


Modern Spectral and Physical Interference Correction in Inductively Coupled Plasma Optical Spectroscopy

Christine Rivera
Product Specialist
Agilent Technologies




ICP-OES Quantitative Analysis - a Relative Technique

Standards solutions prepared with a known concentration are required to quantitatively relate measured response of the element in the working standard to the measured response of an element in a test solution.

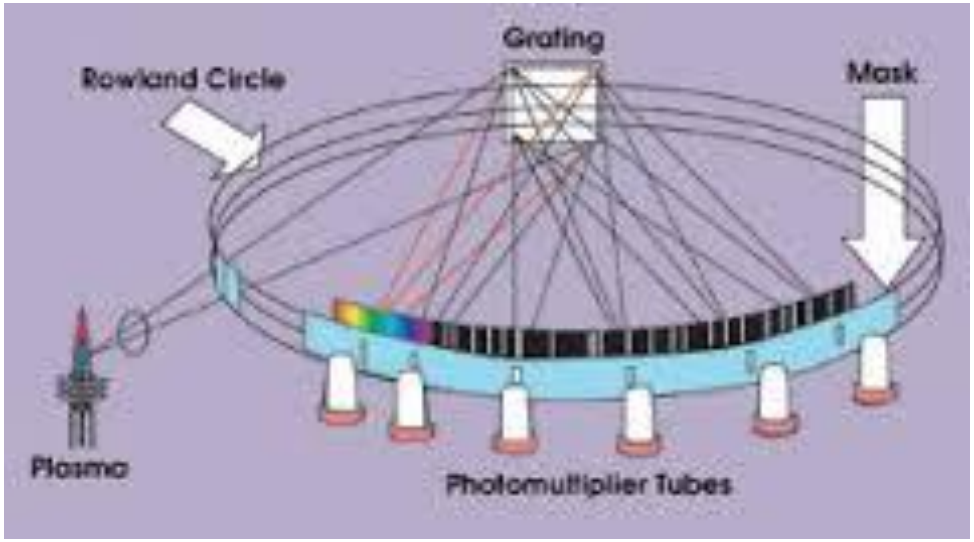
The assumption:

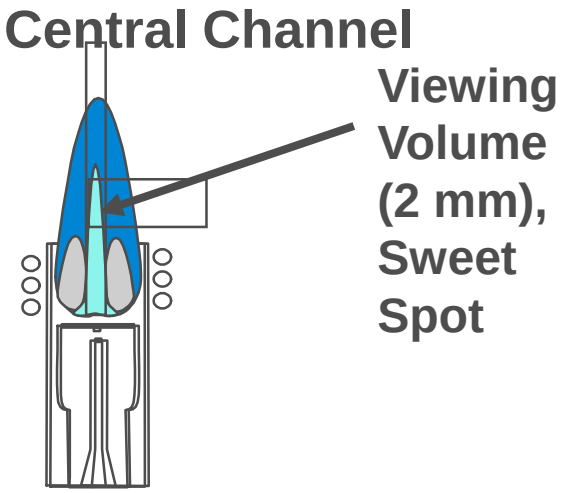
- Measured working standard response for each element is related to the response of the element measured in the test solution which correlates to the concentration of the element in the test solution.

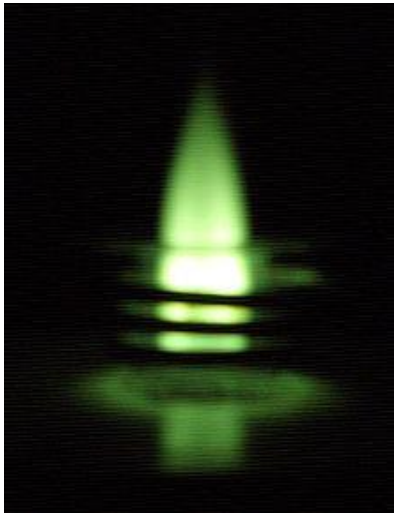
The assumption is only true when the standard solution and test solution are identical (mineral content, viscosity, polyatomic structures).

- Frequently, this is not the case.

History of ICPOES

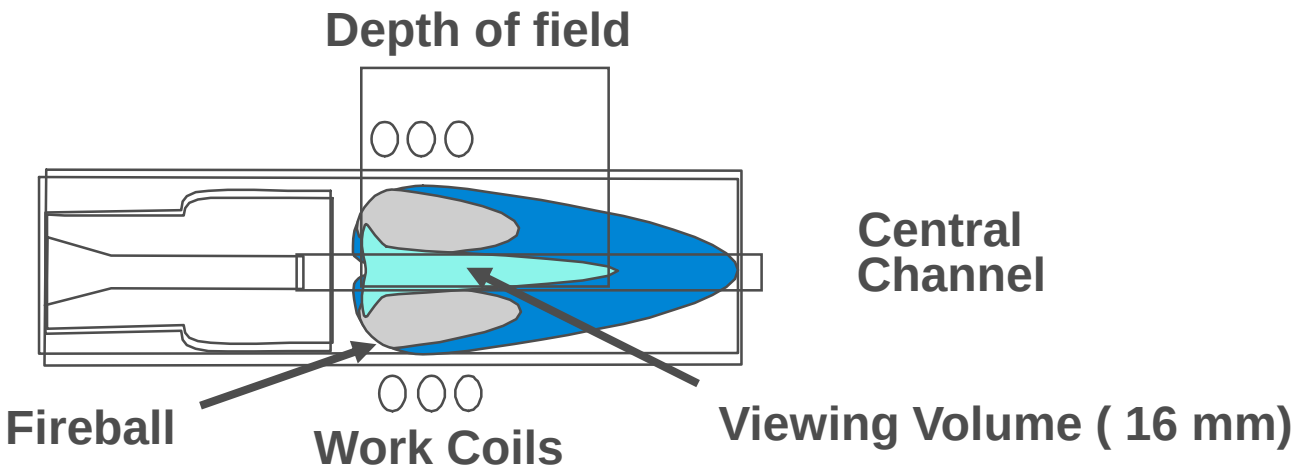






Horizontal Torch – Axial View

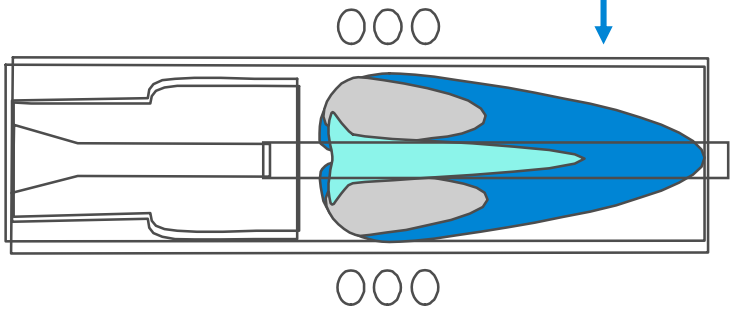
Axial Viewed Plasma (torch horizontal)



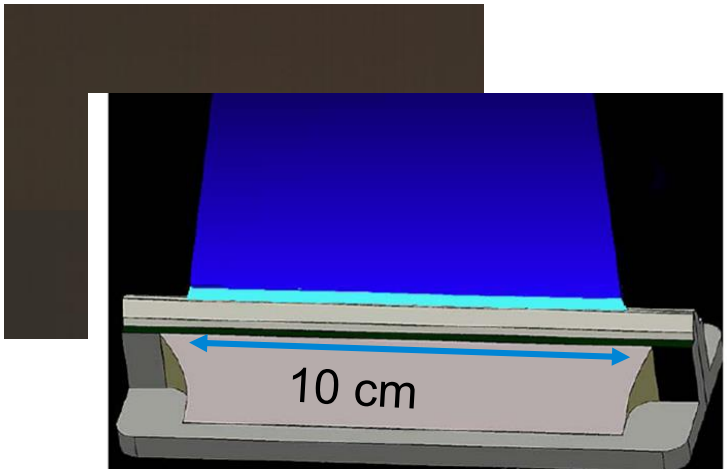
Horizontal Torch with Dual View

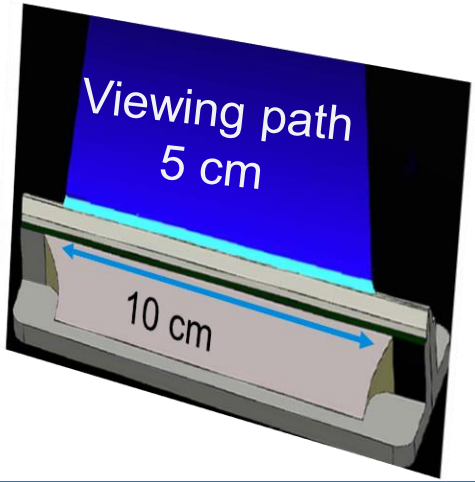
Torch Horizontal

Radial view
2 mm side view - Fixed Position
Reduced Path

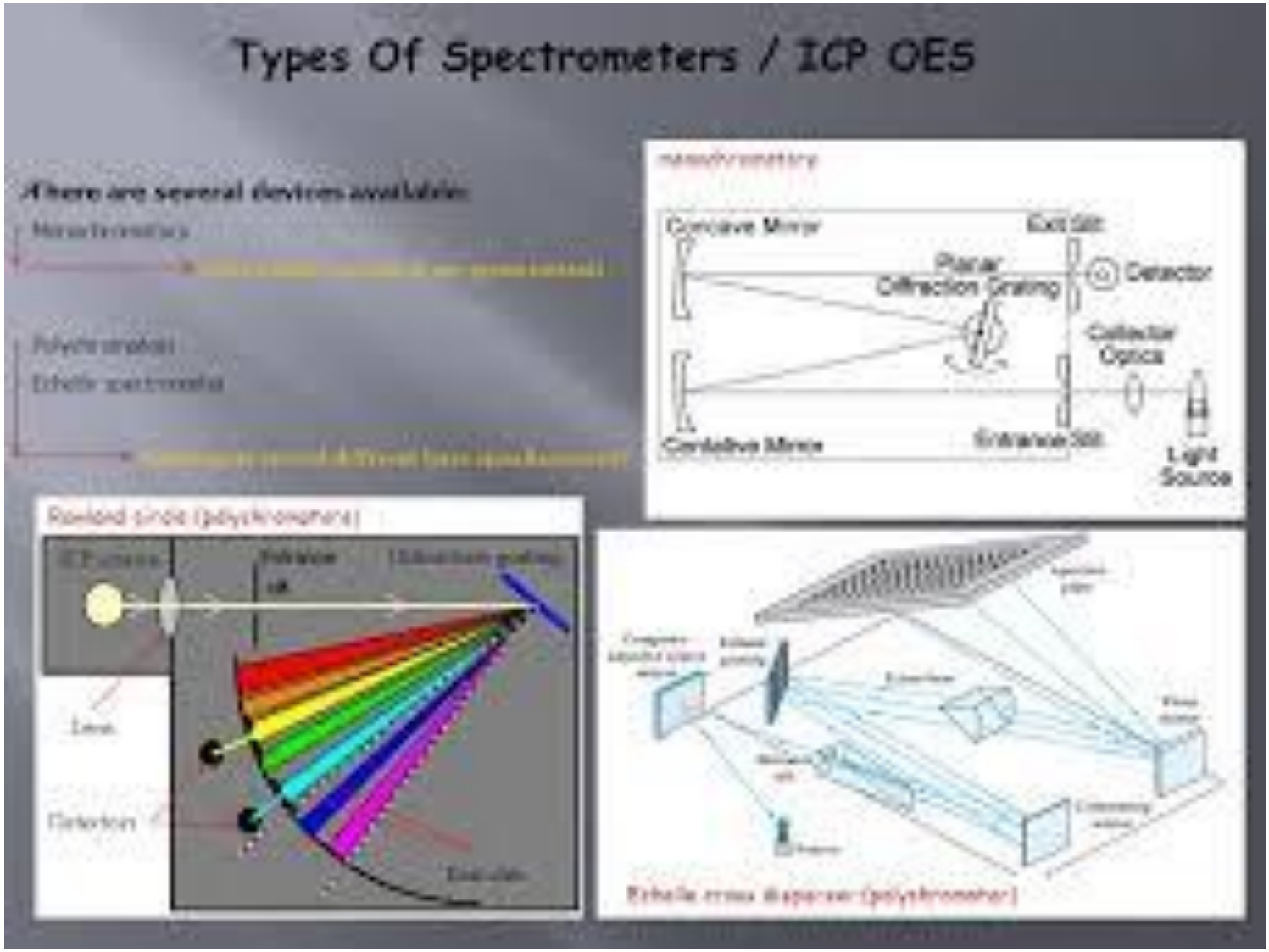


Axial view
Central
Channel



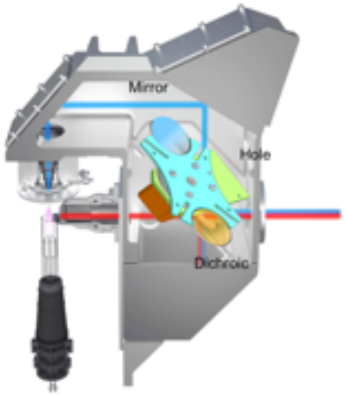


Wavelength Separation

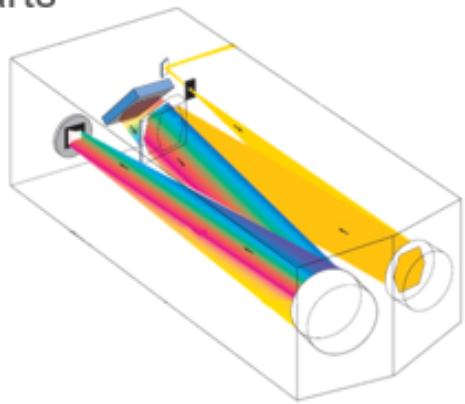


Agilent 5110 SVDV ICPOES

Synchronous Measurement of axial and radial viewed light



Simultaneous Dispersion of all Wavelengths with continuous coverage (167 to 785 nm)– Single Optic with no moving parts



Simultaneous readout of the “Zero Gas Use” CCD continuous wavelength detector

Common Interferences in ICP-OES

Running highly concentrated, complex samples brings a range of new challenges

Physical Interferences

- Aspiration rate differences between standard and sample aerosols

Long Term Drift

- Signal drift over time factors

Spectral Overlaps

- Overlaps from atomic or molecular emissions close to the analytical wavelengths

Difficult Samples

Running highly concentrated, complex samples brings a range of new challenges

Solid Build Up in Torch Injector

- Changes gas flow velocity and observation zone. Manifests as drift

Solid build up in Nebulizer

- Blockage leads to poor precision and signal drift

Organic solvent based samples

- Precision can be impacted

Physical Interferences – Particle Blockage

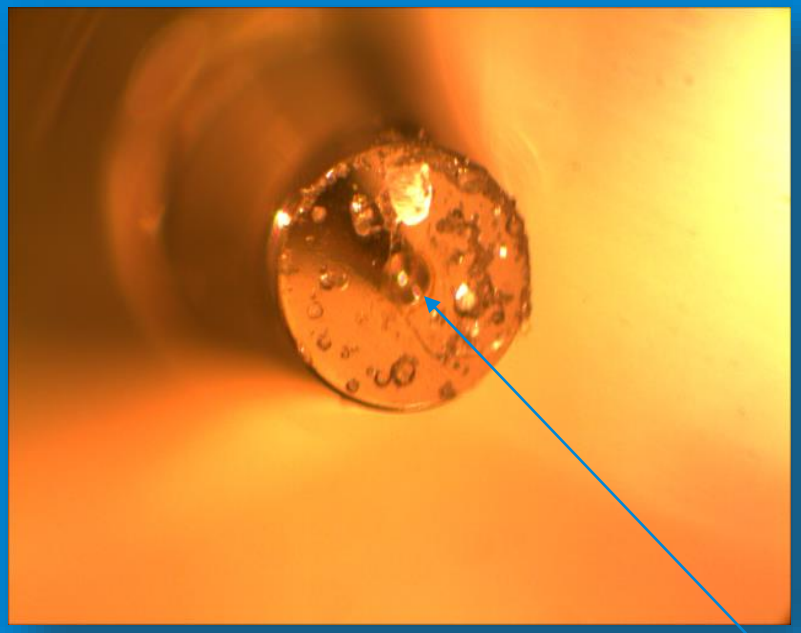
Blockage of sample introduction system

- Results from high levels of particles in samples
  - Blockage can be partial or can be full.

