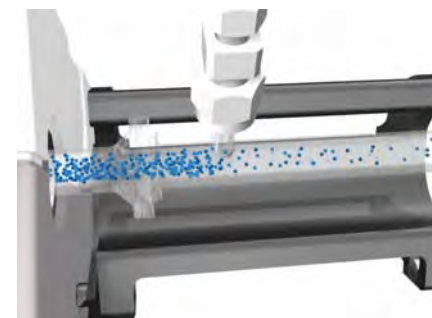




Agilent ICP-MS Maintenance Tips & Tricks



**ASTS 3 Meeting
Houston, TX
June 27, 2013**



Sample Introduction System Problems

Probably 90% of problems with ICP-MS relate to sample introduction system hardware

- Carryover/washout
- Poor precision
- Poor linearity
- ISTD instability
- Plasma ignition problems



Sample Introduction System Problems

Things you can do:

- Make sure all fittings are “true” and no air bubbles in sample stream
- Make sure peri-pump tubing is in good condition
 - Also not too tight, not too loose
 - Old, worn or a “funny” color will probably mean it’s become active
 - IF IN DOUBT, CHANGE IT
- Make sure the nebulizer is clean and functioning correctly
 - Sometimes this will need an alcohol clean (or detergent) to remove built up oils or waxes
 - particularly true for Babington, but bear in mind for other nebs
- Make use of appropriate chemistry – HCl can solve a lot of problems*
- Check all gas fittings, tubing and connectors – damaged fittings cause leaks and gas contamination
- Limit the maximum concentration exposed to the system – keep it appropriate for the analysis
- Keep your instrument clean – no need to “go mad” just be appropriate for your lab and sample types



*Can cause interference problems for non-ORS equipped instruments

“Active Cleaning”

When “sticky” elements (Tl, Sb, Th, Ag...) are taking too long to rinse out, replace autosampler probe and all other tubing leading up to the nebulizer.

Or to clean, with the plasma off, remove Teflon line from back of nebulizer and hang in a beaker. With all of the sample introduction lines hooked up, move the autosampler probe into 50 ml vials of each of the following solutions:

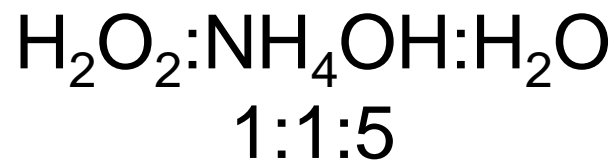
- **~30% NH₄OH (30 min with peri at 0.3 rps)**
- **~30% HCl (30 min with peri at 0.3 rps)**
- **~30% HNO₃ (30 min with peri at 0.3 rps)**
- **De-ionized (a few minutes at 0.3 rps)**

Let each of these solutions flow through the whole sample flow path. You might have to change the Tygon tubing after doing this.

Make sure you empty the 30%NH4OH from beaker before going to acid solutions.

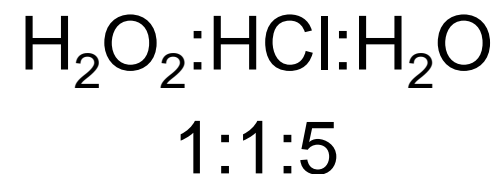
Additional Cleaning Solutions (used in the Semiconductor industry)

SC-1



Use one or both solutions, either as a rinse
or for periodic cleaning

SC-2



Stabilization & Washout Matrix for Hg – a Case Study

Stabilization Matrix

Hg (and many other elements) is MUCH more stable in HCl than in HNO₃, so 0.5% HCl is added to all samples, blanks, standards

Washout Matrix

HCl in the rinse also improves washout of

Even greater washout efficiency can be obtained by a 30s
rinse cycle; e.g. an optimum rinse program might be:

- 5s water (probe tip wash) →
- 15s (or pre-emptive rinse) NH₄OH/EDTA/H₂O₂/Triton-X →
- 15s 5% each HNO₃/HCl (and trace HF) →
- 10s acid mix comparable to acid composition of samples (use Intelligent rinse to monitor the washout)
 - Note: The last step is important, as the spray chamber also takes several seconds to re-stabilize (evaporation rates, vapour pressure, etc)

Basic rinse stock solution

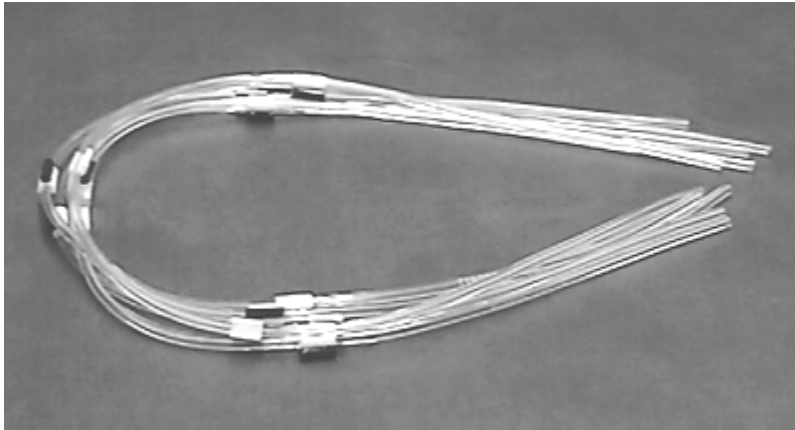
- 2.5g EDTA (as the acid NOT salt)
- 0.2g Triton X-100
- 15g NH₄OH
- 20g H₂O₂ (optional)

