Basic ICP-OES Troubleshooting

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Agenda

- Diagnostic Tests
- Common Issues/Problems
- Preventative Maintainence

Troubleshooting Tips

- Take a moment to review manuals
- Examine the facts and use valid reasoning
- Identify the “root cause” of the problem
- Avoid quick fixes and shotgun approach
- Be patient

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Prodigy7

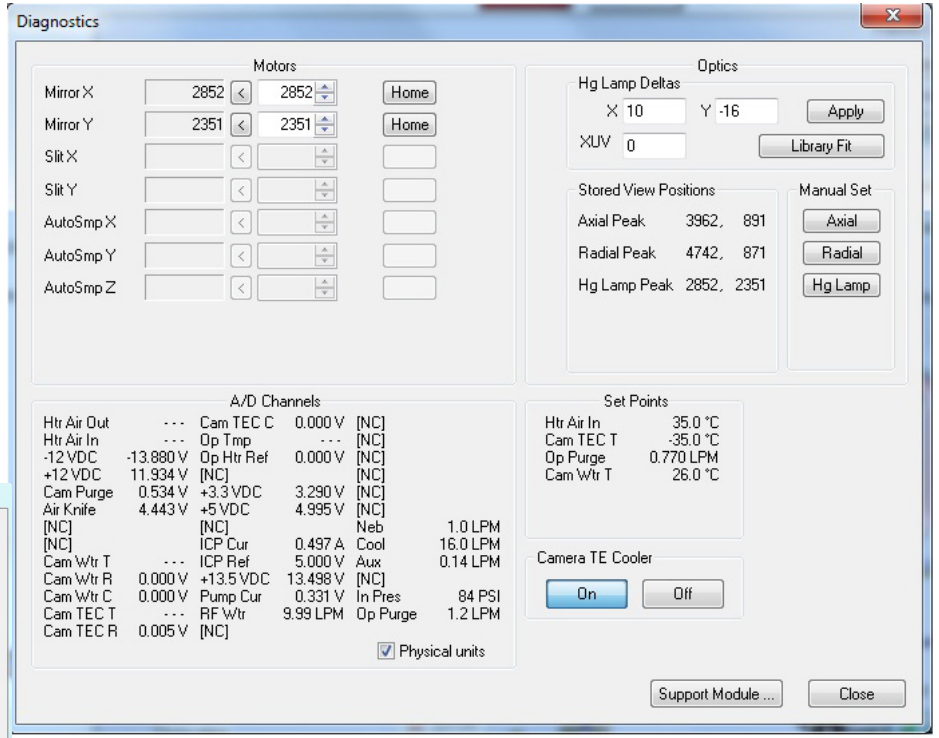
Prodigy

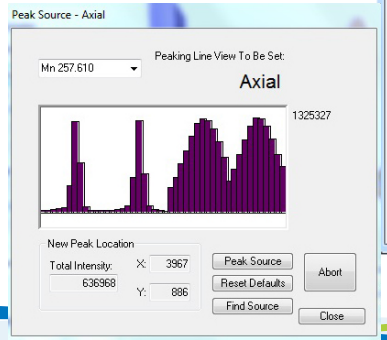
Finding the Root Cause

- Isolate the where the problem is
  - Sample Introduction
  - Spectrometer (Optics)
  - RF system

ICP Troubleshooting

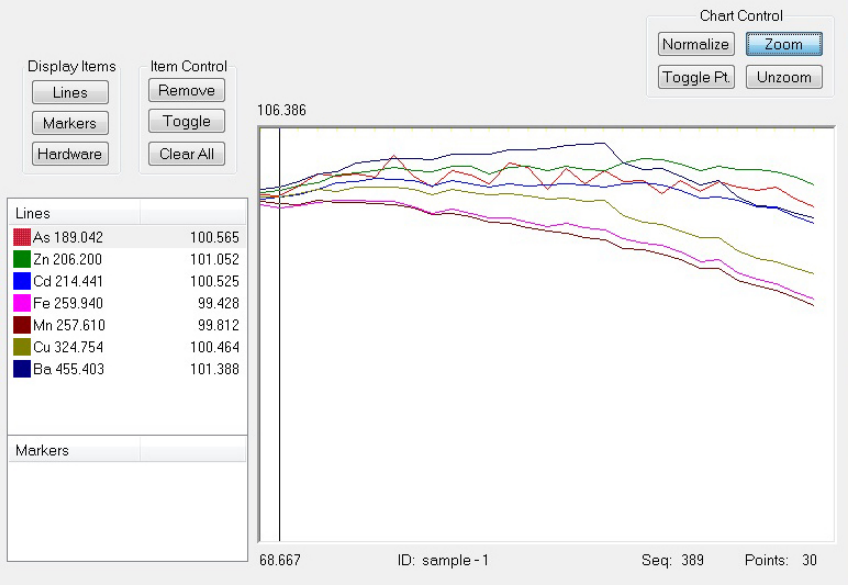
- Keep a record of instrument data
  - Positions
  - Intensities
- Establishes a baseline for comparison





Finding the Root Cause

- Hg Lamp Stability Test
  - Optics
- RF Stability Test
  - RF/power supply
- Analytical QC Test
  - Sample Introduction



Hg Lamp Stability Test

Hg Lamp Stability Test

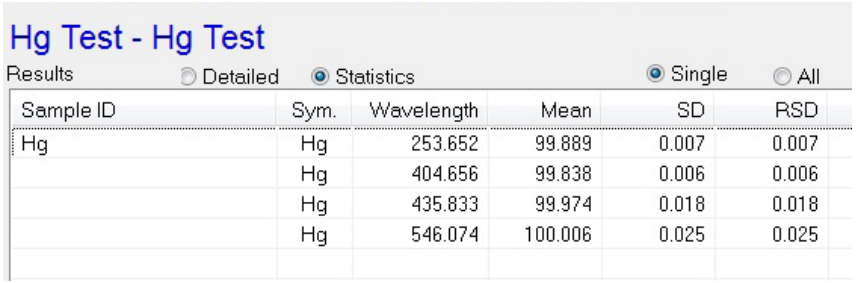
- Tests the optical system
- Eliminates
  - RF
  - Sample introduction system

Hg Lamp Stability Test

- Create a method containing the following Hg lines
  - 253.625
  - 404.656
  - 435.835
  - 546.074
  - Select view to be “Hg Lamp”
  - Make sure the wavelengths are aligned
- Define a standard at a concentration of 100
  - Any drift will be in percent
- Use a 10s integration time with 5 replicates
- Calibrate using 3 replicates
- Run 5 samples
  - 25 individual readings

