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PRODUCT SPOTLIGHT

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Thermo Scientific iCAP PRO Series ICP-OES

Achieving effective interference correction

Inductively coupled plasma optical emission spectrometry (ICP-OES) is a robust and efficient analytical technique for measuring trace elements in a wide variety of sample types. However, the presence of interferences on some of the elements of interest from wavelengths emitted from other elements in the sample is a well-known challenge. These interferences can arise from direct overlap, where the interfering wavelength is the same as that of the analyte wavelength, or from partial overlap, where the interfering wavelength lies within a few picometers of that of the analyte or the analyte peak lies on the edge of another peak that is outside of the sub array window. With the Thermo Scientific™ iCAP™ PRO Series ICP-OES instruments, these interferences can easily and accurately be corrected for by using the interelement correction (IEC) protocol included with the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software suite.

The key requirements for IEC are the ability to correct the interfered element signals on the basis of either the interfering wavelength's signal intensity (in counts per second, cps) or its equivalent concentration relative to the concentration of the element to be measured. Correcting on the basis of signal intensities is a straightforward process of subtracting the interference signal from the signal of the element of interest, after correcting both signals for any drift or suppression, using internal standards. Correcting on the basis of concentration, as stipulated in regulatory protocols such as US EPA Method 200.7: Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry and US EPA Method 6010D (SW-846): Inductively Coupled Plasma – Atomic Emission Spectrometry methods, is more demanding. This requires a clear, systematic approach for measuring the interfering wavelength signal, calculating its apparent concentration relative to the element that it is interfering with, and then subtracting that concentration from the measured concentration of the element. Both approaches can be used in the IEC workflow of the Qtegra ISDS Software, which controls the iCAP PRO Series ICP-OES instruments.

The principal steps for correction based on concentration in the IEC workflow are summarised in the example shown in Figure 1.

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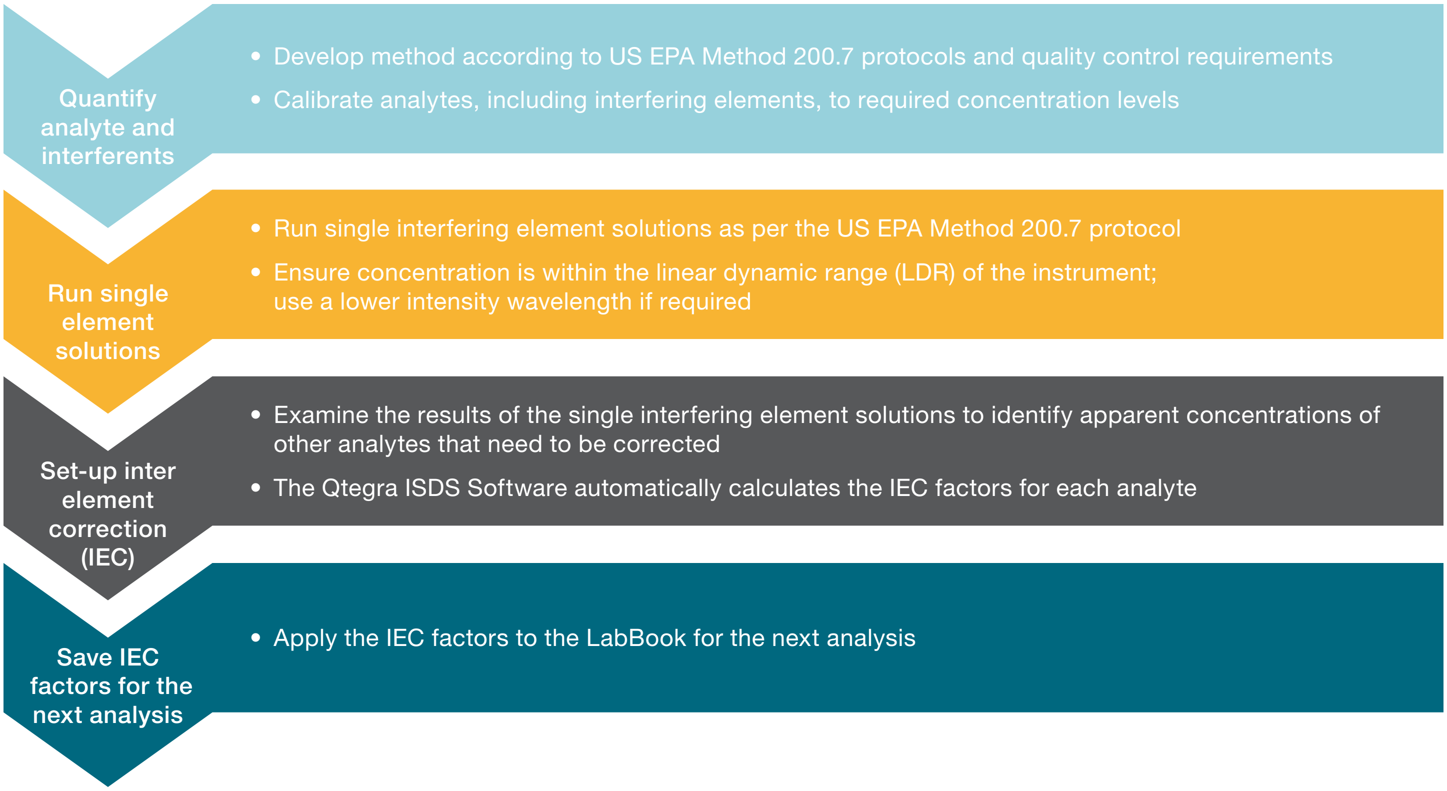


