INORGANIC VENTURE'S PARTNER WEBINAR SERIES: A SPECTRUM OF DIALOGUE

A PRACTICAL GUIDE TO TROUBLESHOOTING THE MOST COMMON ICP-OES/ICP-MS Problems

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GUEST SPEAKER

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THURSDAY, SEPT. 28 | 9:00–9:30AM EST

Common ICP-OES/ICP-MS Problems

- Loss of sensitivity
- Poor precision or high Relative Standards Deviations (RSDs)
- Calibration is good, but the first QC (or Calibration Verification - CV) did not pass

When Something Went Wrong: the Action Plan

- Cleaned the torch
- Cleaned ICP glassware: spray chamber, nebulizer, etc…
- Replaced pump tubing
- Replaced ALL the tubing!
- Replaced the torch
- Replaced ALL the ICP glassware!

Common ICP-OES/ICP-MS Problems

90% - ???

Sample Introduction!

Common ICP Problems: Loss of Sensitivity

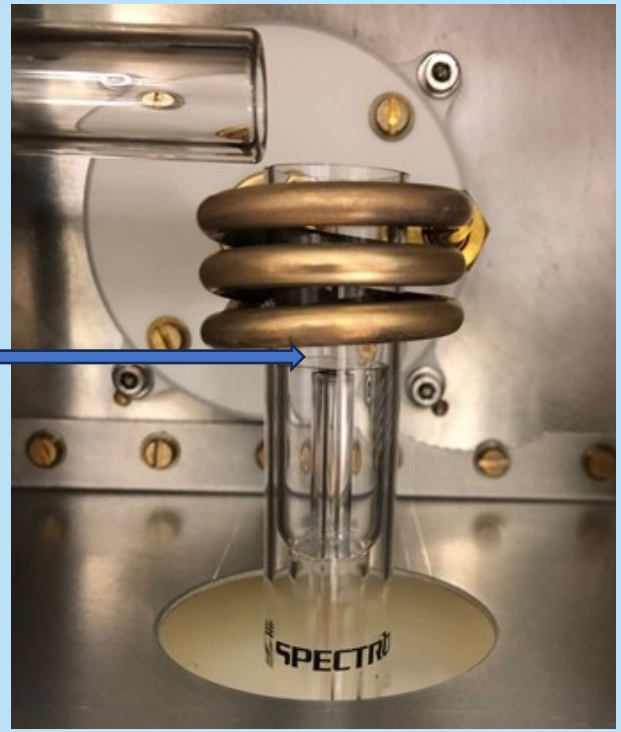
- Torch alignment
- Torch injector ID
- Blockages from the sample:
  - the nebulizer
  - the injector of the torch

Loss of Sensitivity: Torch Alignment

- Automated optimization routines move the torch!
- Use only when the torch is removed or replaced.
- Keep records of the optimization/tuning solution intensities.
- Monitor Mn 257.6 nm line at 5 or 10 ppm.