Hg Lamp Stability Test

Hg	%RSD
253.652	<0.3%
404.656	<0.3%
435.835	<0.3%
546.074	<0.3%



Hg Test - Hg Test

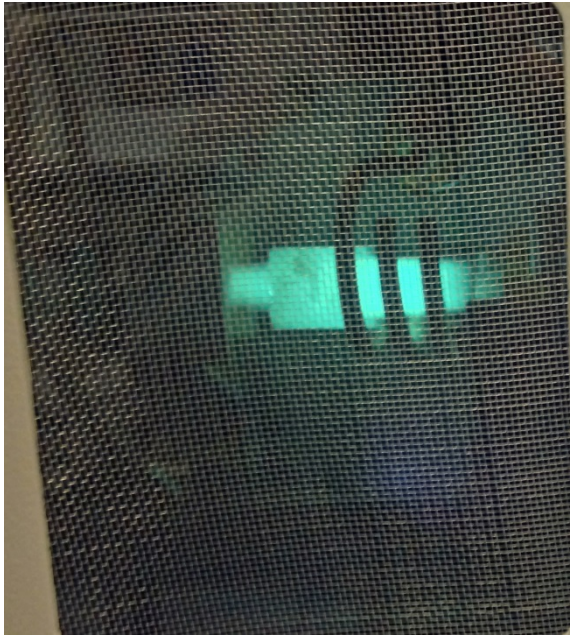
Results ○ Detailed ◉ Statistics ◉ Single ○ All

Sample ID	Sym.	Wavelength	Mean	SD	RSD
Hg	Hg	253.652	99.889	0.007	0.007
	Hg	404.656	99.838	0.006	0.006
	Hg	435.833	99.974	0.018	0.018
	Hg	546.074	100.006	0.025	0.025

RF Stability Test

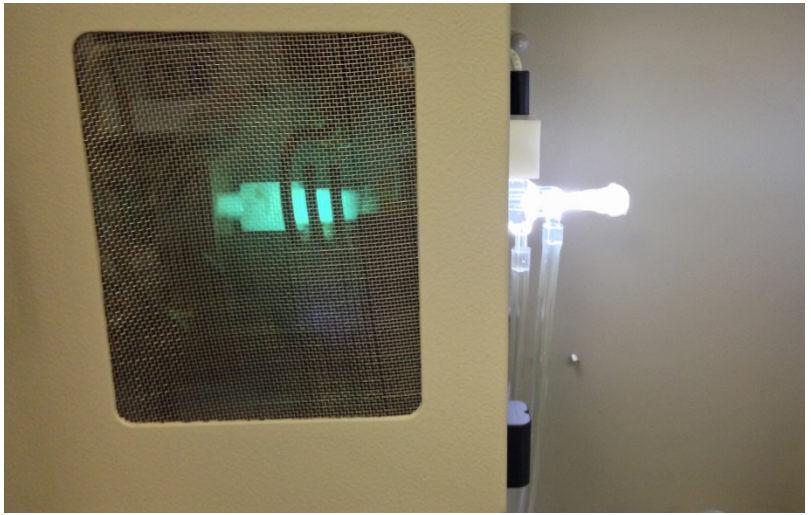
RF Stability Test (Argon test)

- Create a method using the following Ar lines
  - 451.073
  - 404.442
  - Make sure the wavelengths are aligned
- With the plasma off, remove spray chamber and plug torch
  - Parafilm, Teflon tape, etc.
  - Done to remove all traces of water vapor
- Define a standard at a concentration of 100
  - Any drift will be in percent
- Use a 20s integration time with 5 replicates
- Calibrate using 3 replicates
- Run 5 samples
  - 25 individual readings



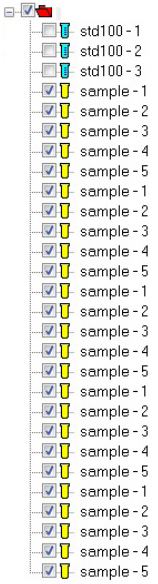
RF Stability Test

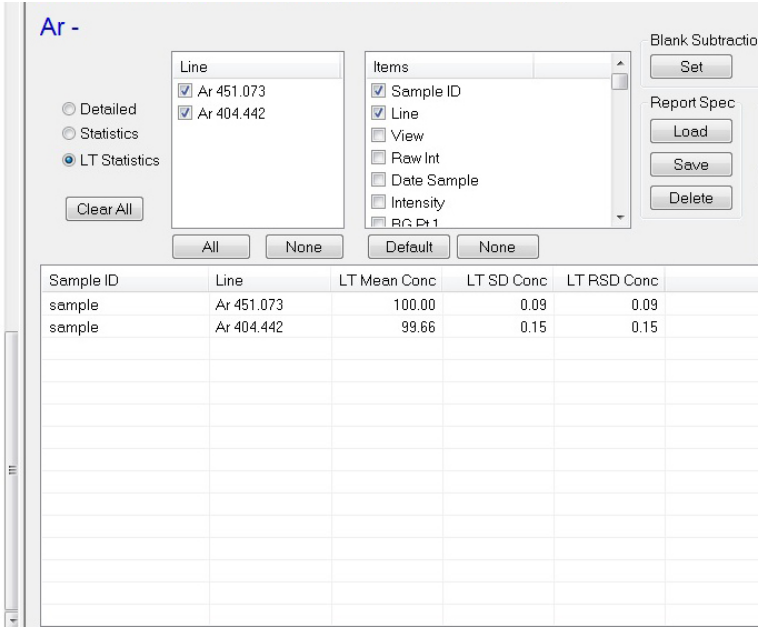
- Spray chamber removed
- Torch Plugged
- Use auxiliary flow to push plasma away from torch
- Wet spray chamber will usually result in a failing test



RF Stability Test (Argon Test)

Ar	%RSD
451.073	<0.3%
404.442	<0.3%





Analytical QC Test

Analytical QC Test

- Designed primarily to test the performance of the sample introduction system
  - Though it can yield information to help isolate problems in other areas
- Uses a specific set of elements/wavelengths based on their spectrochemical characteristics
- Similar to tests used during manufacturing and installation