Quantify analyte and interferents

- Develop method according to US EPA Method 200.7 protocols and quality control requirements
- Calibrate analytes, including interfering elements, to required concentration levels

Run single element solutions

- Run single interfering element solutions as per the US EPA Method 200.7 protocol
- Ensure concentration is within the linear dynamic range (LDR) of the instrument; use a lower intensity wavelength if required

Set-up inter element correction (IEC)

- Examine the results of the single interfering element solutions to identify apparent concentrations of other analytes that need to be corrected
- The Qtegra ISDS Software automatically calculates the IEC factors for each analyte

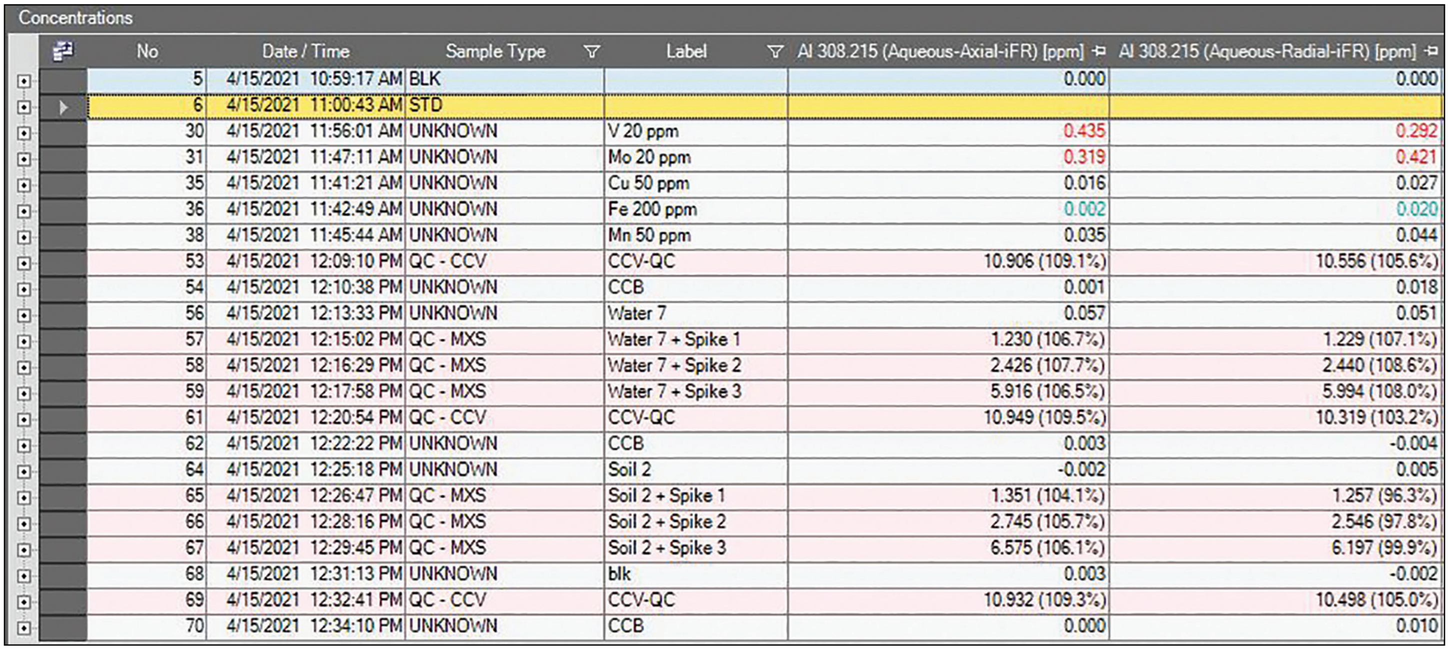
Save IEC factors for the next analysis

- Apply the IEC factors to the LabBook for the next analysis

Figure 1. Example workflow for interelement correction (IEC) in Qtegra ISDS Software for the iCAP PRO ICP-OES for the analysis of samples according to US EPA Method 200.7

In addition to a comprehensive, yet simple to follow IEC workflow within the iCAP PRO Series ICP-OES LabBook environment, the Qtegra ISDS Software allows the user to set pre-defined concentration limits, with color-coded flagging functionality to enable data affected by interelement interference in an analysis to be easily identified in the results table (Figure 2).