Loss of Sensitivity: Torch Alignment

A min 2 mm gap is recommended by ICP manufactures



Good

No gap

Elevated

Loss of Sensitivity. Diagnostics: Observing the Mist

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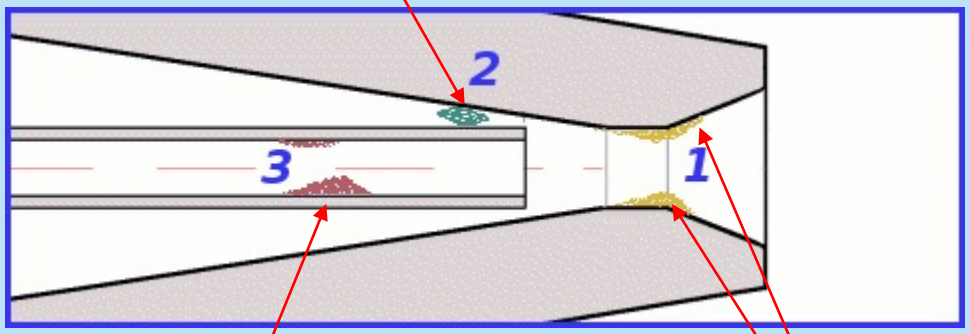
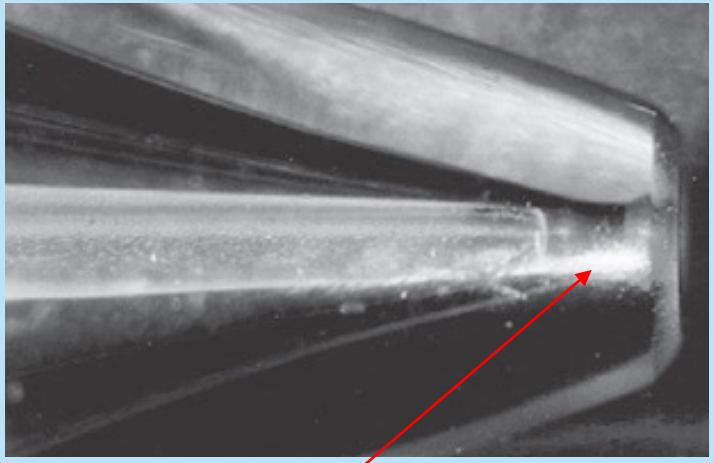
Loss of Sensitivity. Diagnostics: Observing the Mist

No plasma is needed.
Just turn the nebulizer gas flow and peristaltic pump ON

Loss of Sensitivity: Meinhard (Concentric) Nebulizer Typical Blockages

Blocked gas channel



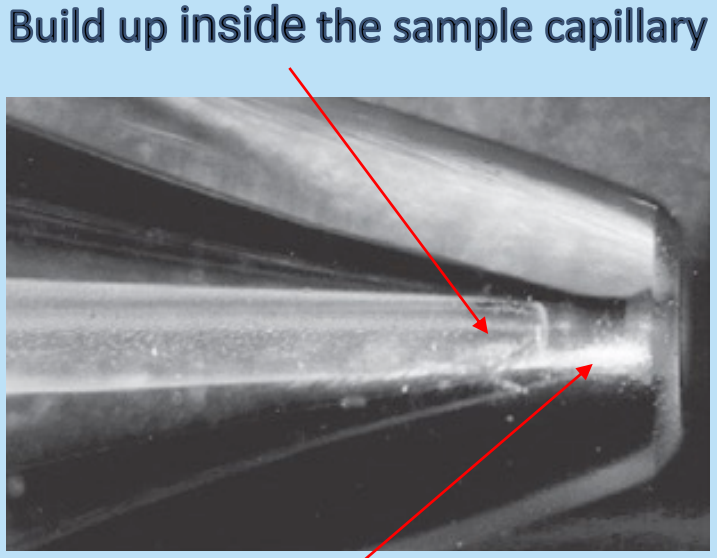


Build up inside the sample capillary

Sample material deposits at the orifice (José-Luis Todoli, Jean-Michel Mermet, Liquid Sample Introduction in ICP Spectrometry: A Practical Guide, 2011).

Loss of Sensitivity: Meinhard (Concentric) Nebulizer Typical Blockages

- Inconsistent and/or “one-sided” mist pattern
- Presence of the large particles in the mist
- Sample tubing may pop off from the join to the peristaltic pump tubing



