Guidelines for Trouble Shooting and Maintenance of ICP-MS Systems

Simple things that will save you time and money

Common ICP-MS Problems Reported by Customers

Sensitivity:

- Sensitivity is worse than it used to be
- How come I can't get the instrument to meet published detection limits?

Precision

- Sensitivity is acceptable but precision not

Accuracy

- Instrument does not give the "right" results.

Need to improve Sample Throughput

- Need to minimize vial to vial run time
- Nebulizer and/or interface cones require frequent maintenance

Causes of Poor ICP-MS Sensitivity

Worn pump tubing

Blocked nebulizer

Partially blocked injector in torch (rare)

Partially blocked interface cones

Poor optimization – especially the neb. flow

Extract/Omega lens needs cleaning

Insufficient argon pressure or flow

Tuning – use of non-standard tune settings

Contaminants in reaction gas (O2, H2O, organics…)

Causes of Poor ICP-MS Precision

Worn pump tubing

Nebulizer condition and performance

Air leaks in transfer tubing

Sample line not grounded

Interface cones not “conditioned” with your sample matrix

Poor optimization – especially the neb. flow

Method setting – appropriate stabilization times?

Nebulizer choice for your samples

Wash-out (memory effects)

Incomplete mixing of online ISTD with sample

Causes of Poor Accuracy in ICP-MS

Old calibration standards (contamination, precipitation, evaporation…)

Standard preparation

Differences in stock solutions between suppliers

Use of AA grade standards

Choice of internal standard (sample may contain ISTD elements)

Insufficient stabilization time

Incomplete digestion – particles in solution

Wash-out (memory effects)

ICP-MS Sample Introduction System Tips

Do:

Check MassHunter performance report at beginning of day

Check/adjust the peri pump tubing

Check the blank reading

Rinse before samples & at the end of the run (multiple blanks at beginning of batch)

- Match rinse and sample acid composition

Clean the cones/extract lens as needed

Condition the cones after cleaning if running high matrix samples

Don't:

Assume system is still optimized

Overtighten the pressure adj. screw on the peripump

Use DI water as rinse water

Wait until the last minute before maintaining system (pump oil, peripump tubing, cone cleaning, lens 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 tube ID

- Chemical compatibility
  - Ensure tubing is resistant to the solvent being used

- Replace frequently
  - Precondition new tubing before analysis (wash, stretch …)
  - Using “old” tubing can lead to problems with precision and stability
  - Typical lifetime is ~5 days based on normal 8 hour working day
    - Detach from tube holder after use – allows tube to “relax”
    - Enable “post peripump rotation” to avoid flat spots

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

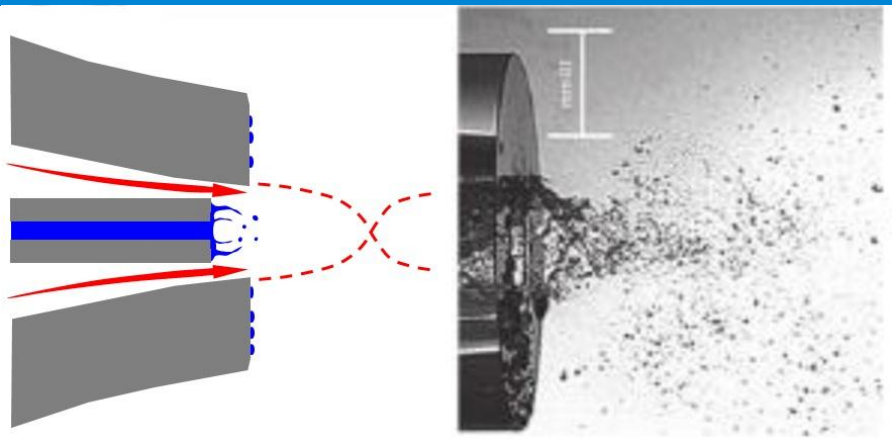
- Maintaining tubes – What to check?
  - Check 2 key things on pump tubing
  - Signal pulsation can be caused by:
    - Roundness of tube – should not be any “flat” spots
    - Tubing should still be elastic – replace if obviously stretched
  - <u>Don’t over tighten</u> – just need smooth and even sample flow

Peri Pump Tubing Tips

Symptoms:

- Peri pump tubing that looks/feel worn or has a strange color
  - IF IN DOUBT, CHANGE IT
- Erratic liquid flow
  - Check peripump clamp tension
- Bubbles in the liquid stream
  - Check sample introduction fittings, tubing and connectors for loose connections
- Spurting Nebulizer or disconnecting tubing segments
  - Plugging in the transfer line (check grounding connector).

