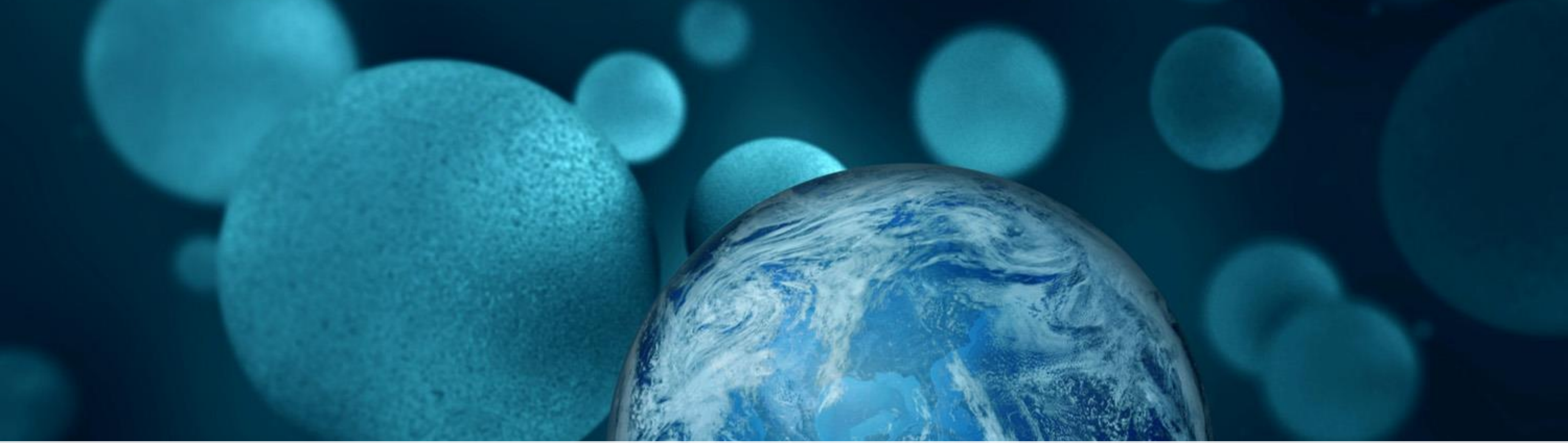

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Kick Interference to the Curb: Tackle ICP-OES Applications with Ease

Maura Rury, Regional Marketing Manager
NEMC 2017

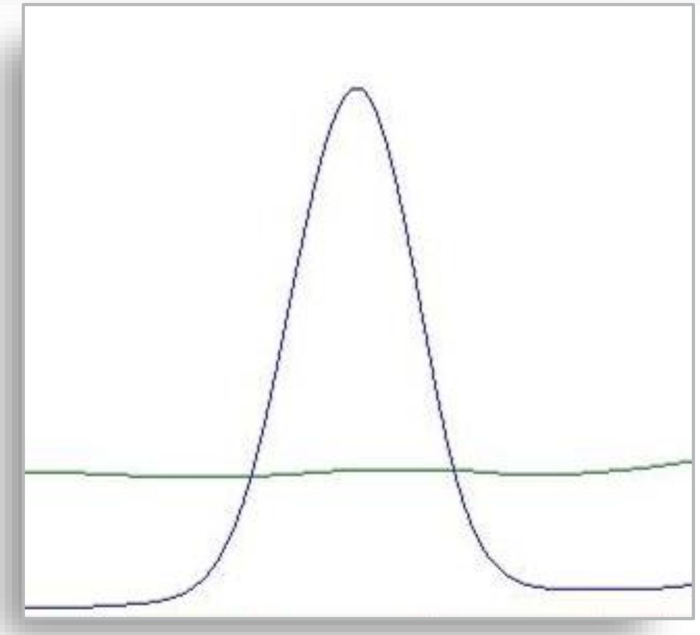
What are spectral interferences?

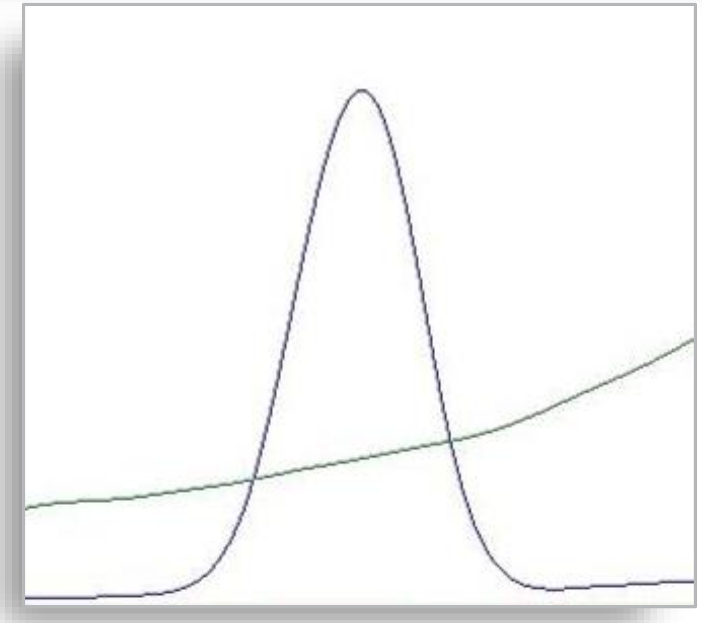
- Produced when emission spectra from matrix components overlap with those of the analyte/s
- They result from interactions between components in the sample and the sample matrix
- The severity of the interferences is dependent on the analyte wavelengths being used

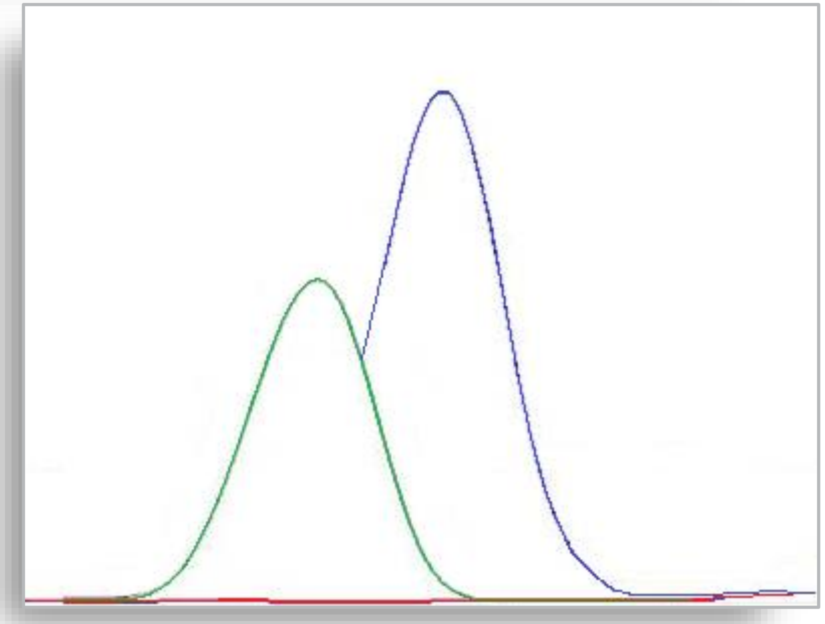
Spectral Interferences

Examples

- Background shift (flat, raised or lowered baseline)
- Background shift (sloped baseline)
- Partial peak overlap
- Full, complete peak overlap







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Spectral Interferences

Do you need to correct for these?

