Wavelength Calibration in ICP Optical Emission Spectroscopy

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Introduction

ICP-OES is a powerful elemental analysis technique which utilizes the UV/Visible spectrum (typically 165 to 800nm) to measure emission from most elements in the periodic table. When configured correctly, today's instruments yield detection limits of 1 to 10 ppb for the majority of elements.

What is Wavelength Calibration?

Minute variations in diffraction gratings cause every ICP-OES instrument to have a unique correlation between emission wavelengths and where each wavelength falls on the instrument's array detector. Wavelength calibration is the process whereby that correlation is established and then stored in the instrument's software for use during subsequent analytical measurements. It is the process used to "teach" the instrument exactly which pixels of its array detector correspond to each emission wavelength.

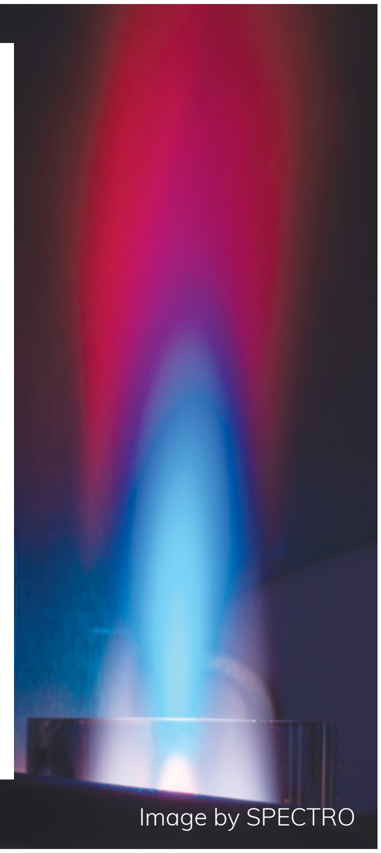

Image by SPECTRO

Why is Wavelength Calibration Important?

The presence or absence of emission lines is used for qualitative identification of each element, whereas emission intensity is used for quantification. Wavelength calibration helps the instrument and analyst ensure that they are measuring each element at the proper location in the spectrum. Below we will see how wavelength calibration impacts virtually every aspect of your ICP's performance, including its sensitivity (detection limits), stability (RSDs), and susceptibility to interferences.

Figure 1 is a typical ICP spectrum for a solution that contains 15 elements. The spectrum is clearly complex and accurate quantitation will require measurement of the emission from each element at exactly the right location in the spectrum. The inset region of the spectrum in **Figure 1** shows the emission line for Cd at 214.44 nm. The vertical black line in the middle of the peak shows where the instrument expects the center of the emission line to be located following wavelength calibration. A small region around the center of the emission line, typically just a few pixels wide, is used to measure emission intensity.   When the emission line is well centered, signal intensity will be maximized contributing to lower detection limits. Further, any slight wavelength drift on the detector elements will have minimal impact on signal stability contributing to more favorable measurement precision (lower RSDs).

Consider for a moment the impact of inaccurate wavelength calibration. For example, what if your ICP was attempting to measure the Cd 214.44 at the vertical green line shown in the inset graph on **Figure 1**. In this case the emission intensity would be roughly half of what it should be. This would contribute to a significant degradation in the instruments detection limit for Cd. Further, in this case, slight wavelength shifts would result in large signal changes which would significantly degrade measurement precision (higher RSDs). Wavelength calibration accuracy also plays a critical role in an instrument's susceptibility to spectral interferences.