Make up to 250ml and store in fridge

Dilute 10x for working solution



Pump - Tubing



Peristaltic Pump
Tubing for Sample
Introduction

- (Tygon Tubing, 1.02mm id, 12/pk)
- P/N : G1833-65569

Peristaltic Pump
Tubing for ISTD

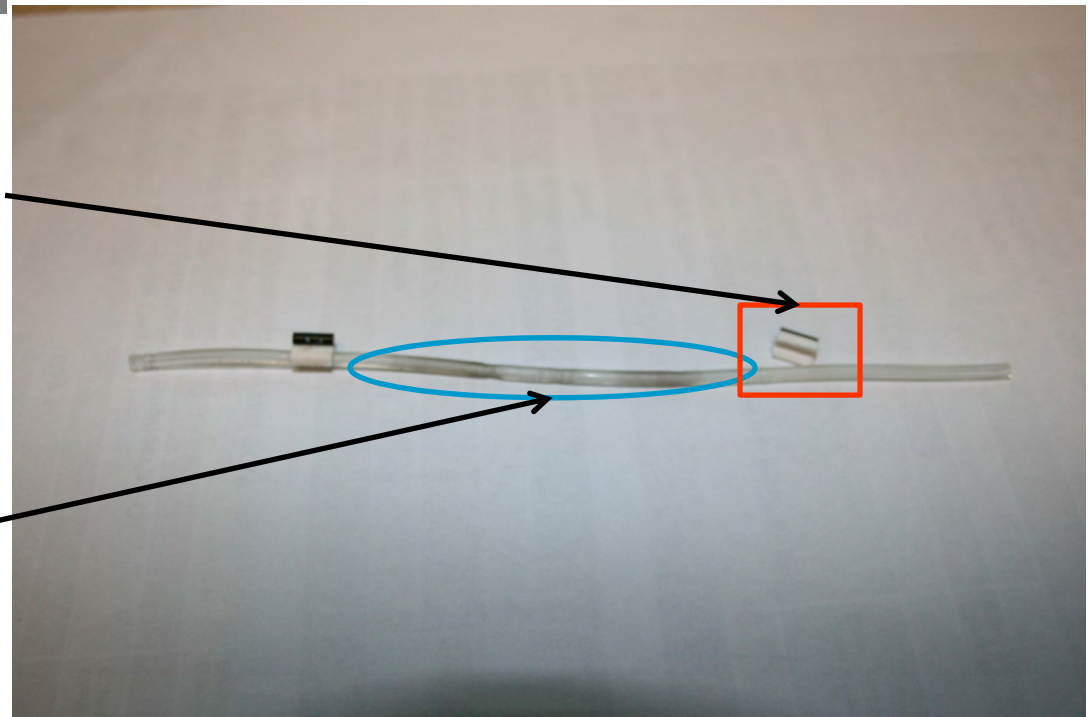
- (Tygon Tubing, 0.25mm id, 12/pk)
- P/N : G3280-67047

Peristaltic Pump
Tubing for Drainage

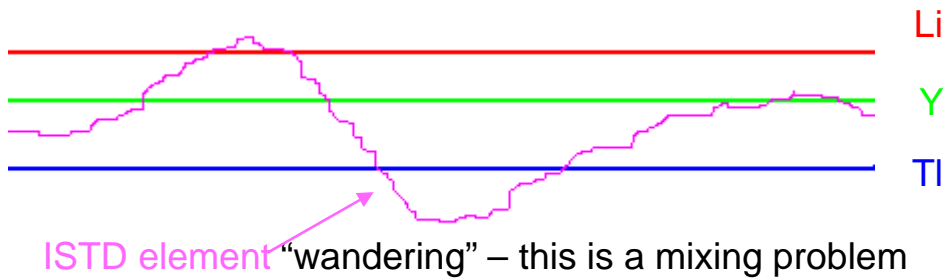
- (PharMed Tubing, 1.52mm id, 12/pk)
- P/N : G1833-65570

The stop has ripped off, this can cause an overnight run to fail. The Agilent part has an additional QC check to minimize this.

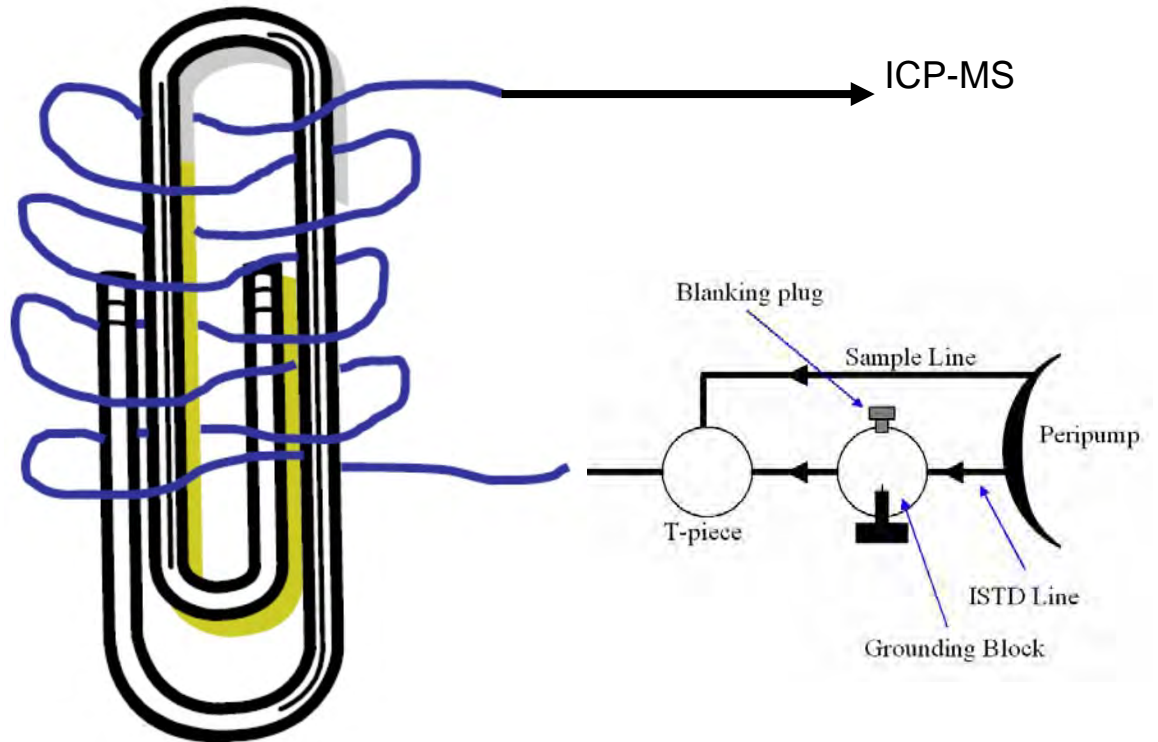
Normal wear & tear (2~5 days), needs replacing