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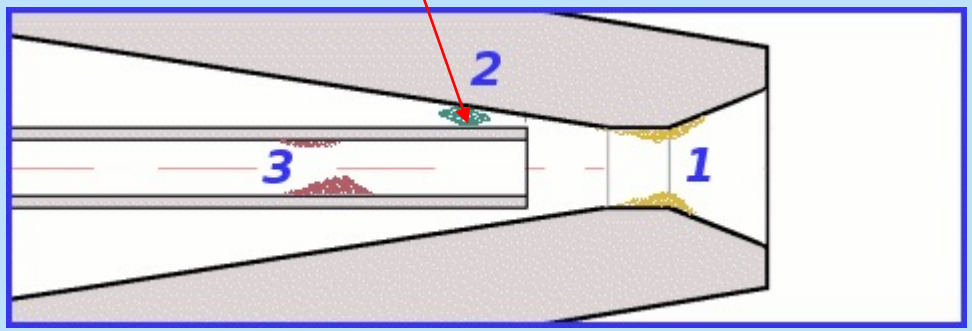
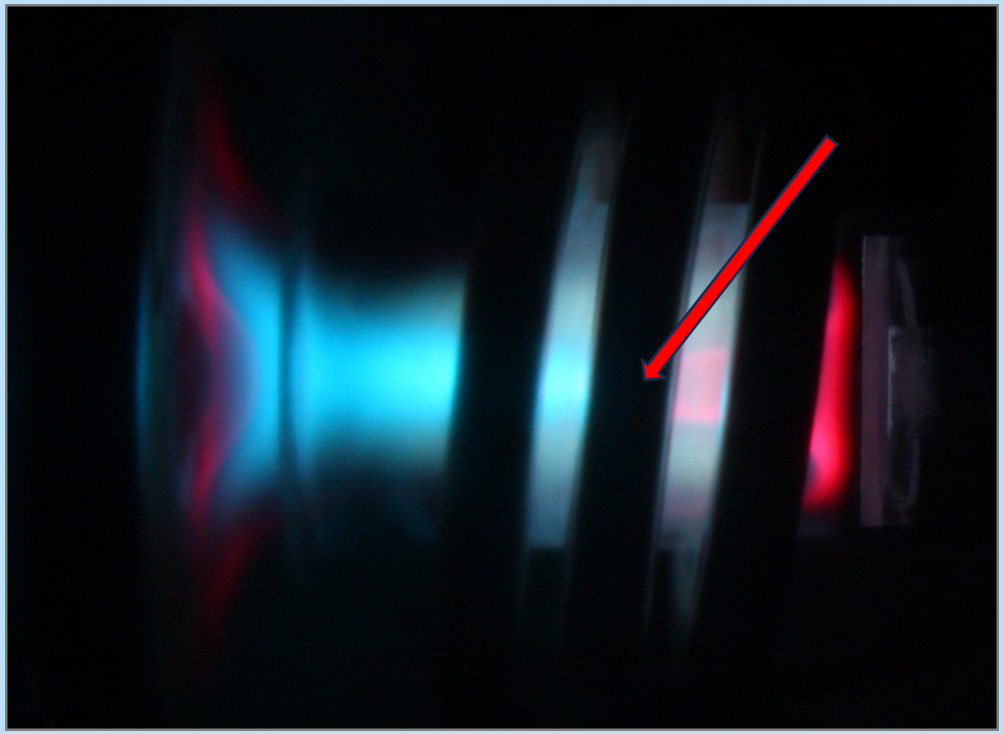


Diagnostics: monitor nebulizer backpressure

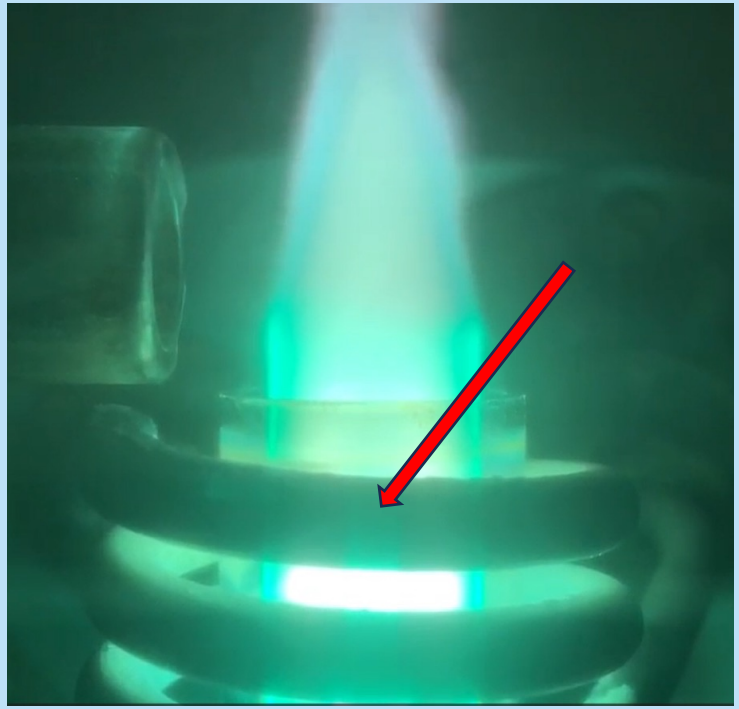
- If increased - the nebulizer is partially clogged
- If decreased - a leak in the sample introduction system