Sample Introduction – Nebulizer Tips



Think "PREVENTION"

- Micro-flow nebulizers
- Zero tolerance to undissolved solids (particulates)
  - Plugging of annulus and/or capillary
  - Filter/Centrifuge/gravitational settling – sampler positioning
  - Use only lint-less 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

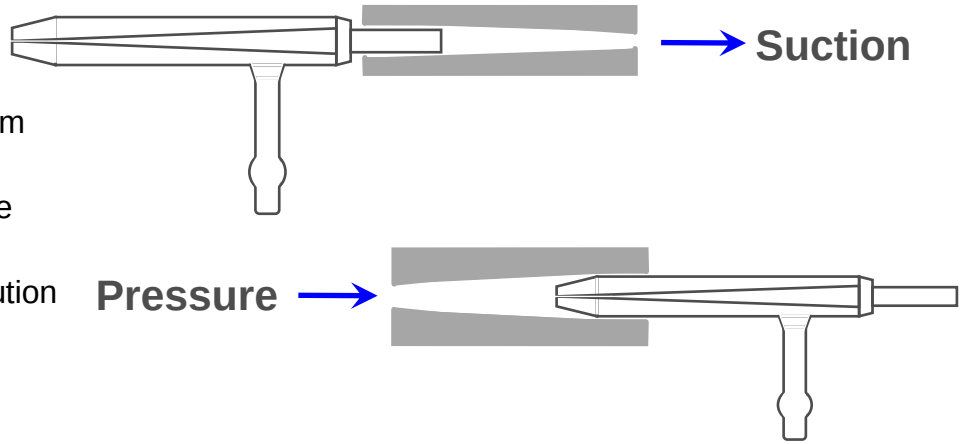
Never sonicate or attempt to clean with wire!

For normal cleaning:

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

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:

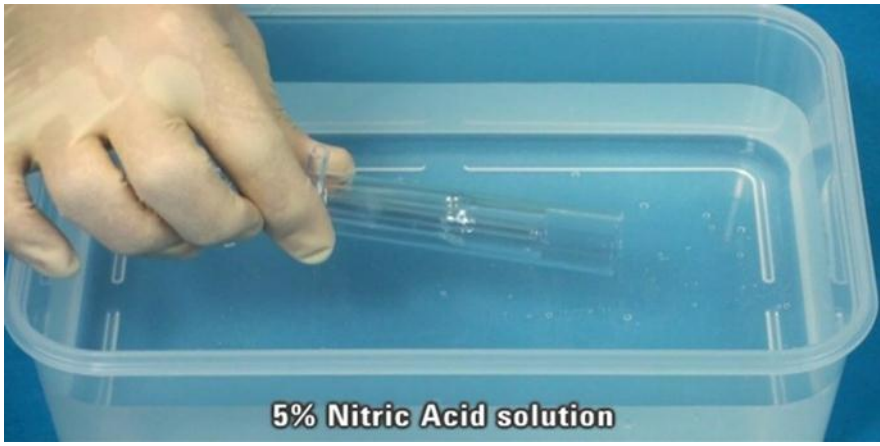
- Soak the nebulizer overnight in conc. nitric acid. Use a pipette to ensure there are no air bubbles in capillary. Rinse with pure water