- Spectral interferences alter the magnitude of the signal that reaches the detector
- The magnitude of the signal is related to the concentration present in the sample
- If the signal increases/decreases as a result of an interference, the instrument will produce an incorrect concentration

Correcting for interferences – probably a good idea!

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Use One or More Correction (IEC) Factors

- Just follow these simple steps:
  - Identify all elements that have interferences
  - Identify all elements that are causing interferences
  - Use carefully-prepared solutions to allow the instrument to measure the spectral overlap and calculate an accurate IEC factor
  - Repeat process for each interference that must be corrected
  - Re-calculate all IEC factors if any conditions change (plasma parameters, sample matrix, analyte mixture)

Isn’t there another way to correct for interferences?

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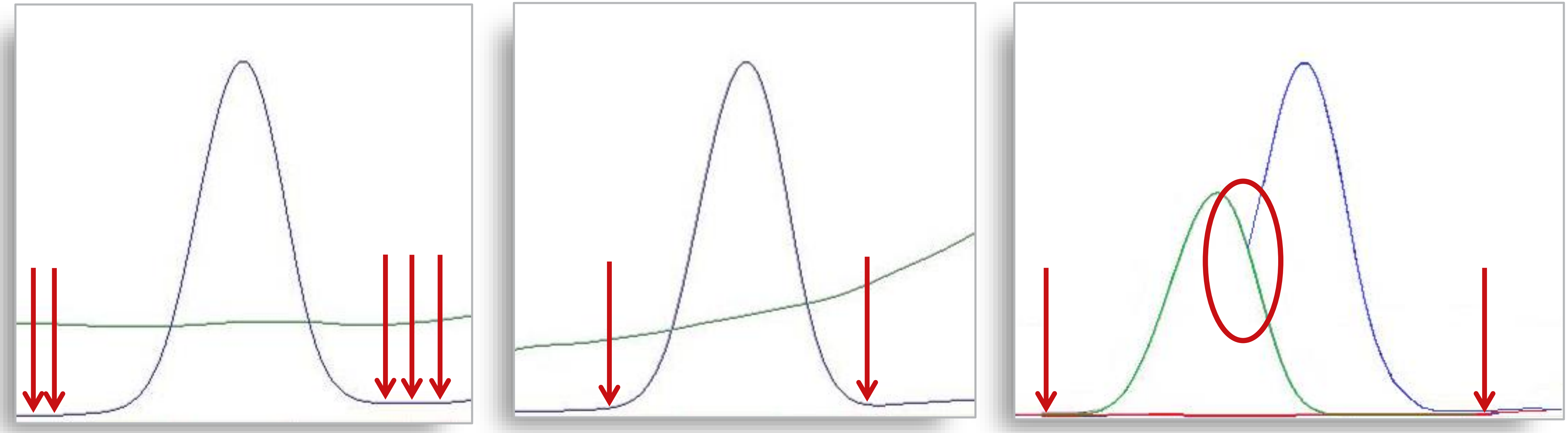
Use Careful Background Point Selection

- Background correction should mimic shape of background emission
- Avoid overcorrection or correction on a nearby emission peak



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Use Careful Wavelength Selection

- Careful wavelength selection – ideally, interference free!
- Element Finder plug-in for Thermo Scientific™ Qtegra™ ISDS Software
  - Automatically selects interference-free wavelengths
  - Eliminates interferences before you know they exist

Analysis of Cd

- Where do we want it?
  - Rechargeable NiCd batteries
  - Paint pigments (“cadium yellow” or “cadmium red”)
  - He-Cd lasers
  - Corrosion-resistant plating materials
- Where don’t we want it?
  - Airborne particles
  - Drinking water
  - Fruits/vegetables grown in Cd-contaminated soil
  - Rice grown in Cd-contaminated fields
  - Food oils

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Analysis of Cd – Theory

Analysis of Cd – Reality

Non-linear calibration curve?!?!

125% Recovery!