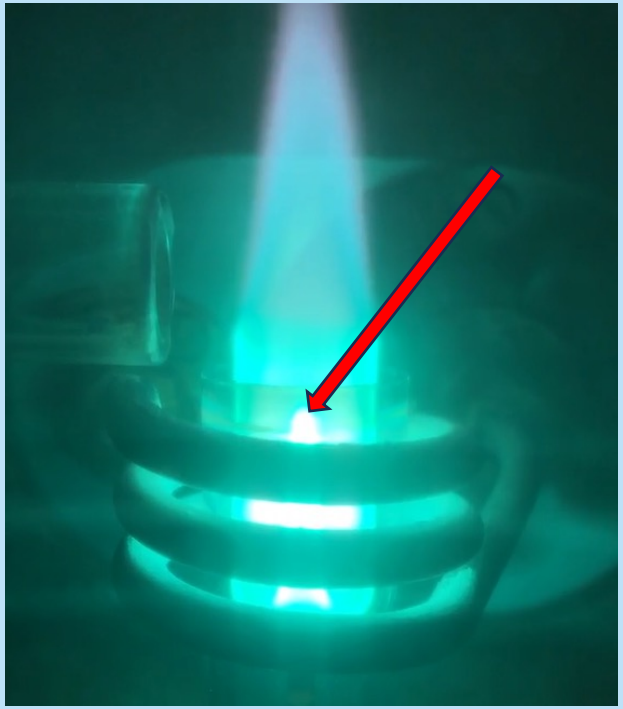
Yttrium “Bullet” Test as an Effective Diagnostics Tool for the Nebulizer Performance



- Needs just Yttrium solution at 5,000-10,000 ppm
- Bullet position relative to the RF coil and torch
- Intensity of colors
- Bullet stability