Nebulizer cleaning tool
P/N G3266-80020

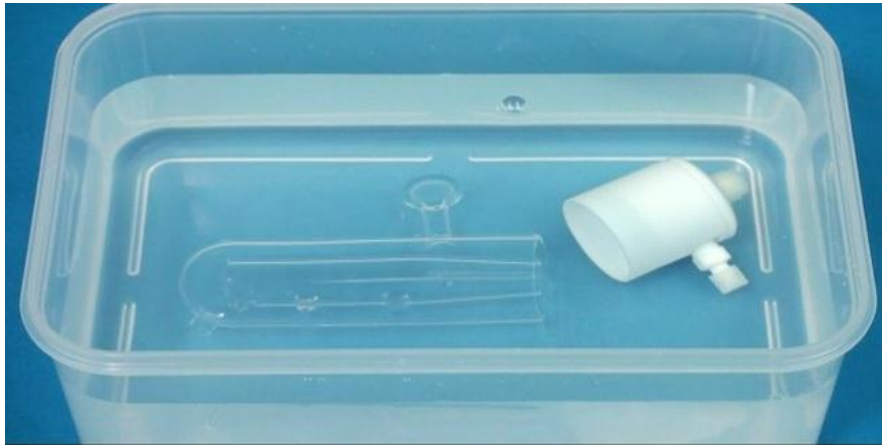
Cleaning the Spray Chamber and Torch

For routine cleaning, soak in 5% nitric acid for ~30 mins

- Rinse, allow to dry and refit
- Do not sonicate torch

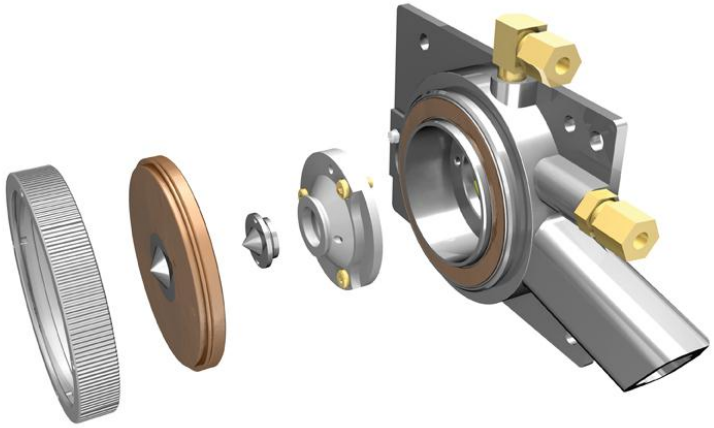


- Other acids may be useful (aqua-regia) for difficult to remove deposits



Interface Cone Choices

- Nickel cones are standard
  - Inexpensive
  - Good thermal and chemical resistance
  - Typically use 2-3/year (based on ~350 samples/day)



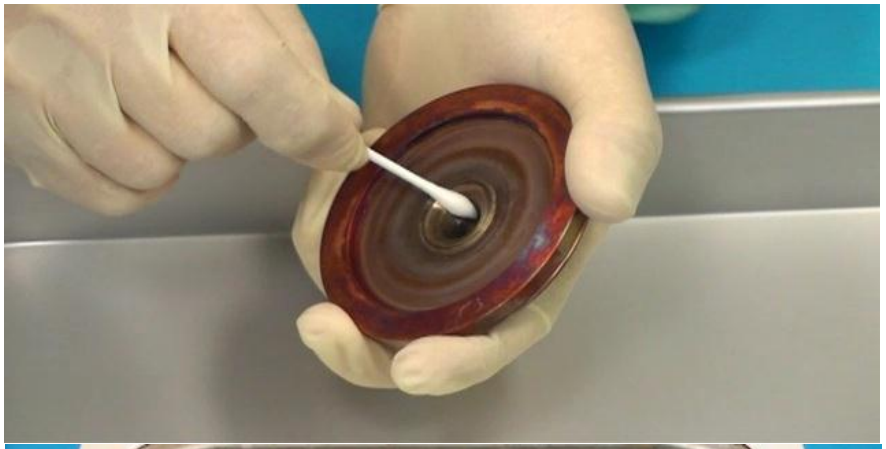
- Pt tipped cones are optional (trade-in program available for spent cones)
  - Essential for users wanting to analyze aggressive acids (esp. HF digests)
  - Also used when O_2/Ar option gas is used to analyze organic solvents
    - Typically use 1/year (based on ~350 samples/year)

Interface Cone Cleaning

For normal contamination:

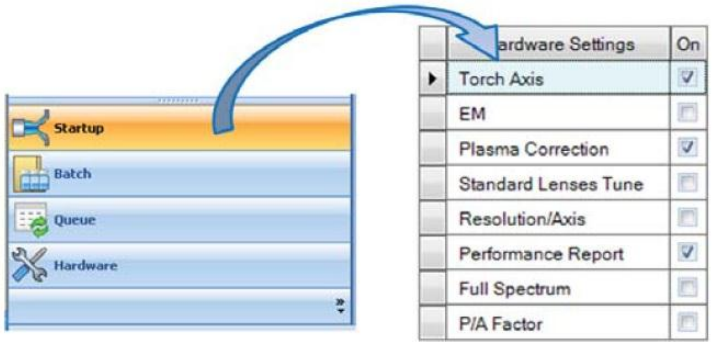
Just need a simple clean with pure water:

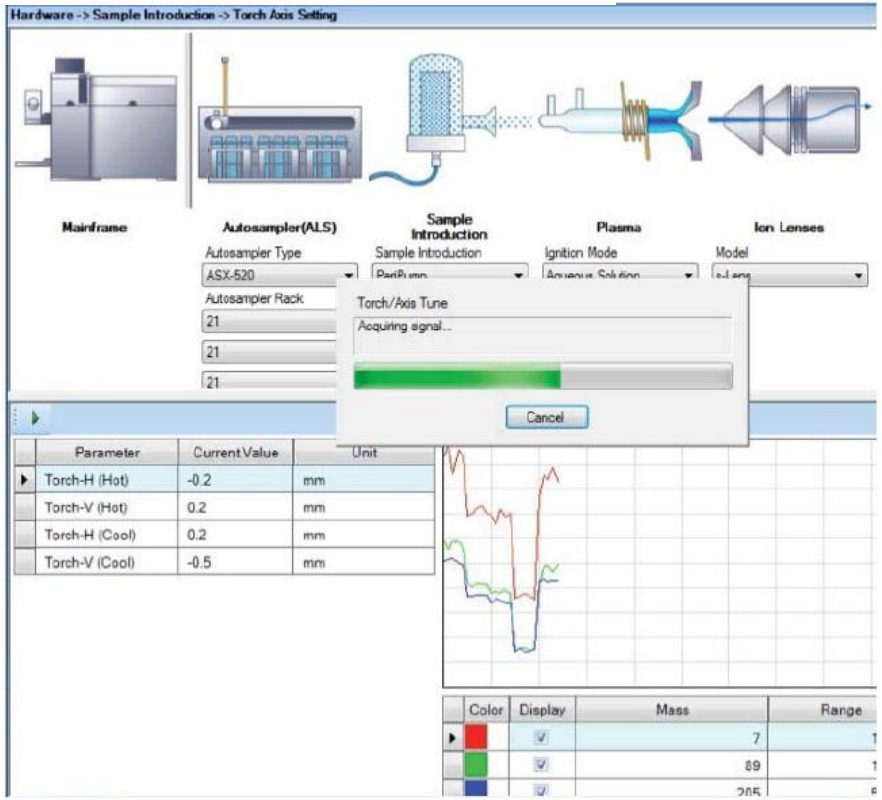
- Dip a cotton swab in pure water and clean both sides of the cone
- Rinse with pure water
- Ultrasonic clean the cones in pure water for more than 5 minutes
- If performance is still not satisfactory, may clean with a 2% Citranox solution
- May require abrasive polishing if deposition is stubborn (polishing paper or “Barkeepers Friend”
- Do not soak in dilute acid solutions for extended time
- Never use concentrated acids for cone cleaning



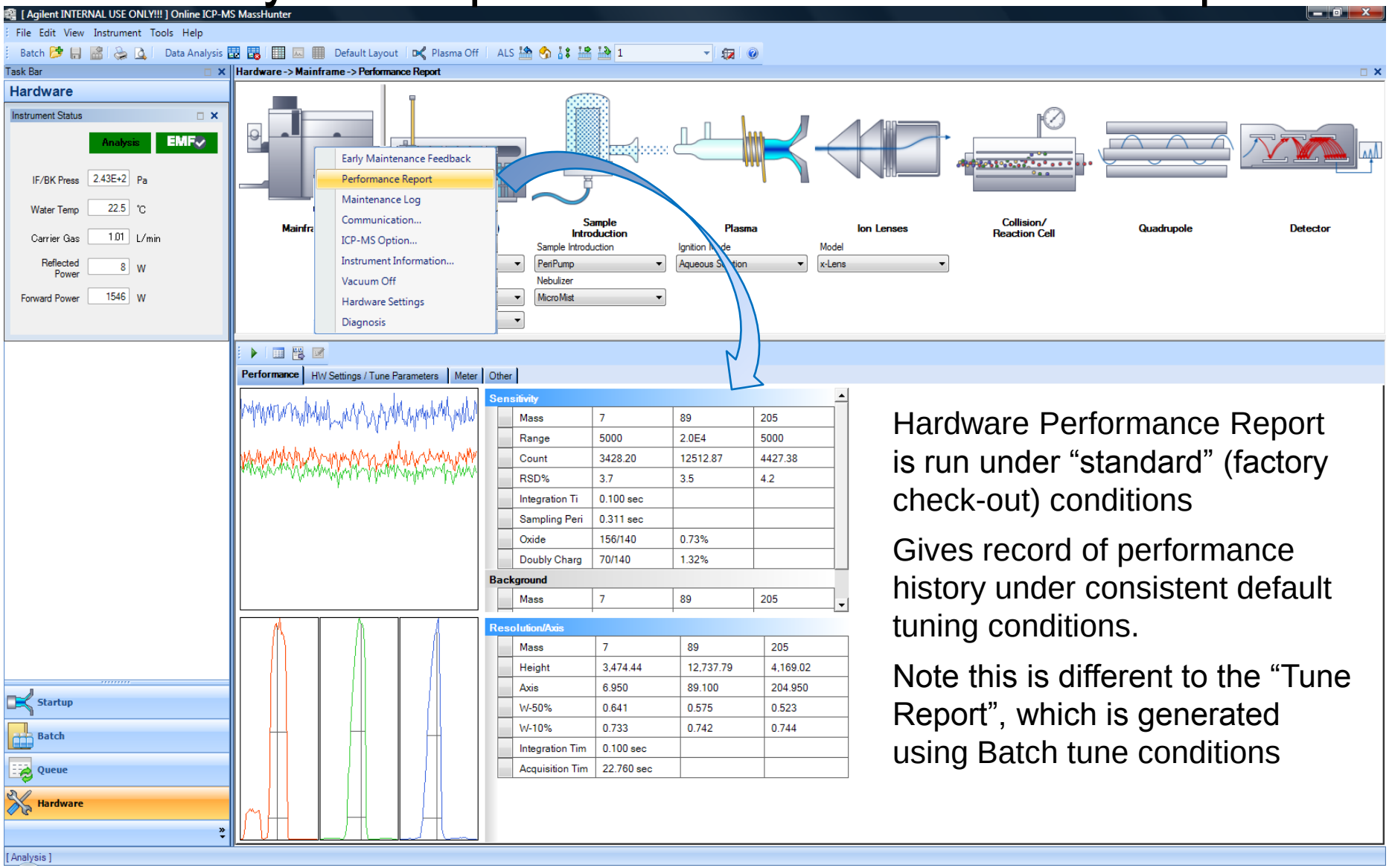
ICP-MS System Optimization – Startup Tune

