Guidelines for Trouble Shooting and Maintenance of ICP-OES Systems

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-Note: Original Agilent document modified by Isabelle Rheault BSc, Chemistry department,UQAM, april 2020

Causes of Poor ICP-OES Sensitivity

Sample introduction system
- Worn pump tubing
- Blocked nebulizer
- Blocked injector in torch

Optimization
- Poor optimization – especially the neb. flow
- Method setting – using right wavelength?
- Wrong tubing type
- Interferences

Standard (& sample) preparation
- High blank level
- Standards prepared correctly?
- Samples prepared correctly? – ionization suppressant

- Optics purge – UV wavelengths only
- Spray chamber type

Causes of Poor ICP-OES Precision

Sample introduction system

- Worn pump tubing
- Beading in spray chamber
- Nebulizer condition and performance
- Air leaks in transfer tubing

Optimization

- Torch alignment
- Poor optimization – especially the neb. flow
- Nebulizer choice for your samples

Standard (& sample) preparation

- Wash-out (memory effects)

Causes of Poor Accuracy in ICP-OES

Sample introduction system
- Worn pump tubing
- Blockage in nebulizer and/or torch

Optimization
- Wrong wavelength choice - 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-OES Sample Introduction System Tips

Do:	Don't:
Check optimization each analysis	Assume system is still optimized
Check/monitor the nebulizer uptake	Assume nebulizer flow rate is the same
Check/adjust the peri pump tubing	Overtighten the pressure adj. screw
Check the blank reading	Use a simple water blank
Rinse between samples & at the end of the run - Rinse should match sample matrix	
Clean the torch/nebulizer regularly - Inspect condition of the nebulizer tip	Wait until you have blockage before cleaning
Follow analytical recommendations in "cookbook"	

Peri Pump Tubing Tips

- 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

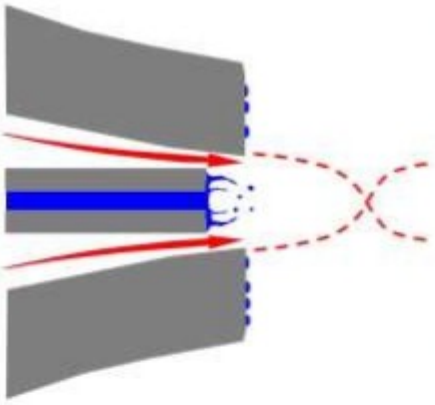
- Replace frequently
  - Using “old” tubing can lead to problems with precision and stability
  - Typical lifetime is 1-2 weeks based on normal 8 hour working day
    - Detach from tube holder after use – allows tube to “relax”

- 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

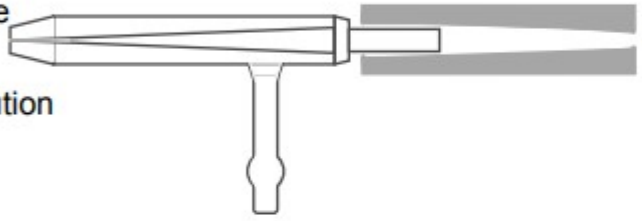
Cleaning the Nebulizer

Never sonicate or attempt to clean with wire!



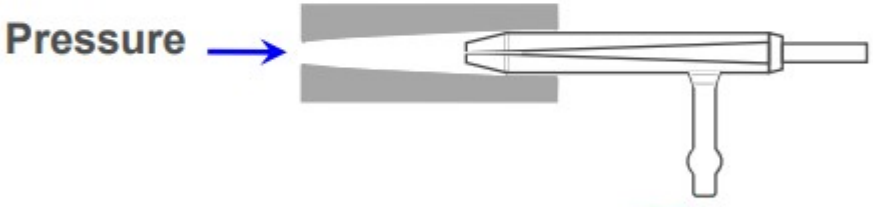
For normal cleaning:

- 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

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
Image provided by Meinhard Glassblowing Products

Performance Characteristics of Common Nebulizers

Nebulizer Type	Aerosol Efficiency	Achieved Precision	Dissolved Solids Tolerance	HF Resistance	Organics Compatibility	Self Aspirates	Ideal Sample Type
OneNeb	Excellent	Excellent	Good (max. 150 um particles)	Excellent	Excellent	No	Handles most samples
SeaSpray concentric	Good	Good	Medium (max. 75 um particles)	Poor	Good	Yes	Environmental, soil & food digests
Conikal concentric	Good	Excellent	Poor to Medium (max. 75 um particles)	Poor	Excellent	Yes	Clean oil samples and organic solvents
V-groove	Medium	Medium	Excellent	Excellent	Medium	No	HF digests, fusions, high TDS or used oil samples

Refer Access Agilent article titled "Tips on choosing – and using – the best nebulizer for your ICP-OES"
http://www.chem.agilent.com/en-US/Newsletters/accessagilent/2013/jul/pages/nebulizer.aspx?cid=7652