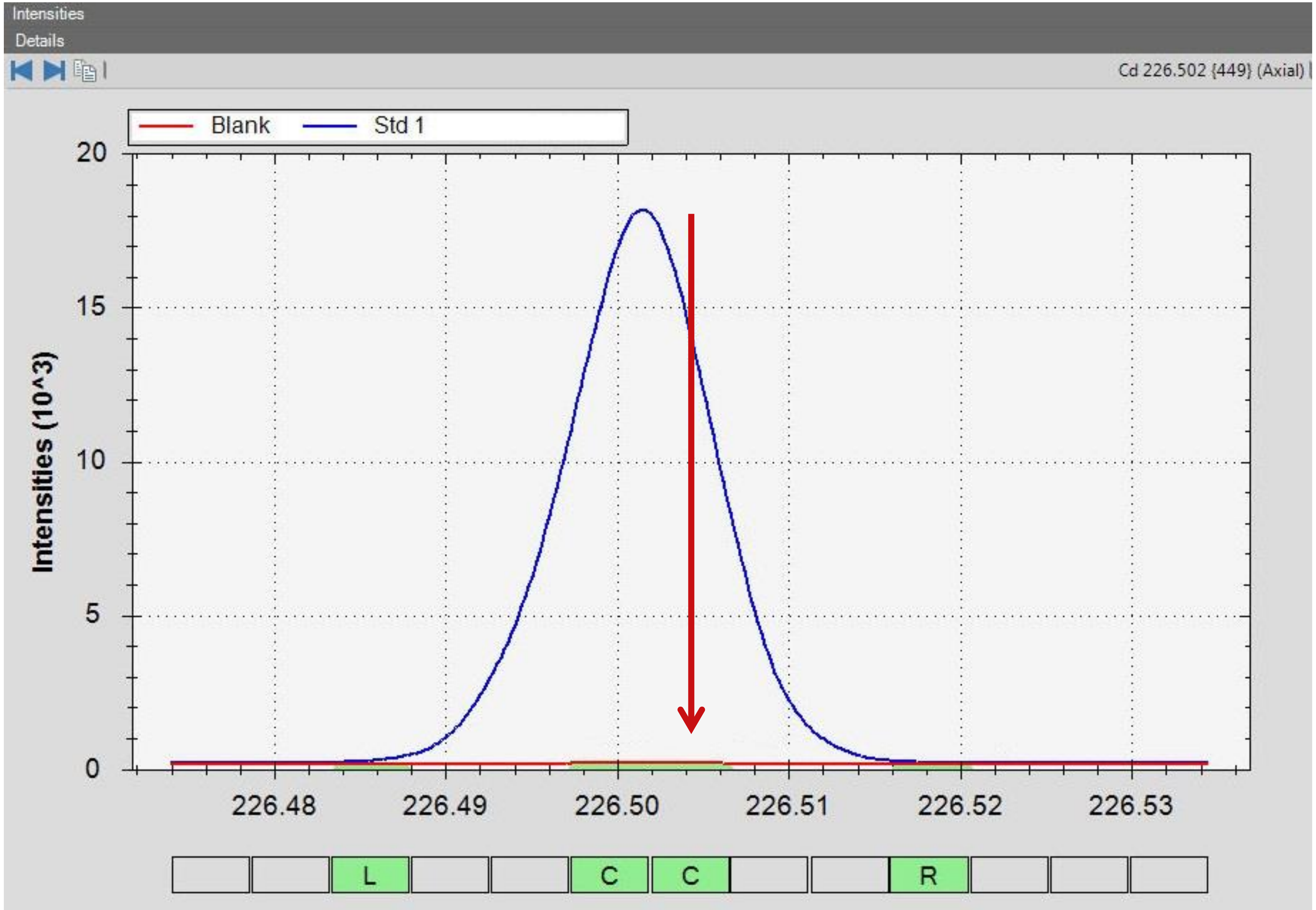
1.5 ppm?
That’s too high!

QC standards don’t pass?!?!

Analysis of Cd – What Happened?

- Wavelength Scan of a Multi-Element Standard

- Single-Element Standard Containing Fe



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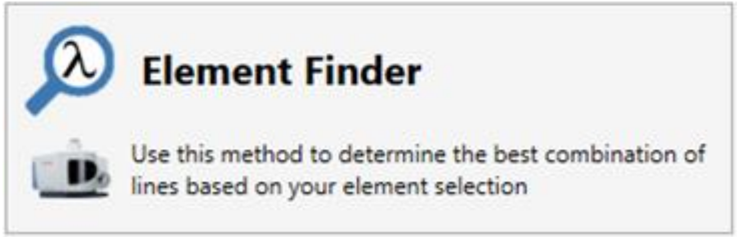
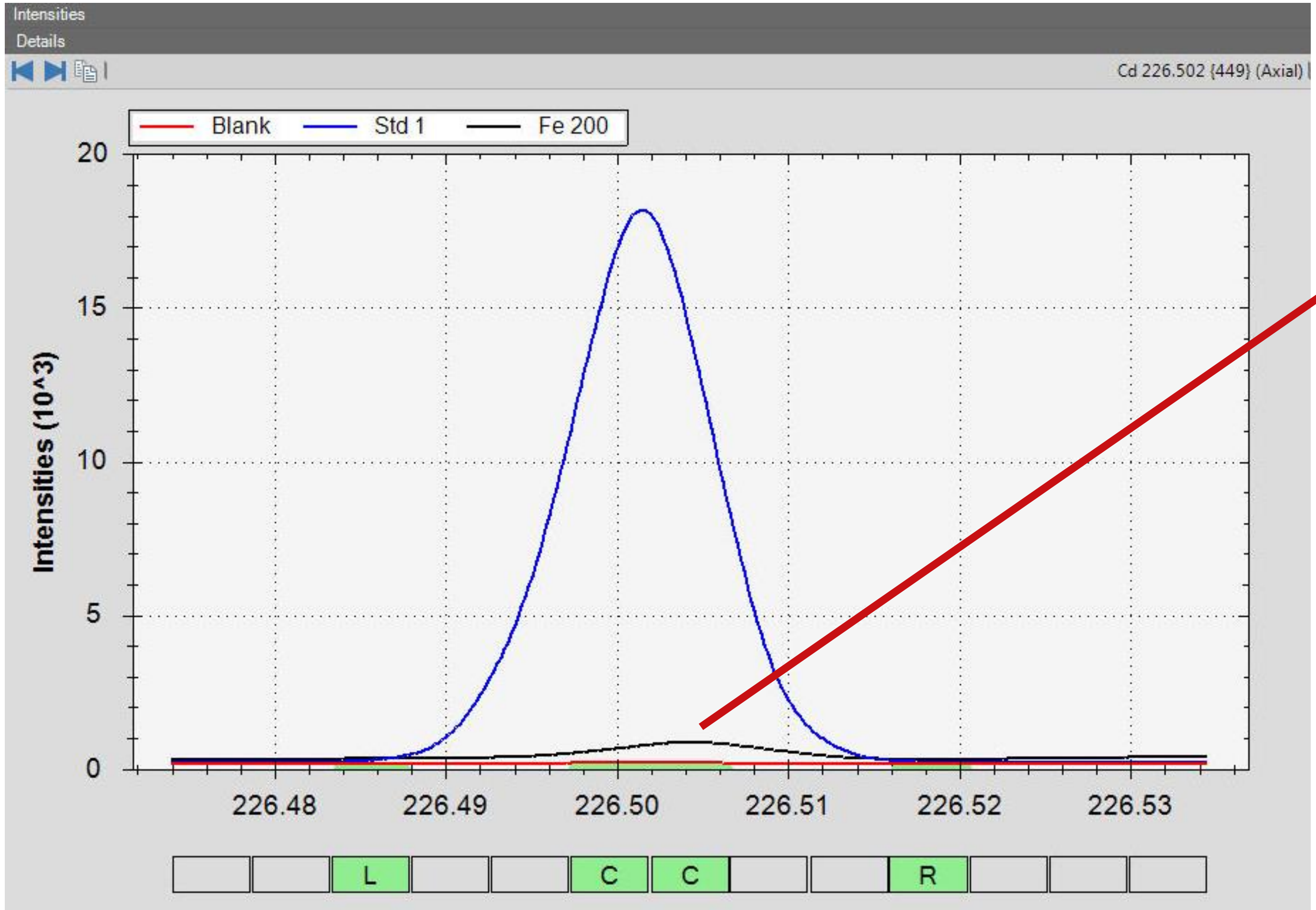