Blockage results in:

- Suppressed emission response compared to standard response
  - Short and long term drift.
- Enhanced emission response compared to standard response
  - Partial blockage reduces solvent to the plasma which can increase response for elements which require hot plasma conditions for ionization.
- Poor precision (% RSD)
- Not always easy to detect
  - Requires microscopic examination of the sample introduction system.

Difficult Samples



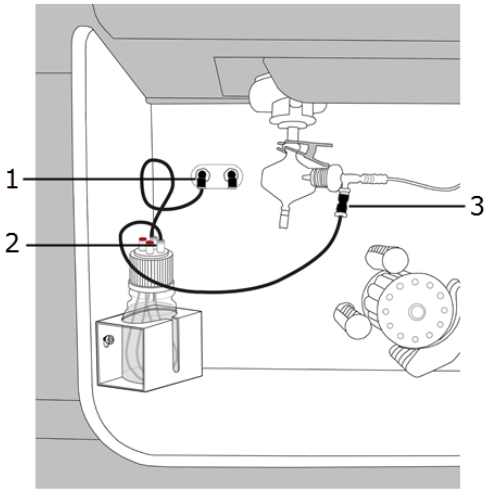
- Desolvation of high TDS sample occurs due to the fast flowing gas through the small nebulizer orifice – leads to crystalline material depositing
- High Total Dissolved Solids samples can lead to poor precision.

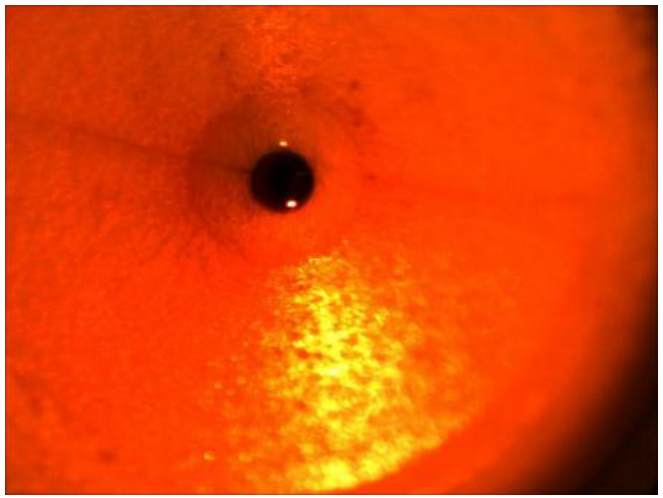
White salt crystal lodged in nebulizer orifice

Difficult Samples

Argon Humidifier

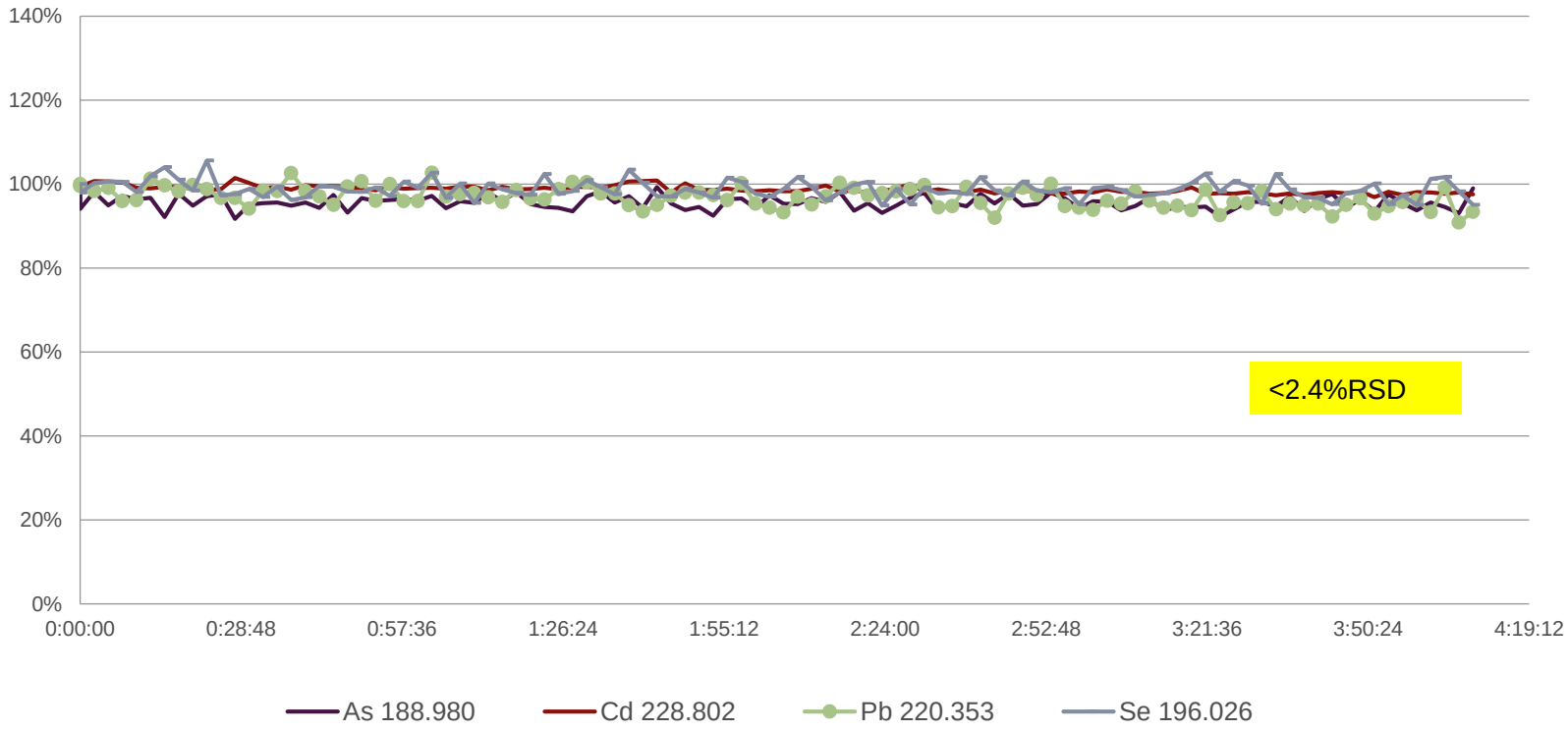
- Argon Humidifier Accessory
  - Permeable PTFE membrane allows H_2O vapor into nebulizer gas
- AHA humidifies nebulizer gas to help reduce
  - Nebulizer blockages owing to salt build-up
  - Long term **drift** from deposits in torch injector





250 ppb Multi-element in 25% NaCl

<2.4% RSD Long Term Stability with VDV Configuration (axial only mode)



OneNeb, Double pass glass cyclonic, Blk/Blk uptake pump tubing, Ar humidifier,1.8mm injector demountable torch, 15rpm peristaltic pump

Agilent – OneNebII Nebulizer

The capillary tubing extends nearly to the tip. The geometry at the tip is carefully dimensioned to allow the carrier gas (argon or nitrogen) to mix with the sample liquid. The result is an aerosol consisting of droplets with a narrow size-distribution range.

Characteristics:

Robust PFA and PEEK construction

- Inert - resistant to strong acids such as HF
- Resistant to breakage
- Molded plastic design provides improved nebulizer to nebulizer reproducibility

- Constant diameter narrow bore tubing through to nebulizer tip
  - Ideal for high solids/particulates
  - Improved tolerance to high TDS samples
- Narrow aerosol size distribution provides improved precision
- Handles a wide flow range from

  0.1 to 2 mL/min.
  - No sensitivity loss at low flow rates

Common Interferences in ICP-OES

Running highly concentrated, complex samples brings a range of new challenges

Physical Interferences

- Aspiration rate differences between standard and sample aerosols

Long Term Drift

- Signal drift over time factors

Spectral Overlaps

- Overlaps from atomic or molecular emissions close to the analytical wavelengths

Physical Interferences

Results from variation between the calibration solutions and the samples due to differences in:

- Surface tension
- Temperature
- Dissolved solids content
- Density and viscosity.

Can lead to suppressed or enhanced emission signals

- Nebulization efficiency changes for physically different solutions.

Overcoming Physical Interferences

Common Correction Choices:

- Matrix Matching between calibration solutions and samples
  - Ensure same concentration of major matrix constituents from the sample are included in the calibration standards
  - Can be difficult if the sample matrix is unknown, or very complex.
- Dilution of samples
  - Easy
  - May reduce the analyte concentration below the Detection Limit.
- Use Internal Standard Correction
- Standard Additions

Internal Standard Method (1)

Used to compensate for the effects of intensity changes due to the sample matrix.

- Variations in atom and ion population may alter the analyte intensity.

The intensity of the internal standard element is monitored in every solution of the analysis.

- Assumes the analyte and the selected Internal Standard are affected by the same amount by the matrix.

Internal Standard Method (2)

A ratio is calculated referencing the intensity of the internal standard element in the samples back to the intensity in first measurement (Blank).

Ratio = Intensity IS(BLANK) / Intensity IS (SAMPLE)

Corrected Concentration Value = C (ANALYTE) x Ratio

- Ratios > 1, analyte concentration results are adjusted lower
- Ratios < 1, analyte concentration results are adjusted higher

An Internal Standard must:

- Be added to the sample and standards in constant concentration and IS NOT inherent to the original sample.
- Be from high purity stock source.
- Not chemically interfere with test sample matrix or target elements.
- Not contribute to spectral overlap to target analyte wavelength.

Criteria for Selecting an Internal Standard

The volume of the added internal standard must be minimal

- For low level determinations, manual spiked additions are preferred.

The internal standard and analyte of interest should have similar excitation potentials

- Where possible, match the state and wavelength to the ionization potential

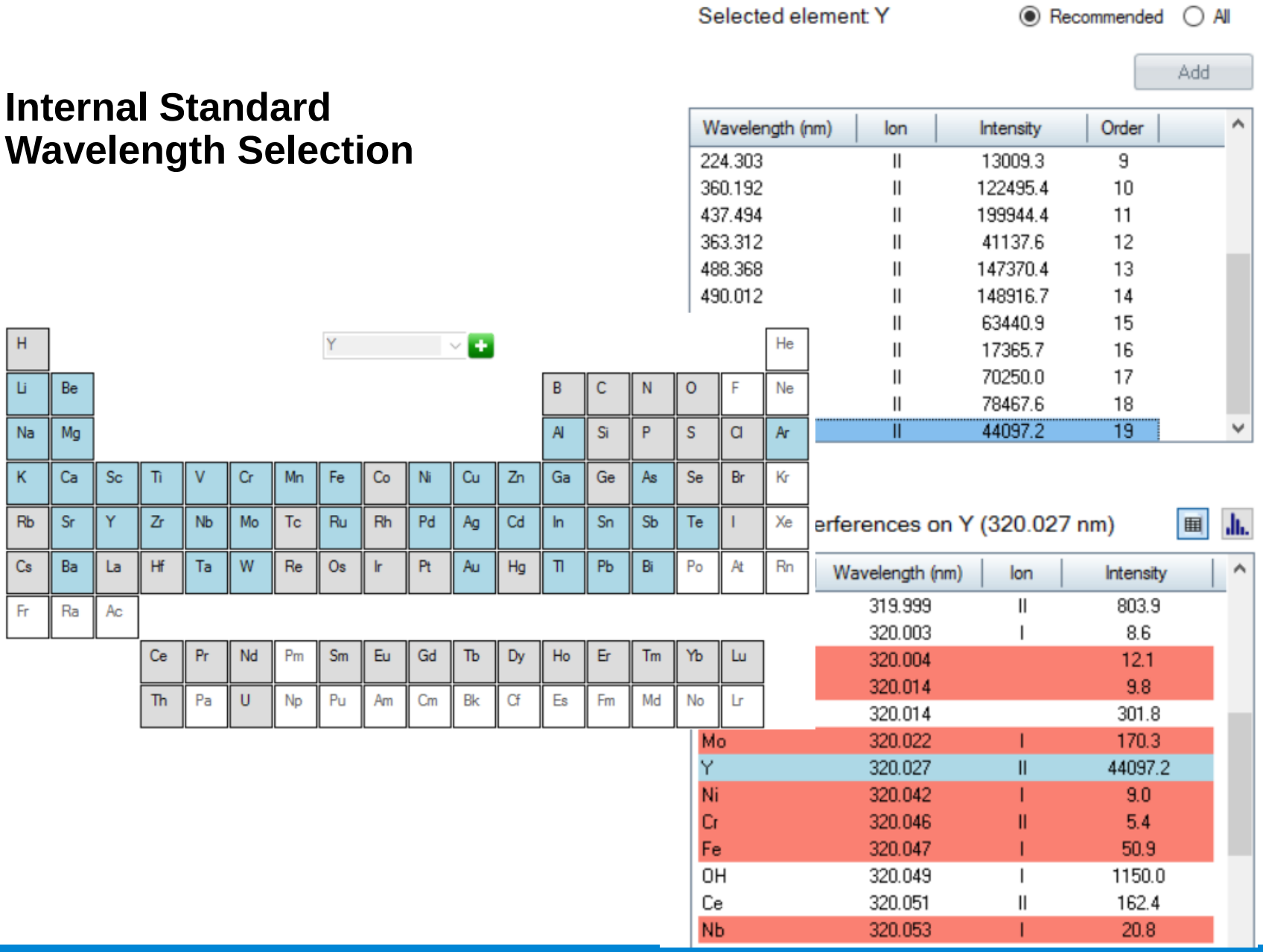
Sc, Y, In and La are commonly used.

- Not commonly present is most sample matrices.
- Multiple ionic and atomic emission lines, allowing correction with most analytes.

Ensure wavelengths are free of spectral interferences.

- Analyte doesn’t interfere with IS wavelength.
- IS doesn’t interfere with analyte of interest wavelength.