With appropriate limits set up, data that are positively biased because of an interference on the analyte peak are highlighted in red, and results that are negative because of incorrect background subtraction due to background interference signals on the left or right side of the peak are highlighted in blue, as shown in Figure 2.

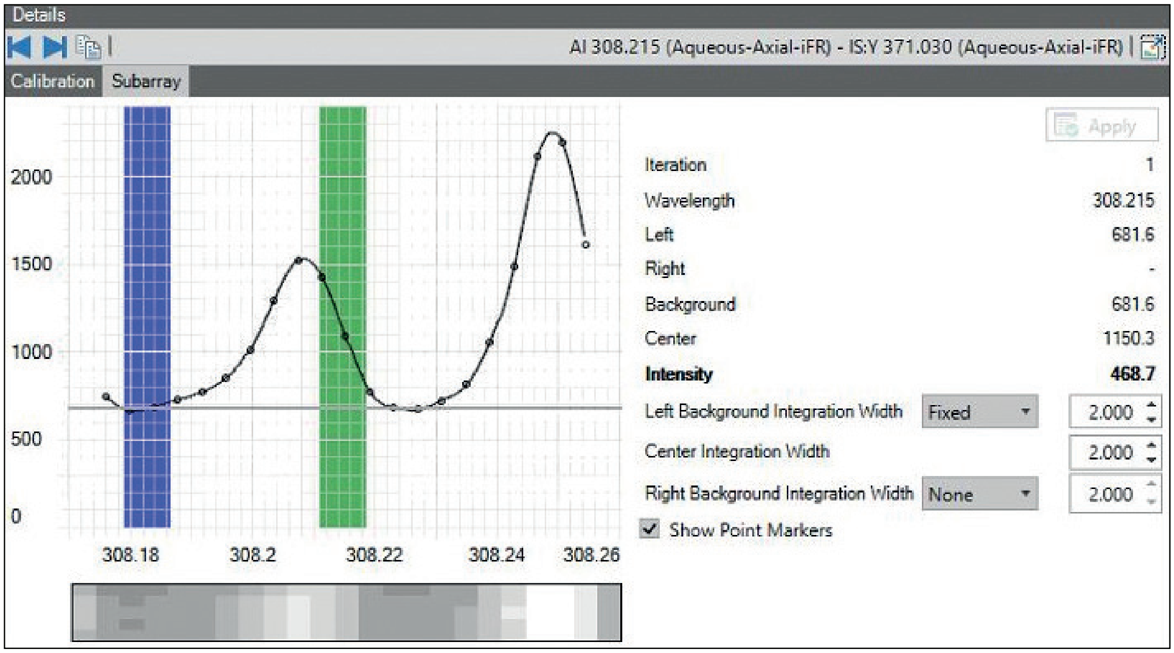


Concentrations

No	Date / Time	Sample Type	Label	Al 308.215 (Aqueous-Axial-iFR) [ppm]	Al 308.215 (Aqueous-Radial-iFR) [ppm]
5	4/15/2021 10:59:17 AM	BLK		0.000	0.000
6	4/15/2021 11:00:43 AM	STD			
30	4/15/2021 11:56:01 AM	UNKNOWN	V 20 ppm	0.435	0.292
31	4/15/2021 11:47:11 AM	UNKNOWN	Mo 20 ppm	0.319	0.421
35	4/15/2021 11:41:21 AM	UNKNOWN	Cu 50 ppm	0.016	0.027
36	4/15/2021 11:42:49 AM	UNKNOWN	Fe 200 ppm	0.002	0.020
38	4/15/2021 11:45:44 AM	UNKNOWN	Mn 50 ppm	0.035	0.044
53	4/15/2021 12:09:10 PM	QC - CCV	CCV-QC	10.906 (109.1%)	10.556 (105.6%)
54	4/15/2021 12:10:38 PM	UNKNOWN	CCB	0.001	0.018
56	4/15/2021 12:13:33 PM	UNKNOWN	Water 7	0.057	0.051
57	4/15/2021 12:15:02 PM	QC - MXS	Water 7 + Spike 1	1.230 (106.7%)	1.229 (107.1%)
58	4/15/2021 12:16:29 PM	QC - MXS	Water 7 + Spike 2	2.426 (107.7%)	2.440 (108.6%)
59	4/15/2021 12:17:58 PM	QC - MXS	Water 7 + Spike 3	5.916 (106.5%)	5.994 (108.0%)
61	4/15/2021 12:20:54 PM	QC - CCV	CCV-QC	10.949 (109.5%)	10.319 (103.2%)
62	4/15/2021 12:22:22 PM	UNKNOWN	CCB	0.003	-0.004
64	4/15/2021 12:25:18 PM	UNKNOWN	Soil 2	-0.002	0.005
65	4/15/2021 12:26:47 PM	QC - MXS	Soil 2 + Spike 1	1.351 (104.1%)	1.257 (96.3%)
66	4/15/2021 12:28:16 PM	QC - MXS	Soil 2 + Spike 2	2.745 (105.7%)	2.546 (97.8%)
67	4/15/2021 12:29:45 PM	QC - MXS	Soil 2 + Spike 3	6.575 (106.1%)	6.197 (99.9%)
68	4/15/2021 12:31:13 PM	UNKNOWN	blk	0.003	-0.002
69	4/15/2021 12:32:41 PM	QC - CCV	CCV-QC	10.932 (109.3%)	10.498 (105.0%)
70	4/15/2021 12:34:10 PM	UNKNOWN	CCB	0.000	0.010