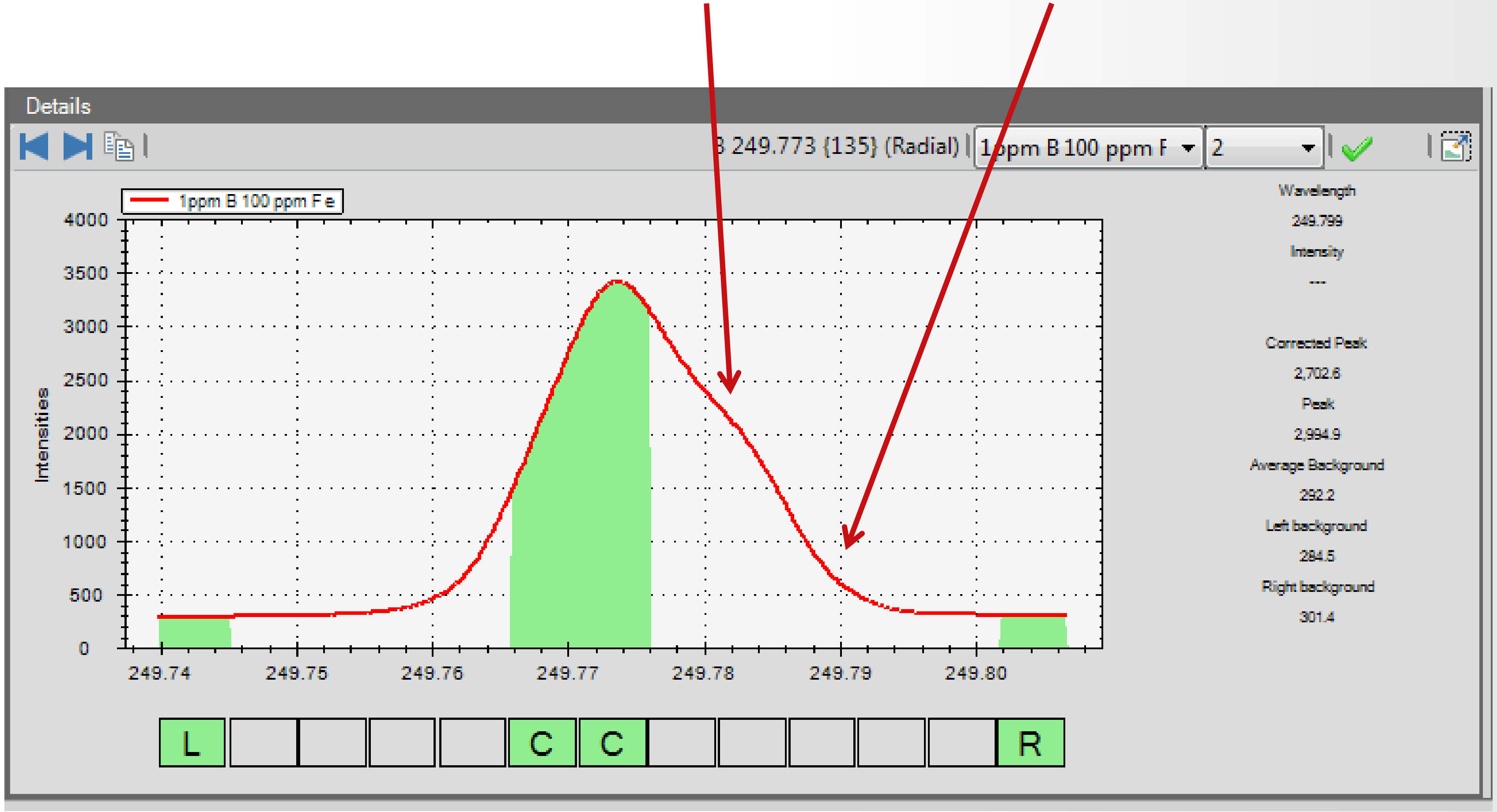


Here's How Element Finder Helps:

1. Hey! You have an interference!
2. It's probably from Fe!
3. What do you want to do about it?
   1. Calculate an IEC? – not recommended!
   2. Find an alternative wavelength? Let me help you!
4. Cd has a strong emission wavelength at 214 nm; Fe doesn't emit there – try that one!

Another Spectral Interference Example

- Analysis of B in a high Fe sample matrix – asymmetric peak for boron at 249.773 nm
- Clear interference on boron peak (visible at 249.780 nm and 249.790 nm)



Manual Interference Identification

Analyze three solutions:

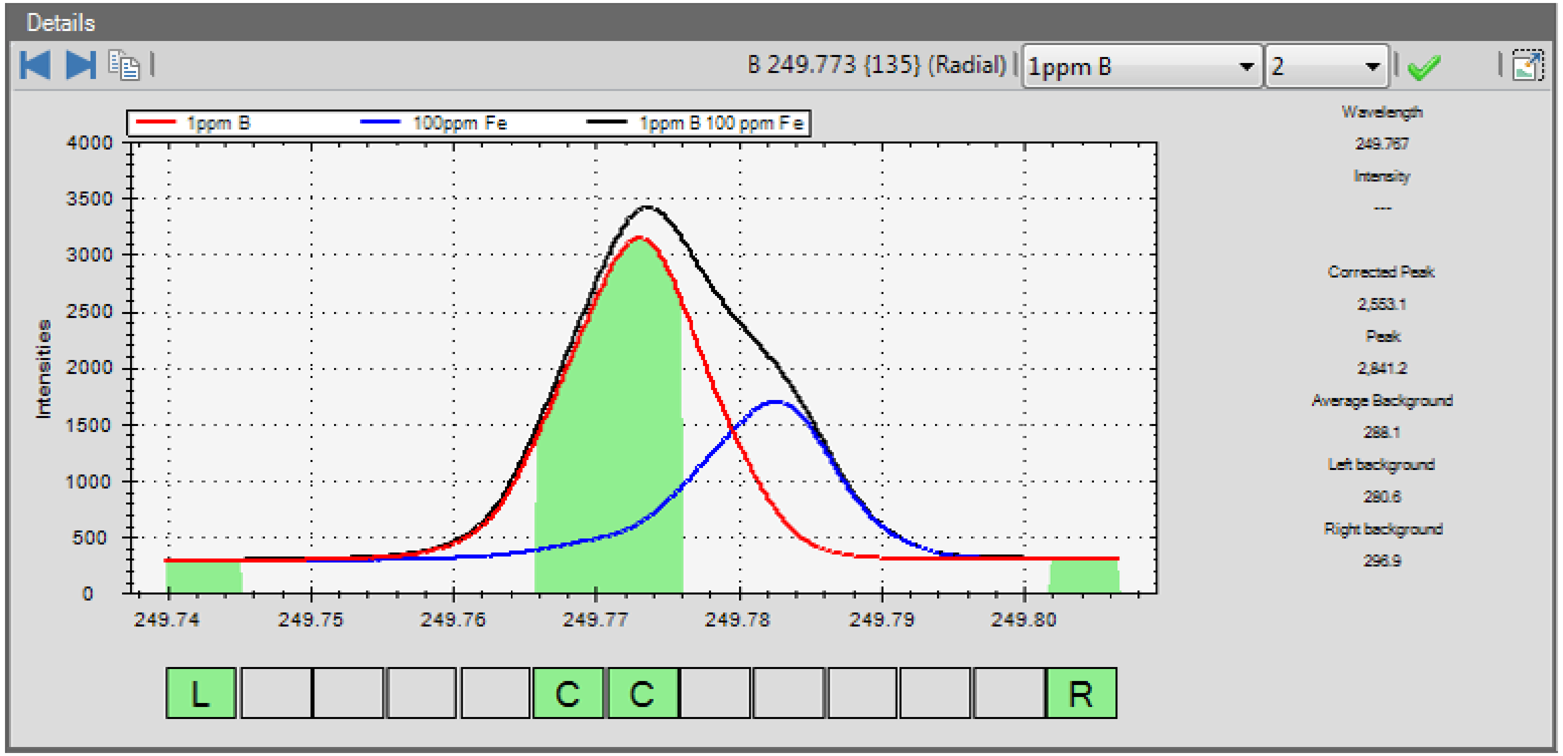
1. The sample
2. A single element solution of the analyte at the concentration in the sample
3. A single element solution of the suspected interference at the concentration in the sample
   - If this is not the interfering analyte, further analysis might be needed to determine this