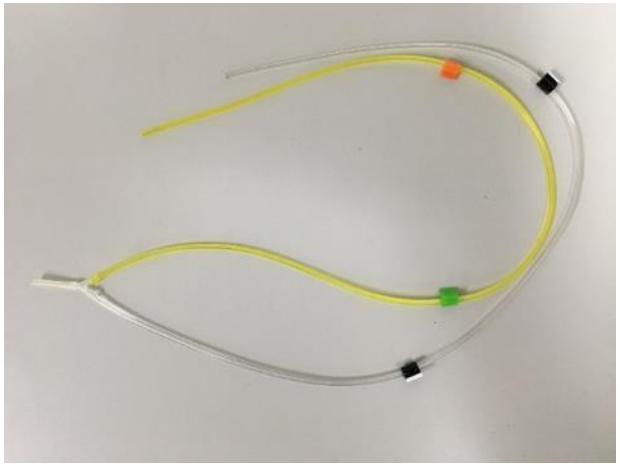
Internal Standard Wavelength Selection

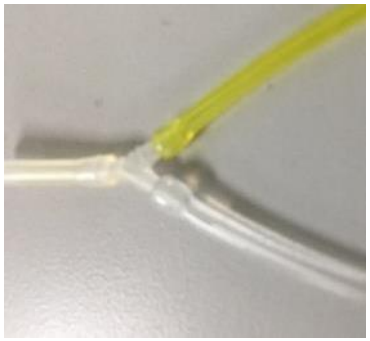


Sample Introduction Configuration

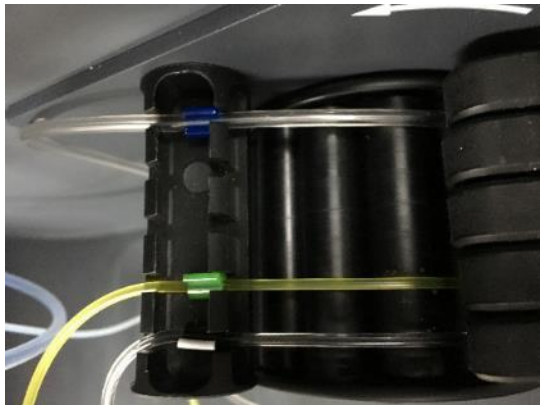
Standard setup, alternate available as needed.

- Internal standard recommended to account for differences in nebulization efficiencies between sample matrix types
  - Addition of IS Solution should be small to avoid diluting sample matrix and decreasing sensitivity

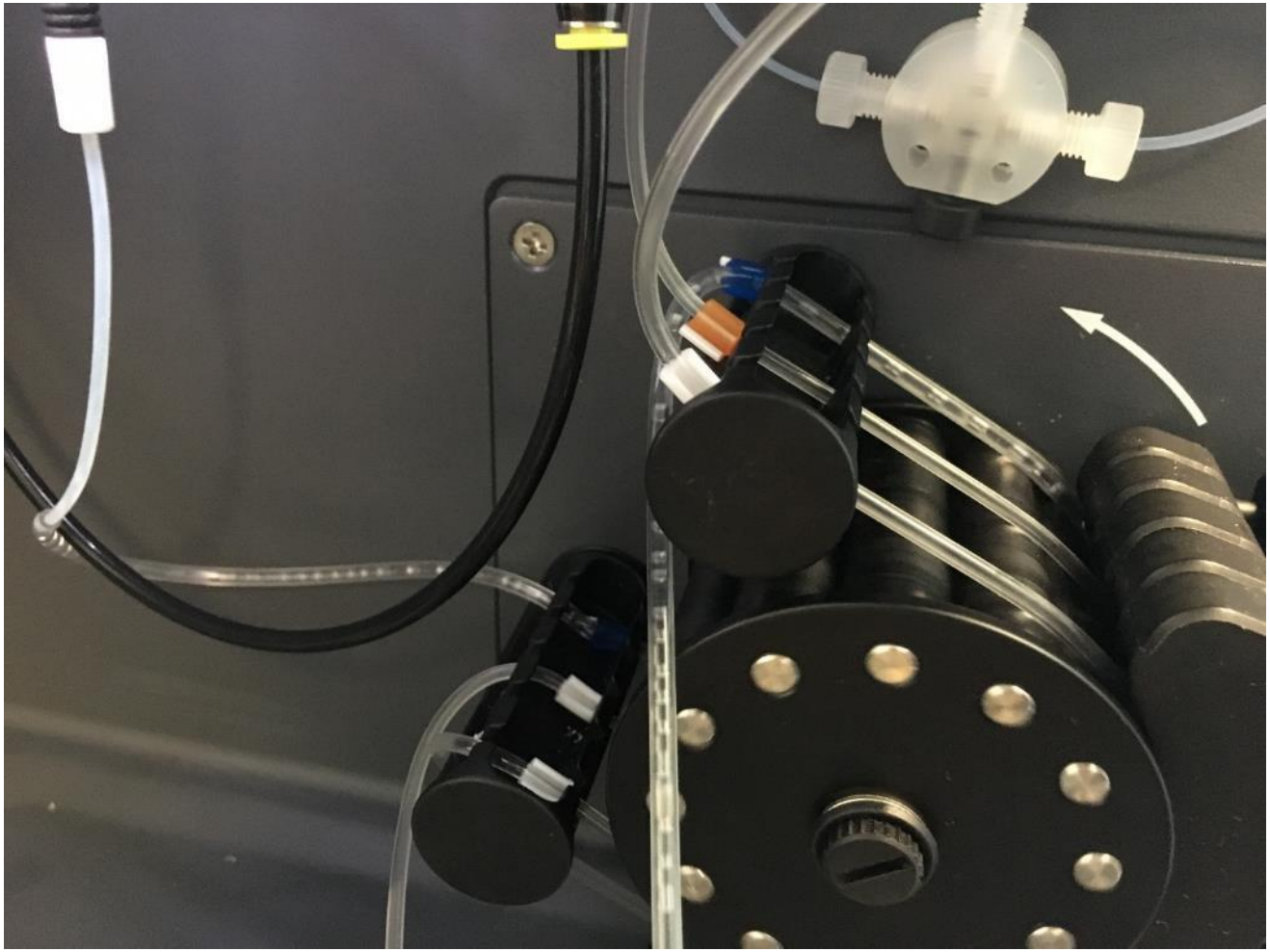




Sample and Internal Standard Tubing



Tubing	Tab color	ID
Sample	black/black	0.76mm
Internal Standard	orange/green	0.38mm



Internal Standard Monitoring without Addition

Argon is an effective internal standard when analyzing samples

1. With high concentrations of rare earth and other common spectral overlapping elements
2. Where sensitivity cannot be jeopardized
   - On-line addition is a dilution of all test solutions including the standards.
   - No dilution factor is applied as all test solutions are treated equally

Assessing Argon as an Internal Standard

Rack:Tube	Solution Label	Ar 420.067 nm Ratio	Sc 361.383 nm Ratio	Sc 364.278 nm Ratio	Y 360.074 nm Ratio	Y 371.029 nm Ratio
S1:1	*Blank*	1.000	1.000	1.000	1.000	1.000
S1:2	*std_1 10 ppb*	0.992	1.000	0.995	1.000	1.000
S1:3	*std_2 25 ppb*	1.000	1.000	0.999	1.000	1.000
S1:4	*std_3 100 ppb*	0.999	0.995	1.000	0.993	0.998
S1:5	*std_4 1000 ppb*	0.991	0.996	1.020	0.991	0.992
1:3	poly Dex 10X_1	0.892	0.911	0.901	0.900	0.908
1:3	poly Dex 10X_2	0.893	0.917	0.908	0.906	0.916
1:3	poly Dex 10X_3	0.895	0.917	0.907	0.906	0.916
1:3	poly Dex 10X_4	0.895	0.917	0.907	0.908	0.912
1:3	poly Dex 10X_5	0.917	0.929	0.922	0.919	0.922
1:3	poly Dex 10X_6	0.912	0.930	0.923	0.920	0.922
1:4	poly Dex 5X_1	0.845	0.935	0.909	0.924	0.935
1:4	poly Dex 5X_2	0.840	0.936	0.911	0.924	0.937
1:4	poly Dex 5X_3	0.841	0.937	0.910	0.925	0.934
1:4	poly Dex 5X_4	0.840	0.941	0.912	0.929	0.935
1:4	poly Dex 5X_5	0.839	0.937	0.912	0.925	0.936
1:4	poly Dex 5X_6	0.836	0.937	0.912	0.925	0.935

Standard Curve, 2% HNO_3

10X dilution Polydextrose 2% HNO_3

5X dilution Polydextrose 2% HNO_3

Argon as an Internal Standard

Great for use when the IS selected in present in the standard or sample

The physical matrix affects can be resolved without repreparation of solutions

Solution Label	Ar 420.067 nm Ratio	Ar 703.025 nm Ratio	Ar 737.212 nm Ratio	Sc 357.634 nm Ratio	Sc 364.278 nm Ratio	Y 360.074 nm Ratio	Y 371.029 nm Ratio
Blank	1.00 !	1.00 !	1.00 !	1.00 !	1.00 !	1.00 !	1.00 !
IQ 1	0.98	0.99	1.00	6.66	6.71	6.18	6.26
IQ 1A	0.96	0.97	0.96	28.75	28.82	26.19	26.13
IQ 2	1.02	1.02	1.02	0.99	1.10	0.99	0.98
IQ 3	1.00	1.01	1.00	1.01	1.00	1.00	1.00
1 ppm qc 27	1.01	1.01	1.02	0.99	1.04	0.98	0.98
1000 ppm Co	0.92	0.92	0.93	0.97	0.82	1.05	1.02
cobalt 1	0.42	0.43	0.45	0.75	0.36	1.17	0.75
cobalt 2	0.43	0.43	0.45	0.75	0.36	1.17	0.75
cobalt 3	0.43	0.43	0.46	0.75	0.36	1.17	0.75
cobalt 4	0.42	0.43	0.45	0.75	0.36	1.17	0.75
cobalt 5	0.43	0.44	0.46	0.74	0.36	1.15	0.74
cobalt spike 1	0.43	0.44	0.46	1.57	0.92	1.93	1.51
cobalt spike 2	0.42	0.42	0.45	1.58	0.92	1.94	1.51
cobalt spike 3	0.43	0.43	0.46	1.57	0.92	1.92	1.51
cobalt spike 4	0.44	0.43	0.46	1.55	0.91	1.91	1.49
cobalt spike 5	0.44	0.44	0.47	1.55	0.91	1.91	1.49
1000 ppm Co	0.94	0.95	0.95	0.93	0.62	1.00	0.97

Interpretation of IS Results (2)

Internal standard ratio for samples

- Close to 1.00

A ratio is calculated referencing the intensity of the internal standard in the samples back to the intensity in first measurement (Blank)

  - Ratio = IIS(BLANK) / IIS (SAMPLE)

When ratio is not 1.00, the intensity of the analyte being corrected is adjusted accordingly

  - Corrected Concentration Value = C (ANALYTE) x Ratio

- Ratios > 1, the analyte concentration results are adjusted lower
- Ratios < 1, the analyte concentration results are adjusted higher

Interpreting Results

Sample Labels	V 309.310 ppm	V 311.070 ppm	V 311.837 ppm	V 311.837... ppm	Sc 335.372
rinse	-0.0013u	-0.0063u	-0.0234u	-0.0101u	0.9930
0.025_1	0.0193	0.0124	0.0028	0.0129	0.9937
0.025_2	0.0190	0.0122	0.0023	0.0132	0.9944
0.025_3	0.0191	0.0125	0.0016	0.0125	0.9940
0.025_4	0.0191	0.0116	0.0018	0.0121	0.9937
0.050_1	0.0655	0.0533	0.0584	0.0627	0.9984
0.050_2	0.0649	0.0537	0.0568	0.0625	0.9944
0.050_3	0.0647	0.0537	0.0570	0.0626	0.9956
0.050_4	0.0649	0.0547	0.0573	0.0624	0.9945
Sample 9	-0.0022u	-0.0066u	-0.0238u	-0.0100u	1.005
AL1_1	14.20	13.01	14.82	14.87	1.001
AL1_2	14.34	13.15	14.95	15.01	1.002
AL1_3	14.38	13.20	15.00	15.07	1.005
AL1_1SPk	25.79	25.43	25.52	26.24	0.9906
AL1_2SPk	25.74	25.37	25.46	26.19	1.001
AL1_3SPk	25.68	25.30	25.42	26.11	1.003
NIST1643c_1	27.24	23.34	29.32	28.81	1.009
NIST1643c_2	27.26	23.37	29.30	28.82	1.012
NIST1643c_3	27.17	23.32	29.08	28.68	1.019
NIST1643c_4	27.15	23.29	29.19	28.72	1.025
Lab Control Sample	2.193	2.064	2.376L	2.270	1.022
Naphtha_Blk_1	0.0052	-0.0091u	0.0017	-0.0066u	0.6575
Naphtha_Blk_2	0.0057	-0.0094u	0.0033	-0.0057u	0.6419
Naphtha_Blk_3	0.0052	-0.0091u	0.0025	-0.0068u	0.6355
Naphtah Spike	0.3974	0.3774	0.5059	0.4573	0.6798
Naphtha Sample	0.0129	-0.0095u	0.0277	-0.0035u	0.4418
Naphtha Sample	0.0130	-0.0096u	0.0265	-0.0021u	0.4516
Distillate	-0.0014u	-0.0069u	-0.0222u	-0.0115u	0.8879
Distillate Spike	0.5767	0.5340	0.6676	0.6189	0.9048
Distillate	-0.0004u	-0.0066u	-0.0186u	-0.0083u	0.8849
Distillate	-0.0027u	-0.0076u	-0.0217u	-0.0116u	0.8865

Matrix # 1

Matrix # 2

Matrix # 3

Matrix # 4

Common Interferences in ICP-OES

Running highly concentrated, complex samples brings a range of new challenges

Physical Interferences

- Aspiration rate differences between standard and sample aerosols

Long Term Drift

- Signal drift over time factors

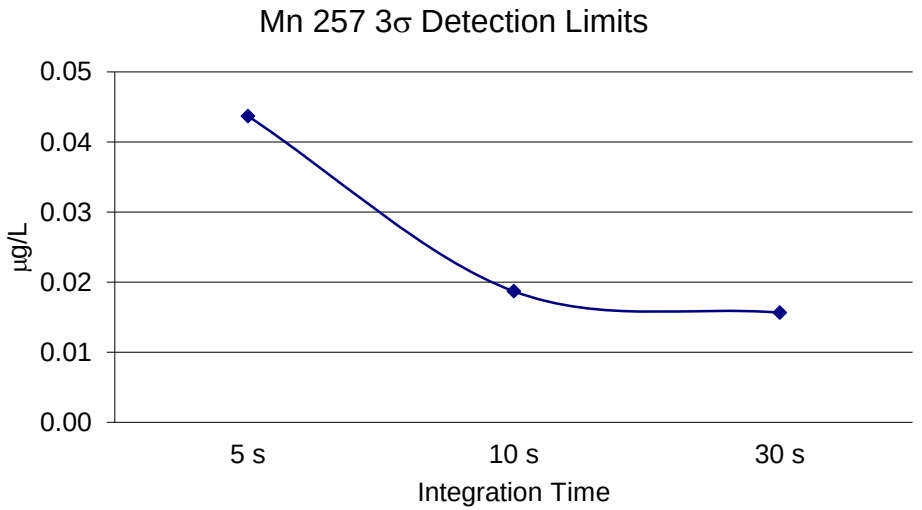
Spectral Overlaps

- Overlaps from atomic or molecular emissions close to the analytical wavelengths