Hard and Soft Emission Lines

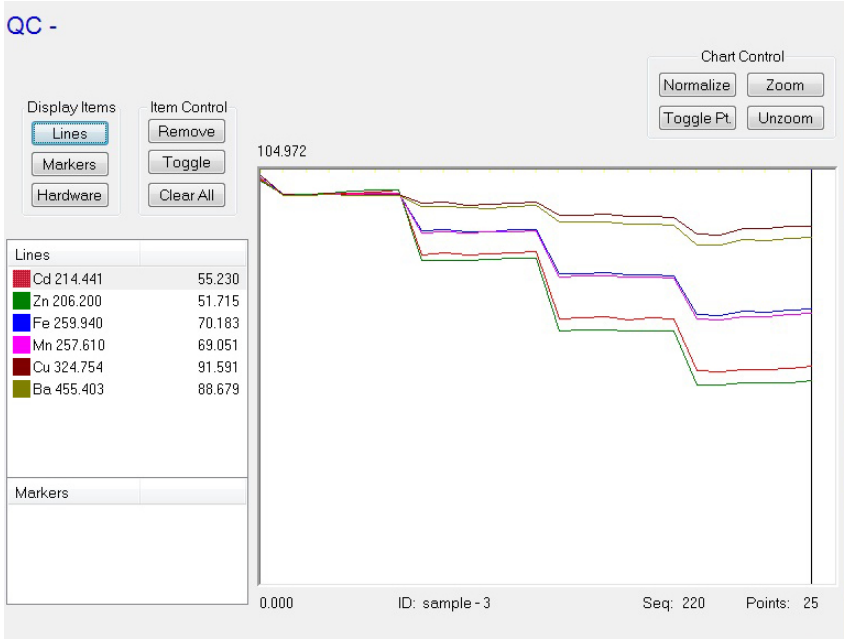
- Hard line
  - Atomic and ionic lines of elements with an ionization potential (IP) > than 8eV
    - Cd 214.441 and Zn 206.200
- Soft Line
  - Atomic lines of elements with an IP of < 8eV
    - Cu 324.754
  - Ionic lines of elements with a low 2nd IP
    - Ba 455.403

Analytical QC Test

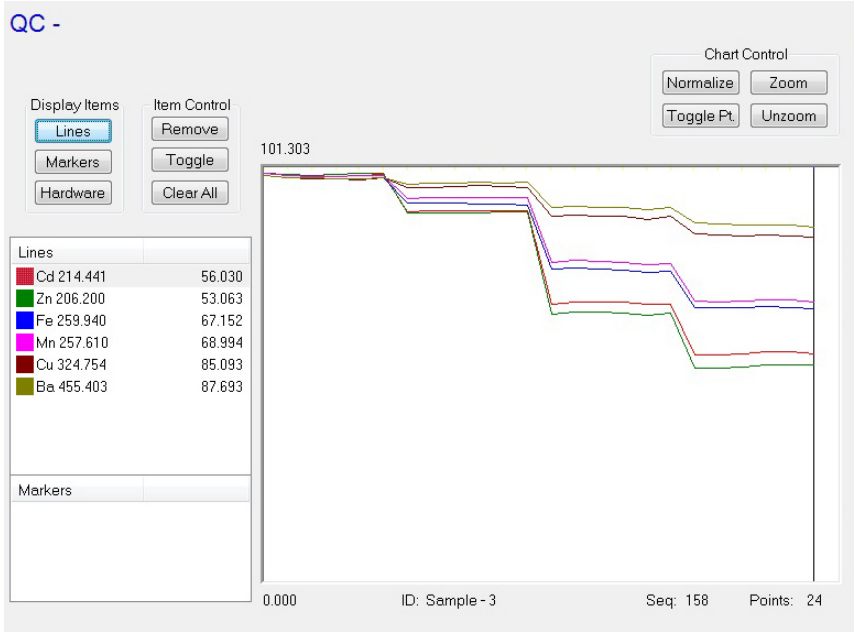
Element	λ, nm	State	1st IP, eV	2nd IP, eV
Cu	324.754	I	7.73	20.29
Ba	455.403	II	5.21	10.0
Fe	259.940	II	7.87	16.18
Mn	257.610	II	7.43	15.64
Zn	206.200	II	9.39	17.96
Cd	214.441	II	8.99	16.91

Analytical QC test

Increasing Nebulizer Pressure



Decreasing RF Power



Analytical QC Test

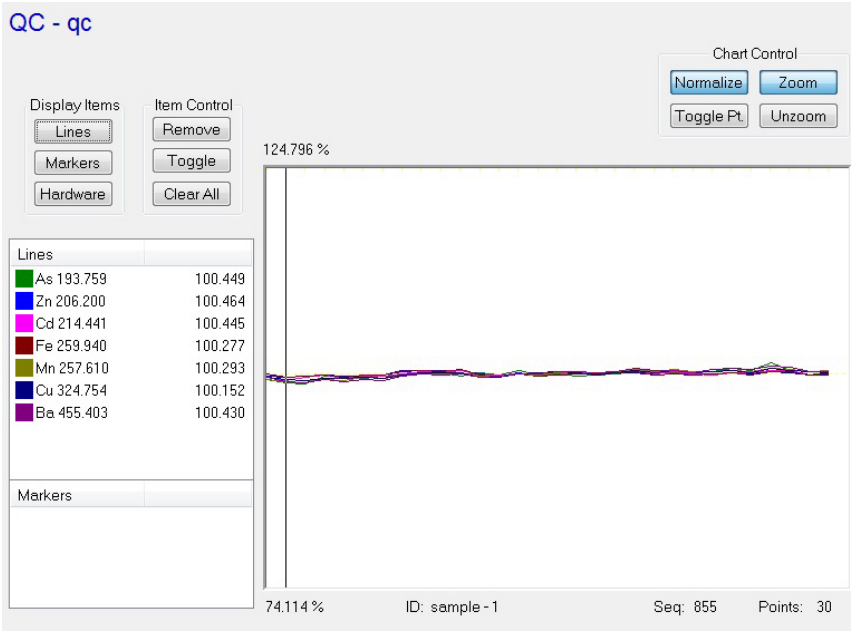
- Create method with the following lines:
  - Cu 324.754 and Ba 455.403
  - Fe 259.940 and Mn 257.610
  - Zn 206.200 and Cd 214.441
  - Make sure they are all aligned
- Define a standard using the concentrations listed on the right
- Position the plasma view on Mn
- Use a 30 second integration
- 20 replicates