Then

- Measure the interference to calculate a correction factor

OR

- Select an alternative wavelength that is free from interferences



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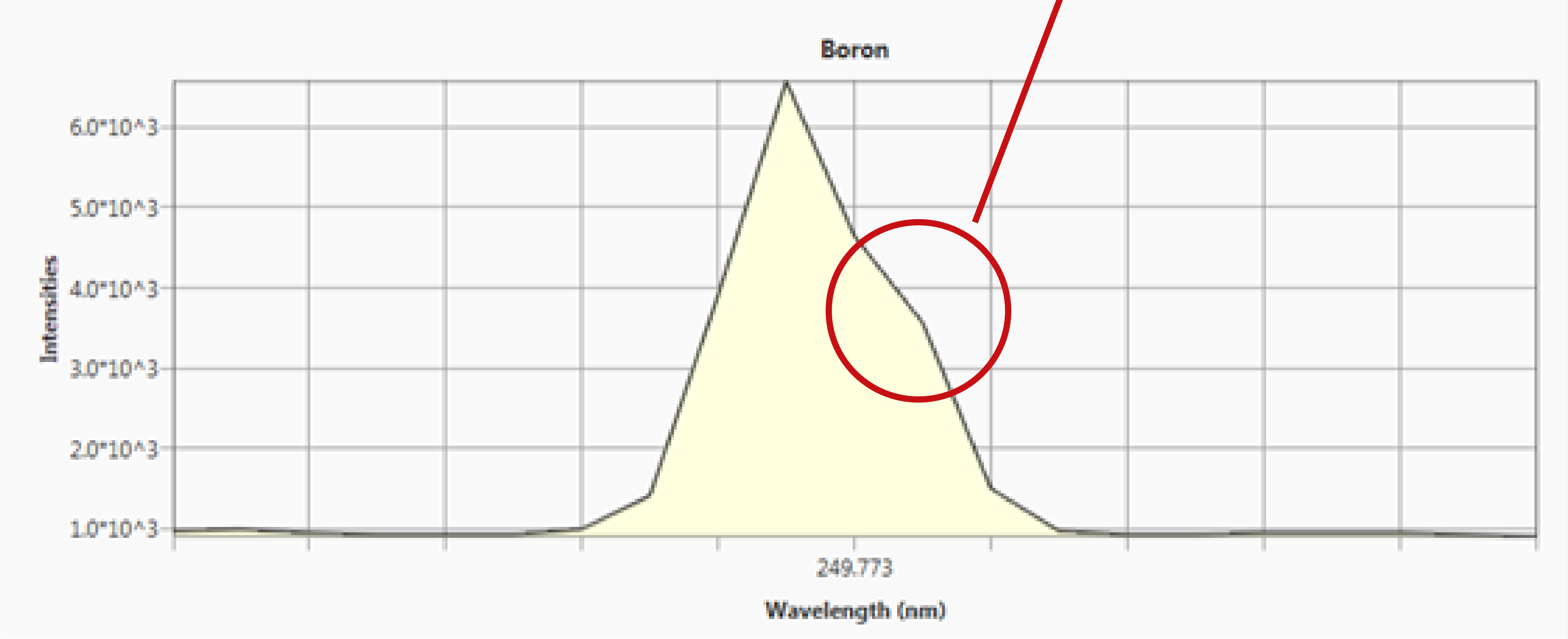
Automatic Interference Identification

- Boron interference automatically detected and identified!

Wavelengths suggested for Analyte Elements that are suited to your analysis

5 **B** Boron Analyte

Wavelength	Order	Intensity	Preference	Available for analysis	Measure Mode	Remarks
249.773	135	4,000,000	Automatic	No	Radial	Interference with: Fe, 249.782; Interfer
249.678	135	2,000,000	Automatic	No	Radial	Interference with: Fe, 249.653; Interfer
208.959	461	1,500,000	Automatic	Yes	Radial	
208.893	461	750,000	Automatic	No	Radial	Interference with: Fe, 208.412;
182.641	484	660,000	Automatic	No	Radial	
182.591	484	290,000	Automatic	No	Radial	
181.837	485	73,000	Automatic	No	Radial	



Available for analysis?

NO!

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Automatic Identification – Additional Interferences Found!