General Sensitivity Improvement

Detection Limits and Integration Time

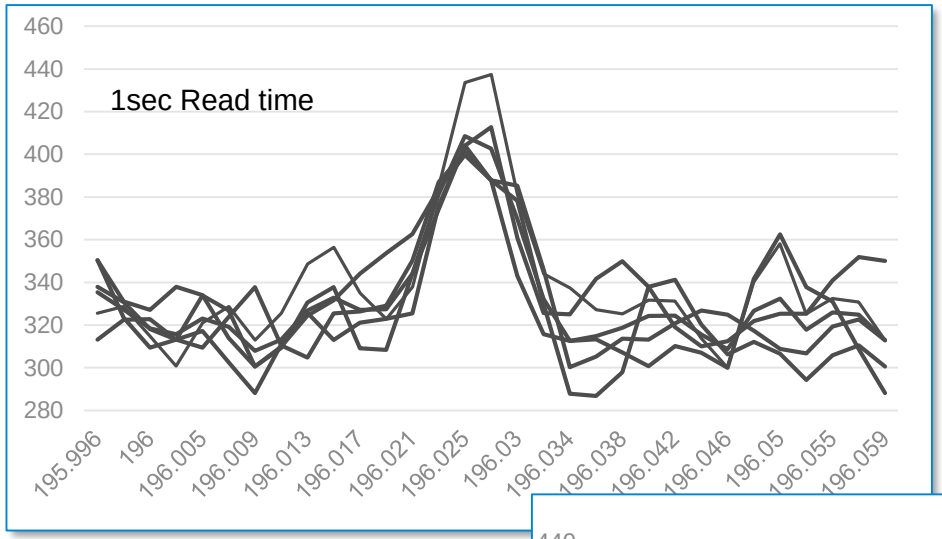
- Solid-state detectors such as the VistaChip^2 CCD detector
  - Improve S/N and detection limits by averaging noise over time
  - DL α 1/√t

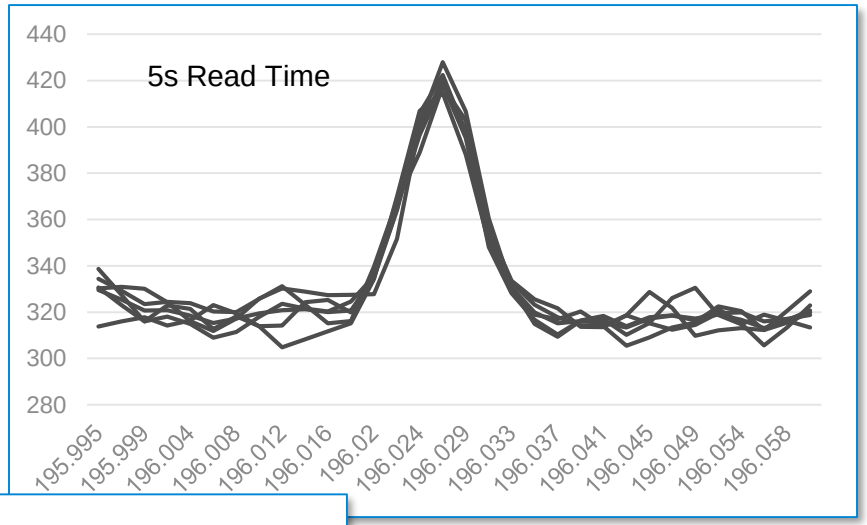


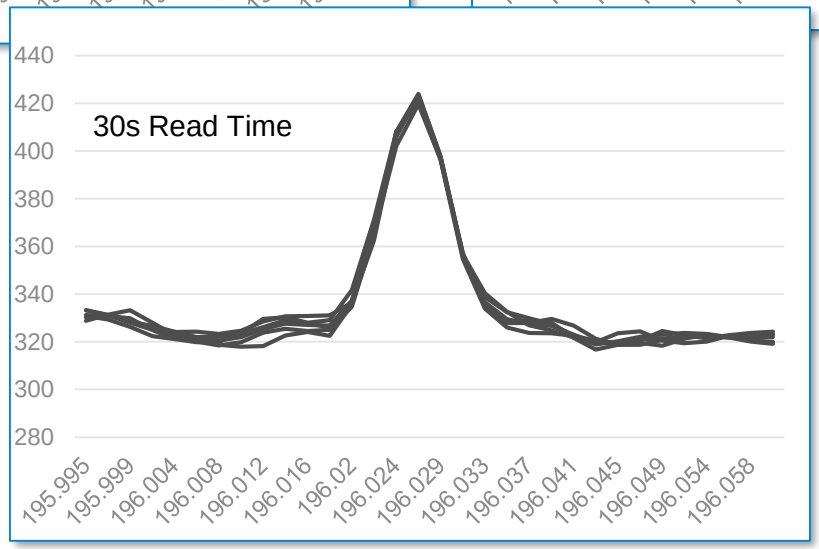
Improve Sensitivity (S/N)

Detection Limits and Read Time

Overlay multiple 50ppb readings of Se at 196.026nm







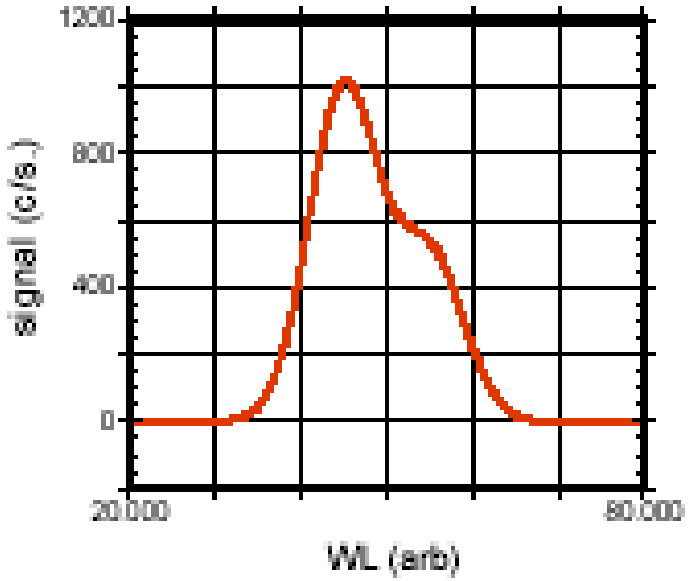
DL α STDEV

Spectral Interferences (1)

Defined as the overlap of emission from more than one atomic or molecular species.

- Spectral interference occurs if wavelength separation of interfering species < instrument resolution.
- Results in enhancement of results (errors) if left uncorrected.

Typical spectral overlap problem



Spectral Interferences (2)

Spectral Interferences arise from

- Other elements present in the samples.
  - Major matrix elements
  - Elements with large number of intense lines in their spectra.

- Occasionally from plasma or solvent
  - Such interferences are typically constant but degrade detection limits.
  - Higher background signal and noise

Spectral Interferences (3)

Concomitant background species

- Argon 300 - >780 nm
- OH 281.0 - 294.5 nm & 306.0 - 324.5 nm
- NH 302.2 - 380.4 nm (336.0 most intense)
- NO 195.6 - 345.9 nm (200 to 280 most intense)

Correcting for Spectral Interferences

- Use Inter-Element Correction (IEC)
  - e.g. Recommended in US EPA Method 200.7 for waters & wastewaters.

- Dilute sample.
  - Only if analyte concentration is high in comparison to interference concentration
- Select an alternative wavelength.
  - Choose a wavelength which is interference free.
  - Previous examples of Cr and Sn

- Apply Background correction.
  - Choice of Off-peak or Fitted.

- Use Fast Automated Curve-fitting Technique (FACT).

Inter-Element Correction, IEC (1)

Preferred correction technique with direct spectral overlap

Must specify the element as an interference when selecting the wavelength.

IEC method determines relationship between interference concentration and response at analyte wavelength.

- Characterized by making measurements at that wavelength on a series of spectroscopic pure solutions
- For samples, must determine concentration of the interfering element, as well the analyte
  - The interference conc. is determined using an un-interfered analyte line

The correction factor is used to subtract the contribution of interference from the apparent analyte concentration.

Inter-Element Correction, IEC (2)

IEC determines both the IEC factors and the analytical calibration of the interference and analyte.

Can calibrate for a range of interference concentrations

No limit to the number of interference elements that can be used in the calculation of the IEC

Run the IEC solutions at the start of the analysis.

IEC factors are calculated automatically.

Inter-Element Correction Formula

IEC factor measures degree to which the interference signal contributes to the analyte.

- The analyte and interference concentrations used to determine the IEC factors are not important
- However, concentrations need to be within the Linear Dynamic range

$$\textit{Corr. Conc.} = \textit{Apparent Conc.} - (F_{IEC} \times \textit{Conc. Interfering Element})$$

$$F_{IEC} = \frac{\text{Apparent Analyte Intensity (Caused by Interference)}}{\text{Interference Concentration}} \times \frac{\text{Analyte Concentration}}{\text{Analyte Intensity}}$$

Inter-Element Correction (IEC)

Interferents

Number of standards: 7

Solution Label	Rack:Tube	Al	Co	Cr	Fe	Mn	Mo
		ppm	ppm	ppm	ppm	ppm	ppm
Al IEC	S1:10	500.00000000					
Co IEC	S1:11		20.00000000				
Cr IEC	S1:12			20.00000000			
Fe IEC	S1:13				500.00000000		
Mn IEC	S1:14					25.00000000	
Mo IEC	S1:15						20.00000000
V IEC	S1:16						

IEC is worksheet based.

IEC solutions are automatically measured and processed via the sample sequence.

Inter-Element Correction Factors

Sample Labels	Cd 214.439 mg/L	Fe 238.204 mg/L
IEC Blank	0.000000	0.000000
Analyte 1	0.100000	
Interferent 1		500.000
Blank	0.000000	0.000000
Standard 1	0.100000	
Standard 2	1.00000	
Standard 3	10.0000	
Sample 1		
Sample 2		
Sample 3		

Measure blank solution.

Measure solution of pure analyte.

Measure solution of pure interference.

- Determine contribution of Fe at Cd 214 nm wavelength – IEC factor
- Generate calibration curve for Fe at interference-free 238 nm line.

IEC Example for Fe Overlap on Cd 214 nm

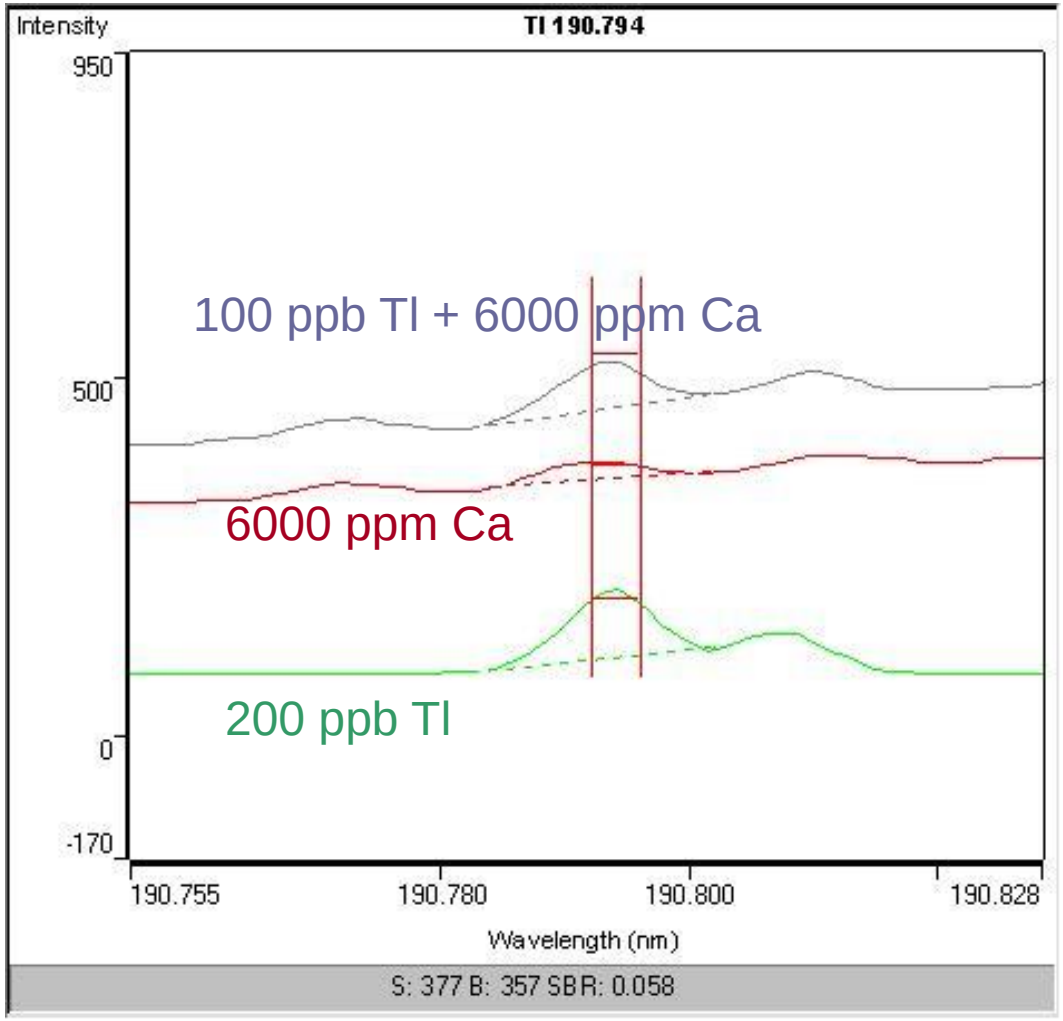
Example:	Interference Conc. (Fe)	= 500 mg/L
	Analyte Conc. (Cd 214 nm)	= 0 mg/L
	Apparent Conc. (Cd)	= 0.5 mg/L
	IEC factor	= 0.002

Corrected Conc = Uncorrected Conc – (IEC factor x Int. Conc)

Example:

Uncorrected Analyte Conc (Cd 214 nm)	= 1.0 mg/L
Measured Interference Conc (Fe 238 nm)	= 750 mg/L
Corrected Conc = 1.0 – (0.002 x 750 mg/L)	= 1.0 – 0.50
	= 0.50 mg/L

Inter-Element Correction - Ca Overlap on Tl at 190.794 nm



Observed concentrations with IEC

99 ppb

4 ppb

204 ppb

Disadvantages of IEC

Tedious and time-consuming work to calculate interference factors before actually running program

There could be more than one interference at the line of interest complicating the factors.

If any parameters in program are altered, the correction factors should be re-examined e.g.

- Pump rate, stabilization time, replicate read time, integration time
- ALL plasma parameters.

Factors are calculated based on the known concentration of the interfering elements.

- Best to work within the linear range of both the analyte and interference to avoid bias.

Internal Standard corrections (where used) must be applied BEFORE IEC factors are calculated.

Overcoming Background Interferences

Background Correction

- Off-peak
  - User selects background points.

Modern Corrections

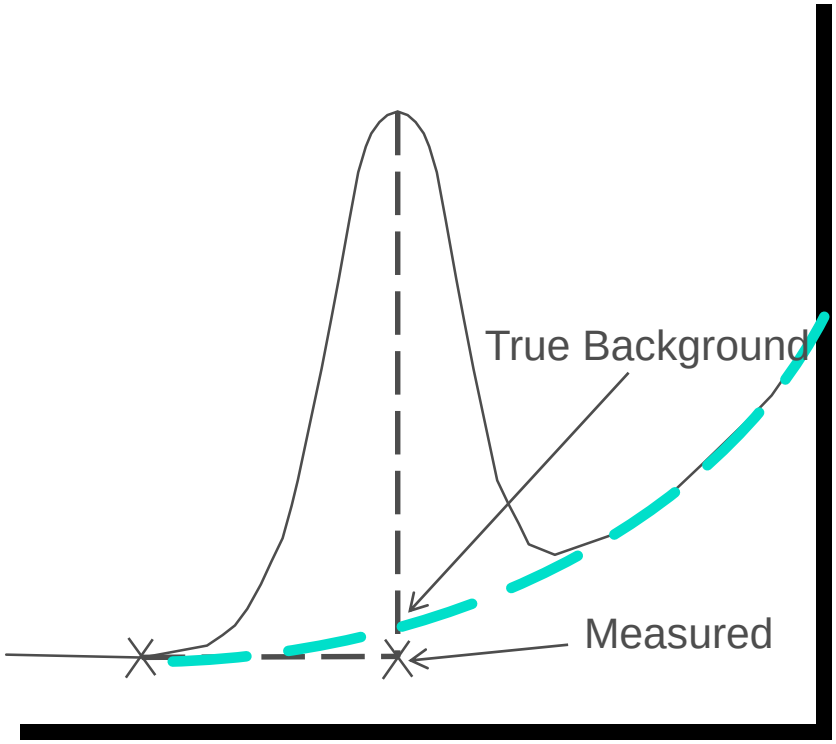
Fitted

  - Peak shaped functions will be fitted to the analyte peak.

