

TROUBLESHOOTING ICP SPECTROMETERS

Glass Expansion February 2014 Newsletter, “Troubleshooting Guide for ICP
Optical Emission Spectrometers.”

Visit: <http://www.geicp.com/cgi-bin/site/wrapper.pl?c1=News>

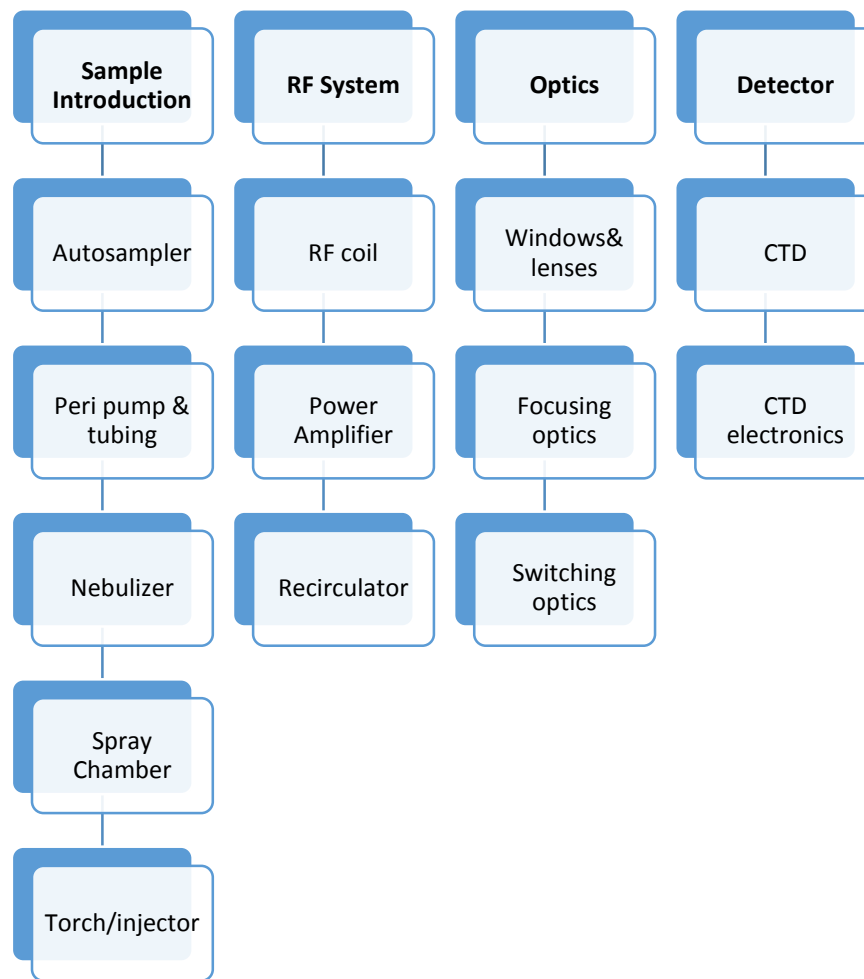
FIVE COMMON PROBLEMS

- Poor precision
- Carryover
- Drift
- Degraded detection limits
- Inaccuracy

COMPONENTS OF AN ICP

Establish a set of controls:

- Reference sample intro
- Expected performance



DIAGNOSTIC TESTS FOR ICP OPTICAL SPECTROMETERS

Simple experiments for the control, evaluation, and diagnosis of Inductively Coupled Plasma Sequential Systems

Test	Measurement	PMT Component	CTD Component
Ba II 233nm line profile	UV resolution	Dispersive system	Camera
Ba II 455nm line profile	VIS resolution	Dispersive system	Camera
Mg II 280/Mg I 285	Atomization/ionization	Generator	Generator
Ar I 404nm intensity	Light Absorption	Collimation/detector	Camera
BKG 400nm/BKG200nm	Lens/Mirror degradation	Optics	Optics
SBR Mg I 285nm	Transport efficiency	Nebulizer/Spray Chamber	Nebulizer/Spray Chamber
RSD Mg I 285nm	Nebulizer precision	Nebulizer/Spray Chamber	Nebulizer/Spray Chamber
SD BKG at 190nm	Background noise	Detector	Camera
SD BKG Plasma off	Detector noise	Detector	Camera
RSD Ar I 404nm	Drift	Generator Stability	Generator Stability

E. Poussel, J.M. Mermet, and O. Samuel,, Spectrochimica Acta Part B, **48**, 1993, 743-755.

POWER FLUCTUATIONS

RF Coil

- Regular replacement of corroded coils reduces the load on the RF generating system
- Changing corroded coils increases energy transfer, resulting in a more robust plasma and generally higher analytical line intensities
- Reduce chance of arcing



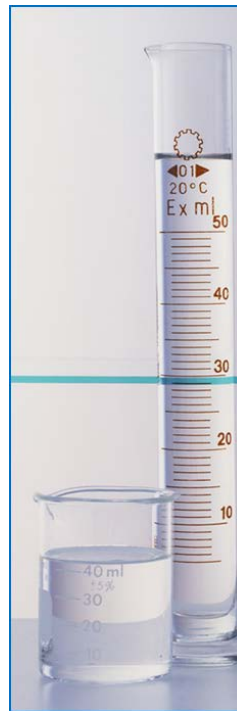
GE RF Coil

- Unique plating process
- Coil former, protects and maintains spacing
- Do-it-yourself installation tool

CAUSES OF UPTAKE FLUCTUATIONS

- Peristaltic pump
 - Worn pump tubing
 - Worn roller
 - Improper tension
 - Faulty pump