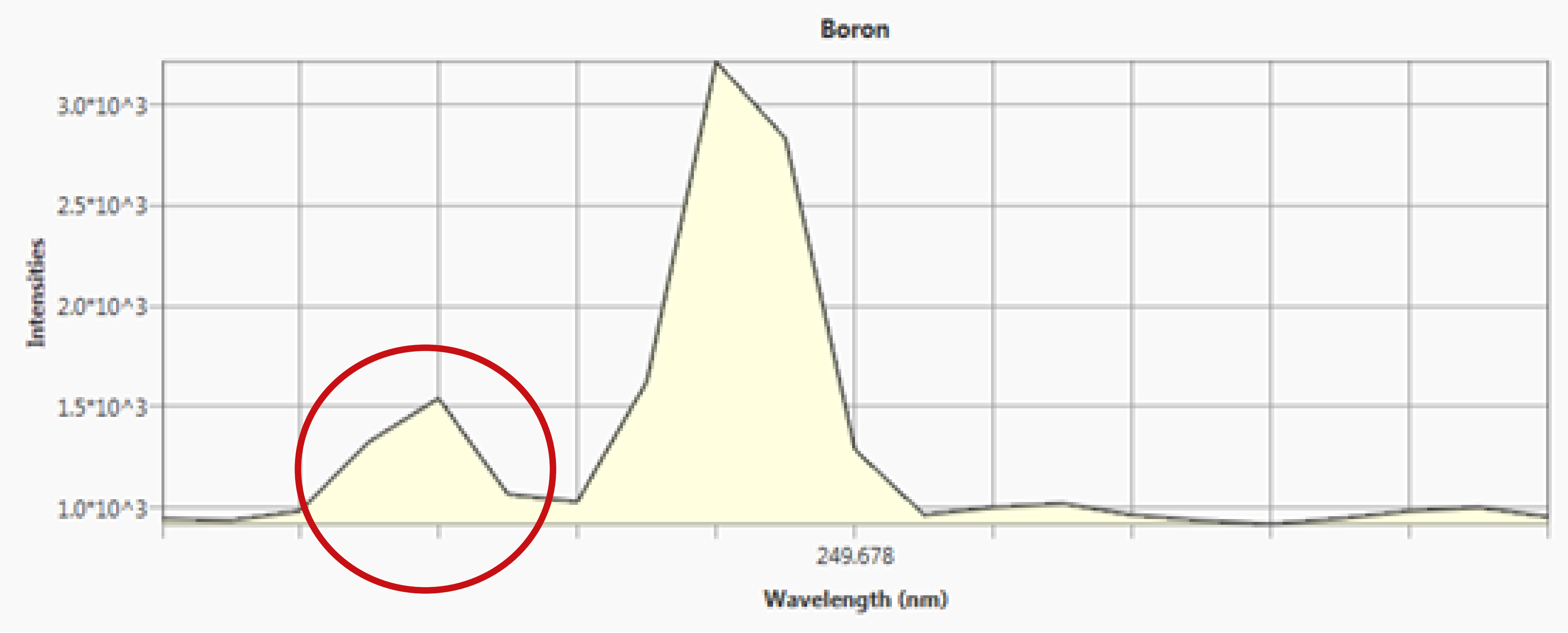
Wavelengths suggested for Analyte Elements that are suited to your analysis

5
B
Boron
Analyte

Wavelength	Order	Intensity	Preference	Available for analysis	Measure Mode	Remarks
249.773	135	4,000,000	Automatic	No	Radial	Interference with: Fe, 249.782; Interfer
249.678	135	2,000,000	Automatic	No	Radial	Interference with: Fe, 249.653; Interfer
208.959	461	1,500,000	Automatic	Yes	Radial	
208.893	461	750,000	Automatic	No	Radial	Interference with: Fe, 208.412;
182.641	484	660,000	Automatic	No	Radial	
182.591	484	290,000	Automatic	No	Radial	
181.837	485	73,000	Automatic	No	Radial	

Available for analysis?

NO!
YES!



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Automatic Interference Identification

Wavelengths suggested for Analyte Elements that are suited to your analysis

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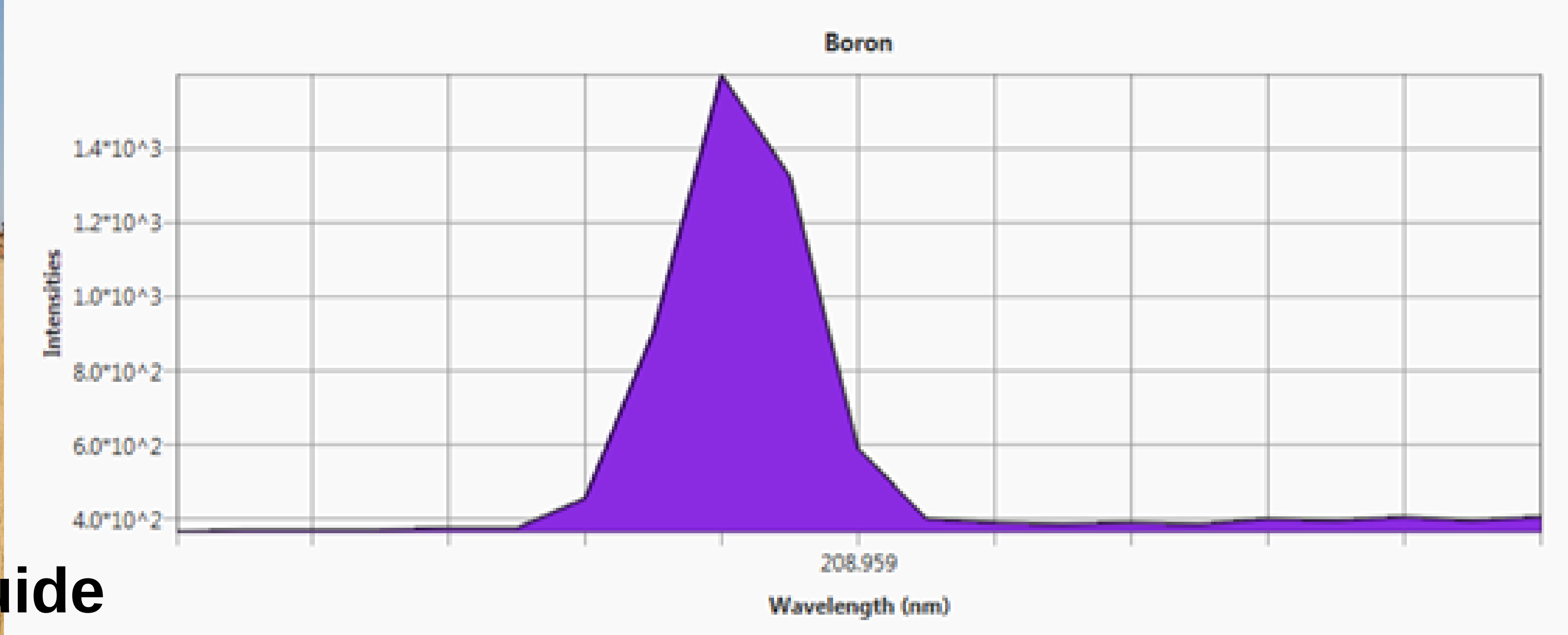