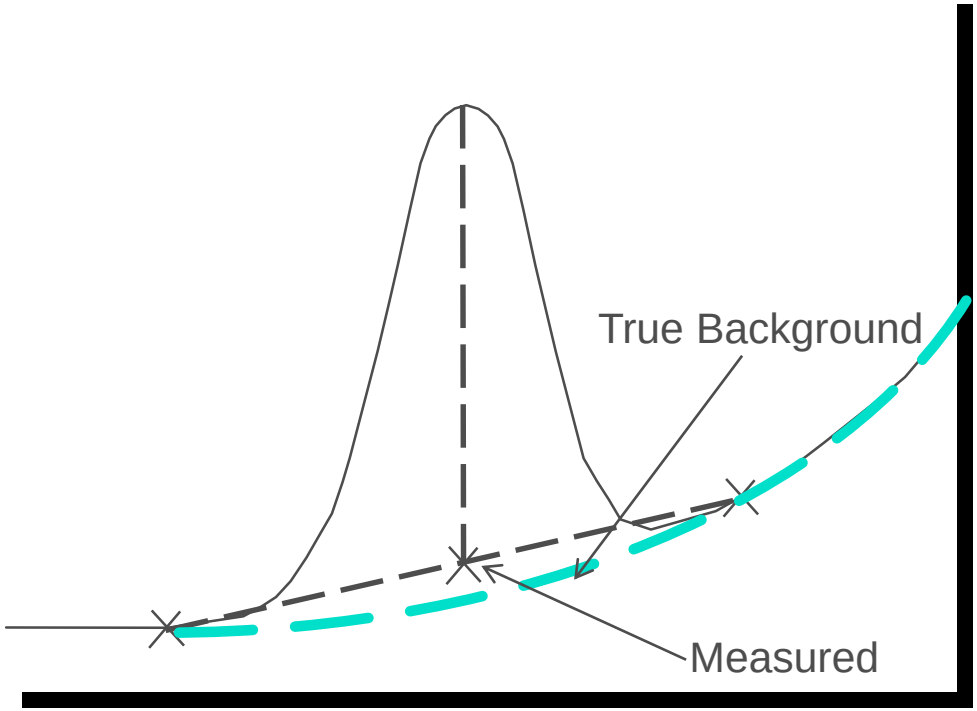
- Fast Automated Curve-fitting Technique (FACT)
  - Provides real-time spectral correction using Gaussian peak models.

Select an alternate wavelength for measurement.

Off-peak Background Correction (2)

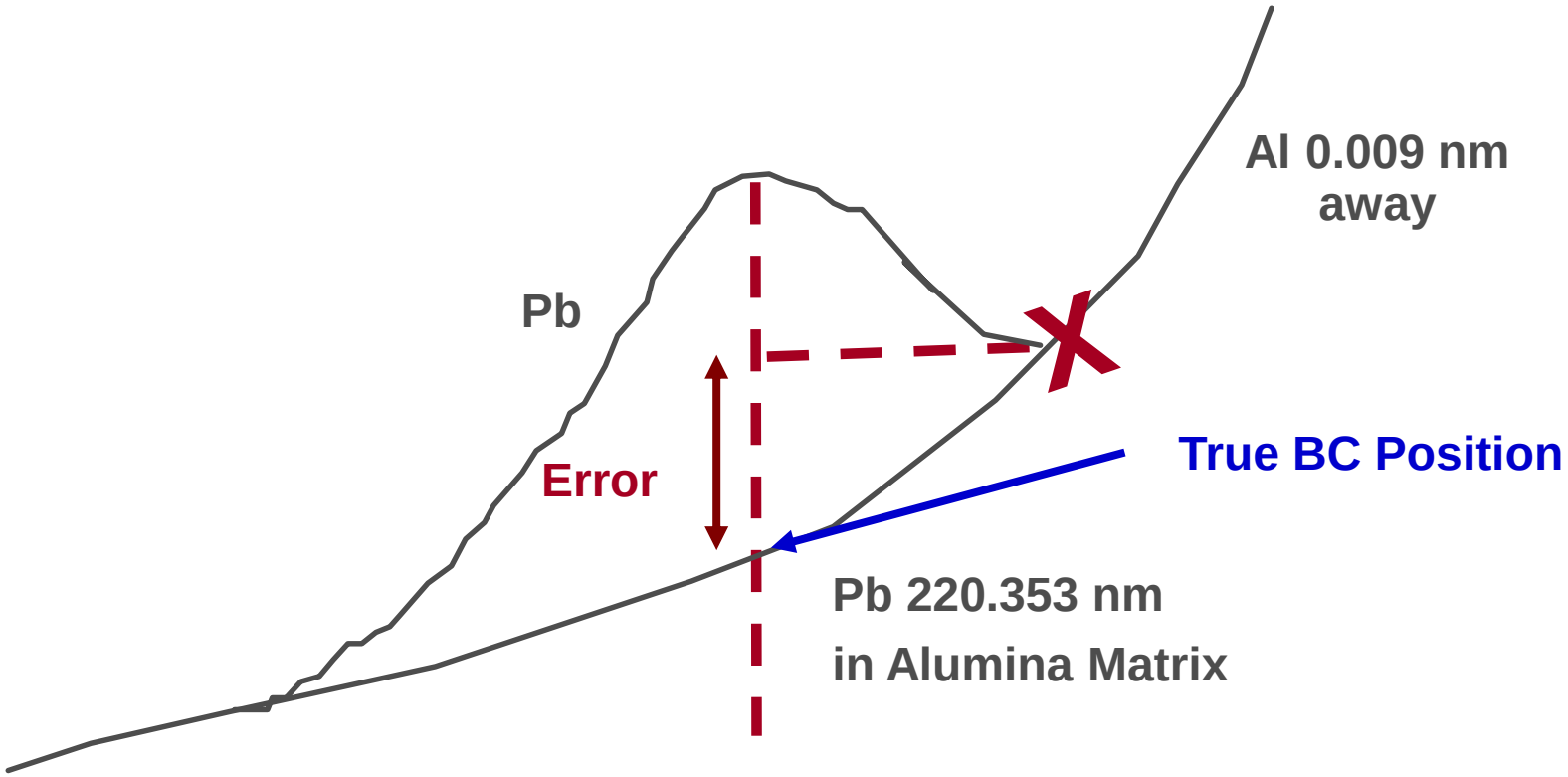


Single Point Off-peak



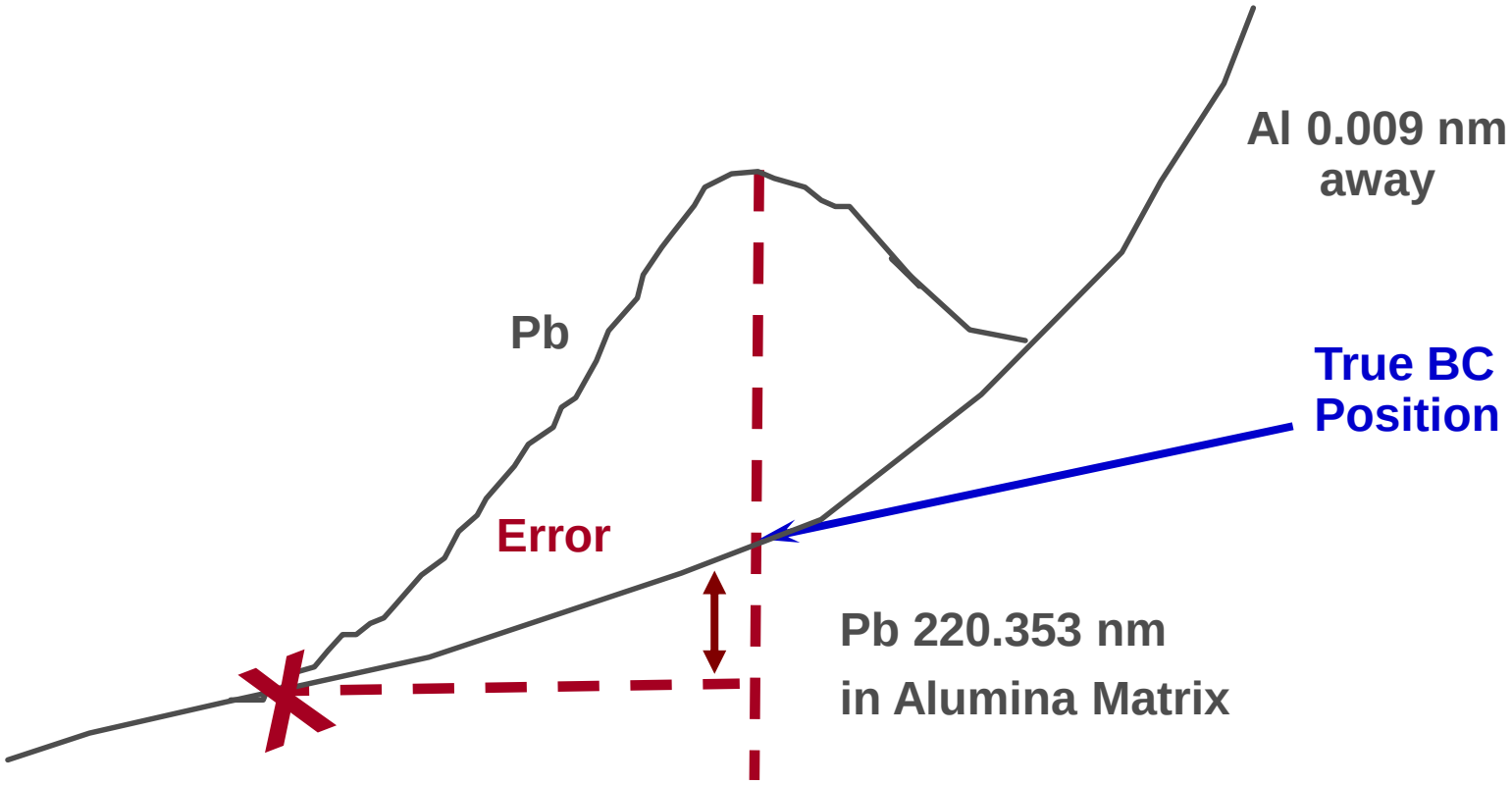
Off-peak using Both Sides

Off Peak Background Correction (3)



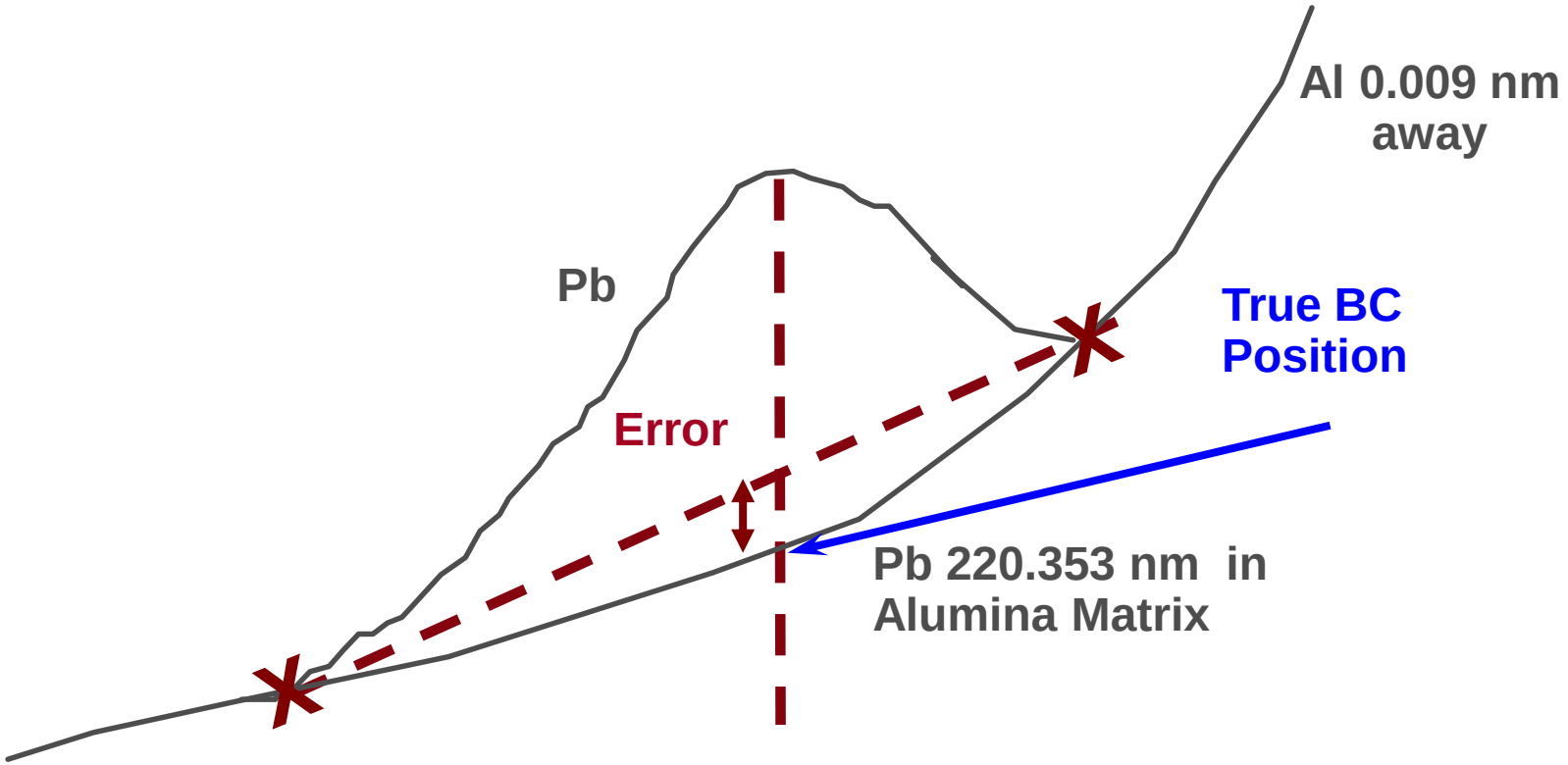
Over Correction with OPB - Right Only

Off Peak Background Correction (4)



Under Correction with OPB - Left Only

Off-Peak Background Correction (5)



Over-correction with OPB – left and right

Fitted Background Correction (1)