Volumetric measurement Vs Real-time continuous measurement



TruFlo P/N: 70-803-0643

CAUSES OF UPTAKE FLUCTUATIONS

Clogged Nebulizer



Fluka RBS-25 (P/N FLUKA25)



Part number: 70-ELUO

1. Flush your nebulizer with warm water using the Eluo.
2. Soak the nebulizer tip in a 25% solution of Fluka for 24 hours. Use the Eluo to make sure the Fluka solution fills the nebulizer.
 - An initial flush of 25% Fluka using the Eluo may be required.
3. Flush 3 times with warm water using the Eluo.
4. Stubborn deposits may require an additional soaking for 2 hours with 5% nitric acid.
5. Flush 3 times with warm water using the Eluo.
6. For faster drying, flush again with methanol.

SPRAY CHAMBER NOISE

- Blocked drain line
- Worn drain pump tubing
- Dirty spray chamber surface (clean with Fluka)
- Droplet buildup on transfer tube (axial or DV)



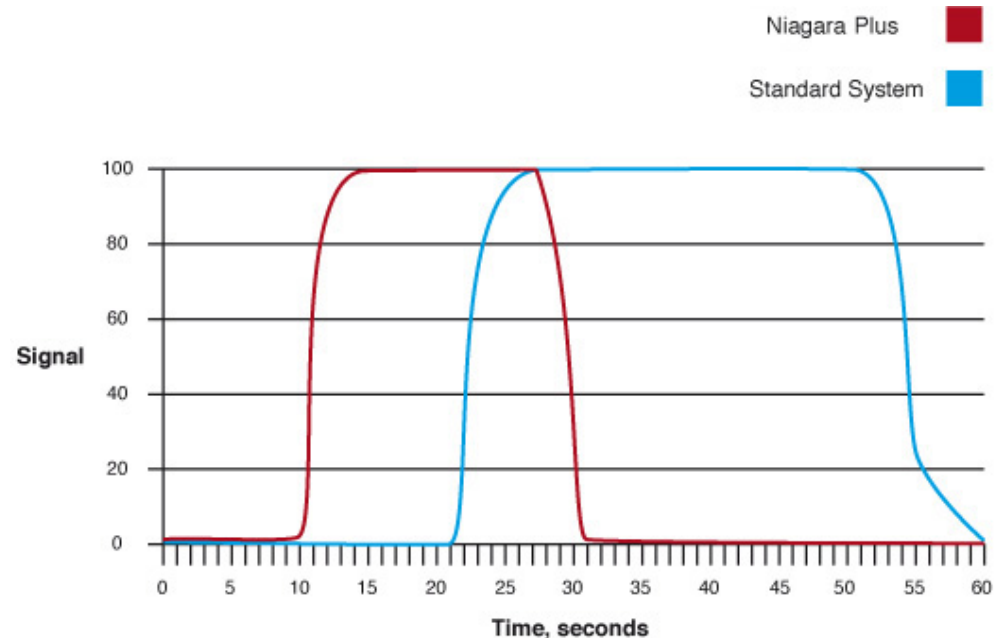
TORCH ISSUES

- Torch alignment
- Injector alignment
- Torch condition
 - Devitrification
 - Coil concentricity



WASHOUT

- 0.1% is typical goal
- Run 100ppm standard
 - < 0.1ppm on subsequent blank
- Varies between elements
- Rinse time and solution
- Switching valve-Niagara Plus CM



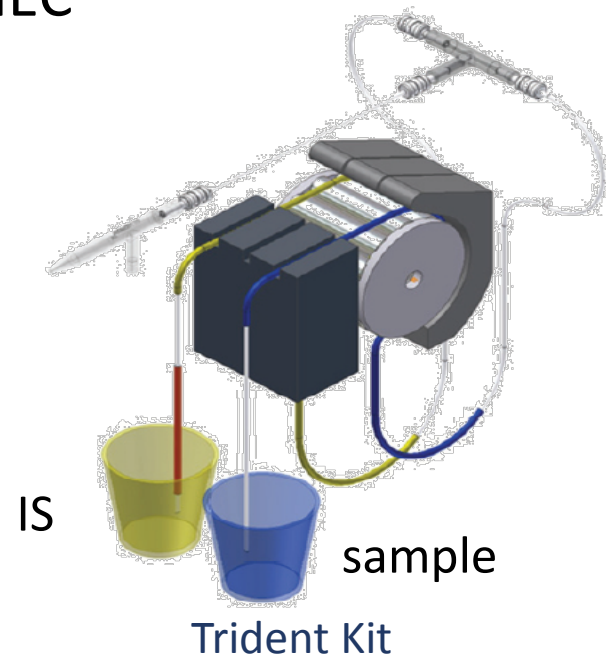
CAUSES OF LONG-TERM DRIFT

- Build-up on nebulizer tip - up or down
- Build-up on injector tip - usually down
- Room temperature drift - IsoMist
- Faulty gas regulator or MFC
- RF system-use Ar I 404test
- Optics - UV lines more severe
- Detector - use BKG test at 190



ACCURACY PROBLEMS

- Sample preparation
 - Contamination
 - Inaccurate dilution
 - Incomplete dissolution
 - Loss of analyte
- Physical interferences
 - Density
 - Viscosity
 - Surface tension
- Chemical interferences
 - EIE
- Inter-element interferences
 - Line selection
 - BKG correction
 - IEC



SUMMARY

- Establish baseline
- Eliminate spectrometer
- Isolate sample introduction culprit