

Overcoming Interferences with the Thermo Scientific iCAP 7000 Plus Series ICP-OES

Key Words

Overcoming Interferences, Element Finder plug-in

Introduction

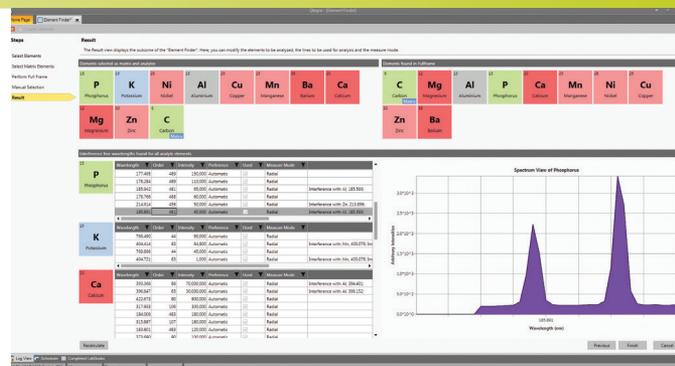
Interferences are common in Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES) for many different reasons; matrix, sample transport, ionization, suppression or spectral overlaps. These are commonly encountered in environmental, metallurgy and geological applications where there is a mixture of very high and low concentrations of elements resulting in a spectrally rich matrix. Ideally, physical and chemical effects should be experimentally eliminated and the use of a solid state detector should allow the analyst to select an interference free wavelength for analysis, but if this is not possible then alternative techniques must be used.

There are three main types of interferences that are encountered during ICP-OES analysis: physical, chemical and spectral.

Physical interferences are characterized as a difference between the samples and calibration standards which affect the sample transport or nebulization – these differences can be viscosity, density, matrix (from the sample itself or the digestion/preservation procedure).

Chemical interferences occur when there is a difference in the way that the sample and the calibration standard react in the plasma such as ionization, molecular formation and plasma loading.

Finally, spectral interferences are characterized by an overlap (either partial or direct) of the analyte of interest by an interfering element. These interferences are normally apparent during method development and can lead to suppression or enhancement of signals, and thus, false negative or positive results which ultimately degrade the accuracy and precision of the method. This technical note will focus on spectral interferences and how to overcome these using analytical approaches and software tools.



Spectral Interferences

Spectral interferences are sometimes severe in ICP-OES due to the effectiveness of the plasma as an excitation source and the complexity of the emission spectrum of the elements and their ions. These interferences can be a shoulder on a peak, a partial or direct spectral overlap. It is a challenge to soundly analytically correct for a direct spectral interference, so a wavelength experiencing this uncommon phenomenon would be eliminated at the early stages of method development. Partial and peak overlaps are dealt with by either off-peak background correction (as per Figures 1 and 2) or with the use of an experimentally derived mathematical correction.

The correct use of background correction is paramount when developing a method and can be used to alleviate or negate the effect of a spectral interference – as shown below in Figure 1. The sample contains 1 mg·L⁻¹ boron, which is being interfered with at the B 249.678 nm wavelength by the presence of 100 mg·L⁻¹ iron.

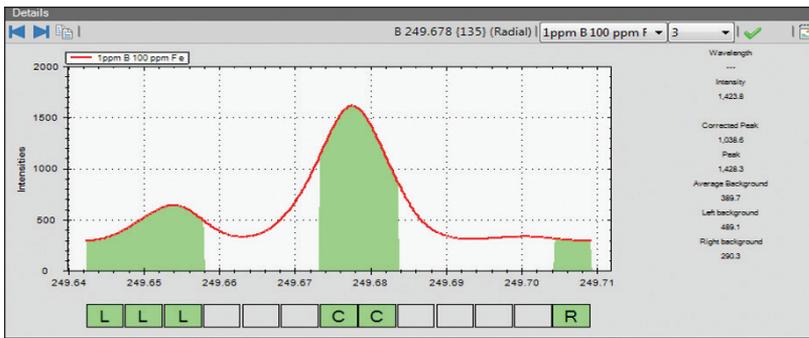


Figure 1. 1 mg·L⁻¹ Boron in the presence of 100 mg·L⁻¹ Iron.

This background interference is visible on the smoothed subarray display and the interferent is identified by the Interferences list as Fe 249.653 nm. Due to this background interference (and minor wing overlap), the 1 mg·L⁻¹ boron is reading as 0.4804 mg·L⁻¹. By turning off the left background point, the method can be salvaged and the interference negated – see Figure 2 below where the boron now reads at 1.004 mg·L⁻¹. If the analyst prefers to use two background points, it is possible to extend the width of the subarray in the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution (ISDS) software to accommodate this.