Element	Radial, ppm	Axial, ppm
Cu, Fe, Mn, Zn, Cd	10	1
Ba	1	0.1

Analytical QC Test

Element	Wavelength	% RSD
Cu	324.754	<0.6
Ba	455.403	<1.0
Fe	259.940	<0.6
Mn	257.610	<0.6
Zn	206.200	<0.6
Cd	214.441	<0.6

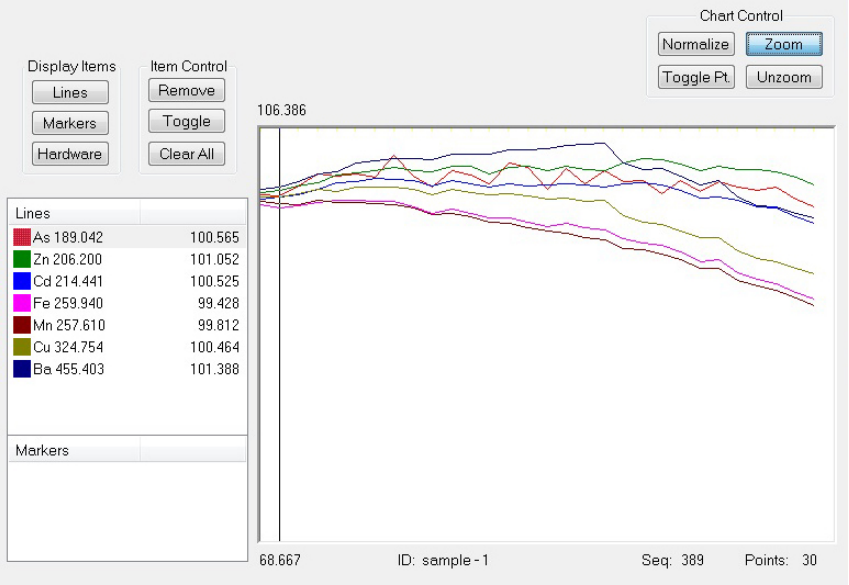
Analytical QC Test



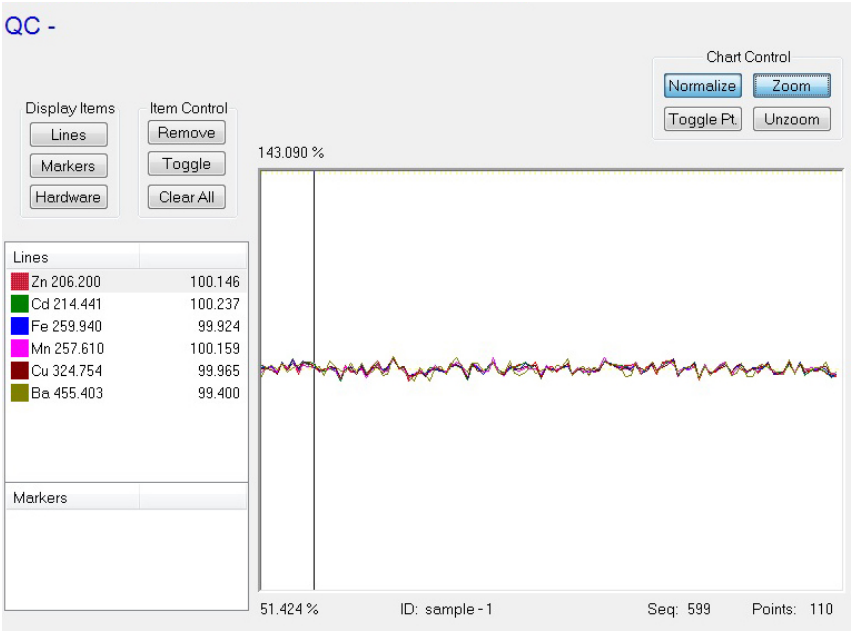
Sample ID	Line	LT Mean Conc	LT SD Conc	LT RSD Conc
sample	As 193.759	101.500	0.499	0.491
sample	Zn 206.200	100.915	0.257	0.254
sample	Cd 214.441	101.222	0.304	0.300
sample	Fe 259.940	100.712	0.244	0.243
sample	Mn 257.610	101.269	0.470	0.464
sample	Cu 324.754	100.886	0.387	0.384
sample	Ba 455.403	101.361	0.559	0.552

Analytical QC Test – What we're looking for

- Trends
  - All elements moving together
  - Like elements moving
- Random



Analytical QC Test – short (1s) integration



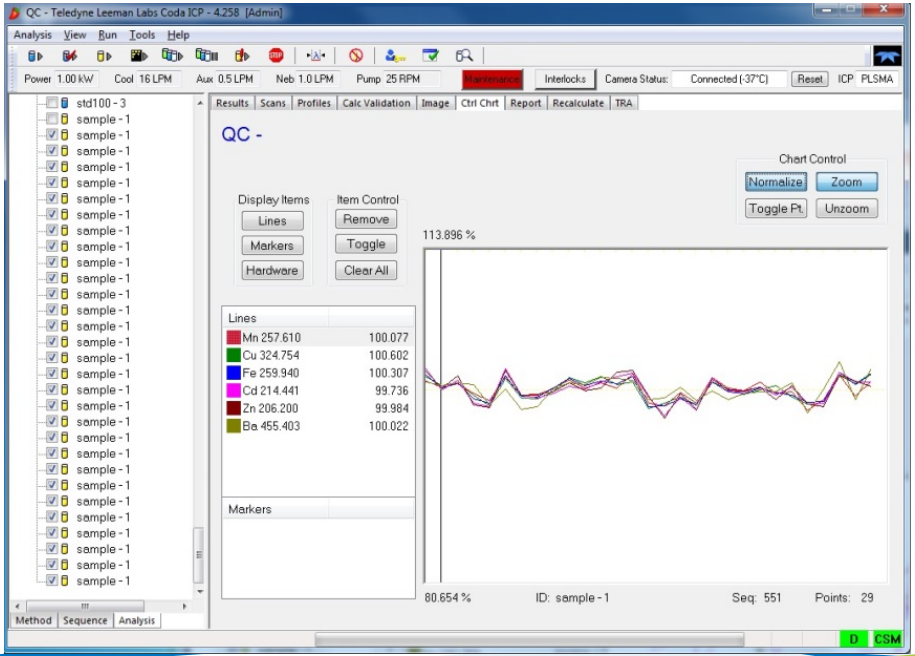
Sample ID	Line	LT Mean Conc	LT SD Conc	LT RSD Conc
sample	Zn 206.200	99.299	1.114	1.122
sample	Cd 214.441	99.100	1.044	1.054
sample	Fe 259.940	99.177	0.987	0.995
sample	Mn 257.610	99.599	0.957	0.960
sample	Cu 324.754	99.378	0.963	0.969
sample	Ba 455.403	99.472	1.157	1.163