Universal inert OneNeb

Routine usage concentric type

High Solids capable (V-Groove)

Performance Characteristics of ICP Spray Chambers

Spray Chamber Type	Aerosol Efficiency	Achieved Precision	Dissolved Solids Tolerance	HF Resistance	Organics Compatibility	Ideal Sample Type
Single pass glass cyclonic	Excellent	Good	Poor to Medium	Poor	Good	Environmental, food digests
Double pass glass cyclonic	Good	Excellent	Good	Poor	Good	Soil digests, clean oil samples and organic solvents
Inert double pass cyclonic	Good	Excellent	Excellent	Excellent	Excellent	HF digests, fusions, high TDS or used oil samples
Cooled spray chamber	Good	Good	Poor to Medium	Poor	Excellent	Highly volatile organic solvents e.g. gasolene

Single pass glass cyclonic

Double pass glass cyclonic

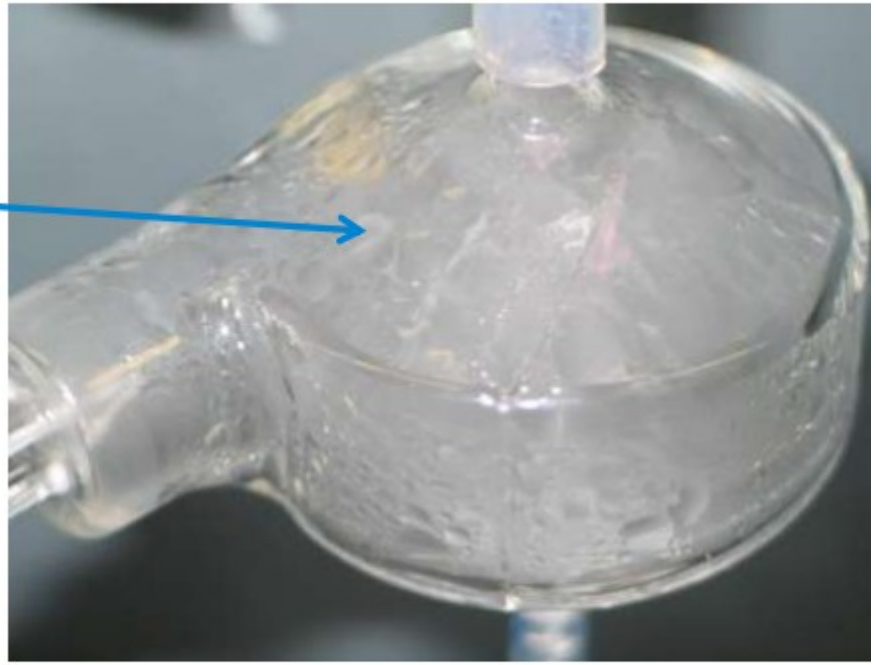
Inert for high solids & HF capable (Sturman-Masters)

Cleaning the ICP Spray Chamber

Can sonicate in a detergent solution (with care), rinse and dry

Must clean the spray chamber when you see “beading” or droplet formation on the walls (or if precision degrades):

- 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



5100 Series ICP Torch Selection

Features moulded torch base designed for “plug and play” loading

- Easy to install (3 steps)
- Automatic torch alignment - no torch scanning required
- Automatic gas connection to eliminate confusion around gas line fitting
- Not compatible (interchangeable) with 700 Series ICP-OES torch designs

One piece quartz Easy-fit torches

- Simple to use
- 1.8mm id injector (SVDV and VDV models)
  - 1.4mm id injector (Radial)
- Recommended for most applications

Demountable Easy-fit torches

- Gives greater flexibility & lower costs
  - 1.8mm injector (Standard - aqueous applications)
  - 2.4mm injector (High solids samples)
  - 1.4mm injector (Organics; also standard with radial)
  - 0.8mm injector (Volatile organics)
  - Alumina injector 1.8mm (Fusions and HF digests)

SVDV= Simultaneous Vertical Dual View

Demountable torches have replaceable outer tube

5100 Series ICP-OES Torch Cleaning

3 easy steps to cleaning the torch (see the torch cleaning guide (Agilent pub# G8000-90019) for more details):

1. Soak in 50% aqua regia for 1 hour
2. Thoroughly flush inside & outside using de-ionized water
3. Blow clean compressed air or nitrogen through the three gas supply ports

CAUTION

Ensure torch is dry before re-installation!