Figure 2. Main results table with data affected by interelement interference highlighted (blue = below the pre-defined lower limit, red = above the pre-defined higher limit). Only Al data is shown in this example, for clarity.

Selecting a highlighted result opens up the calibration and sub-array measurement for the interfered wavelength, enabling the user to clearly see the effect of the interference on the target analyte and also determine if there are any interferences on the background signal on either side of the analyte peak. Interfered background points can be moved to an interference-free region or either the left or right background correction (whichever is interfered) can be removed (Figure 3). IEC correction on the target wavelength is then subsequently applied by first importing the set of single element solution data into the IEC tab, and then selecting each element interference in turn for those results where interference has been highlighted by color-coded flags (Figure 4) and finally applying the corrections to produce the interference corrected results for the single interference check solutions (Figure 5). At the same time, the interference corrections are automatically applied to all the other samples in the analysis, as illustrated in Figure 6.

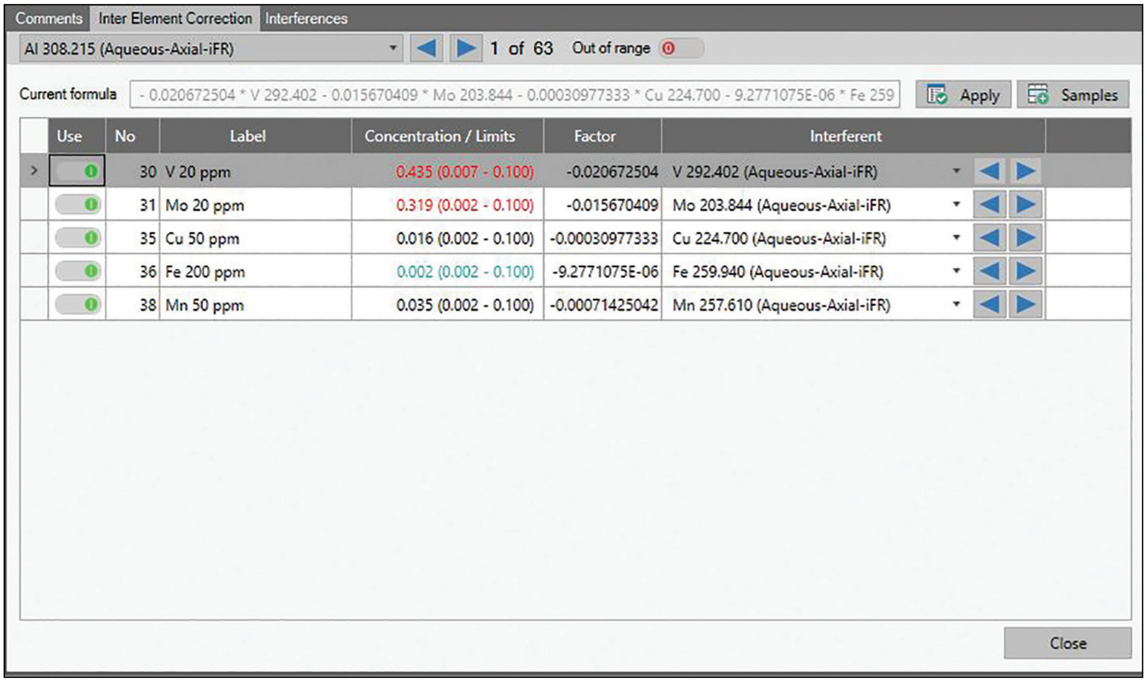


Al 308.215 (Aqueous-Axial-iFR) - IS:Y 371.030 (Aqueous-Axial-iFR)

Iteration		1
Wavelength		308.215
Left		681.6
Right		-
Background		681.6
Center		1150.3
Intensity		**468.7**
Left Background Integration Width	Fixed	2.000
Center Integration Width		2.000
Right Background Integration Width	None	2.000

☑ Show Point Markers

Figure 3. Application of background interference correction and single element solution data import. (The peak at 308.211 nm is due to vanadium interference on the Al 308.215 nm emission line.)