Internal Standard Mixing Problems

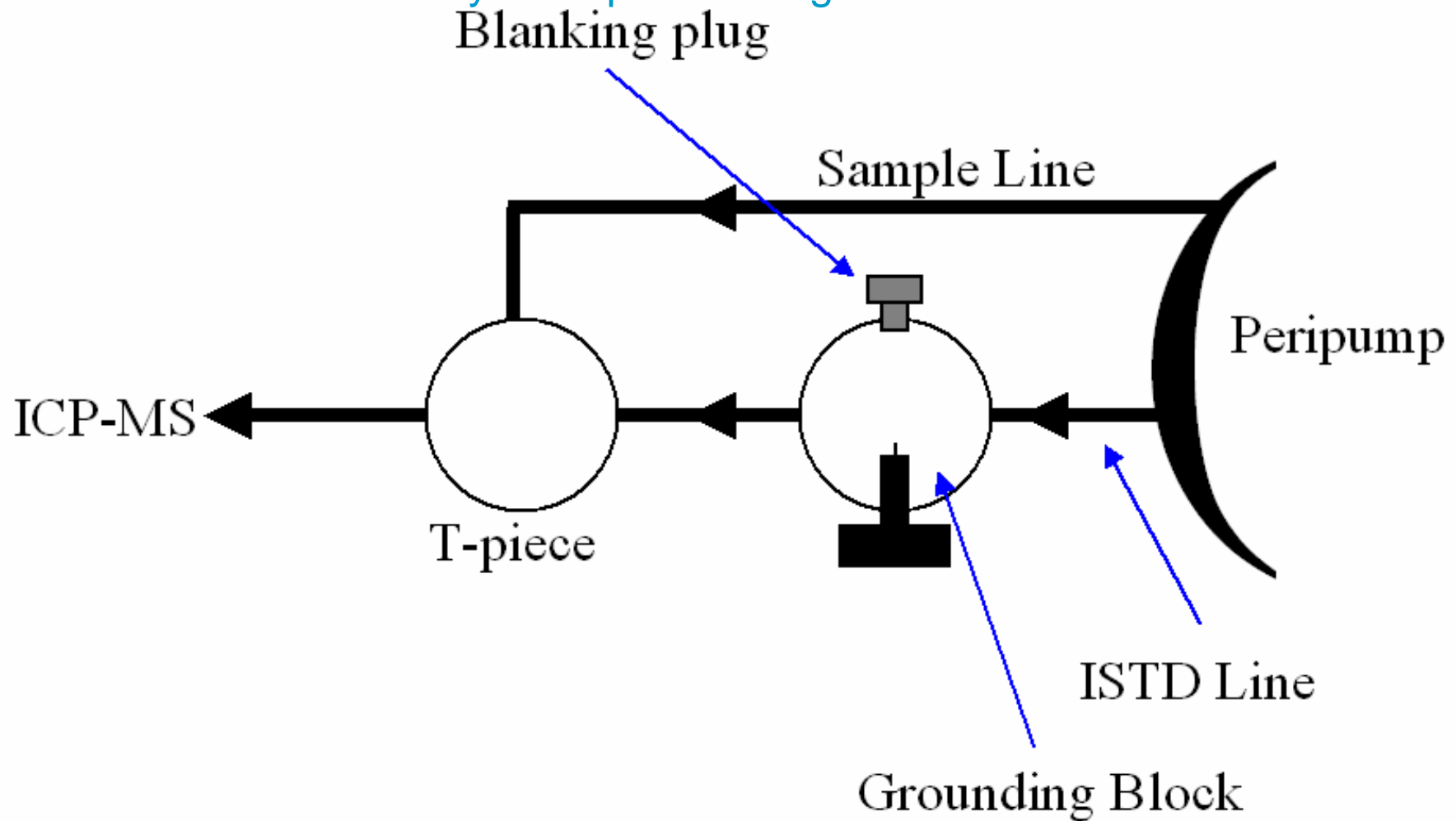


Make a "serpentine" mixer by threading uptake tube in and out of a paperclip or similar item (forming a figure of 8 shape) – this improves turbulence & mixing



Carryover problem can be related to Pt wire!

Some elements (e.g. Ag) can electroplate on the Pt grounding wire
This can be overcome by a simple reconfiguration of the ISTD line



Pump damaged by pump tubing



G1833 - 65577 – Teflon Tape

Nebulizers



Babington Nebulizer (G1820-60453)

- The Babington nebulizer can be used for introducing samples with high matrix and high TDS (total dissolved salts) level samples.



MicroMist Nebulizer (G3266-65003)

- The MicroMist nebulizer can be used for introducing samples with high matrix and high TDS (total dissolved salts) level samples.



Agilent Micro Flow Nebulizer

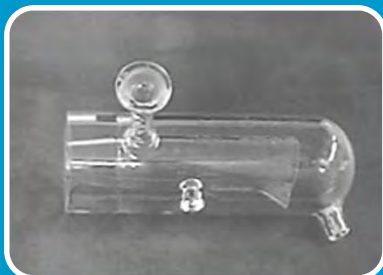
- This is an all fluoropolymer, low-flow, reliable nebulizer. This nebulizer is used for many sample types including low volume samples, aggressive chemicals and solvent, organic solvent, etc.