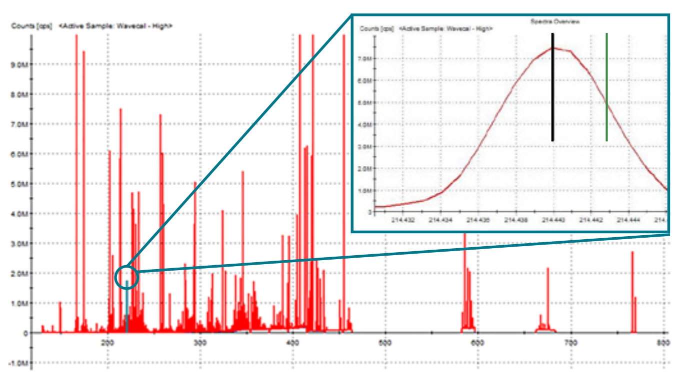

Figure 1. Typical ICP-OES Spectrum – 15 Elements at 50 ppm

Wavelength Calibration and Spectral Interferences

An example of the critical role that wavelength calibration plays in an instrument's susceptibility to spectral interferences is shown in **Figure 2**. In this spectrum a shoulder is clearly visible on the right side of our analyte peak indicating the presence of a spectral interference. In this case our analyte emission is from boron at 249.773 nm and the interfering emission is from iron at 249.785 nm. The cause of the shoulder becomes quite clear when the spectrum from the mixture is overlaid with those for single-element solutions containing 1 ppm B, and seperately 100 ppm Fe. This is a real-world example of an analytical challenge associated with attempting to measure the amount of boron contained in steel. In this case if the instruments wavelength calibration is good and we are measuring our analyte peak at exactly the correct wavelength (reference the vertical black line in the spectrum) there will be relatively little contribution from iron on our analyte peak. In fact, this magnitude of interference can be easily handled with appropriate interelement corrections which are part of most instruments' software. Once again, even a very small amount of wavelength drift will have relatively little impact on the quality of our measurements. However, if we are working with a less favorable wavelength calibration, say the instrument is attempting to measure the boron emission at the spectral location associated with the vertical red line, then the interference from iron will be far more severe and any wavelength drift will magnify the problem significantly.