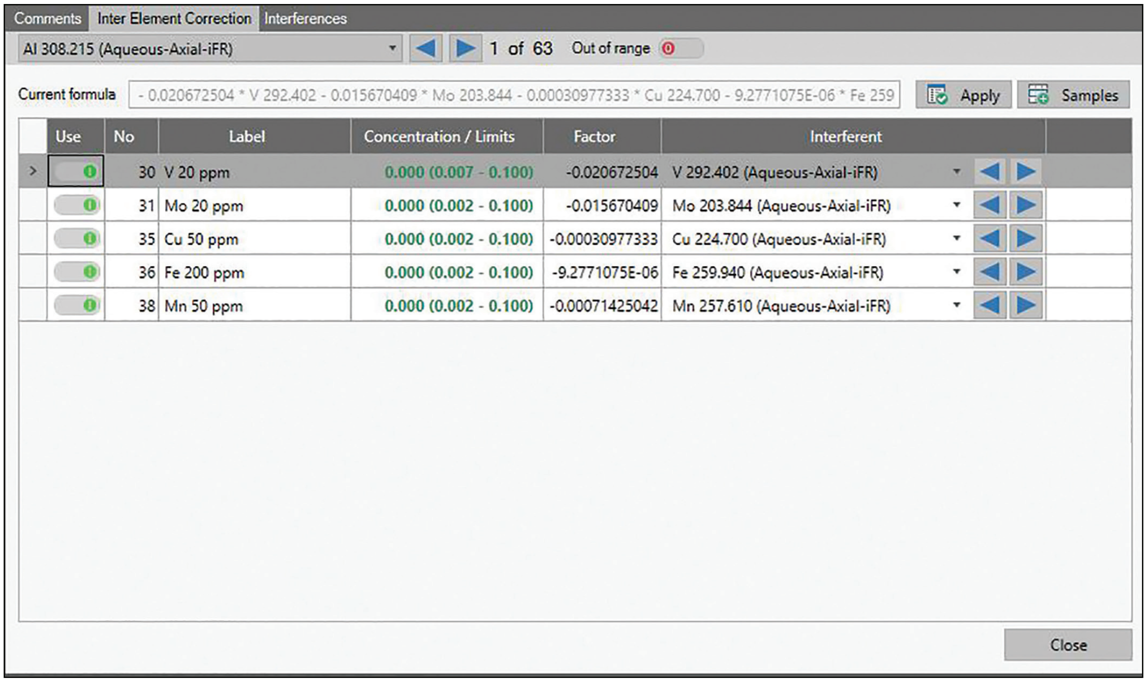


Al 308.215 (Aqueous-Axial-iFR) 1 of 63 Out of range 0

Current formula - 0.020672504 * V 292.402 - 0.015670409 * Mo 203.844 - 0.00030977333 * Cu 224.700 - 9.2771075E-06 * Fe 259

Use	No	Label	Concentration / Limits	Factor	Interferent
	30	V 20 ppm	0.435 (0.007 - 0.100)	-0.020672504	V 292.402 (Aqueous-Axial-iFR)
	31	Mo 20 ppm	0.319 (0.002 - 0.100)	-0.015670409	Mo 203.844 (Aqueous-Axial-iFR)
	35	Cu 50 ppm	0.016 (0.002 - 0.100)	-0.00030977333	Cu 224.700 (Aqueous-Axial-iFR)
	36	Fe 200 ppm	0.002 (0.002 - 0.100)	-9.2771075E-06	Fe 259.940 (Aqueous-Axial-iFR)
	38	Mn 50 ppm	0.035 (0.002 - 0.100)	-0.00071425042	Mn 257.610 (Aqueous-Axial-iFR)

Figure 4. Application of interference correction to results that are highlighted as being interfered (using the single element solution data)