Analytical QC Test

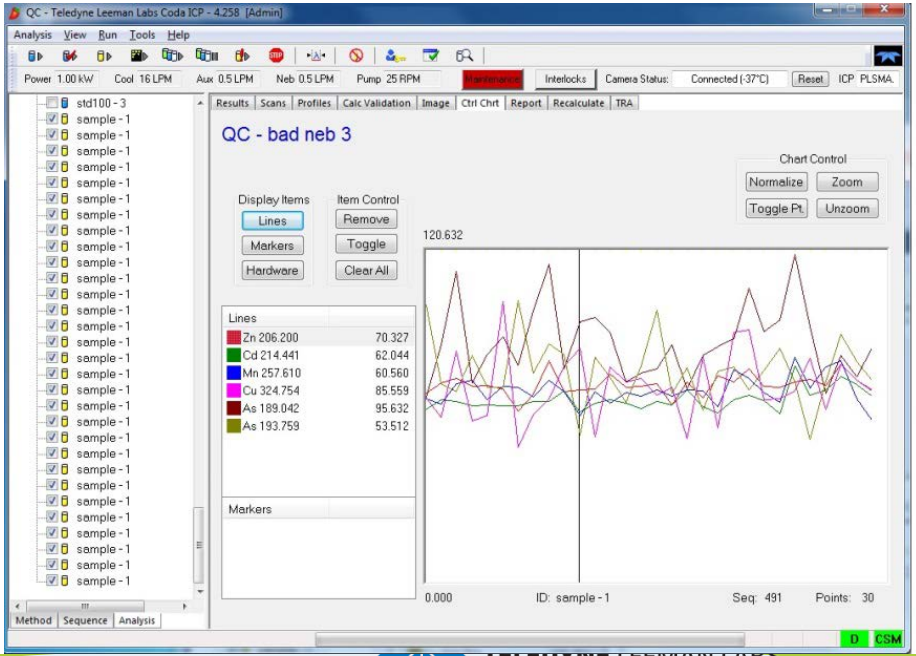
- Can use shorter integration times
- Longer test to look for intermittent problems
- Hg 312 nm and Ar 404 lines can be added
  - Hg tests spectrometer
  - Ar tests the gas control

Example QC Test Results

Pump Problem

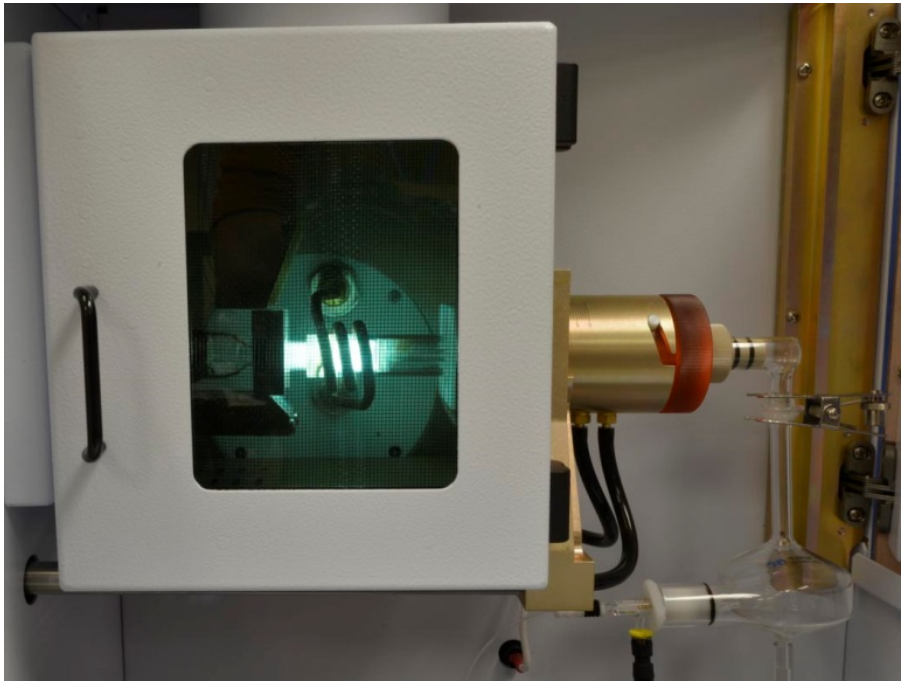


Nebulizer Problem



Finding the Root Cause

- Majority of problems will be related to the sample introduction system
  - Peristaltic Pump
  - Pump tubing
  - Spray chamber
  - Torch
  - Nebulizer
- Important to understand operation and set-up of sample introduction system



ICP Trouble Shooting

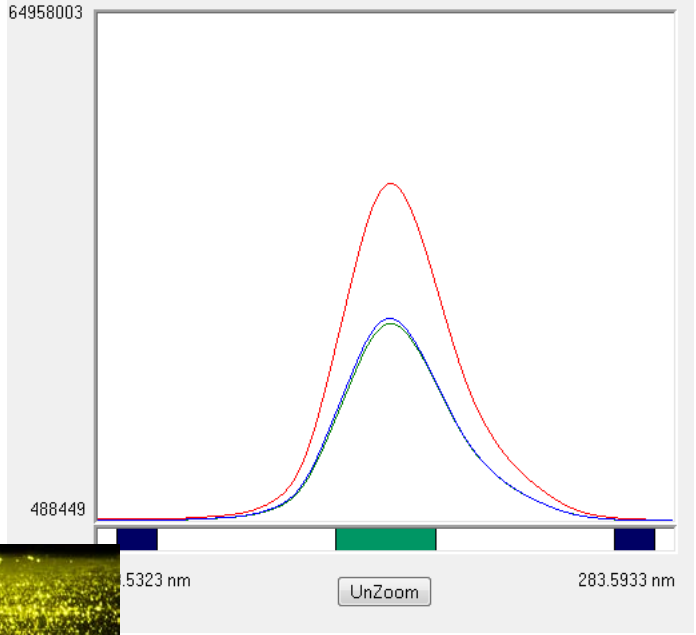
Common Problems

ICP-OES Troubling Shooting

- No Sensitivity
- Low Sensitivity
- Poor Precision
- Drift
- Plasma Won’t Light
- Melting Torches
- Check Standards Failing