- Startup provides a simple, user-configured schedule of system optimization and performance checks
- One-click 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 (great for troubleshooting)





ICP-MS System Optimization – Performance Report



Hardware Performance Report is run under “standard” (factory check-out) conditions

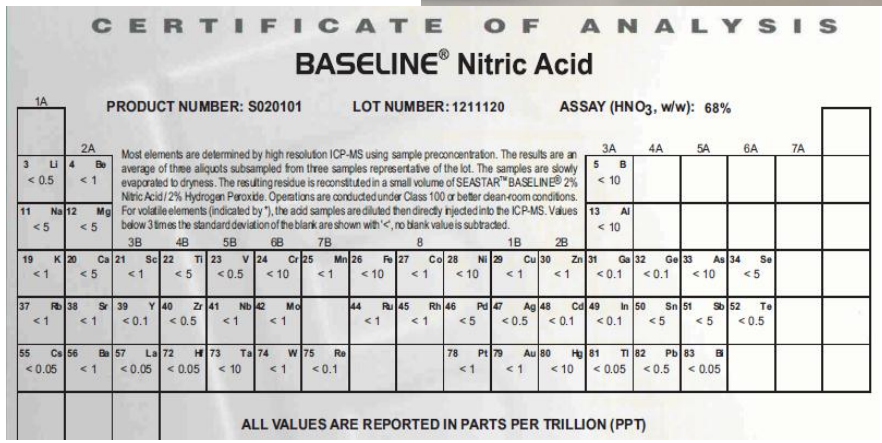
Gives record of performance history under consistent default tuning conditions.

Note this is different to the “Tune Report”, which is generated using Batch tune conditions

Tips to Reduce Contamination

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

- Always buy the best reagents necessary for your application – use high purity or trace metal grade
- 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)