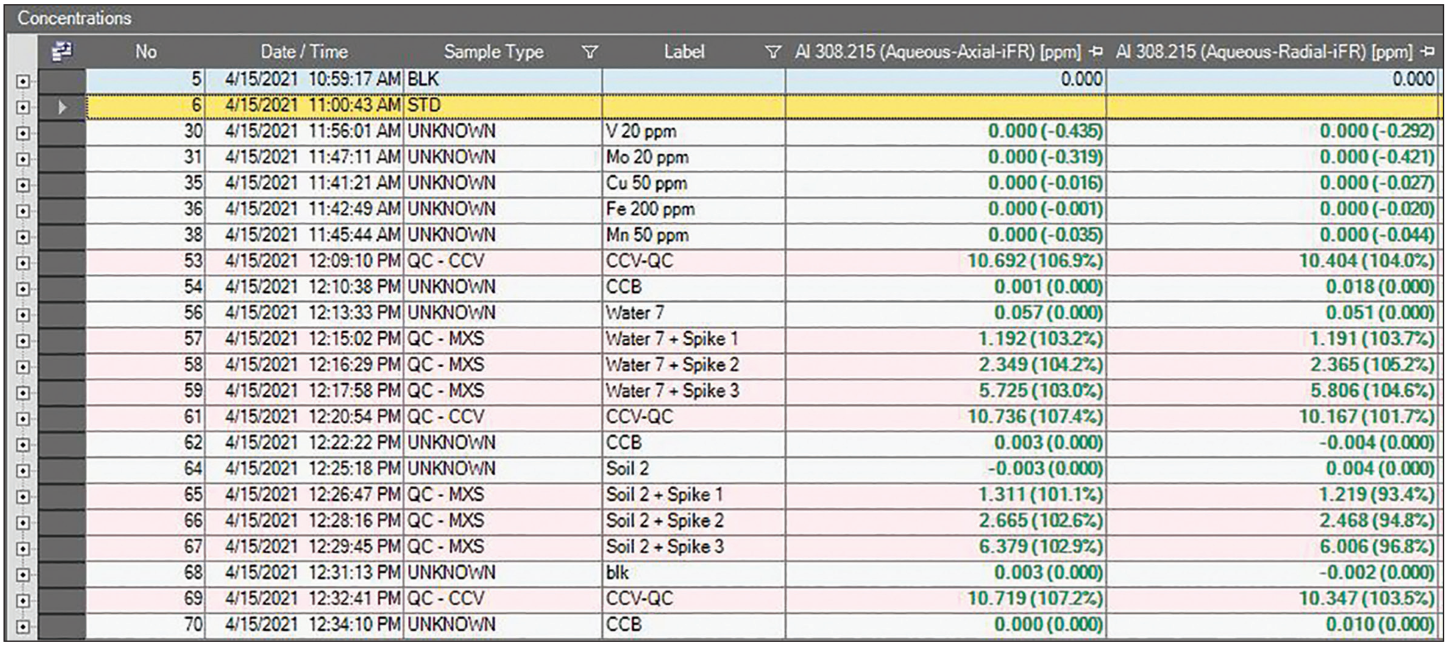


Al 308.215 (Aqueous-Axial-iFR) 1 of 63 Out of range

Current formula - 0.020672504 * V 292.402 - 0.015670409 * Mo 203.844 - 0.00030977333 * Cu 224.700 - 9.2771075E-06 * Fe 259

Use	No	Label	Concentration / Limits	Factor	Interferent
●	30	V 20 ppm	0.000 (0.007 - 0.100)	-0.020672504	V 292.402 (Aqueous-Axial-iFR)
●	31	Mo 20 ppm	0.000 (0.002 - 0.100)	-0.015670409	Mo 203.844 (Aqueous-Axial-iFR)
●	35	Cu 50 ppm	0.000 (0.002 - 0.100)	-0.00030977333	Cu 224.700 (Aqueous-Axial-iFR)
●	36	Fe 200 ppm	0.000 (0.002 - 0.100)	-9.2771075E-06	Fe 259.940 (Aqueous-Axial-iFR)
●	38	Mn 50 ppm	0.000 (0.002 - 0.100)	-0.00071425042	Mn 257.610 (Aqueous-Axial-iFR)