Problems

- The main problem with nebulisers is sample contamination or physical damage, contamination can normally be cleaned by the customer.
- Replacement 1 every 1~2 years.

Spray Chamber



Spray Chamber (Quartz)-G1820-65337

- This quartz spray chamber is the standard spray chamber for the Agilent 7700. This spray chamber should not be used with hydrofluoric acid.



Spray Chamber (Polypropylene, Sapphire Tube)-G1833-65463

- This polypropylene spray chamber is the standard spray chamber for the inert sample introduction kit, and is used for introducing hydrofluoric acid.



Problems

- The main problem with spray chambers is sample contamination or physical damage, contamination can normally be cleaned by the customer.
- Replacement 1 every 1~2 years.
- 3rd party suppliers do not have Agilent drawings, different manufacturing techniques, tolerances can cause catastrophic failures this can result in instrument / analytical problems. (Plasma ignition, signal stability, elemental contamination..)

Plasma Torch



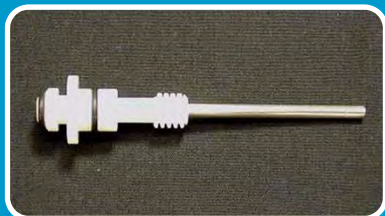
Torch (Quartz)

- The torch (2.5mm id) is the standard torch for the Agilent 7500. The torch (taper type) is used for introducing organic solvent, etc. These torches cannot be used for introducing hydrofluoric acid.



Inert Torch (without Injector, Quartz)

- This torch is used with the platinum or sapphire injector for the inert torch assembly. The inert torch assembly is used for introducing hydrofluoric acid.



Injector for Inert Torch (Platinum or Sapphire)

- This injector is used with the inert torch for the inert torch assembly. The inert torch assembly is used for introducing hydrofluoric acid.



Problems

- The main problem with plasma torches is physical damage when customers handle them or during multiple bad ignitions of the plasma.
- Replacement 1 every 1~2 years.
- 3rd party suppliers do not have Agilent drawings, different manufacturing techniques, tolerances can cause catastrophic failures this can result in instrument / analytical problems. (Plasma ignition, signal stability, elemental contamination..)

Discoloration of the Torch



Figure 1. Heavy brown deposit on inside of plasma torch after hundreds of hours use. Argon gas was delivered through the standard plasticized PVC tubing.



Figure 2. Plasma torch after at least 1,000 hours of use. Clear except for a slight "feather" of brown at the coolant gas entry tube, and a general darkening at the open end of the torch. Argon gas was delivered through the plasticizer-free polyurethane tubing.

How to Remove the Brown Deposits

Masahiko Endo

ICP-MS Product Marketing, Agilent Technologies, Tokyo Analytical Division

There are a number of methods for cleaning the brown deposit from the torch. One requires soaking the torch in a 10 - 30% bleach solution (hypochlorous acid $\text{Ca}(\text{OCl})_2$) for 1 or 2 nights. If the deposit is heavy, it may need more time or a higher concentration of cleaning solution.

Another method uses a 5% TMAH solution. Heat the required volume of deionized water to about 50°C , add concentrated TMAH (25%) and submerge the torch for about 48 hours. Rinse and the brown deposit should be removed. Remember to wear gloves and safety glasses when handling TMAH.

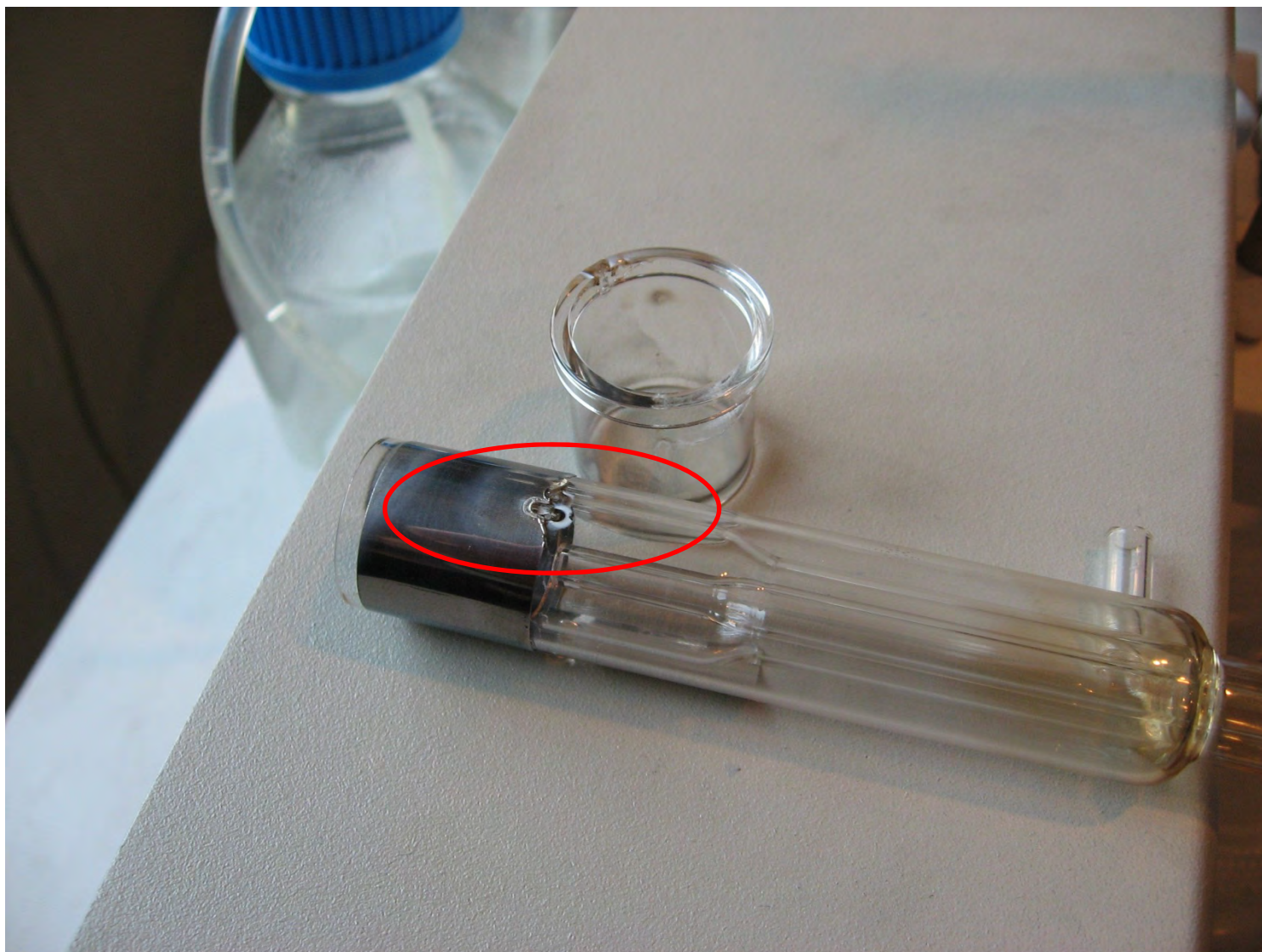
An alternative method is to boil the torch in a 10% solution of Decon90 for 30 minutes.