CERTIFICATE OF ANALYSIS

BASELINE® Nitric Acid

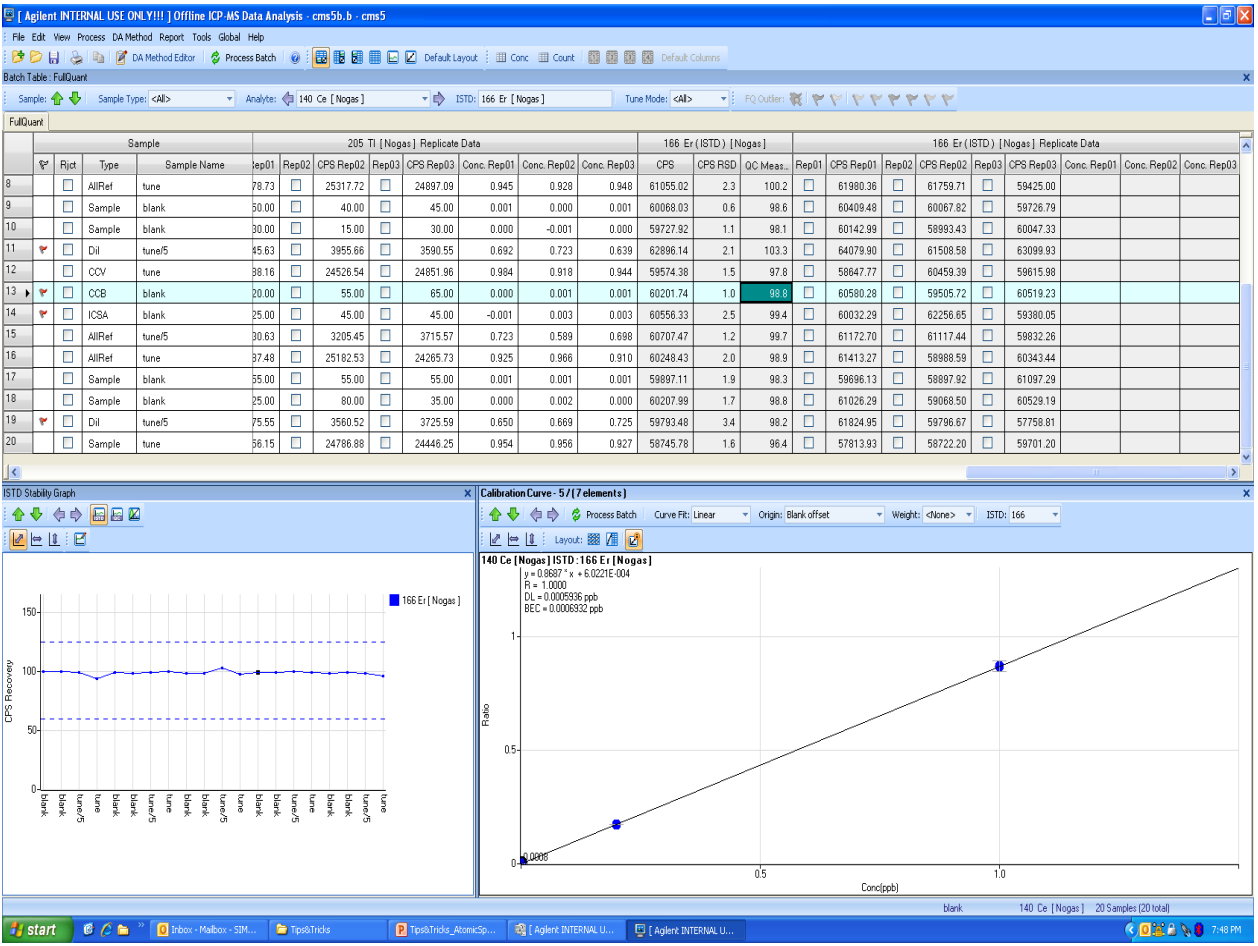
PRODUCT NUMBER: S020101 LOT NUMBER: 1211120 ASSAY (HNO_3, w/w): 68%

Most elements are determined by high resolution ICP-MS using sample preconcentration. The results are an average of three aliquots subsampled from three samples representative of the lot. The samples are slowly evaporated to dryness. The resulting residue is reconstituted in a small volume of SEASTAR™ BASELINE® 2% Nitric Acid / 2% Hydrogen Peroxide. Operations are conducted under Class 100 or better clean-room conditions. For volatile elements (indicated by *), the acid samples are diluted then directly injected into the ICP-MS. Values below 3 times the standard deviation of the blank are shown with '<', no blank value is subtracted.

1A	2A	3B	4B	5B	6B	7B	8	1B	2B	3A	4A	5A	6A	7A
3 Li < 0.5	4 Be < 1									5 B < 10				
11 Na < 5	12 Mg < 5									13 Al < 10				
19 K < 1	20 Ca < 5	21 Sc < 1	22 Ti < 5	23 V < 0.5	24 Cr < 10	25 Mn < 1	26 Fe < 10	27 Co < 1	28 Ni < 10	29 Cu < 1	30 Zn < 1	31 Ga < 0.1	32 Ge < 0.1	33 As < 10	34 Se < 5	
37 Rb < 1	38 Sr < 1	39 Y < 0.1	40 Zr < 0.5	41 Nb < 1	42 Mo < 1		44 Ru < 1	45 Rh < 1	46 Pd < 5	47 Ag < 0.5	48 Cd < 0.1	49 In < 0.1	50 Sn < 5	51 Sb < 5	52 Te < 0.5	
55 Cs < 0.05	56 Ba < 1	57 La < 0.05	72 Hf < 0.05	73 Ta < 10	74 W < 1	75 Re < 0.1			78 Pt < 1	79 Au < 1	80 Hg < 10	81 Tl < 0.05	82 Pb < 0.5	83 Bi < 0.05		

ALL VALUES ARE REPORTED IN PARTS PER TRILLION (PPT)

Tips to Improve Accuracy – Internal Std. Recovery



- Internal standard may not be matched to analyte (IP, Mass, chemistry…)
- Always include “quick-scan” in your acquisition
- ISTD may be present in sample
- Make sure alternate ISTDS are available in your ISTD mix
- Reprocess data post run using an alternate internal standard