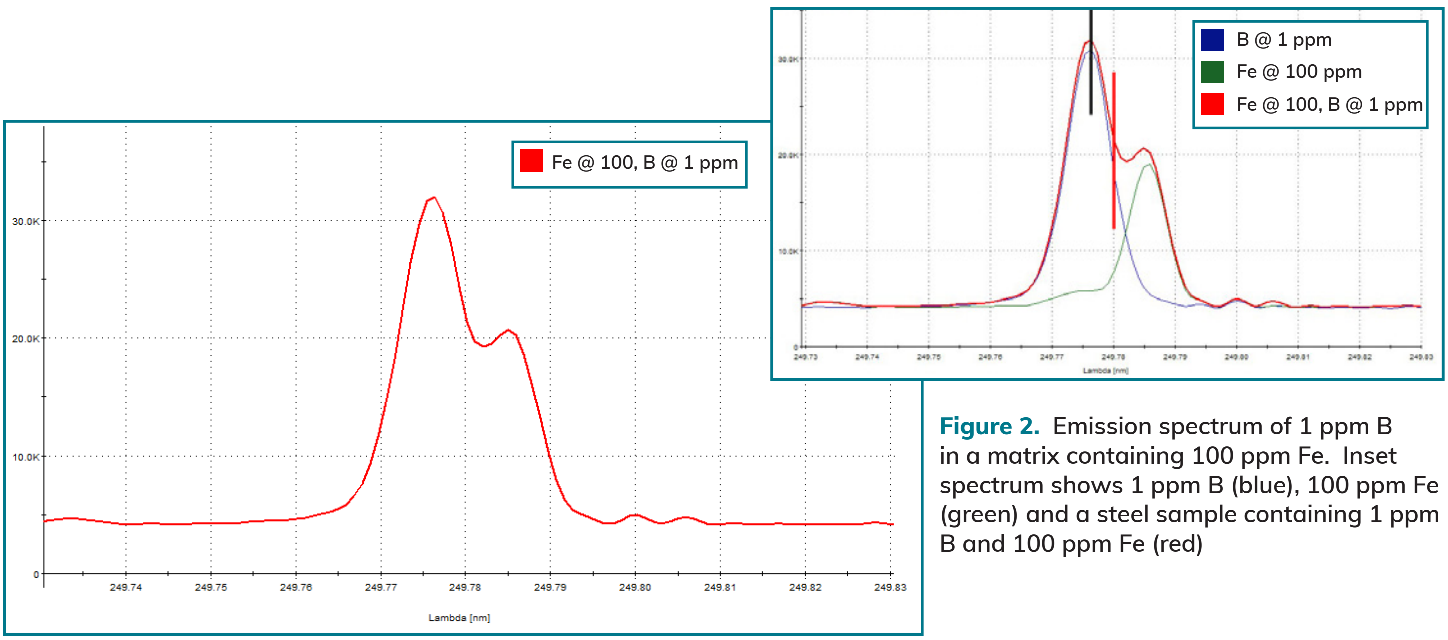

Figure 2. Emission spectrum of 1 ppm B in a matrix containing 100 ppm Fe. Inset spectrum shows 1 ppm B (blue), 100 ppm Fe (green) and a steel sample containing 1 ppm B and 100 ppm Fe (red)

How Wavelength Calibration is Carried Out

Wavelength calibration in ICP-OES is typically achieved by aspirating a multielement solution which will produce elemental emission for each element contained in the solution. The specific elements are chosen to produce relatively simple spectra while also covering the relevant wavelength range of the instrument.

All modern ICP-OES instruments incorporate an automated wavelength calibration routine. The instrument operator simply loads the wavelength calibration solution designed for use with the their model instrument and the software does the rest.

VHG™, an LGC Standards company, manufactures a complete line of wavelength calibration solutions for ICP-OES and tuning solutions for ICP-MS. Many are are listed in the table on the following page. For a full list of ICP-OES and ICP-MS Start Up Solutions, please visit www2.lgcgroup.com/VHG_StartUpSolutions.

Wavelength Calibration Solutions

Wavelength Calibration and Related Solutions for ICP-OES				
Description	Composition	Product No.	mL	Suitable for use with
Low UV Wave Cal Solution	Al, P, S @ 10 µg/mL in 2% HNO_3	VHG-ISUPE-LOW-250	250	PerkinElmer® ICP-OES
VIS Wave Cal Solution	K @ 50 µg/mL; La, Li, Mn, Na, Sr @ 10 µg/mL; Ba, Ca @ 1 µg/mL in 2% HNO_3	VHG-ISUPEVIS-250	250	PerkinElmer® ICP-OES: Optima® / Avio®
UV Wave Cal Solution	K, P, S @ 100 µg/mL; As, La, Li, Mn, Mo, Na, Ni, Sc @ 20 µg/mL; Ca @ 1 µg/mL in 5% HCl	VHG-ISUPEUVW-500	500	PerkinElmer® ICP-OES: Optima® / Avio®
Multi-Element Setup Standard	As, K @ 50 µg/mL; La, Li, Mn, Ni, Sr, Zn @ 10 µg/mL; Ba, Mg @ 1 µg/mL in 2% HNO_3	VHG-ISUPEOPTME-500	500	PerkinElmer® ICP-OES: Optima®
Instrument Check Standard 3	K, P, S @ 100 µg/mL; As, La, Li, Mn, Mo, Na, Ni, Sc @ 20 µg/mL in 5% HCl	VHG-ISUPECHKSTD3-250	250	PerkinElmer® ICP-OES
Instrument Calibration Standard 4	As, Tl @ 100 µg/mL; Cd, Pb, Se @ 50 µg/mL in 5% HNO_3	VHG-ISUPECAL4-100	100	PerkinElmer® ICP-OES: Optima® / Avio®
ICAL Solution	S @ 50 µg/mL; Ce, Cu, Eu, Fe, In, K, Ni, P, Si, Ti, V, Y, Zr @ 10 µg/mL; Mn, Mo, Na, Sc @ 5 µg/mL; Be, Li, Sr @ 2 µg/mL; Ca @ 1 µg/mL in 2% HNO_3, 2% HCl	VHG-ISUSPCTICAL-250	250	SPECTGRO ICP-OES: Genesis®, ARCOS®, Blue®