Carbon “Bullet” in Organic Matrix. Diagnostics Tool





Common ICP Problems: Poor Precision

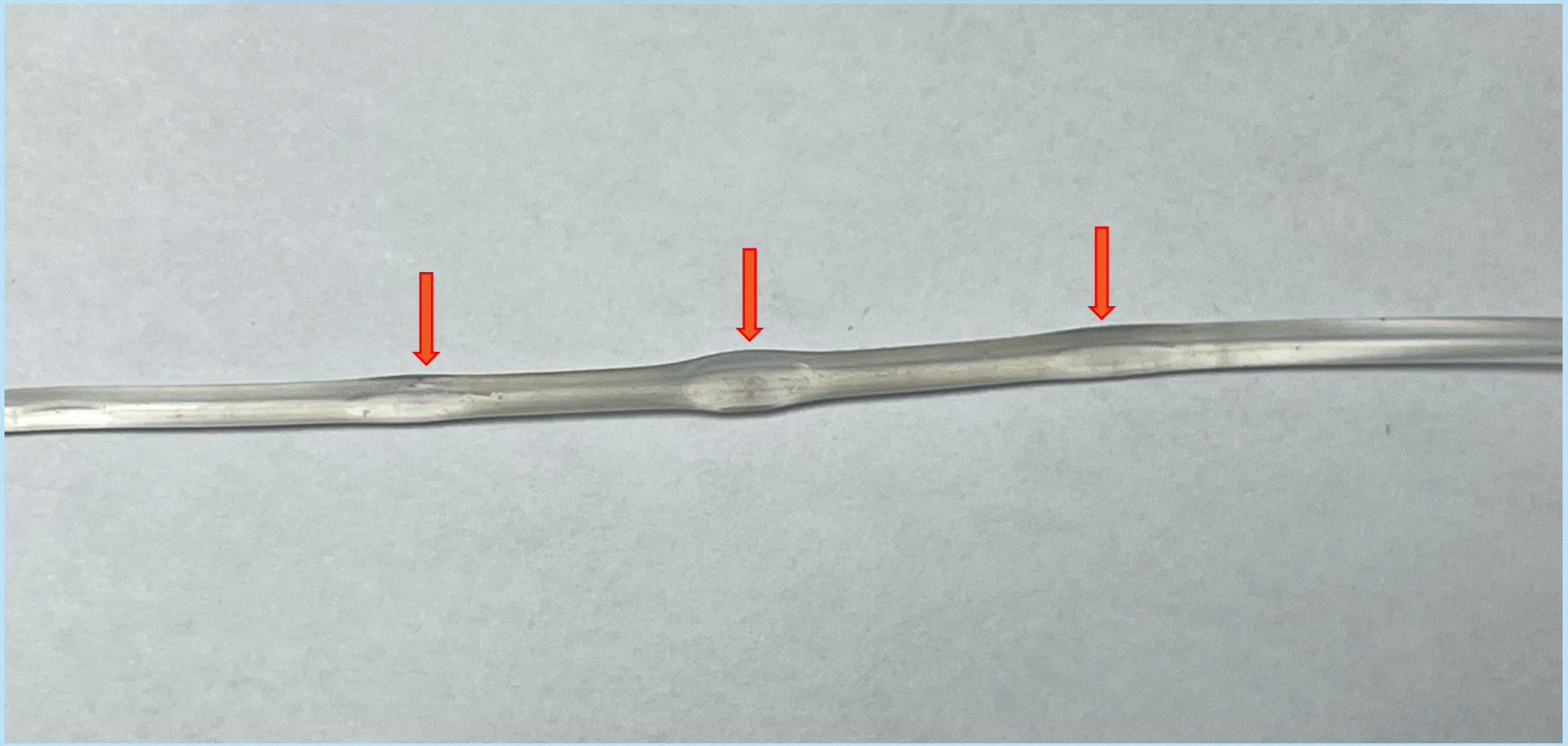
- Nebulizer blockage
- Spray chamber needs cleaning
- Pump tubing is worn
- Pump tubing is overtightened
- Injector of the torch is worn

Poor Precision:
Spray Chamber “Health”
Diagnostics - Proper Draining

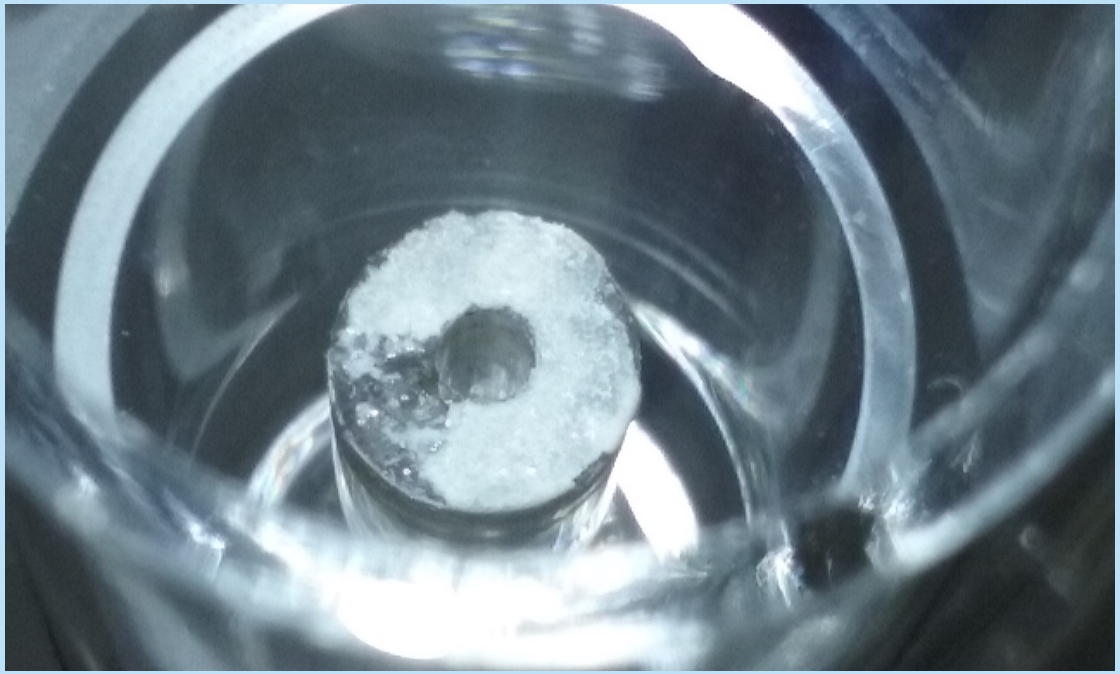
Poor Precision:
Spray Chamber “Health”
Diagnostics - Droplets Formation