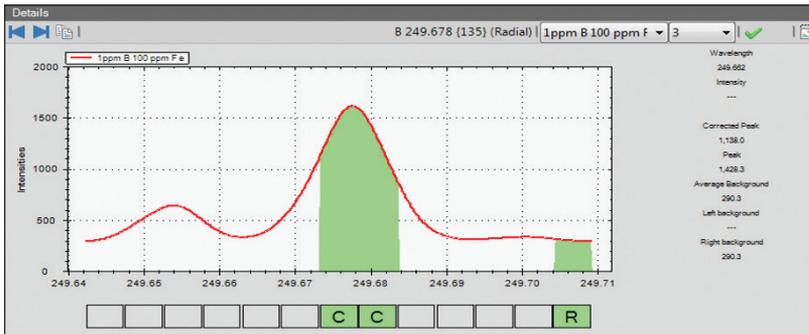


Figure 2. Use of flexible background correction.

Figure 3 below displays the interferent - 100 mg·L⁻¹ iron, a 1 mg·L⁻¹ boron solution, and the sample, which contains both boron and iron to demonstrate the spectral overlap contributions.

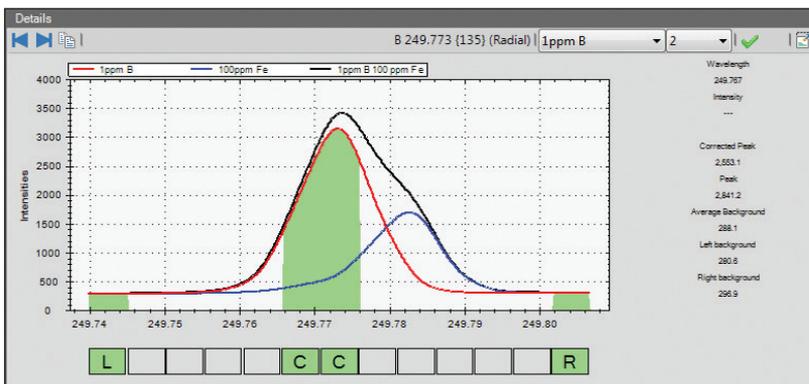


Figure 3. Spectral overlap.

Qtegra ISDS can use an Inter-Element Correction (IEC) calculation to correct for specific interferences. This is a ratio of the intensity of the interfered analyte wavelength divided by the intensity of an undisturbed wavelength of the interfering element. Figure 4 below shows a sample of boron being interfered with by iron, which causes a false result for a 1 mg·L⁻¹ boron, solution of 1.208 mg·L⁻¹. This is due to the shoulder overlap from Fe 249.782 nm on B 249.773 nm.

To set up an IEC for a method, the analyst must first identify the contaminant – this is done by clicking on the interferent peak and using the *Interferences* list (see below Figure 4) to correctly identify what element is causing the overlap.

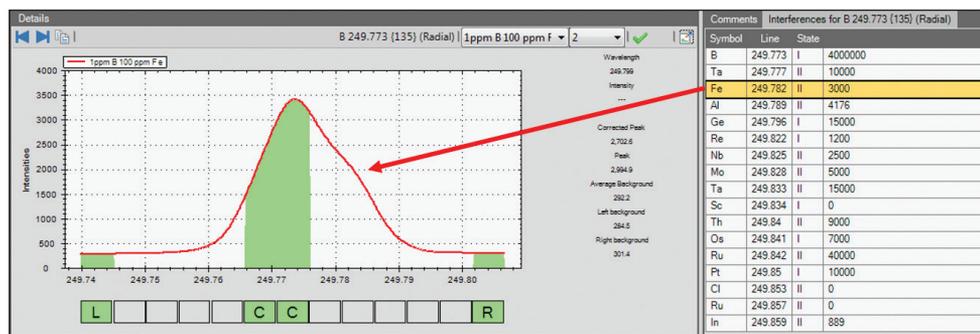


Figure 4. Use of interferences list to identify the interfering element.

When the interfering element has been identified, the analyst adds a primary wavelength (in this case Fe 259.940 nm) to the method and analyses a single element solution (to avoid further interferences, it must be a single element solution) as a sample, which allows the apparent intensity of iron to be read as boron. Right-clicking on the false positive intensity allows you to select the interfering element and IEC correction factor will automatically be calculated.