ICP Trouble Shooting

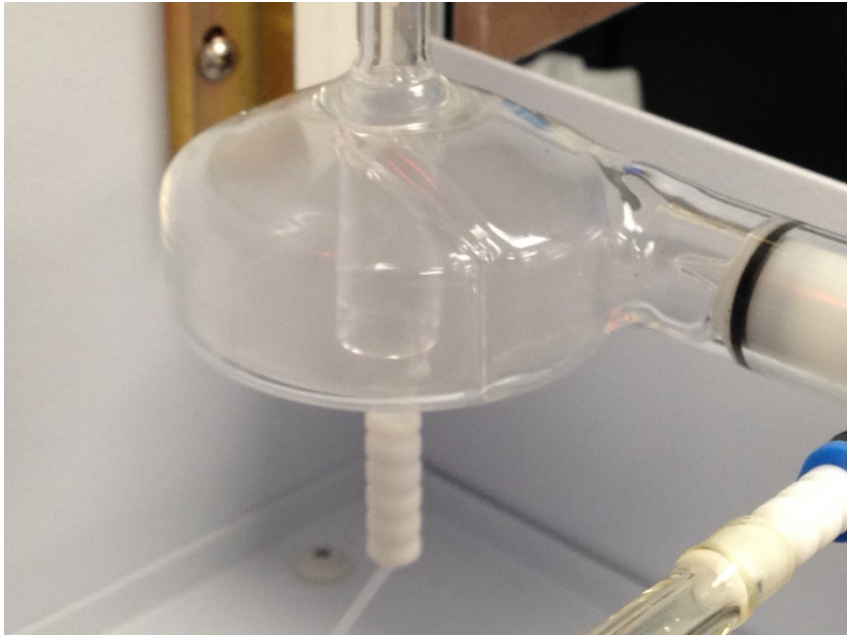
- Use wavelength scans and full frame images
- They show you what the instrument is actually seeing.



No Sensitivity or Signal

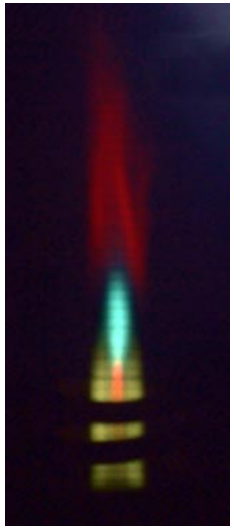
No Sensitivity or Signal - Is aerosol being generated?

- Quick visual inspection
  - Is nebulizer aspirating?
  - Check Pump tubing
  - Drain hooked up?



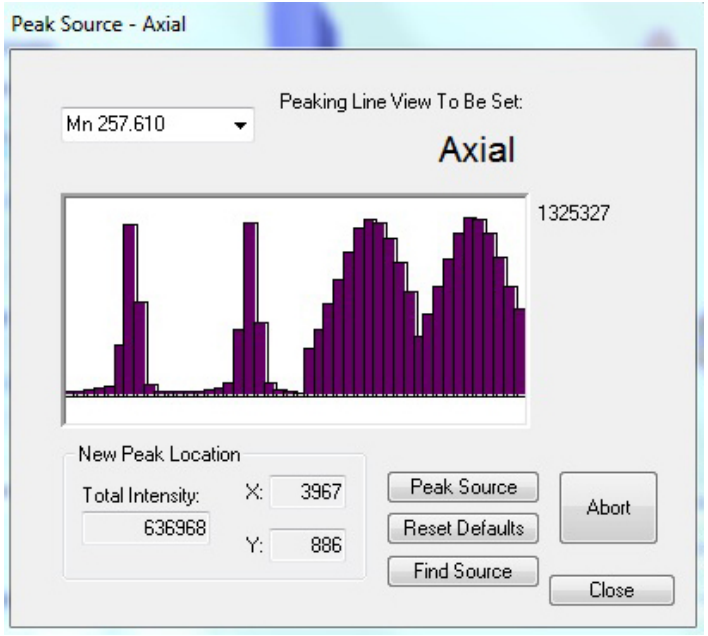
No Sensitivity or Signal

- Is aerosol getting to the torch?
  - Yttrium
  - Sodium



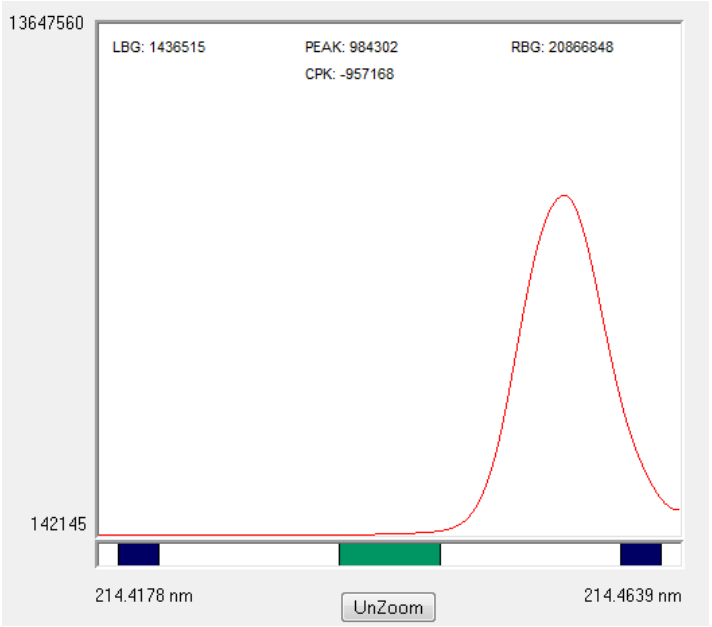
No Sensitivity or Signal

- Source Mirror correctly aligned?
- Aligned on a blank solution?



No Sensitivity or Signal

- Optics aligned?
- Wavelengths Aligned?