Online Internal Standard Addition Considerations

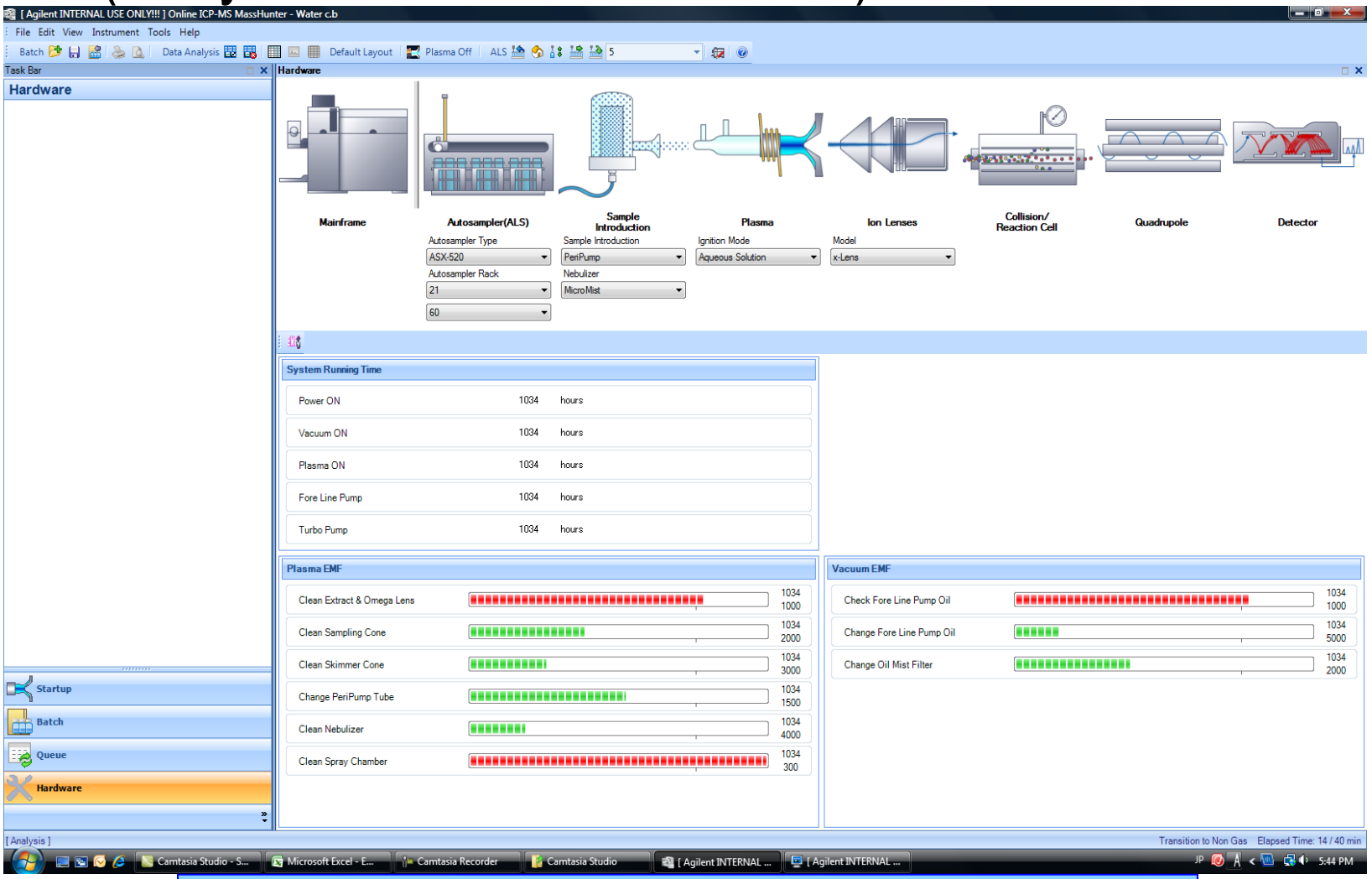
Most common problems:

1. Long ISTD stabilization times
   - Disruption of flow ratios (ISTD/Sample) Tubing from ISTD mixing tee to nebulizer is too narrow - use 0.5mm id tubing on ISTD and sample lines (if not using ISIS)
     - Restrictions downstream from mixing Tee change flows during high uptake flow.
       - Use “monitor mass” function to identify problem before acquisition begins
2. Sporadic high RSD on ISTD replicates
   - Inconsistent mixing (ISTD/Sample)
     - Add 0.1% Triton X-100 to internal standard mix to enable mixing of ISTD with sample
3. ISTDs increase with higher concentration calibration points
   - Improperly grounded flow path
     - Insure grounding Tee is correctly grounded