Finally, a dirty torch can be cleaned by simply placing it in a muffle furnace at $\sim 500^\circ\text{C}$ for about 15 minutes.

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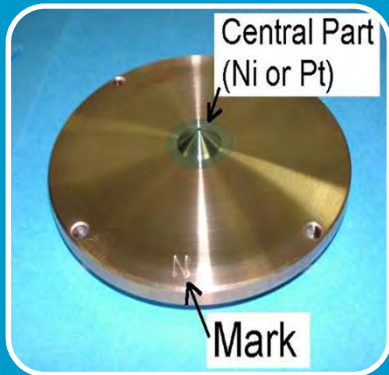


Damaged Shield / Bonnet / Torch – This was caused by poor installation
(The torch was still wet from earlier cleaning)



Sampling Cone

1mm Orifice Nickel or Platinum Centers



- Nickel
 - Physically stronger
 - Tip runs hotter – less sample deposition
- Platinum
 - Chemically stronger
 - Used for higher Acid Conc.
 - 1.0mm diameter
 - Higher cost

Problems



- The main problem with sample cones is, physical damage to the orifice when customers clean them (too aggressive). Sample matrix (acid strength & type) and number of samples analysed each day.
- Nickel - Replacement 3 or 5 each year. (based on 250~350 environmental samples each day)
- Platinum - Replacement 1 or 2 each year. (based on 20~50 semiconductor samples each day)
- 3rd party suppliers do not have Agilent drawings, different manufacturing techniques, tolerances can cause catastrophic failures this can result in instrument / analytical problems. (Signal stability, elemental contamination)

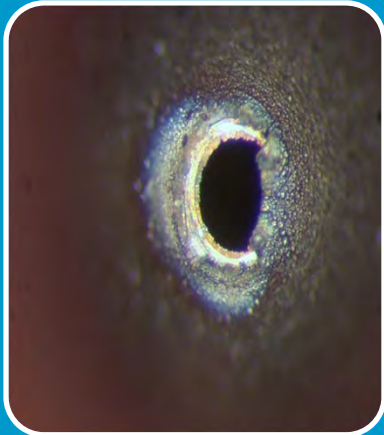


Skimmer Cone



0.4mm Orifice Nickel or Platinum

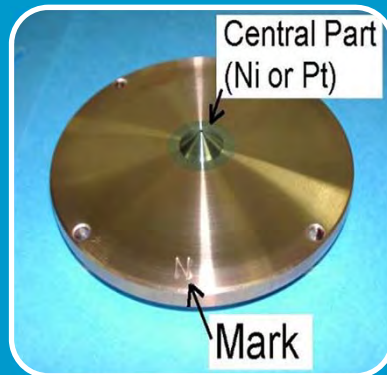
- The Nickel skimmer cone is used as the standard interface of the Agilent 7700x and s models. This skimmer cone is relatively robust to samples containing high concentrations of matrix and salts, and is suitable for a wide variety of samples. It is usually used with the Nickel sampling cone.
- The Platinum skimmer cone is usually used for introduction of hydrofluoric acid and other reagents for the 7700s and x. It is usually used with the Platinum sampling cone.