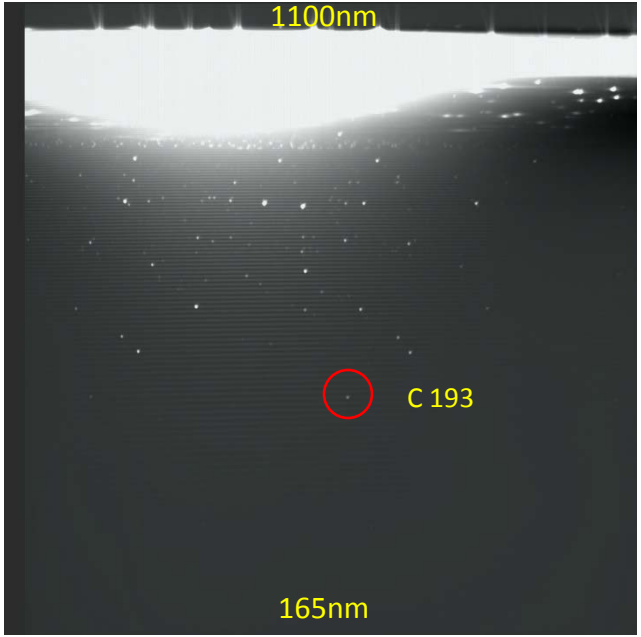


No Sensitivity or Signal (especially <190nm)

- Purged Optical Path (POP)
- Windows clean?
- Optics Purge Gas
  - On?
  - Leaks
  - Right Gas?

No Sensitivity or Signal (<190nm)

Purge Off



Purge On

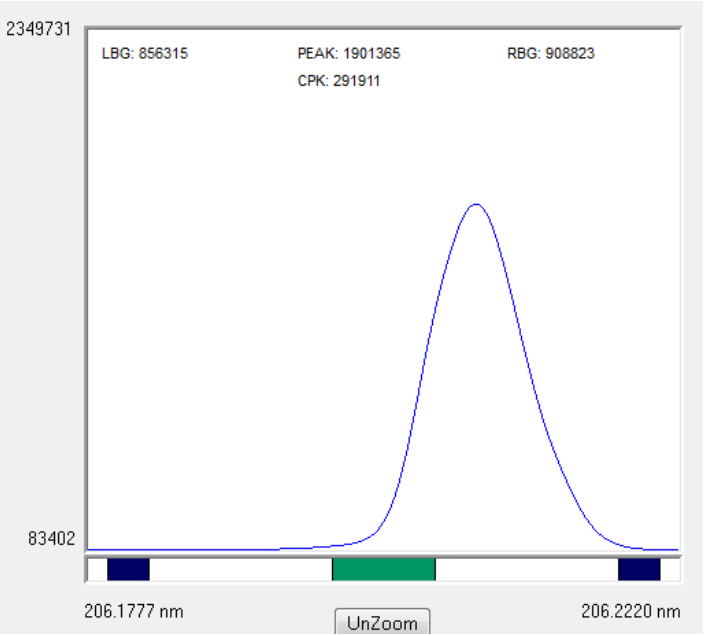
Low Sensitivity

Low Sensitivity

- Nebulizer operating pressure/flow correct?
  - Look at the yttrium, sodium
  - Green finger on organics
- Viewing Position Correct?
  - Correct element used?
  - Especially on radial system
- Torch positioned correctly in RF Coil?

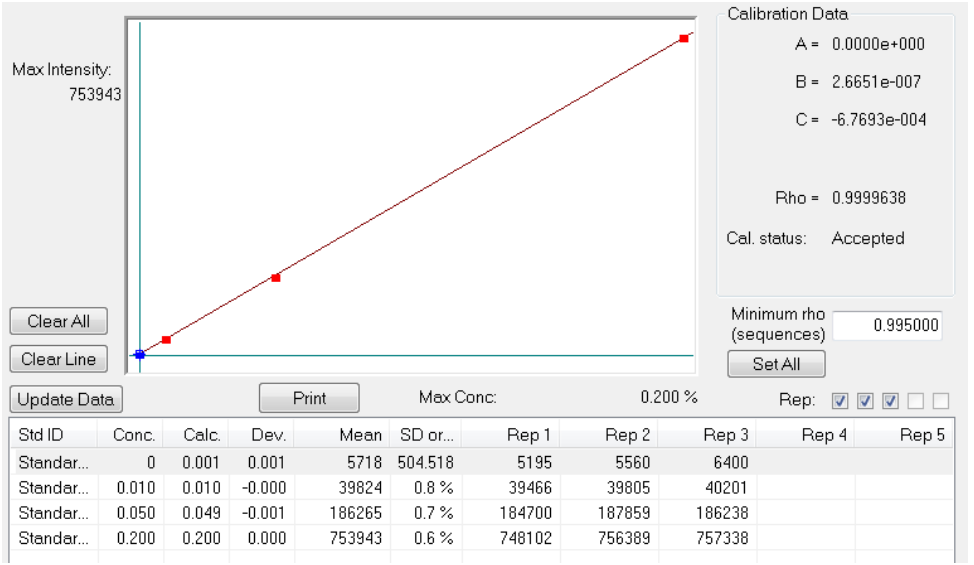
Low Sensitivity

- Optics not aligned
- Wavelengths misaligned



Low Sensitivity

- Correct standard concentrations?
  - The 10ppm is really 1ppm
- Old standards?



Std ID	Conc.	Calc.	Dev.	Mean	SD or...	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5
Standar...	0	0.001	0.001	5718	504.518	5195	5560	6400		
Standar...	0.010	0.010	-0.000	39824	0.8 %	39466	39805	40201		
Standar...	0.050	0.049	-0.001	186265	0.7 %	184700	187859	186238		
Standar...	0.200	0.200	0.000	753943	0.6 %	748102	756389	757338		

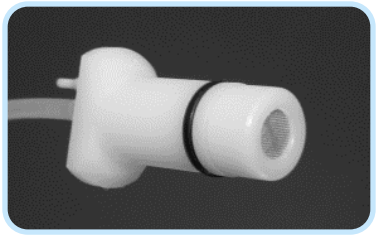
Low Sensitivity

- Purged Optical Path (POP)
- Windows clean?
- Optics Purge Gas
  - Leaks
  - Stable purge established