Steps to determine and automatically calculate an IEC using Qtegra ISDS

1. Isolate the interferent
2. Add the primary wavelength for the interferent to the method
3. Analyze an appropriate concentration of the interferent (high purity, single element) as a sample
4. Right click on the false positive intensity of the interfered wavelength
5. Select the wavelength of the interferent and let Qtegra ISDS calculate the IEC correction factor

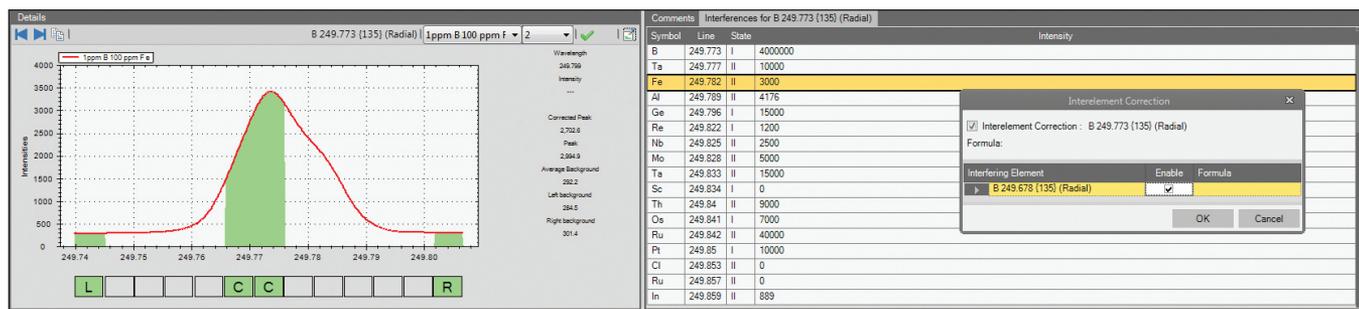


Figure 5. Selection of interfering element in the Inter-Element Correction table.

6. The IEC correction factor will be applied to each sample that is analyzed in this LabBook and can easily be switched on and off via an icon in the headline of Qtegra ISDS.

IECs should be re-assessed if there is a change in the sample type or matrix, which may affect the correction.

Element Finder plug-in for the Qtegra ISDS software

The use of an IEC as described is a well-accepted correction technique and prescribed by many standard protocols such as United States EPA Method 200.7 Trace Elements in Water, Solids, and Biosolids by Inductivity Coupled Plasma-Atomic Emission Spectrometry. However, selection of wavelengths that are interference free would be an ideal solution in all cases this is especially true when carrying out IEC corrections close to the detection limit of the instrument (as the small uncertainty associated with any measurement can be critical). However, given the spectral complexity of many matrices wavelength selection is a challenge for many analyst as many components of the sample are unknown leading to unexpected interferences.

The Element Finder plug-in for the Qtegra ISDS software is a method development tool that allows for the selection of analytical wavelengths that are free from spectral interferences taking into account the analytes present in the sample and the sample matrix. By utilizing Fullframes (an image of the entire sample spectrum, within the wavelength of the instrument) of the sample, the software intelligently selects the wavelengths that are interference free for the elements you have selected. In this way the Element Finder plug-in helps to save time and cost associated with long and cumbersome method development.

Steps to utilize the Element Finder plug-in for Qtegra ISDS

1. Select the element you wish to analyze in the sample (in this case boron); we can see there are, potentially, seven wavelengths for analysis.

Figure 6. Selection of analyte elements.

2. Optional step - Select the matrix elements in the sample (if they are known). Based on this the Element Finder plug-in will rationalize the wavelengths used for the analysis (if the matrix elements will interfere with the analyte elements).

3. Run a Fullframe using a typical sample containing your matrix and analyte elements (the matrix is iron and the analyte is boron). The Fullframe will be measured and then analyzed for all elements present in the sample.