Let Element Finder be your guide

Final Spectral Interference Example

Trace Elements in Pentanol

- Multiple interference challenges

1. Physical
   - Differences in nebulization and/or transport efficiency between the standards and samples
2. Chemical
   - Differences between the behavior of standards and samples when in the plasma
     - Easily ionized element (EIE) effects
     - Plasma loading
3. Spectral
   - Atomic emission overlaps from other elements in the sample
   - Molecular emission from solvent

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Final Spectral Interference Example

Trace Elements in Pentanol

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1. Physical
   - Differences in nebulization and/or transport efficiency between the standards and samples
2. Chemical
   - Differences between the behavior of standards and samples when in the plasma
     - Easily ionized element (EIE) effects
     - Plasma loading
3. Spectral → Element Finder
   - Atomic emission overlaps from other elements in the sample
   - Molecular emission from solvent

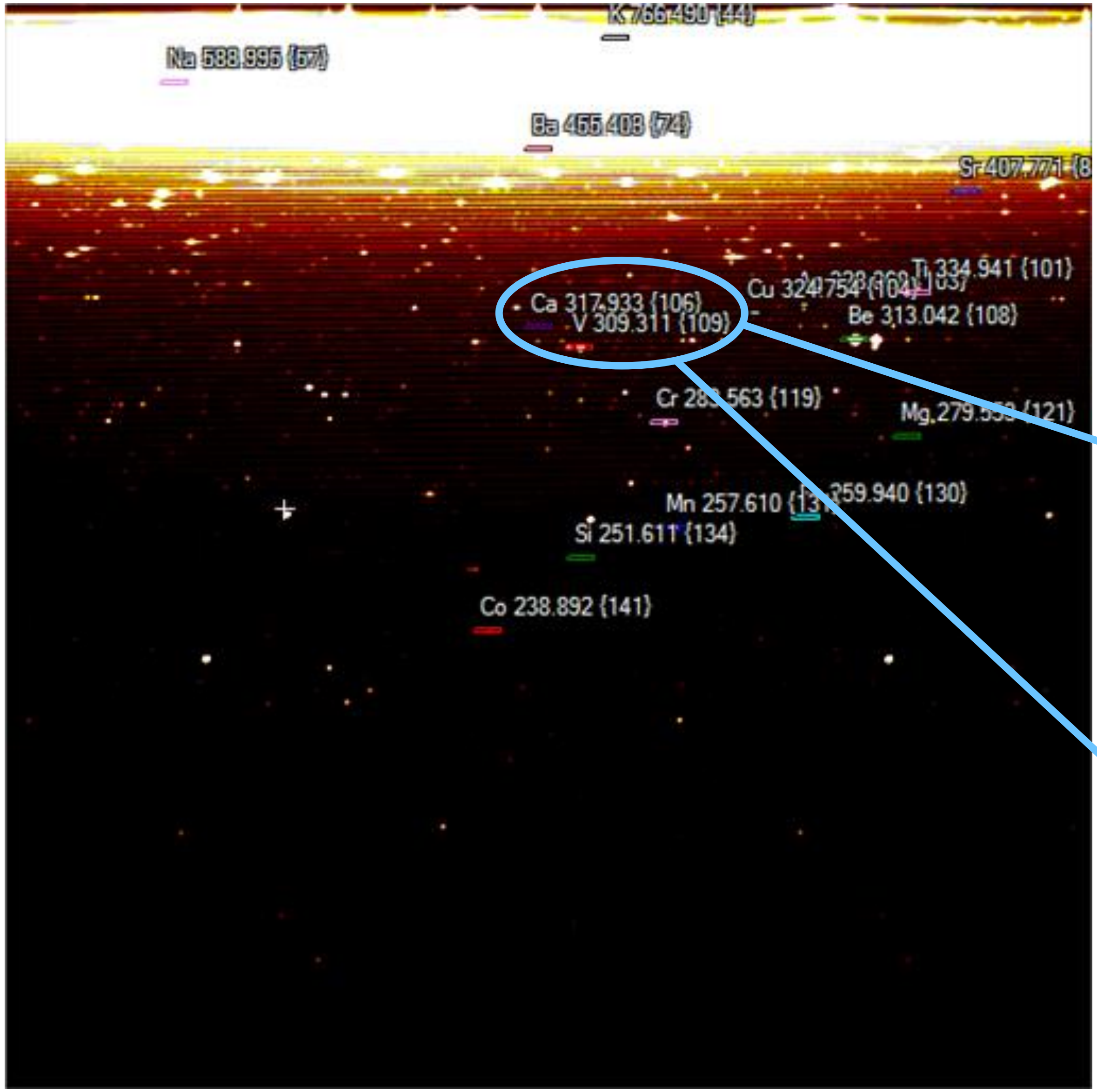
Matrix match standards to samples

Use an internal standard

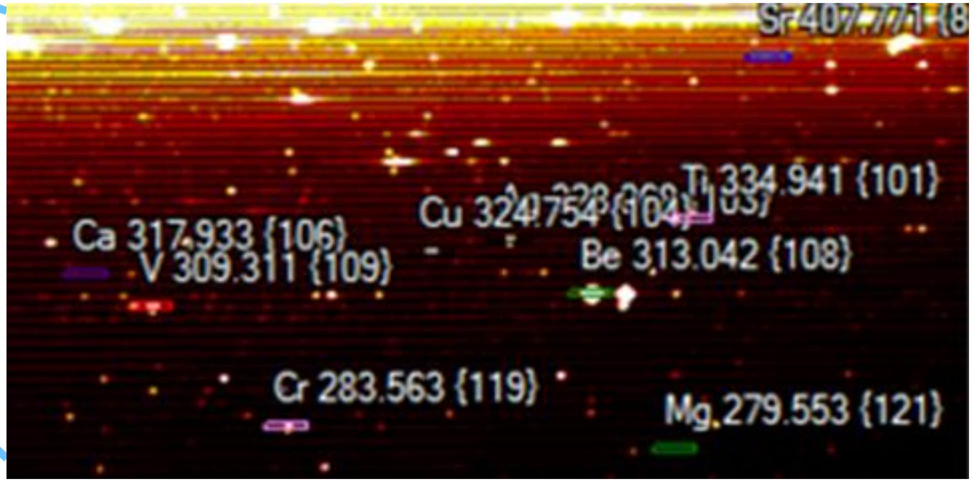
Carefully select sample introduction

Reduce sample uptake to reduce matrix loading

Full Frame of Pentanol Sample



- Emission from other analytes
- Emission from sample contaminants
- Molecular emission from solvent (CO, CO_2, CN)