Problems

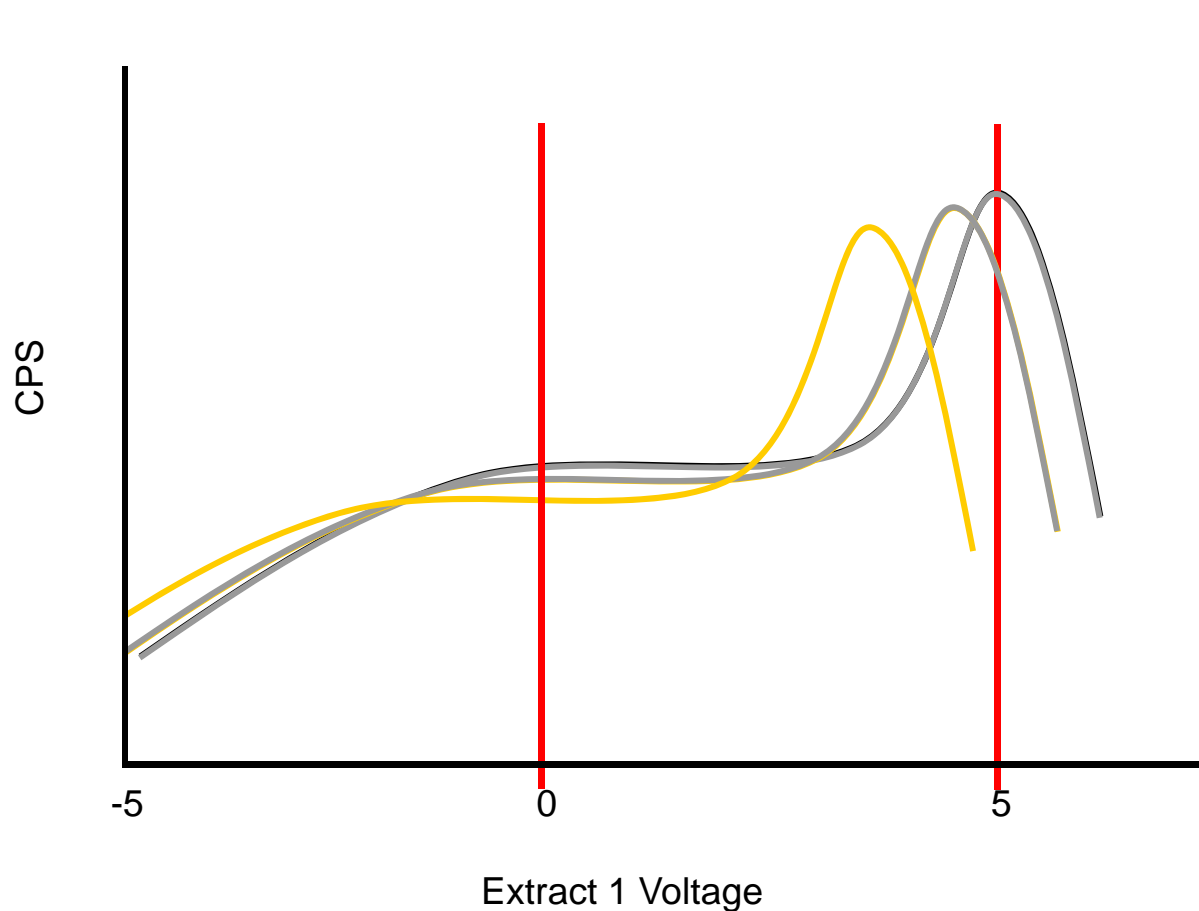
- The main problem with sample cones is, physical damage to the orifice when customers clean them (too aggressive). Sample matrix (acid strength & type) and number of samples analyzed each day.
- Nickel - Replacement 2 or 4 each year. (based on 250~350 environmental samples each day)
- Platinum - Replacement 1 or 2 each year. (based on 20~50 semiconductor samples each day)
- 3rd party suppliers do not have Agilent drawings, different manufacturing techniques, tolerances can cause catastrophic failures this can result in instrument / analytical problems. (Signal stability, elemental contamination)

Cleaning



- Sonicate in 2 % Nitric Acid or Citrinox
- Rinse well with Deionized water
- Dry with nitrogen before installing

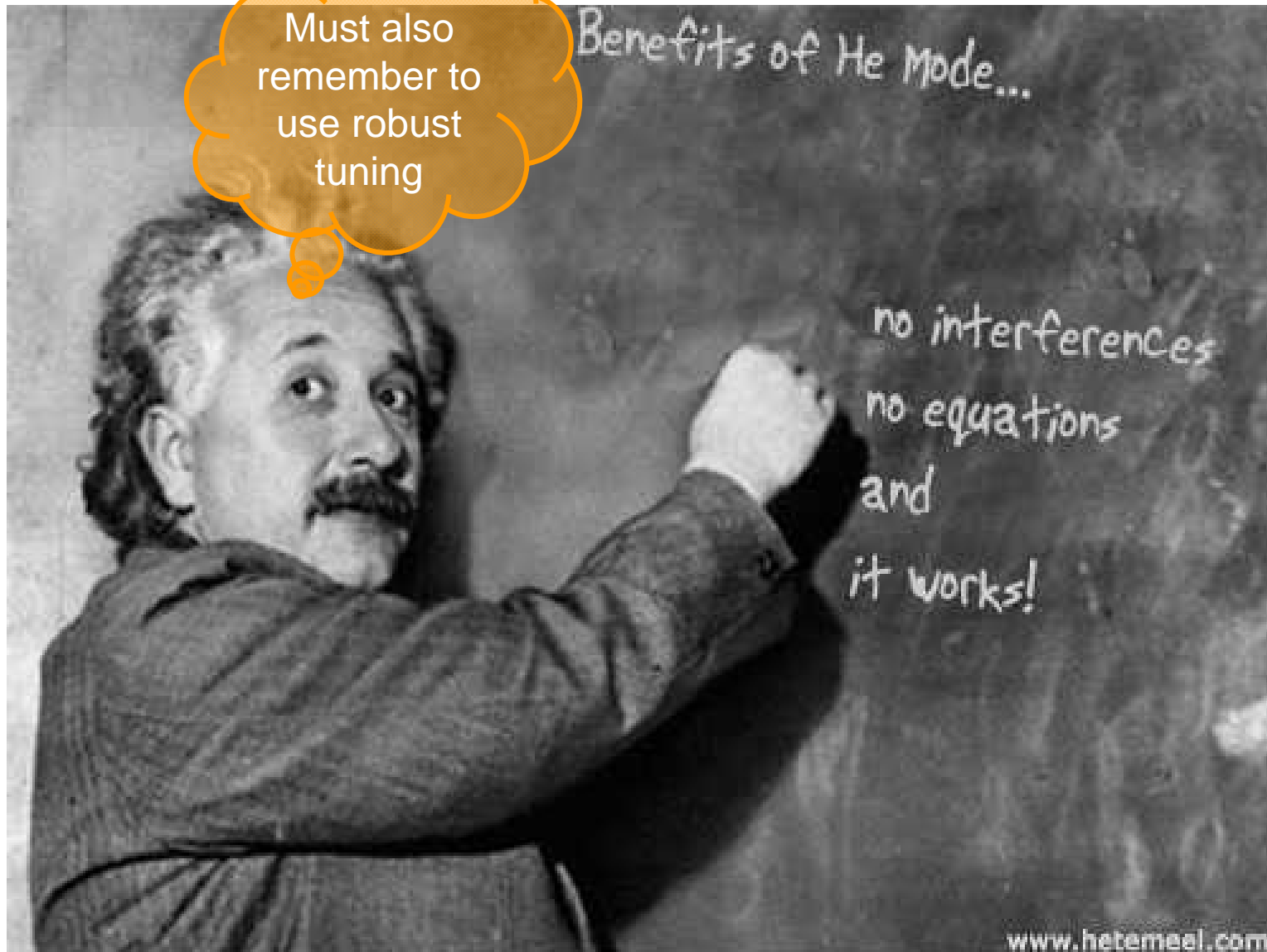
Why should the extract 1 lens be set to 0V?