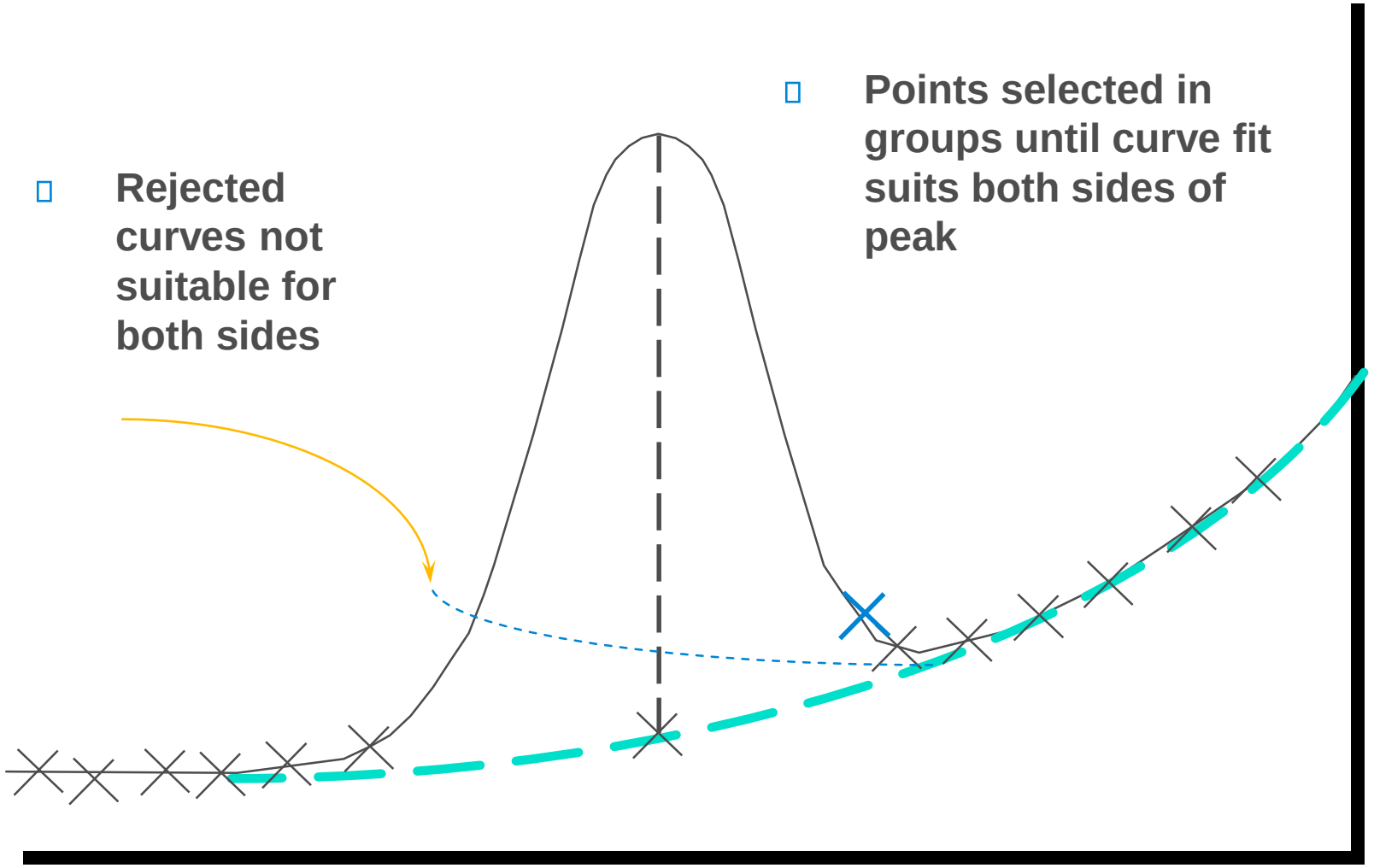
Works by creating a model for the measured spectrum.

Model is composed of:

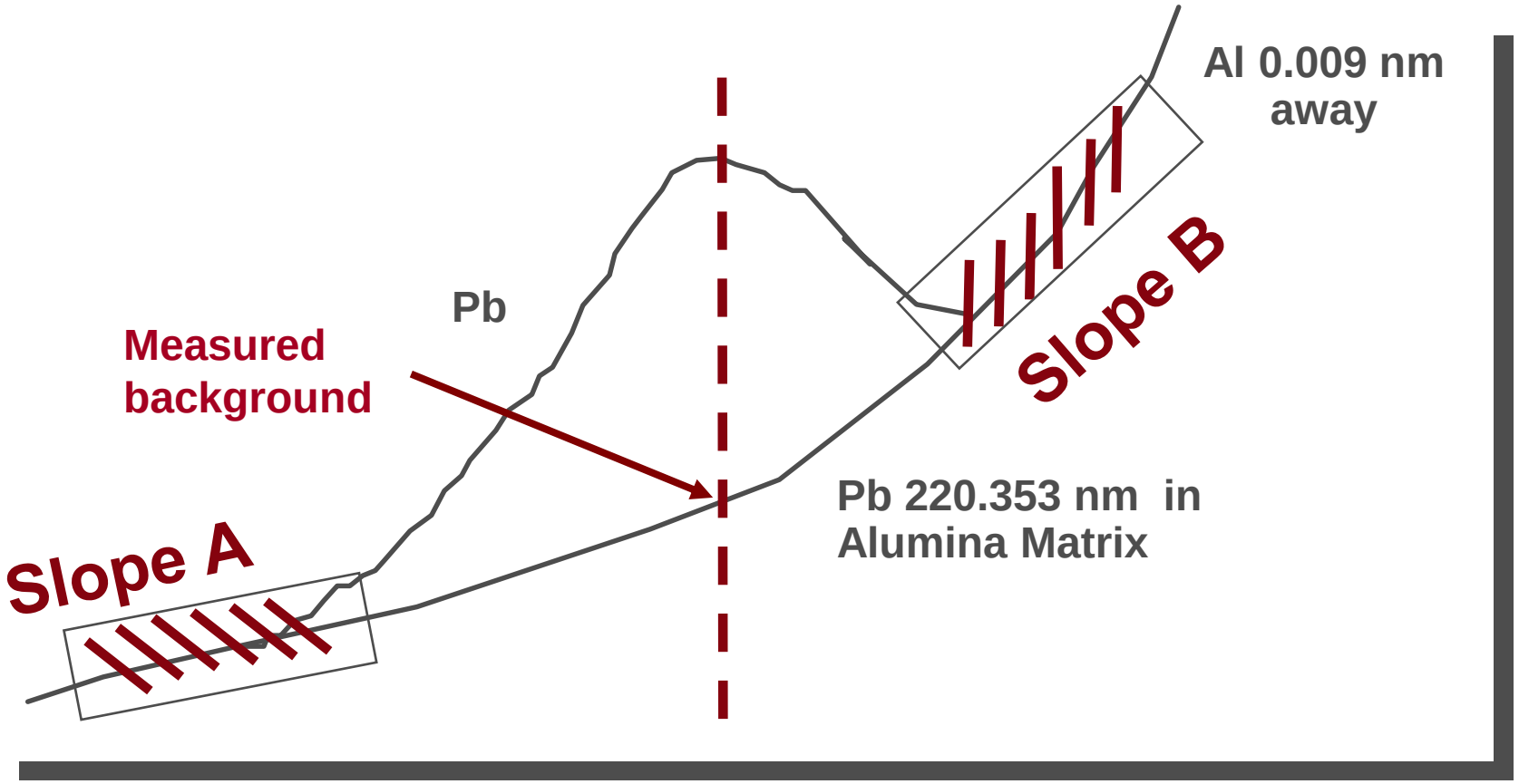
- An offset component to model unstructured plasma background.
- A slope component to model wings of large distant peaks.
- Three Gaussian peak components to model
  - Analyte peak
  - Possible interference peak to the left
  - Possible interference peak to the right.
- Uses an iterative procedure to estimate the width and positions of peaks.
- Combined with least squares technique to estimate the magnitudes of offset, slope and peak heights.
- Once model has been fitted, the analyte peak component is removed from the equation
  - Model for background remains.

Fitted Background Correction (2)

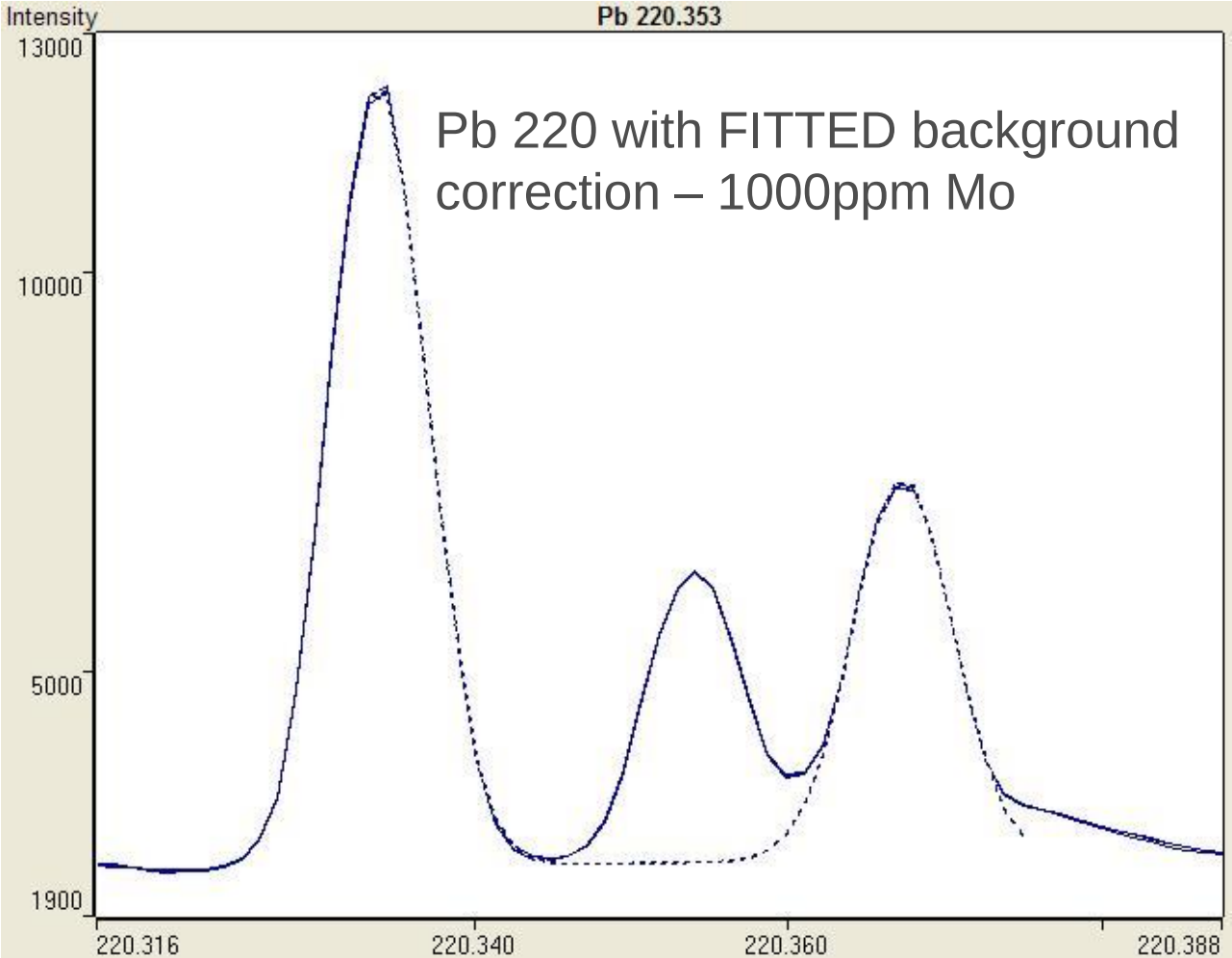
- Points selected in groups until curve fit suits both sides of peak
- Rejected curves not suitable for both sides



Fitted Background Correction (3)



Off-Peak Versus Fitted



Results – Target Elements Requested

Units = Mg/L	Agilent 5110 VDV ICP-OES axial mode		
Solution Label	Wastewater	Matrix Spike Wastewater	%REC
Al 167.019 nm	< 0.025	0.99	97
Al 394.401 nm	< 0.025	1.06	103
Al 396.152 nm	< 0.025	1.08	106
B 249.678 nm	1.13	2.16	103
B 249.772 nm	1.13	2.16	103
Na 588.995 nm	0.58	1.59	101
Na 589.592 nm	0.56	1.58	102
Si 251.611 nm	0.07	1.12	105
Si 288.158 nm	0.07	1.12	105
Sn 189.925 nm	< 0.050	1.02	102
Sn 283.998 nm	< 0.050	1.82	182
Ti 334.941 nm	0.49	1.42	93
Ti 336.122 nm	0.49	1.42	93

- Confirmatory wavelength for each element with NO time penalty
- Confidence in the measurement
- Matrix Spike recovery 100 ± 6%

Wavelength Confirmation

Simultaneous instruments typically have more than one wavelength available for each target element

Confirmatory wavelengths are helpful

Sn concentrations determined at both 189.925 nm and 283.998 nm were less than detection limit

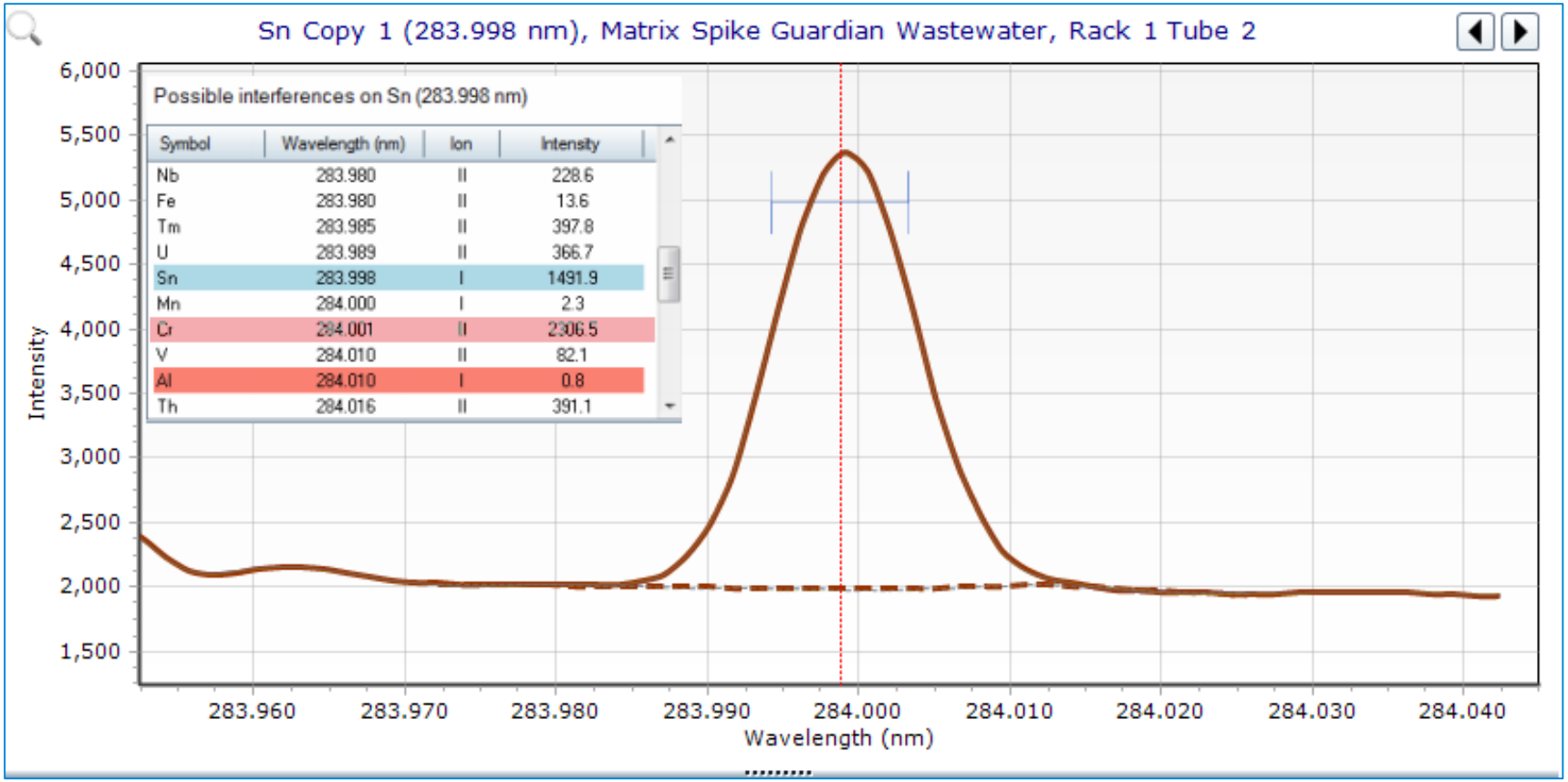
Matrix Spike of 1.0 ppm was determined to be 1.82 ppm at the Sn 283.998 nm