Soaking in laboratory detergent

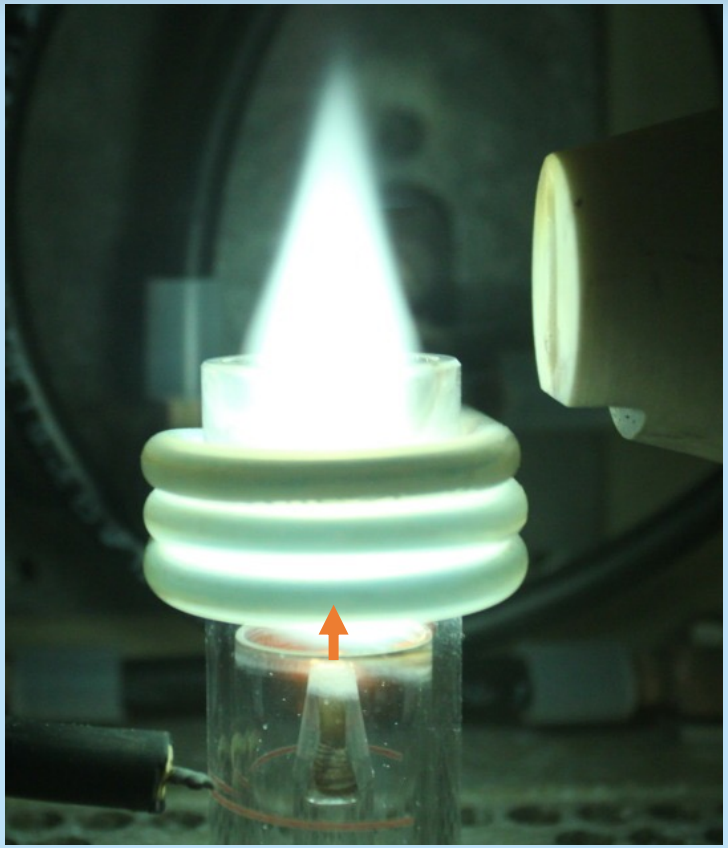
Poor Precision:
Worn (Flatten) Pump Tubing