Poor Precision

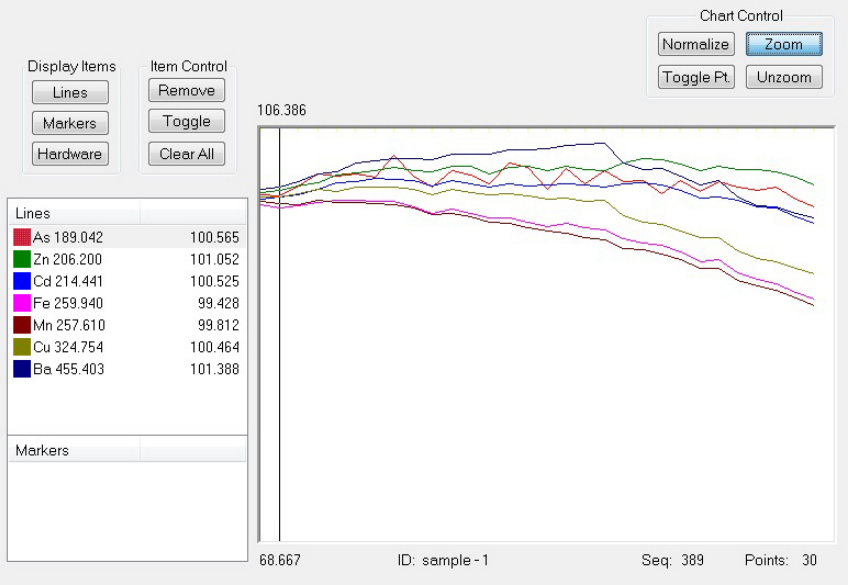
Poor Precision

- Uptake time too short
- Intermittent spray from nebulizer
  - Remove from spray chamber and observe spray with **deionized** water
- Stuck/dragging roller on peristaltic pump
- Poor drainage from spray chamber
- Solution beading up in spray chamber



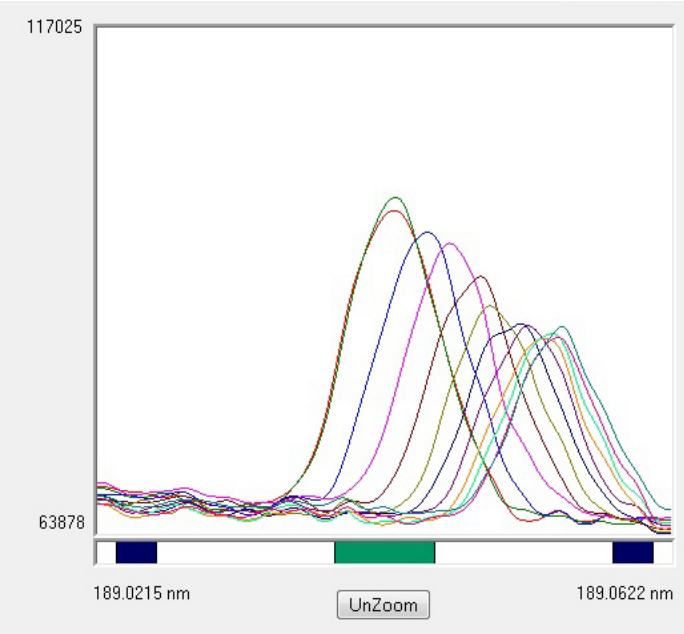
Drift

- Sample Intro sufficiently warmed up?
- Pump tubing broken in?
- Nebulizer clogging



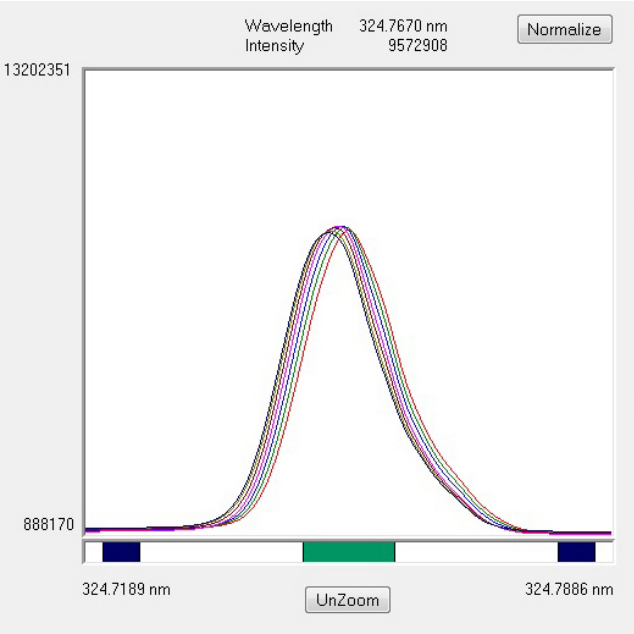
Drift

- Optical drift due to optics coming up to temperature



Drift

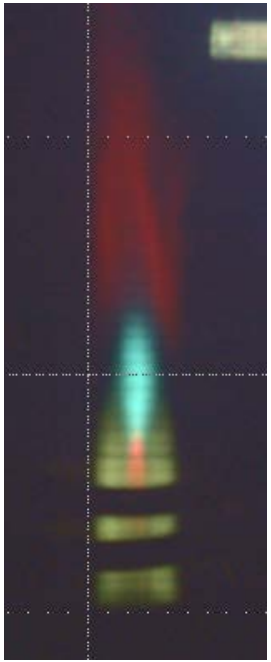
- Optical drift due to temperature change
  - Optics heater not at equilibrium; still changing
  - Torch exhaust flow too low



Plasma Won’t Light

Plasma Won’t Light (assuming no interlocks)

- Sample introduction leaks
  - Air getting in
- Gas lines reversed
- Igniter lead not attached
- Old or damaged torch
- Defective torch
- Bad gas



Torch Melts

Torch Melts

- Gas lines reversed
- Torch too deep in load coil
- Torch not centered in load coil
- Coolant flow not high enough for application
- Not enough auxiliary flow

Check Standards Fail

Check Standards Fail - Immediately After Calibration

- Inaccurate/Incorrect standards
- Inaccurate/Incorrect Check Standards
- Sample Introduction system not set up well
- Carry Over from High Standard
- Poor calibrations accepted
- Instrument not stable during calibration

Check Standards Fail – During analysis

- Drift
- Nebulizer clogging
- Sample carry over
- Rinse not long enough
- Check Standards not stable
- Poor calibrations accepted

Preventative Maintenance

Preventative Maintenance

- Clean/replace air filters
- Clean purge/POP tube windows
- Change water in chillers and recirculators
- Replace any o-rings in sample introduction system
- Change Pump Tubing
- Thoroughly rinse sample introduction at end of analytical runs

Summary

- Diagnostic Tests
  - Isolate Problem's Location
  - Hg Test for optics
  - RF Test for power
  - QC Test for sample introduction
- Common Issues/Problems
- Preventative Mountainous
  - Reduce the likelihood of problems

Questions?

Thank you!