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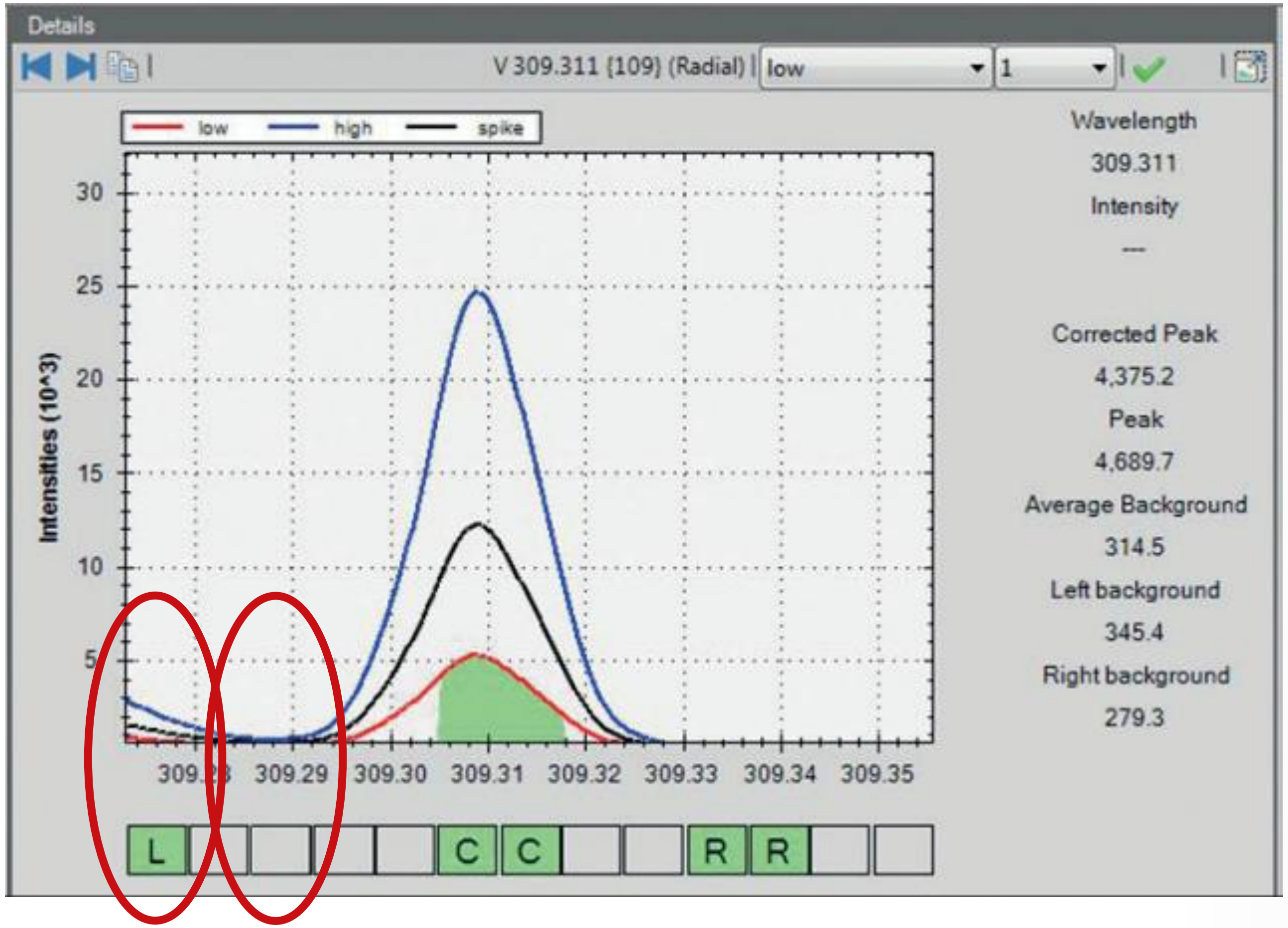
- Vanadium wavelength scan



Possible Interferences

Interferences for V 309.311 {109} (Ra

Symbol	Line	State	Intensity
Cd	309.234	II	100000
Sc	309.249	II	112500
Pu	309.259	II	10000
Al	309.271	I	600000
Na	309.273	II	6188
Nd	309.273	II	10000
Al	309.284	I	0
Sc	309.288	I	18750
Nd	309.292	II	0
Mg	309.298	I	277778
Mg	309.299	I	2037037
U	309.301	II	8000
Th	309.305	II	12000
V	309.311	II	2500000
Tm	309.311	II	25000
Er	309.314	II	18000
V	309.324	I	833
Mn	309.335	I	5185
U	309.337		2500
Ar	309.34	II	3571
W	309.35	I	1500
Os	309.359	I	9310

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Pentanol Results

- Successful tactics to address interferences
- Results: Good precision and accuracy, and spike recoveries ~100%

Element and Wavelength (nm)	View	Spike Concentration (mg kg⁻¹)	Measured Spike Concentration (mg kg⁻¹)	Spike Recovery (%)	RSD on three Replicates of the Spike (%)	MDL (µg kg⁻¹)
Ag 328.068	Radial	2.47	2.45	99.2	1.6	3.5
Al 167.079	Axial	2.47	2.82	114.2	0.2	1.4
As 189.042	Axial	2.49	2.58	103.6	0.2	7.6
Ba 455.403	Radial	2.47	2.53	102.4	1.8	0.3
Ca 393.366	Radial	2.47	2.46	99.6	1.9	0.2
Cd 214.438	Axial	2.47	2.69	108.9	0.3	0.3
Cr 267.716	Radial	2.47	2.48	100.4	1.6	4.8
Cu 324.754	Radial	2.47	2.41	97.6	2.0	2.4
Fe 238.204	Radial	2.47	2.50	101.2	1.6	4.8
Hg 184.950	Axial	2.48	2.59	104.4	0.6	2.6
K 766.490	Radial	2.47	2.41	97.6	2.4	55
V 309.311	Radial	2.47	2.46	99.6	1.7	2.1

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Kick Interference to the Curb for Successful ICP-OES Results

- Thoughtful method development will ensure that physical, chemical and spectral interferences are identified and corrected
- Wavelength scans and full frame images (captured in seconds) will help illustrate nearby emission from other analytes and sample matrix components and the solvent
- Background shifts and nearby spectral interferences can often be addressed with careful background correction
- When background correction is insufficient on its own:
  - Spend your valuable time and lose productivity calculating IEC factors

    OR
  - Let Element Finder identify possible interferences and automatically select alternative wavelengths

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