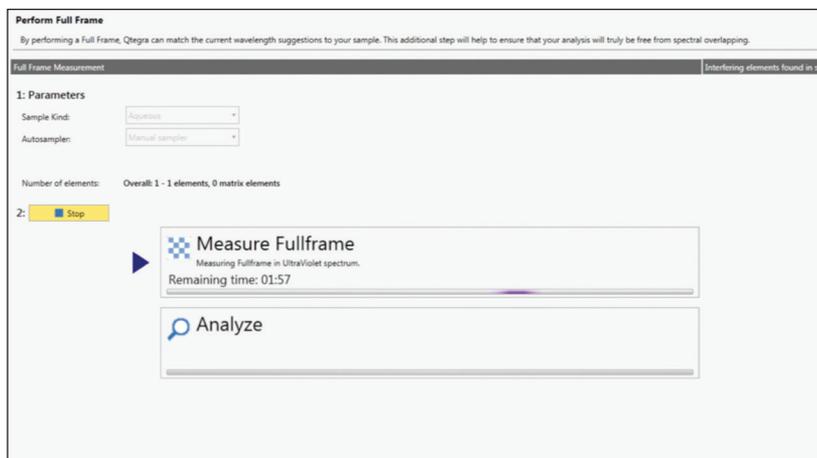


Figure 7. The measurement of a Fullframe within the Element Finder plug-in.

In this case we can see that the Fullframe analysis took place for 76 elements and two were identified in the sample. Elements that are not measurable by ICP-OES are excluded from the Fullframe analysis (elements with a short half-life etc).

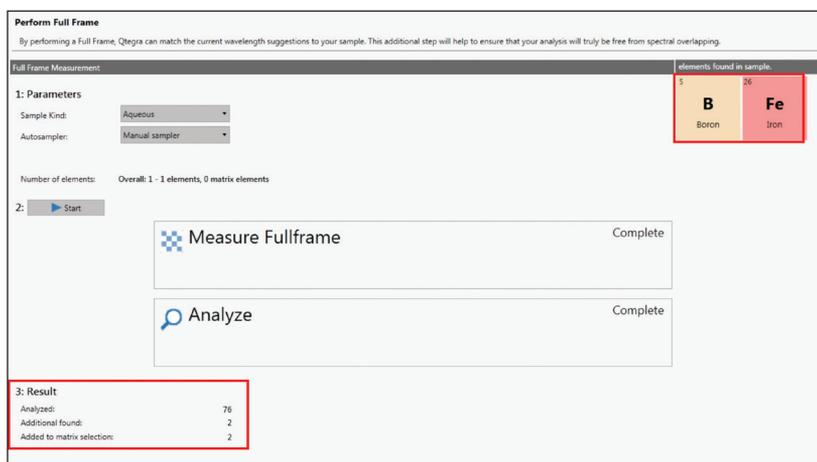


Figure 8. Results of the Fullframe analysis show there are two elements present in the sample (the analyte and the matrix).

4. Taking into account the matrix elements found during the Fullframe analysis the Element Finder plug-in will then suggest the wavelengths to be used for the analysis of the selected analyte. In this case we can see that B 208.959 nm has been selected for the analysis. The suggested wavelengths can then be imported into a LabBook and used for the analysis.

According to the selections on the previous screens, Qtegra has suggested the wavelengths displayed here for your analysis. They have been determined to be free from spectral overlapping. If desired

		Show 1 Line	Show Matrix Elements	Recalculate			
5 B Boron	Wavelength	Intensity	Preference	Used	Measure Mode	Remarks	
	249.773	4,000,000	Automatic	<input type="checkbox"/>	Radial	Interference with: Fe, 249.782; Interference with: Fe, 249.653;	
	249.678	2,000,000	Automatic	<input type="checkbox"/>	Radial	Interference with: Fe, 249.653; Interference with: Fe, 249.782;	
	208.959	1,500,000	Automatic	<input checked="" type="checkbox"/>	Radial		
26 Fe Iron Matrix	Wavelength	Intensity	Preference	Used	Measure Mode	Remarks	
	249.653	4,000	Include	<input type="checkbox"/>	Radial	Interference with: B, 249.678; Interference with: B, 249.773;	
	249.782	3,000	Include	<input type="checkbox"/>	Radial	Interference with: B, 249.678; Interference with: B, 249.773;	
	208.412	7,000	Include	<input checked="" type="checkbox"/>	Radial	Included because this is a matrix element.	

Conclusion

Through careful method development and understanding of interferences, precise and accurate results can be achieved for almost any sample. This process is simplified with the Thermo Scientific Qtegra ISDS Element Finder plug-in which correctly identifies interfering elements, and enables the analyst to automate their method development. The iCAP™ 7000 Plus Series ICP-OES also offers the inherent benefits of CID detector technology, and with over 55,000 wavelengths to choose from, complementing the benefits of the Element Finder plug-in.

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