Solution Label	Sn FBC 189.925 nm ppm	Sn FBC 283.998 nm ppm
Blank	0.00	0.00
Standard 1		
Standard 2		
Standard 3		
Standard 4	0.10	0.10
Standard 5	1.00	1.00
Standard 6	10.00	10.00
blank	0.00	0.01
Wastewater	0.00	0.01
Matrix Spike Wastewater	1.02	1.82

Sn 283.998 nm

Potential interferences from Cr and Al

When Cr is present and Sn is NOT, an unexpected positive result for Sn can be measured. Peaks are separated by 3 picometers and the Cr intensity is greater than the Sn intensity



Confirmation of Interference

Analyze a solution with target analyte only

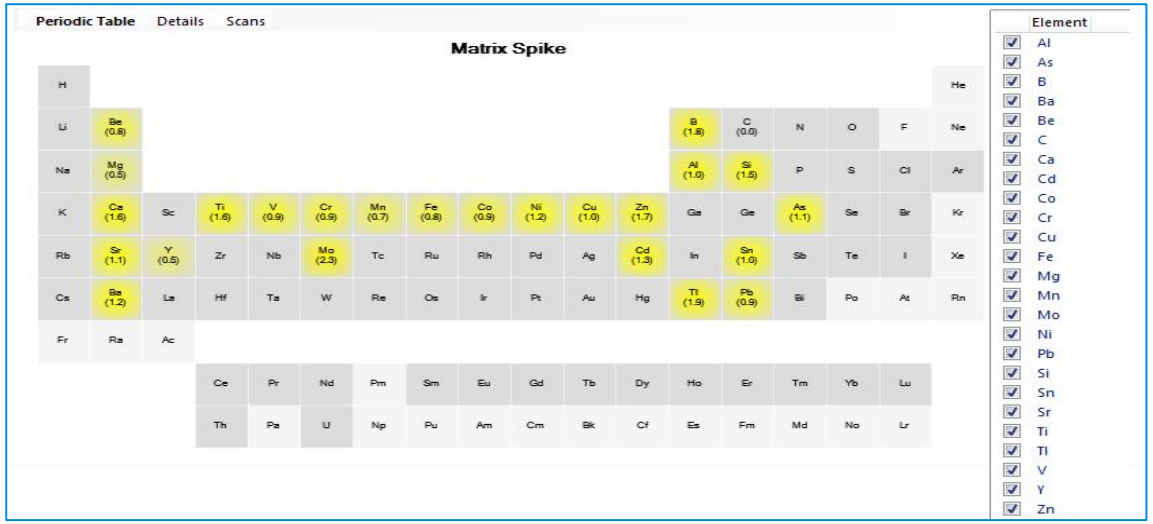
Analyze solution of interference only

Solution Label	Sn FBC 189.925 nm ppm	Sn FBC 283.998 nm ppm
Blank	0.00	0.00
Standard 1		
Standard 2		
Standard 3		
Standard 4	0.10	0.10
Standard 5	1.00	1.00
Standard 6	10.00	10.00
blank	0.00	0.01
Wastewater	0.00	0.01
Matrix Spike Wastewater	1.02	1.82
Std 2	0.00	0.78
Std 5 Sn	1.00	0.99
Cr	0.00	12.72 o

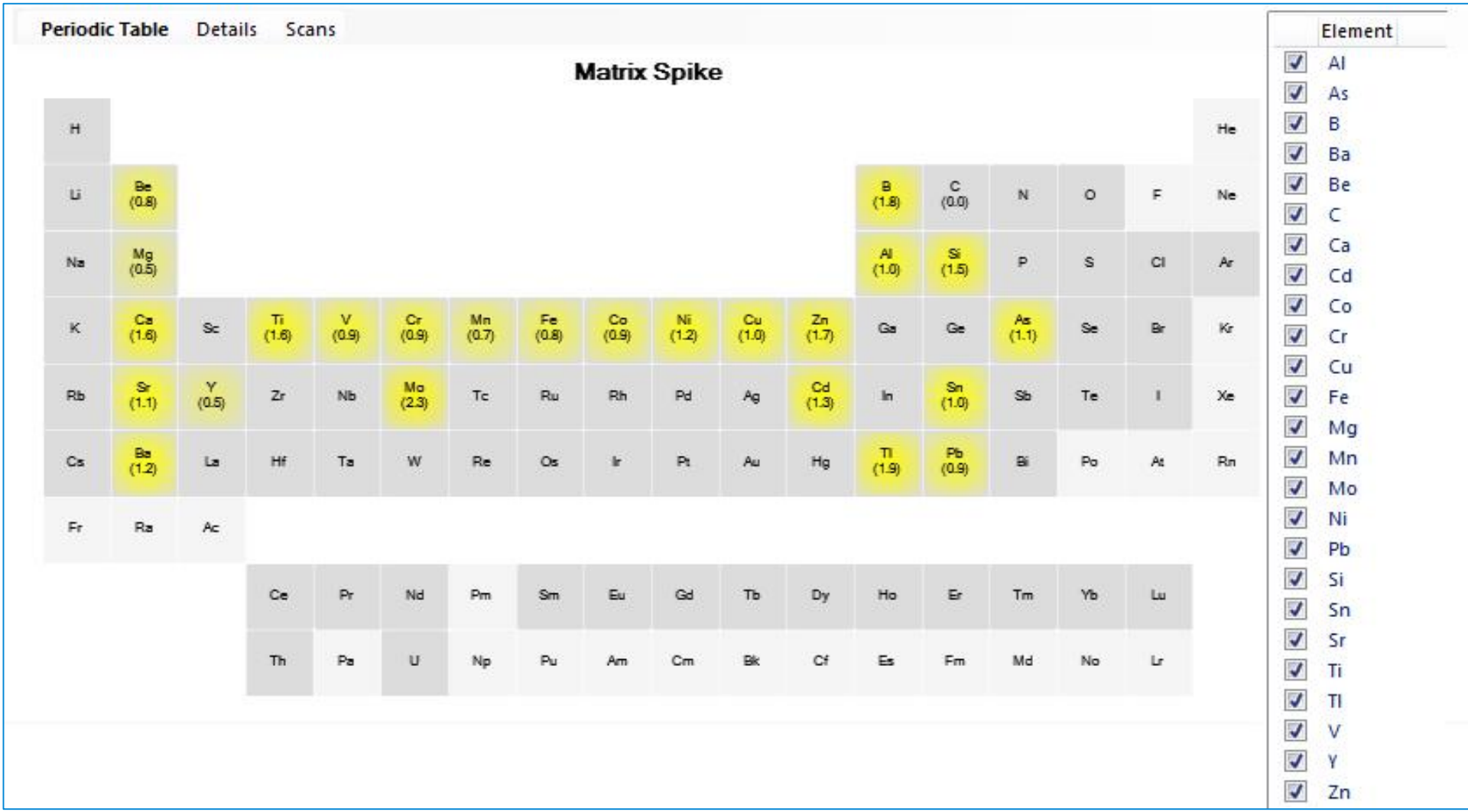
IntelliQuant Summary of Heat Map

- Heat Map Wastewater indicates few elements near the 1 ppm concentration

- Heat Map - Matrix Spike identifies most of the elements present in the multi-element standard utilized to prepared the 1.0 ppm spike



IntelliQuant Summary of Heat Map



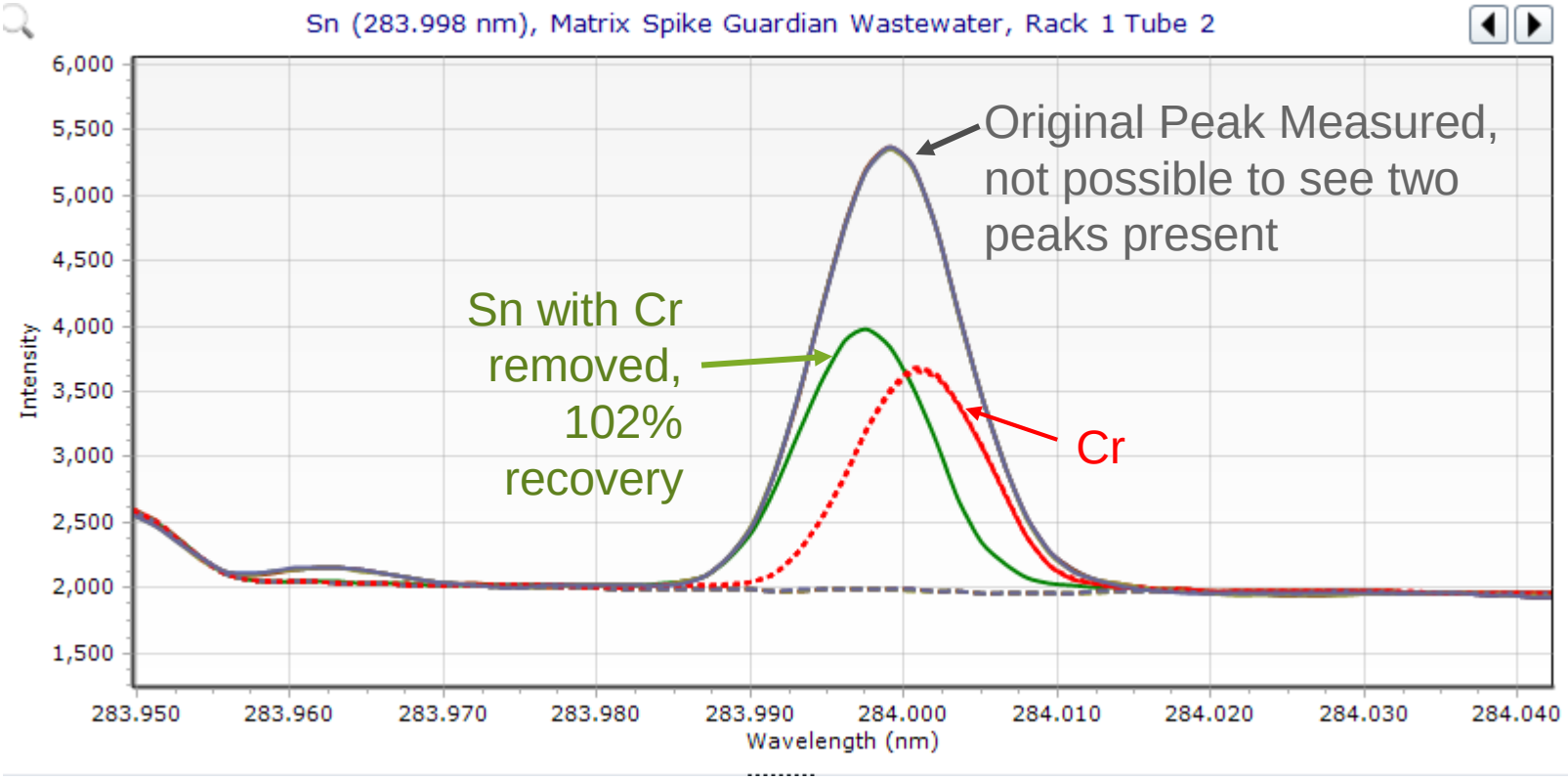
Results

IntelliQuant

Recoveries

Label	Wastewater	Matrix Spike	%REC
Al		0.95	95
As		1.05	105
B	0.91	1.77	86
Ba		1.18	118
Be		1.04	104
Ca	0.62	1.63	101
Cd		1.00	100
Co		1.17	117
Cr		0.88	88
Cu		1.02	102
Fe		1.14	114
Mg	0.02	1.24	122
Mn		1.04	104
Mo		1.01	101
Ni		1.18	118
Pb		0.86	86
Si		1.48	148
Sn		1.04	104
Sr		1.15	115
Ti	0.54	1.62	108
Tl		1.10	110
V		0.89	89
Zn	0.51	1.60	109

Sn 283.998 nm Correction with FACT



Models can be stored in the library or analyzed in a sequence to be used repeatedly without additional setup

Removing Carbon Structures

Deconvolution can also be used to remove molecular, matrix structures

Low UV wavelengths suffer from these overlaps, however, there are no alternate wavelengths for use