***Note**: Aqua regia 50% solution is prepared with 45 ml HCl conc. + 15 ml HNO_3 conc + 60 ml de-ionized water*

5100 Series ICP-OES Torch Cleaning

Agilent's torch cleaning stand (pn G8010-68021) provides a convenient, alternative way for users to clean their torches

- Provides a stable platform to enable either the complete torch assembly or the bare injector/base assembly to be suspended in the acid cleaning solution
- Encloses the container for the acid cleaning solution, reducing the chance of accidental spillage
- Prevents damage to the fragile end of the outer tube/injector from contact with the base of the container
- Reduces premature degradation of the elastomer materials on the torch body, by limiting exposure to acid fumes

ICP-OES System Tips – Wavelength Calibration

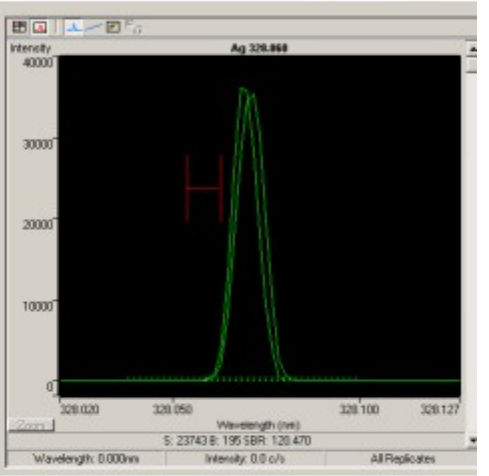
- "Wavelength Calibration" co-relates actual emission peak to the pixels on the detector chip

- Periodic calibration required – typically once/month
  - Monitor peak position w.r.t. peak marker to determine if calibration required

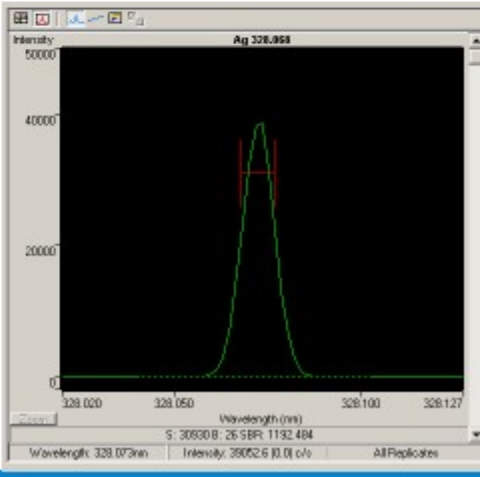
- For best results:
  - Use Agilent's prepared wavelength calibration solution (pn 6610030100)
    - Improves productivity –no "missing " components to troubleshoot
    - Improve reproducibility
    - Convenience – the pre-mixed solution has long shelf life, so you can use it as required
    - Also suitable for use when completing torch alignment
  - Plasma should be warmed up and stable
  - Optics boost purge should be enabled and stable (also requires the snout purge on the radial ICP)

- If the percentage value for "Calibration lines used" is < 100%:
    - Check that tuning solution had reached plasma
    - Check that purge enabled and stable – then repeat

Needs wavelength calibration



Peak display after successful wavelength calibration



ICP-OES System Tips – Plasma Ignition Problems

In most cases, the plasma will ignite first time.
If not, check the following (failure usually indicates presence of air):

- First, try repeating the ignition step again
- Have you changed argon cylinders recently?
  - Check the grade of argon
  - Try another cylinder
- Check all connections in the sample introduction system for cracks, loose fittings, missing or damaged items
  - Check that the plasma and auxiliary gas connections to torch were not reversed
- Check that the torch is sitting correctly w.r.t. RF coils (top of intermediate tube should be 2-3mm from edge of coil)
- Are you running a different sample type? e.g. higher RF power required for organic solvents
- Check the emergency off button on the front of the instrument
- Check the RF supply circuit breaker (on the rear panel of the instrument)

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 ICP Expert S/W running

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

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 $\geq$ 18 MΩ/cm^3)
    - 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

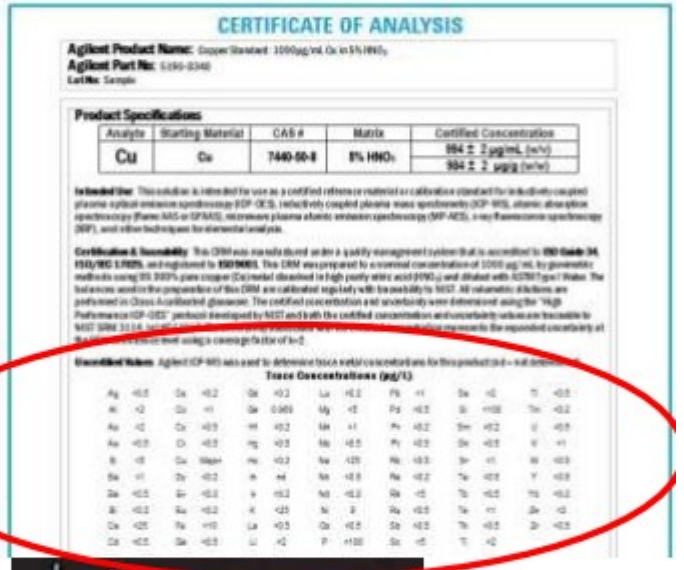
- What concentration are they?
  - Low concentration standards have a finite life
    - Prepare ppb (ug/L) concentration standards daily from high conc. stock
    - Prepare low ppm (mg/L) concentration standards weekly