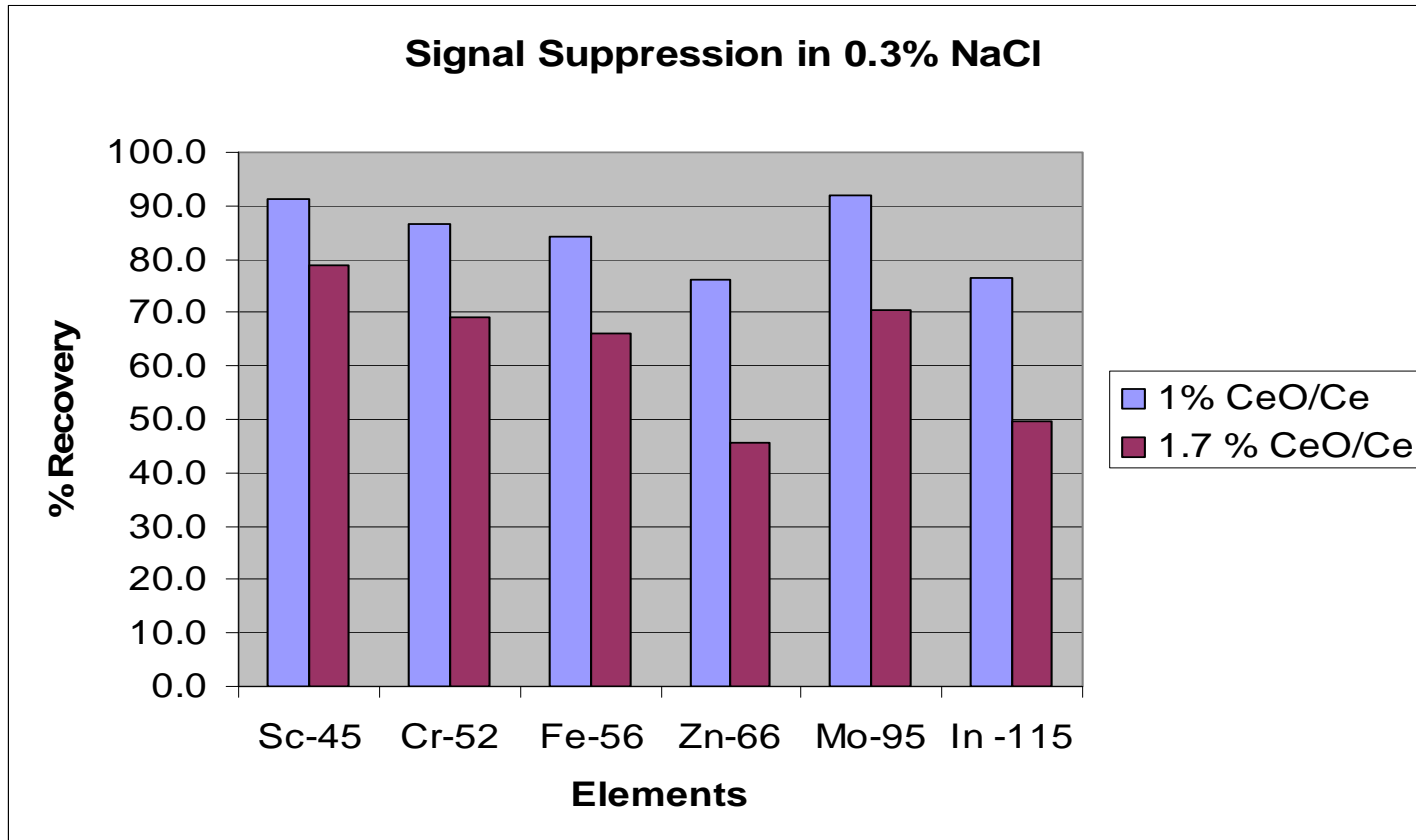
Clean lens has sensitivity profile similar to Black Line
Sensitivity optimized at about 5-6V

By fixing the lens at 0V there is a wide sample sensitivity optimization but resolution is much lower and is not recommended for optimized peak at high trap and large gas drift appears to be severe

