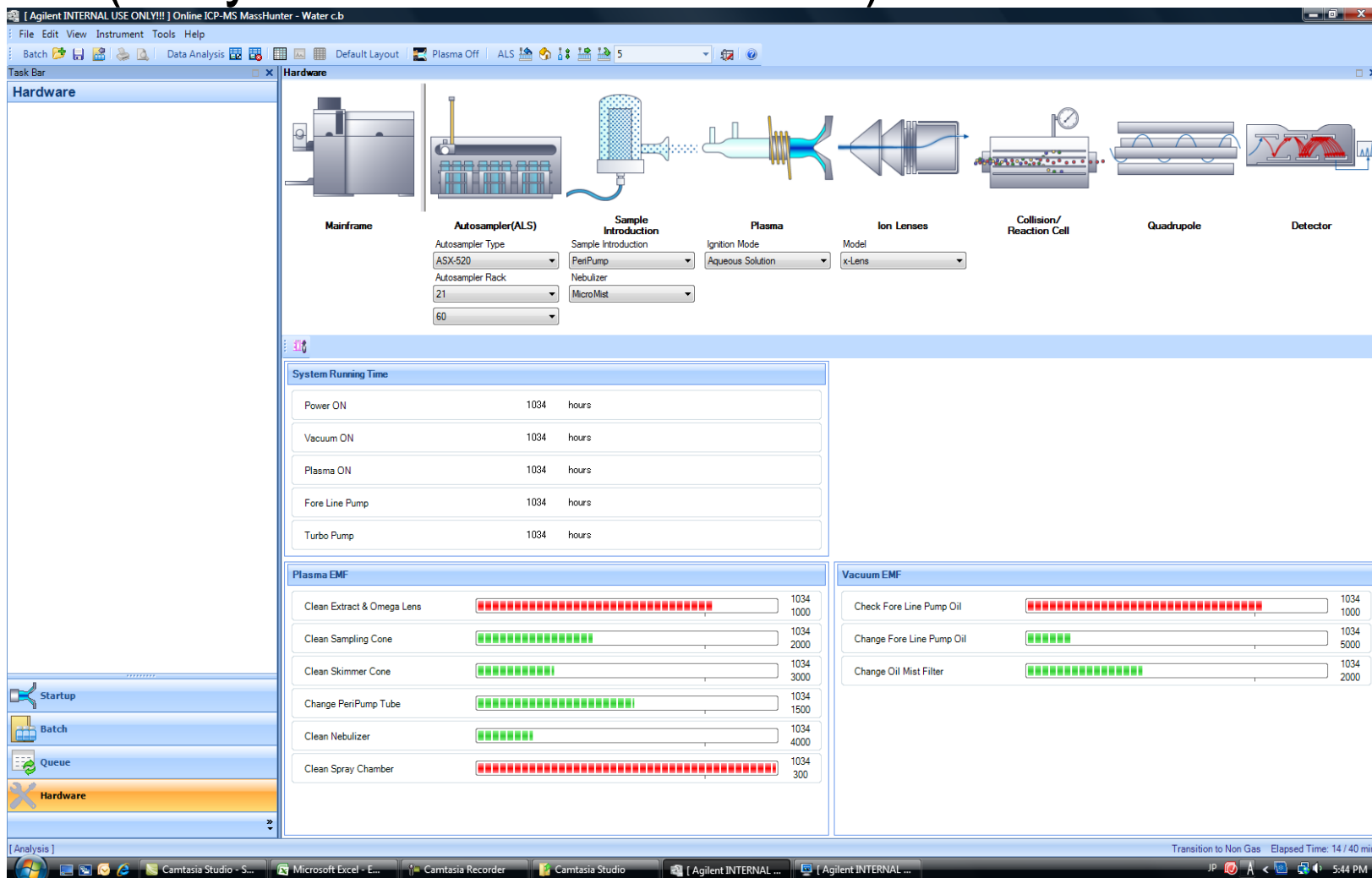
EMF window shows usage of various components and predicts when to perform maintenance (user definable)

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

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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

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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 0.5 mm) Torch (2.5 mm)	Graphite gasket for sampling cone Rotary pump oil (I L)
G3280-67004	Comprehensive Spares Kit for 7700x/e	Peristaltic pump tubing Sample tubing (id 0.5 mm) 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)

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

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