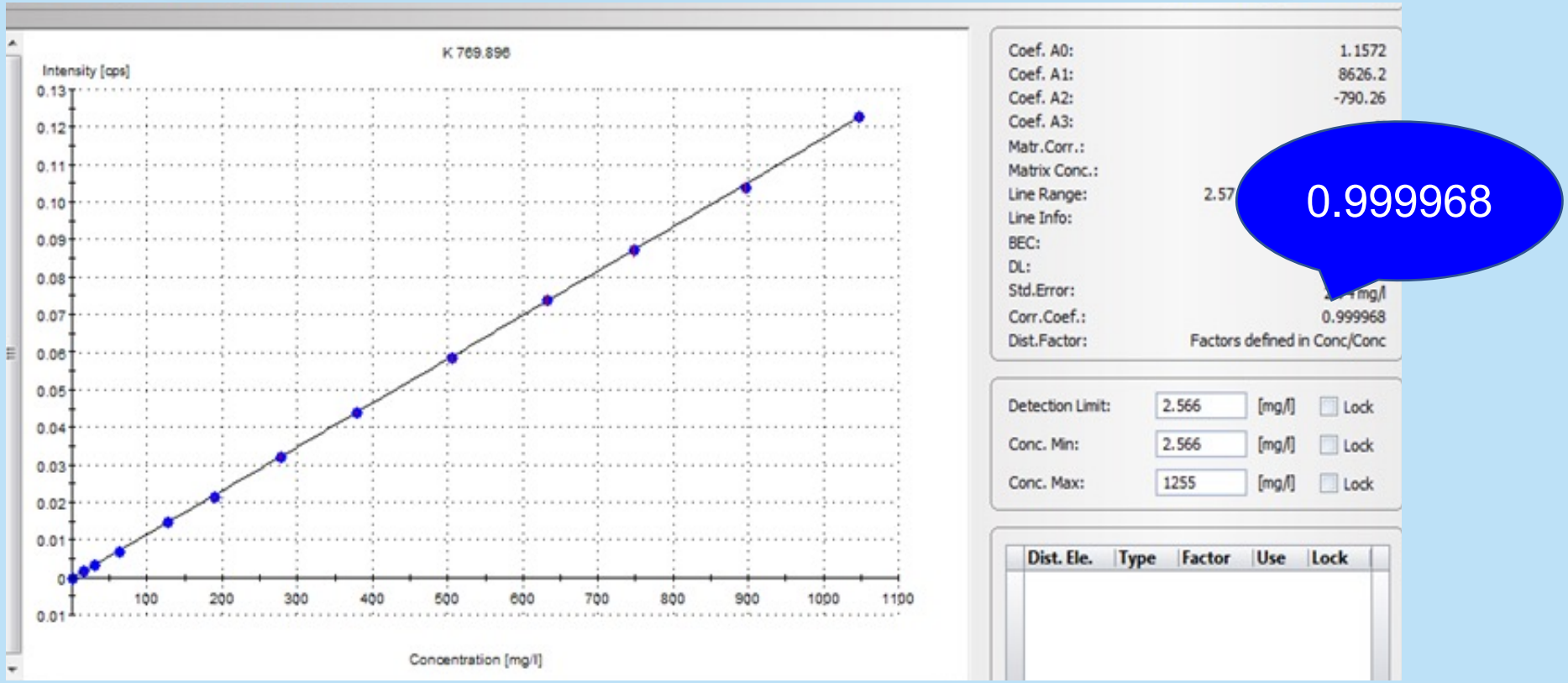
Poor Precision:
Worn Injector of the ICP Torch



Worn Injector or Sample Deposition on the Injector Tip: Increasing the Auxiliary (Intermediate) Plasma Argon Flow



Calibration Curve for K 769.89 nm Analytical Line



Calibration Data: What is More Important?

Cert.Conc. [mg/l]	Calc.Conc. [mg/l]	Diff. [mg/l]	Rel.Diff. [%]
0.0000	0.81666	0.81666	--
15.1500	15.3915	0.2415	1.59
30.3000	30.1847	-0.1153	-0.38
63.1000	61.3427	-1.7573	-2.78
126.0000	127.9853	1.9853	1.58
189.0000	186.8101	-2.1899	-1.16
278.0000	278.3932	0.3932	0.14
379.0000	379.8001	0.8001	0.21
505.0000	502.4845	-2.5155	-0.50
631.0000	635.8527	4.8527	0.77
747.0000	748.3224	1.3224	0.18
897.0000	890.3221	-6.6779	-0.74
1046.0000	1048.8439	2.8439	0.27

Common ICP-OES/ICP-MS Problems: Summary of Simple and Effective Diagnostic Tools

- Keeping a record of the tuning solution intensities.
- Monitoring torch alignment.
- Nebulizer mist observation.
- Nebulizer backpressure monitoring.
- Yttrium or Carbon “bullet” test.
- Spray chamber “health” monitoring: proper draining.
- Calibration data - focus on relative difference %.

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Thank you for your attention!

TSP
TEXAS SCIENTIFIC PRODUCTS

ICP-OES as a Viable Alternative to ICP-MS for Trace Analysis: Meeting the Detection Limits Challenge

Sergei Leikin and Autumn Phillips

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