ICP-MS – Potential Autosampler Issues

- 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
    - Dust can introduce contamination
    - Sample evaporation may occur during long unattended runs
    - Sediment in the sample may settle out
  - Ensure transfer line to ICP-MS is clean
    - Indicated if Th, Tl or Sb carryover is a problem
    - May require ~ half hour cleaning with SC1 and SC2 (plasma off, nebulizer removed from spray-chamber)
      - SC1 = 1:1:5 ammonium hydroxide: H2O2:DI water
      - SC2 = 1:1:5 HCl:H2O2:DI water

EMF (Early Maintenance Feedback)

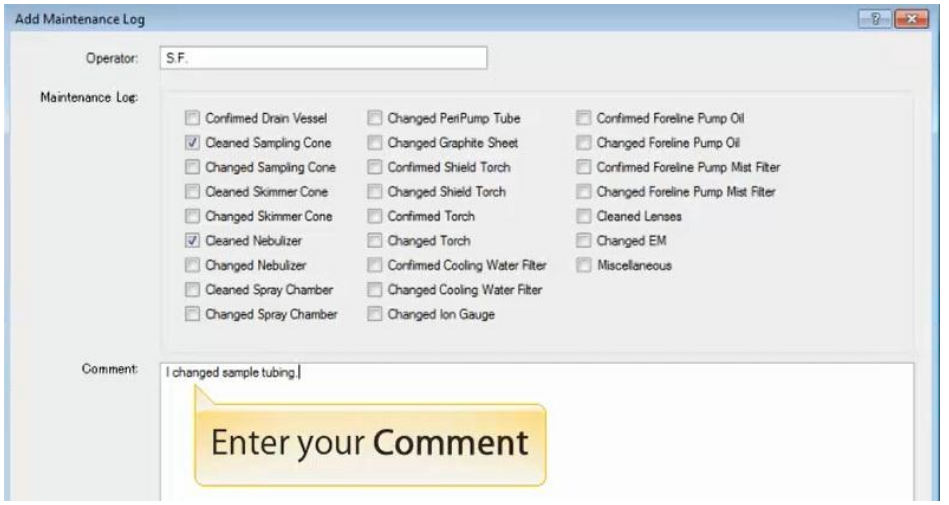


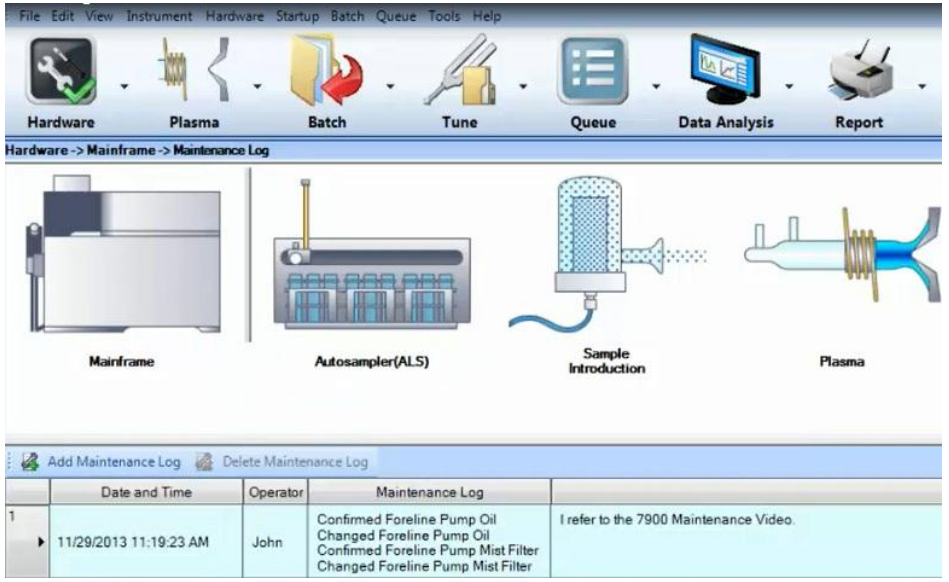
EMF window shows usage of various components and predicts when to perform maintenance (user definable)

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





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

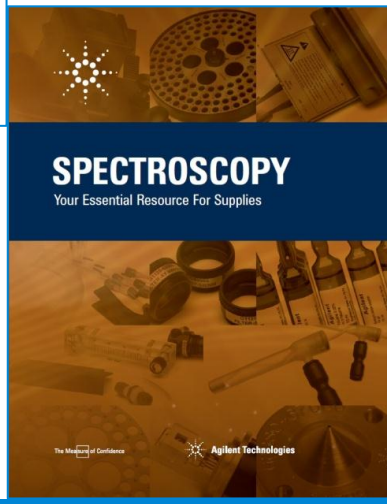
Atomic Spectroscopy Application Notes

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