- 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
  - 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


CERTIFICATE OF ANALYSIS

Agilent Product Name:

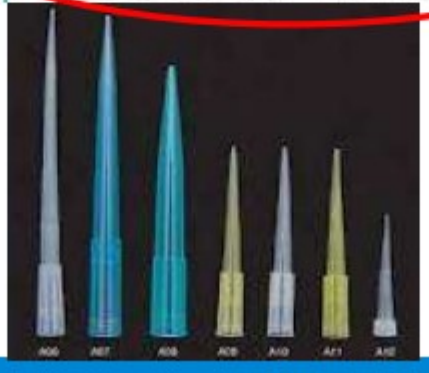
Agilent Part No:

Lot No:

Product Specifications

Analyte	Starting Material	CAS #	Matrix	Certified Concentration
Cu	Cu	7440-50-8	5% HNO_3	994 ± 2 µg/mL (w/v) 994 ± 2 µg/g (w/w)

Trace Concentrations (µg/L)



- Other common contamination sources
  - Reagent water
  - Clean glassware?
  - 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)

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

ICP-OES – Recommended Maintenance Schedule

Daily:

- Check exhaust system operating (smoke test)
- Inspect torch for injector blockage/other damage
- Check nebulizer for blockage/pulsation
- Inspect peristaltic pump tubing for stretching or flatness
- After analysis is complete:
  - Aspirate rinse solution for 5-10 mins. before shutting down (minimizes sample deposits)
  - Release pressure bar and detach peristaltic pump tubes from holder
  - Empty waste vessel
  - Leave ICP-OES in stand-by mode (gas and power on; software shutdown)

Weekly:

- Clean torch (or earlier if required)
- Check the other sample introduction tubing and O-rings
- Inspect cone (axial) or snout (radial); clean if req. by sonicating in dilute detergent
- Inspect torch bonnet for cracks or sample deposition (radial)
- Wipe down exterior surfaces of your ICP-OES (esp. sample compartment)

ICP-OES – Recommended Maintenance Schedule

Monthly:

- Clean spray chamber (or earlier if "beading" in spray chamber)
- Clean nebulizer
- Check the other sample introduction tubing and O-rings
  - Look for excessive wear, poor sealing or kinks and replace as necessary
  - Especially look at the transfer tube from spray chamber to torch and the spray chamber waste outlet
- Inspect/clean cone (axial ICP)
- Inspect/clean bonnet and/or snout (radial ICP)
- Inspect the state of induction coil
- Clean/check the air filter for the cooling air inlet (behind chimney)
- Clean/check air filter on the water chiller/recirculator
- Check the water level in the water chiller/recirculator & top-up if required

Periodically (every 6-12 months?):

- Clean water particulate filter on instrument
- Replace the water in the water chiller and dose with algaecide
- Change argon filters on argon gas supply (if using gas cylinders)

These functions (and more!) are completed as part of a Preventative Maintenance program by an Agilent Field Service Engineer

Overview – Key Consumables for ICP-OES

Sample introduction:

- Peristaltic pump tubing
- Torches
- Transfer and drain tubing
- Nebulizers
- Spray chambers
- Application kits (adapt your instrument to a new application)
- ICP standard solutions
- Ionization suppressant / buffer solutions

Autosampling:

- Sample tubes, racks, probes and transfer tubing

Vapor generation systems:

- Peristaltic pump tubing
- Connecting tubing

Links to Useful ICP-OES Resources

- ICP-OES parts and supplies (On-line Store):
  http://www.chem.agilent.com/store/en_US/Cat-SubCat1ECS_30364/ICP-OES?navAction=push&navCount=0
- ICP-OES troubleshooting videos:
  http://www.agilent.com/en-us/products/icp-oes/icp-oes-supplies/nebulizers/icp-oes-nebulizers-accessories/overview
- Agilent atomic spectroscopy application notes:
  http://www.chem.agilent.com/en-US/search/library/Pages/LibrarySearchResult.aspx?k=atomic&a=Scope:"Library"&w="Language"%20=%20'English'
- ICP-OES supplies inventory checklist:
  http://www.agilent.com/cs/library/brochures/OwnersChecklist_ICP_OES_LR.pdf
- Agilent Quick Reference Guides (lists most common consumables items):
  http://www.agilent.com/search/?Ntt=quick reference guides&N=164
- 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