Figure 5. Results after IEC correction applied



Concentrations

No	Date / Time	Sample Type	Label	Al 308.215 (Aqueous-Axial-iFR) [ppm]	Al 308.215 (Aqueous-Radial-iFR) [ppm]
5	4/15/2021 10:59:17 AM	BLK		0.000	0.000
6	4/15/2021 11:00:43 AM	STD			
30	4/15/2021 11:56:01 AM	UNKNOWN	V 20 ppm	0.000 (-0.435)	0.000 (-0.292)
31	4/15/2021 11:47:11 AM	UNKNOWN	Mo 20 ppm	0.000 (-0.319)	0.000 (-0.421)
35	4/15/2021 11:41:21 AM	UNKNOWN	Cu 50 ppm	0.000 (-0.016)	0.000 (-0.027)
36	4/15/2021 11:42:49 AM	UNKNOWN	Fe 200 ppm	0.000 (-0.001)	0.000 (-0.020)
38	4/15/2021 11:45:44 AM	UNKNOWN	Mn 50 ppm	0.000 (-0.035)	0.000 (-0.044)
53	4/15/2021 12:09:10 PM	QC - CCV	CCV-QC	10.692 (106.9%)	10.404 (104.0%)
54	4/15/2021 12:10:38 PM	UNKNOWN	CCB	0.001 (0.000)	0.018 (0.000)
56	4/15/2021 12:13:33 PM	UNKNOWN	Water 7	0.057 (0.000)	0.051 (0.000)
57	4/15/2021 12:15:02 PM	QC - MXS	Water 7 + Spike 1	1.192 (103.2%)	1.191 (103.7%)
58	4/15/2021 12:16:29 PM	QC - MXS	Water 7 + Spike 2	2.349 (104.2%)	2.365 (105.2%)
59	4/15/2021 12:17:58 PM	QC - MXS	Water 7 + Spike 3	5.725 (103.0%)	5.806 (104.6%)
61	4/15/2021 12:20:54 PM	QC - CCV	CCV-QC	10.736 (107.4%)	10.167 (101.7%)
62	4/15/2021 12:22:22 PM	UNKNOWN	CCB	0.003 (0.000)	-0.004 (0.000)
64	4/15/2021 12:25:18 PM	UNKNOWN	Soil 2	-0.003 (0.000)	0.004 (0.000)
65	4/15/2021 12:26:47 PM	QC - MXS	Soil 2 + Spike 1	1.311 (101.1%)	1.219 (93.4%)
66	4/15/2021 12:28:16 PM	QC - MXS	Soil 2 + Spike 2	2.665 (102.6%)	2.468 (94.8%)
67	4/15/2021 12:29:45 PM	QC - MXS	Soil 2 + Spike 3	6.379 (102.9%)	6.006 (96.8%)
68	4/15/2021 12:31:13 PM	UNKNOWN	blk	0.003 (0.000)	-0.002 (0.000)
69	4/15/2021 12:32:41 PM	QC - CCV	CCV-QC	10.719 (107.2%)	10.347 (103.5%)
70	4/15/2021 12:34:10 PM	UNKNOWN	CCB	0.000 (0.000)	0.010 (0.000)

Figure 6. Example of interference corrected data in the main results table. Only Al data is shown in this example, for clarity.

To make the process more streamlined, only those element wavelengths that are out of range of the defined concentration limits in any sample in the analysis can be selected using the “Out of range” toggle button shown at the top of Figures 4 and 5. With this option enabled, clicking the forward arrow to the left of the “Out of range” button jumps sequentially to each element wavelength that has an out of range result, bypassing all the other, non-interfered wavelengths.

Once all the interelement interference and background corrections have been identified and applied for the single element solutions, the corrections are stored in the LabBook and automatically applied to all other samples in the analysis.

For subsequent analytical runs, the IEC factors and background correction settings determined from the above procedure can then be saved as a LabBook template or copied over into a new LabBook created from the one used to generate the corrections. The user can then verify that the corrections are still valid by running a Spectral Interference Check (SIC) solution as described in sections 7.12 and 7.13 of US EPA Method 200.7, after running the calibration blank and standard solutions and before running the sample batch. For more information about the practical application of interference correction in accordance with the requirements of US EPA Method 200.7, see Thermo Scientific iCAP PRO ICP-OES Application Note 000056.[1]

The exceptional optical stability of the Thermo Scientific iCAP PRO ICP-OES enables highly stable IEC factors to be achieved. Examples of peak position stability (for four overlaid axial and radial readings of the Mg 279.079 nm and Ca 315.887 nm wavelengths in a surface water sample, with each reading taken at 4-hour intervals during a 16 hour analytical sequence) are shown in Figure 7, and the stability of 11 measured IEC factors over a 20 day period is shown in Table 1. If/when the existing factors are no longer sufficiently accurate, updated factors can be obtained by running the single element standards again within the same LabBook as the SIC solution, following the steps described above.

With this advanced interelement correction protocol, consistently accurate results can be confidently achieved with the Thermo Scientific iCAP PRO Series ICP-OES instruments for elemental analysis in a diverse range of applications.