As = 188.98 nm and 193.7 nm

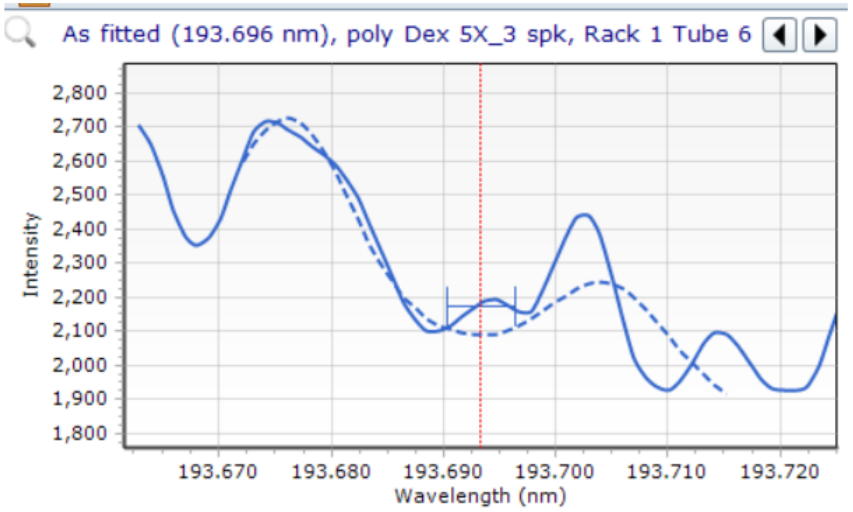
Se = 196.096 nm

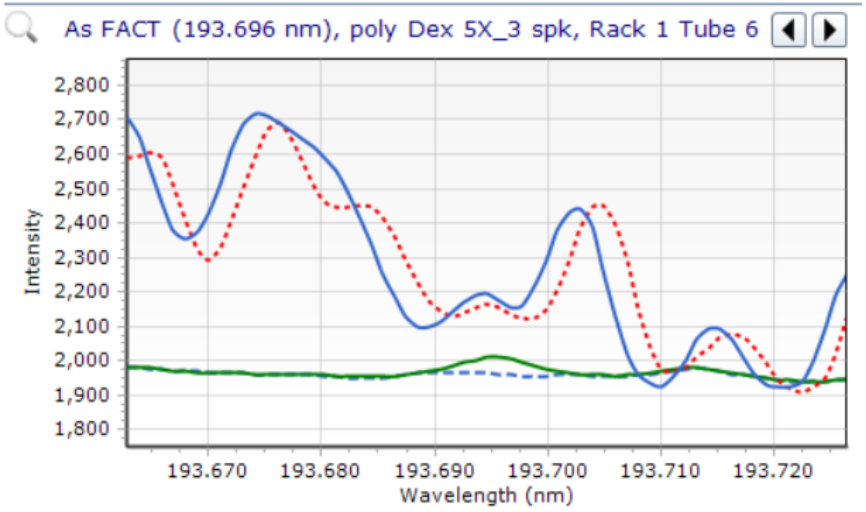
Tl = 190.794 nm, 276.789 nm

Sb = 206.834 nm, 217.582 nm

Pb = 220.353 nm

Carbon on Arsenic at 193.696 nm





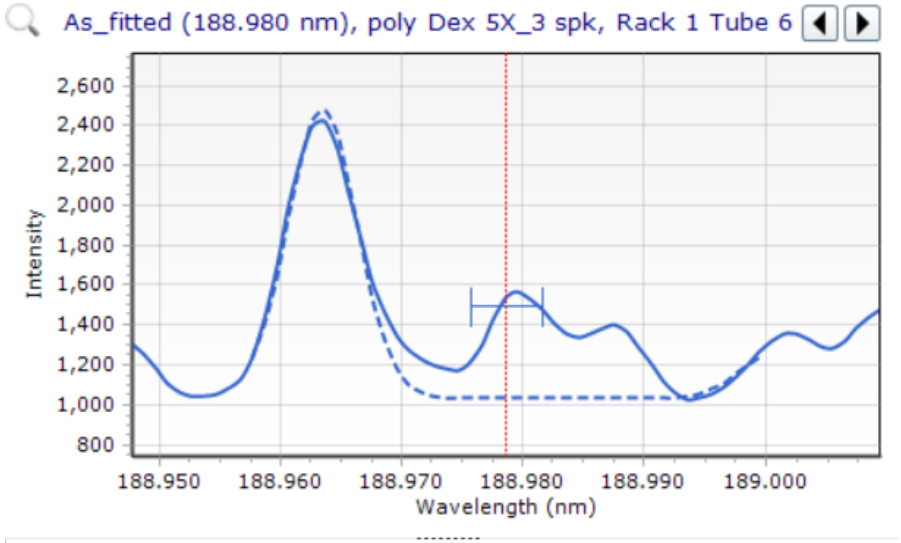
As fitted (193.696 nm) Calibration

Standards	Intensity	Method Concentration	Calculated Concentration	% Error
Blank	13.6463	0.0000	0.0027	N/A
std_1 10 ppb	24.1039	0.0100	0.0114	13.8167
std_2 25 ppb	39.8689	0.0250	0.0244	2.3675
std_3 100 ppb	126.6039	0.1000	0.0961	3.9239
std_4 1000 ppb	1221.0386	1.0000	1.0004	0.0393

As FACT (193.696 nm) Calibration

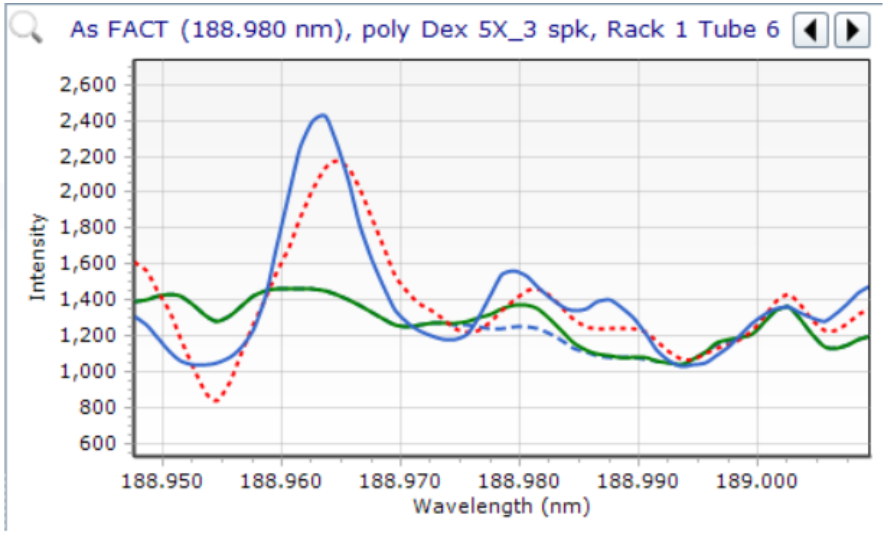
Standards	Intensity	Method Concentration	Calculated Concentration	% Error
Blank	1.35649	0.00000	0.00040	N/A
std_1 10 ppb	18.48870	0.01000	0.01066	6.59608
std_2 25 ppb	43.58050	0.02500	0.02568	2.72065
std_3 100 ppb	164.53908	0.10000	0.09809	1.91114
std_4 1000 ppb	1671.45960	1.00000	1.00017	0.01675

Carbon on Arsenic at 188.980 nm



As_fitted (188.980 nm) Calibration

Standards	Intensity	Method Concentration	Calculated Concentration	% Error
Blank	5.86572	0.00000	0.00089	N/A
std_1 10 ppb	17.45646	0.01000	0.00990	0.97483
std_2 25 ppb	36.38408	0.02500	0.02462	1.52933
std_3 100 ppb	132.74268	0.10000	0.09953	0.46899
std_4 1000 ppb	1291.06060	1.00000	1.00006	0.00574



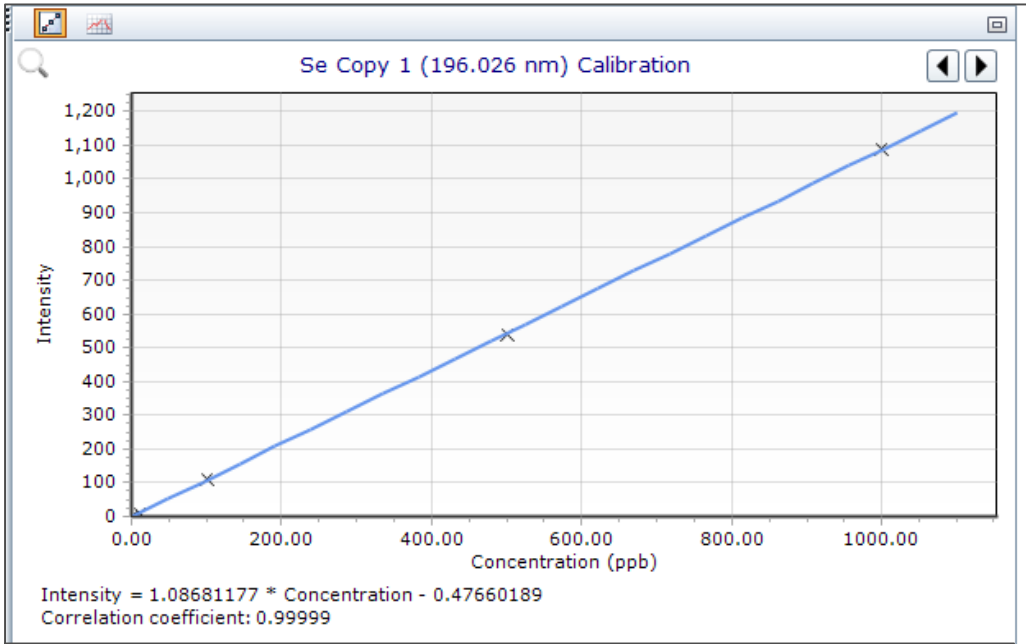
As FACT (188.980 nm) Calibration

Standards	Intensity	Method Concentration	Calculated Concentration	% Error
Blank	1.3566	0.0000	0.0015	N/A
std_1 10 ppb	13.3317	0.0100	0.0091	8.8809
std_2 25 ppb	36.6695	0.0250	0.0239	4.3072
std_3 100 ppb	157.2725	0.1000	0.1005	0.4635
std_4 1000 p...	1574.6375	1.0000	1.0000	0.0011

Arsenic Result Comparison

	As FACT 193.696 nm ppm	As fitted 193.696 nm ppm	As off peak 193.696 nm ppm	As FACT 188.980 nm ppm	As_fitted 188.980 nm ppm	As off peak 188.980 nm ppm
poly Dex 10X_1	0.0021	0.015	0.063	0.025	0.146	0.081
poly Dex 10X_2	0.0068	0.008	0.057	0.023	0.140	0.077
poly Dex 10X_3	0.0050	0.007	0.059	0.022	0.142	0.078
poly Dex 10X_4	0.0033	0.010	0.064	0.024	0.144	0.074
poly Dex 10X_5	0.0056	0.006	0.062	0.025	0.140	0.077
poly Dex 10X_6	0.0065	0.013	0.057	0.024	0.143	0.075
Average	0.0049	0.010	0.060	0.024	0.143	0.077
SD	0.0018	0.0037	0.0030	0.0011	0.0023	0.0024
%RSD	38	37	5	4	2	3
poly Dex 10X_1 spk	0.025	0.047	0.095	0.051	0.167	0.101
poly Dex 10X_2 spk	0.022	0.033	0.086	0.054	0.171	0.106
poly Dex 10X_3 spk	0.022	0.031	0.088	0.053	0.168	0.102
Average	0.023	0.037	0.090	0.053	0.169	0.103
SD	0.00	0.01	0.00	0.00	0.00	0.00
%RSD	8.6	23.3	5.0	3.0	1.1	2.6
%REC	93%	110%	118%	115%	104%	104%

Selenium



Se Copy 1 (196.026 nm) Calibration

Standards	Intensity	Method Concentration	Calculated Concentration	% Error
blank	0.117	0.000	0.546	N/A
2 ppb	1.870	2.000	2.159	7.950
10 ppb	9.822	10.000	9.476	5.241
100 ppb	109.600	100.000	101.300	1.328
500 ppb	539.900	500.000	497.200	0.553
1000 ppb	1088.000	1000.000	1001.000	0.125

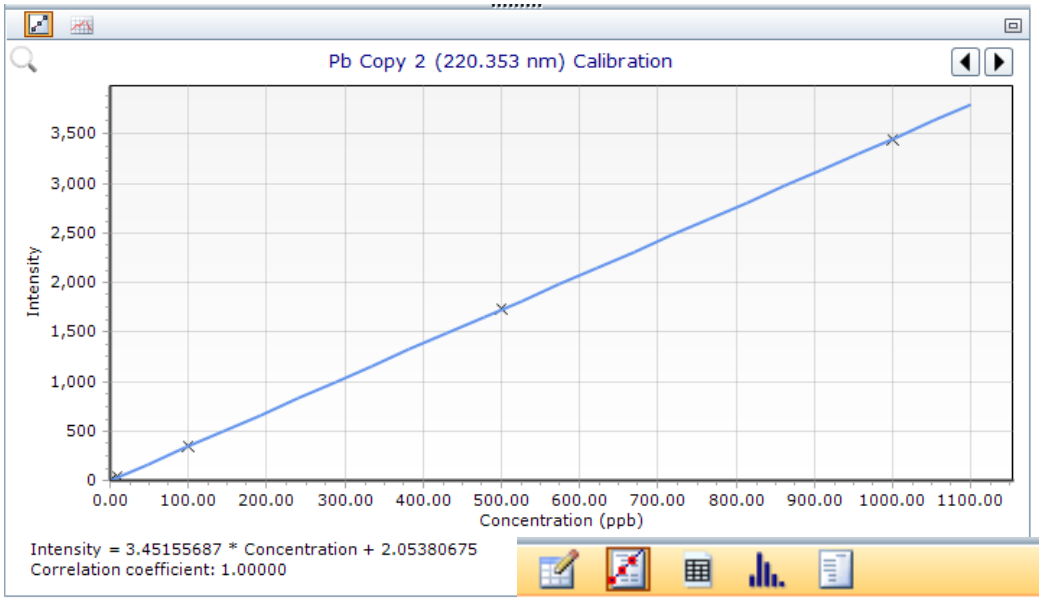
Rack:Tube	Solution Label	Se Copy 1 196.026 nm ppb	Se Copy 1 196.026 nm ppb
		uncorrected	corrected
1:1	TMDW	9.454 !	9.454 !
1:2	TMDW_10X	0.893	8.933
1:3	TMDW A	10.380	10.380
1:4	TMDWA_10X	1.613	16.130
1:5	5 ppm Pb	1.571	1.571
1:6	Blank	2.181	2.181
1:7	TMDW	9.696	9.696
1:8	TMDW 10X	1.191	11.910

CRM value = 10 µg/L

	Concentration	Intensity
Average:	1.191	0.913
SD:	0.063	0.069
%RSD:	5.33	7.55
Background:	N/A	-0.481

☑	Replicate	Concentration	Intensity
☑	1	1.146	0.865
☑	2	1.236	0.962

Lead



Pb Copy 2 (220.353 nm) Calibration

Standards	Intensity	Method Concentration	Calculated Concentration	% Error
Blank	0.043	0.000	-0.583	N/A
2 ppb	8.818	2.000	1.960	2.018
10 ppb	37.930	10.000	10.390	3.945
100 ppb	346.000	100.000	99.640	0.360
500 ppb	1732.000	500.000	501.100	0.222
1000 ppb	3452.000	1000.000	999.500	0.052

Lead

	Concentration	Intensity
Average:	2.400	10.000
SD:	0.500	1.700
%RSD:	20.97	16.81
Background:	N/A	-0.001

	Replicate	Concentration	Intensity
▶	1	2.800	12.000
	2	2.000	9.100

	Concentration	Intensity
Average:	4.100	16.000
SD:	0.070	0.240
%RSD:	1.73	1.51
Background:	N/A	-0.001

	Replicate	Concentration	Intensity
▶	1	4.100	16.000
	2	4.000	16.000

Rack:Tube	Solution Label	Pb Copy 2 220.353 nm ppb
S1:1	*Blank*	0.000
S1:2	*2 ppb*	2.000
S1:3	*10 ppb*	10.000
S1:4	*100 ppb*	100.000
S1:5	*500 ppb*	500.000
S1:6	*1000 ppb*	1000.000
1:5	TMDW A	19.000
1:6	TMDWA_10X	2.400
1:7	5 ppm Pb	5730.000 o
1:8	Blank	1.132
1:9	TMDW	36.250
1:10	TMDW 10X	4.069

CRM Value ppb

20

2

40

4

FACT advantages

FACT Models for Interference Correction

Number of interferents: 2 Edit model names Show library Read

Worksheet models

	Label (Wavelength nm)	Blank	Matrix	Analyte	Interferent 1	Interferent 2	Test
▶	P Copy 2 (177.434)	Blank	---	<Standard 7>	<cu int>	QC_27 _ 100	
	Cu (213.598)	Blank	---	<Standard 3>	Fe_1500	---	
	P Copy 1 (213.618)	Blank	---	<Standard 7>	<cu int>	QC_27 _ 100	
	Cu (223.009)	Blank	---	<cu>	Fe 500 Ni 250 Cr 1000	---	
	Cu (224.700)	Blank	---	<cu>	Fe_1500	---	
	Co (230.786)	Blank	---	QC_27 _ 1.0	1000 ppm Cr_500 ppm...	---	
	Co (231.160)	Blank	---	QC_27 _ 1.0	500 ppm Cr_250 ppm_...	---	
	Co (236.379)	Blank	---	QC_27 _ 100	Fe 500 Ni 250 Cr 1000	---	
	Co (237.863)	Blank	---	QC_27 _ 1.0	Fe_1500	---	
	Nb Copy 1 (269.706)	Blank	---	Std3_4_10	Fe_1500	---	
	Cr (283.563)	Blank	---	Fe 500 Ni 250 Cr 1000	Fe_1500	---	

Summary

- ICP-OES used for routine inorganics analysis
- Drift and poor precision can be caused by SIS blockage
- Internal Standard Correction using Argon
- IEC Correction Had it's place before modern technology
- Advanced deconvolution techniques

LEADING THE WAY IN ATOMIC SPECTROSCOPY INNOVATION

Questions?