He Mode – Quite Brilliant



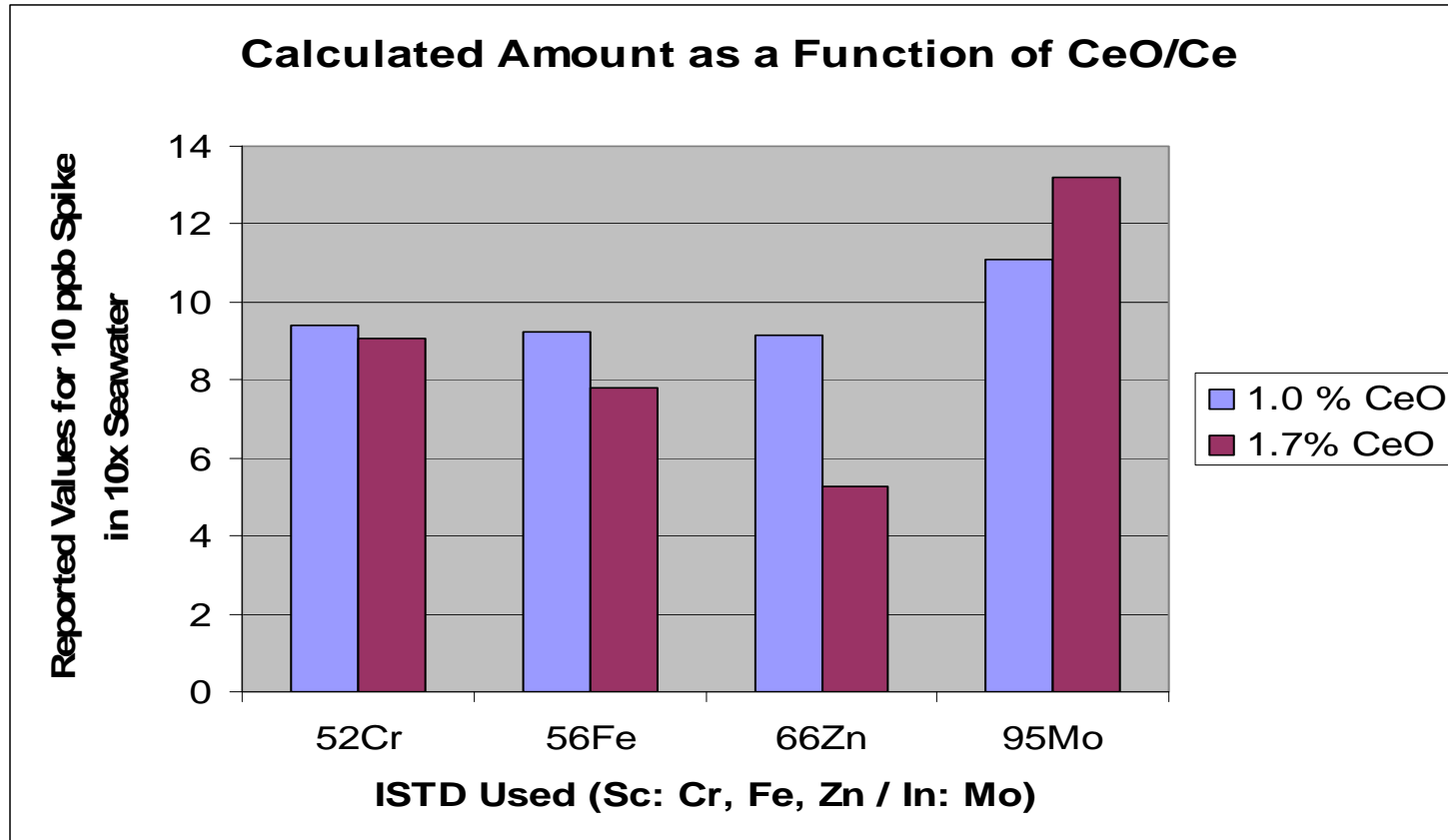
Why should the CeO% be optimized?



***When compared to 1% HNO₃, there is significantly more ionization and signal suppression when the CeO/Ce ratio is higher**

Internal Standardization is not Enough to Overcome Effects of Ionization Suppression When CeO is High

-Calibration was prepared in 1% HNO₃



**All determinations under 1%CeO conditions are within 10% of actual.
Under 1.7 CeO settings results are either too high or too low.**

Oxides in Gas mode

General rule:

The CeO/Ce ratio % in **Helium** mode should be AT THE WORST CASE the same as the CeO% in NoGas mode and usually around a factor of 2 lower

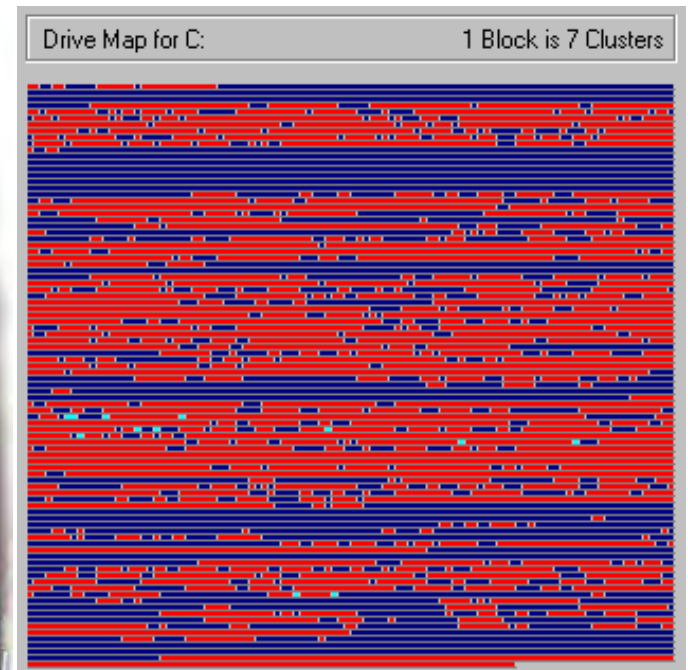
If the CeO% is higher in **He** mode, then the He is contaminated, the lines are not purged or there is a leak or some other problem

The Ce⁺⁺ might be slightly higher in **He** mode (the Ce⁺⁺ ion has a smaller cross-section than the Ce⁺ ion so is not affected by KED)

CeO% may be higher in **Hydrogen** mode but the Ce⁺⁺ is usually lower (the 2⁺ ion induces a greater dipole in the H-H bond increasing its reactivity). However, if the CeO% is much higher (e.g. >2x this indicates gas contamination).

Also remember that the **H₂** lines MUST be high quality Stainless Steel as coatings can contaminate the cell and OP and copper is porous

Don't forget your WorkStation !!!



Fragmentation occurs during the normal operation of windows this can eventually slow disc access down

- Use a defragmentation utility to reorder files and remove fragmentation

Thank You for staying awake!