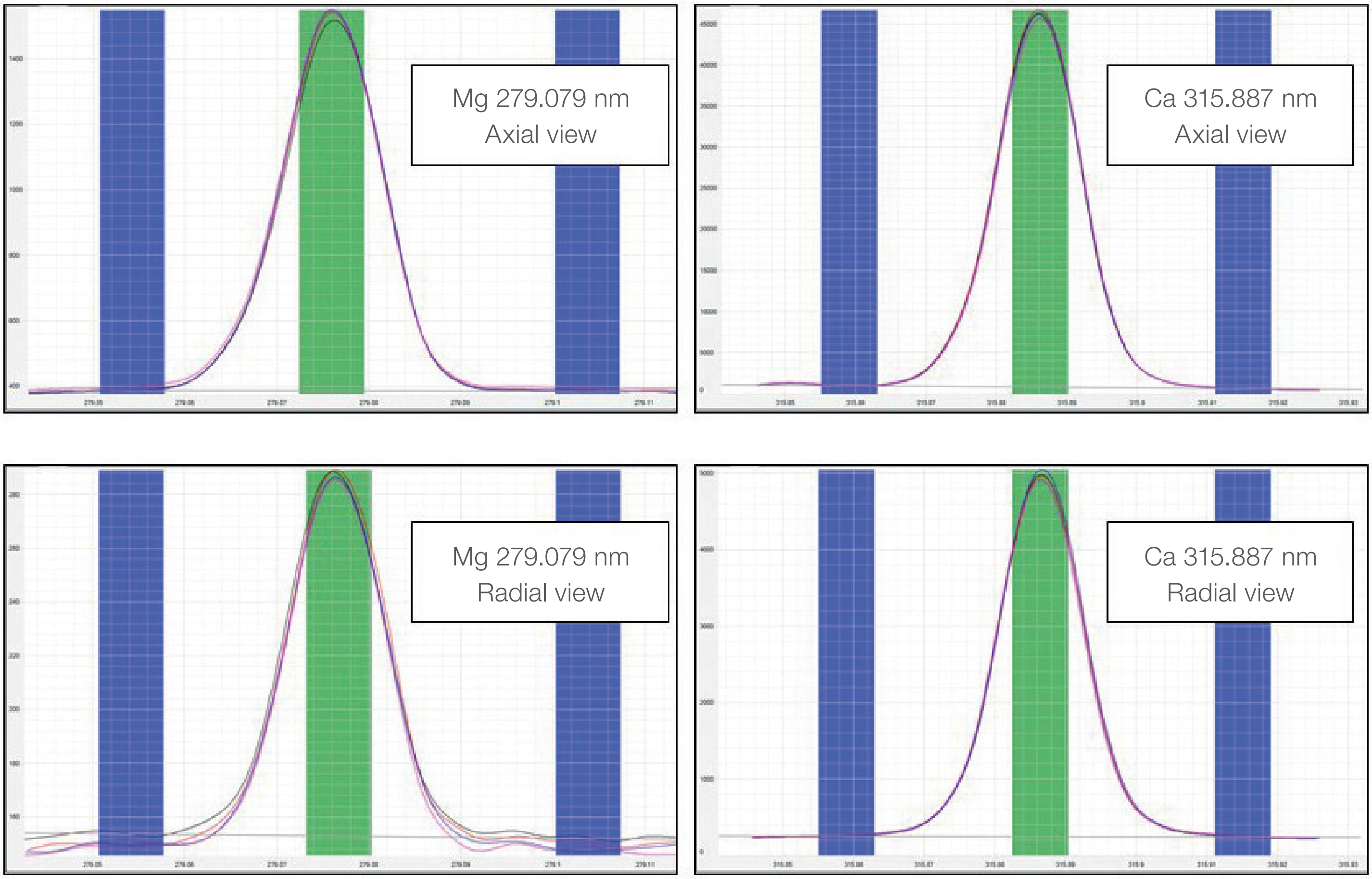


Figure 7. Peak position stability with the Thermo Scientific iCAP PRO ICP-OES

Table 1. Normalized IEC factor stability for a range of interferences described in US EPA Method 200.7

Analyte	Interferent	Day 1	Day 8	Day 14	Day 20	% RSD
Ag	Ce	1.00	1.03	1.04	1.08	3.0
Al	Ce	1.00	0.99	0.97	1.02	1.9
Al	Mo	1.00	1.00	0.98	0.98	1.3
Al	V	1.00	0.96	1.03	1.02	3.2
As	Al	1.00	1.01	1.00	1.00	0.5
P	Cu	1.00	1.02	1.04	0.98	2.7
Sb	Cr	1.00	0.95	0.98	1.00	2.3
Tl	Co	1.00	0.98	1.02	1.01	1.7
Tl	Ti	1.00	1.09	1.07	1.03	4.0
V	Ti	1.00	1.07	0.99	1.08	4.6
Pb	Al	1.00	0.91	0.96	1.00	4.4

Reference

1. Thermo Scientific Application Note 000056: Rapid, interference-free analysis of water according to US EPA Method 200.7. https://assets.thermofisher.com/TFS-Assets/CMD/Application-Notes/an-000056-tea-water-analysis-epa200.7-icap-pro-icp-oes-an000056-na-en.pdf

Find out more at **thermofisher.com/ICP-OES**