Application Example –
PlasmaQuant® PQ 9000 Elite

Composition Analysis of Animal Feed by HR ICP-OES

Abstract

The High-Resolution Array ICP-OES PlasmaQuant® PQ 9000 Elite equipped with Standard-Kit was used for the analysis of Al, B, Ca, Co, Cr, Cu, Fe, I, K, Na, Mn, Mg, Mo, Ni, P, S, V and Zn in an animal feed sample.

The sample was digested in 3 mL HNO_3 and 0.5 mL HCl by microwave (TOPWAVE®), filled up to a total volume of 20 mL, subsequently diluted by factor 50 to give a nominal matrix concentration of 0.5 g/L and measured with an aqueous standard calibration routine without matrix matching using 1000 mg/L multiple and single element standards from SIGMA ALDRICH. Method robustness was further tested from comparison of multiple lines for elements prone to spectral interferences (e.g. Cu, Ni, P, V and Zn) and a spike recovery test.

Trace impurities and matrix-elements including Calcium, Iodine, Magnesium, Sodium and Potassium were determined from the same sample with excellent precision using Dual View PLUS. That is agreement of results, good recovery rates and RSD values about 1% were realized using interference-free emission lines for all elements. However, for Iodine prominent Phosphorous interferences on the I178.215 nm emission line were successfully removed by the CSI-Tool, which rules out overestimation of Iodine in Phosphorous-rich matrices.

Matrix-specific detection limits smaller than 1 µg/L (ppb) for all metals and 5 ppb for Iodine were achieved here, which highlights the unique sensitivity of the PlasmaQuant® PQ 9000 Elite benefiting QC labs dealing with animal feed samples.

Challenge

Interference-free Dual View PLUS method, reduction of detection limits

Matrix

Animal feed (HNO_3, HCl)

Purpose

Method development for QC lab

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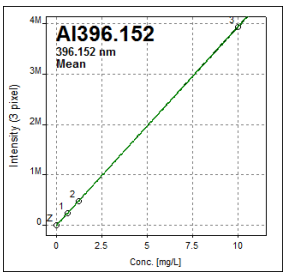
Materials and Methods

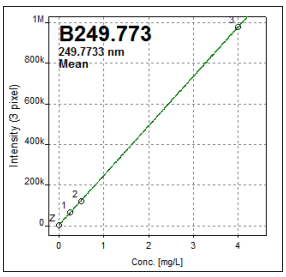
Calibration

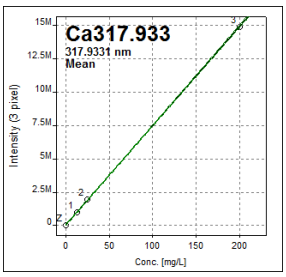
Table 1: Concentration of calibration standards

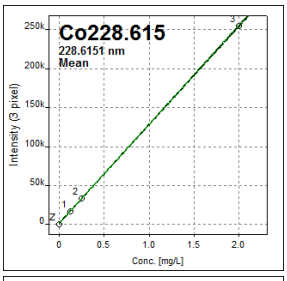
Element	Unit	Cal.0	Cal.1	Cal.2	Cal.3	Cal.4
Al	mg/L	0	0.625	1.25	10	-
B	mg/L	0	0.25	0.5	4	-
Ca, K	mg/L	0	12.5	25	200	-
Co, Cr, Mo, Ni, V	mg/L	0	0.125	0.25	2	-
Cu, Zn	mg/L	0	0.1875	0.375	3	8
Fe	mg/L	0	0.5625	1.125	9	-
I	µg/L	0	0.2	-	-	-
Mg	mg/L	0	2.5	5	40	-
Mn	mg/L	0	0.1875	0.375	3	5
Na	mg/L	0	5	10	80	200
P	mg/L	0	10	20	160	-
S	mg/L	0	3.125	6.25	50	-

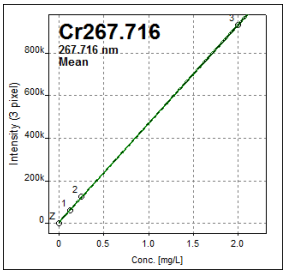
Calibration Curves

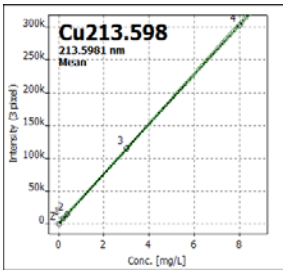


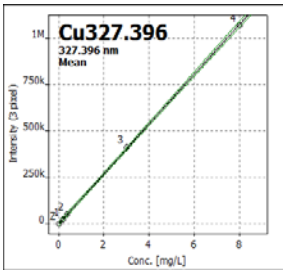


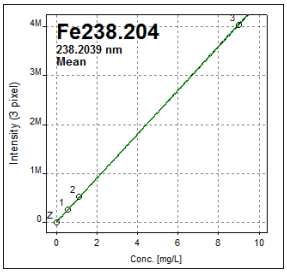












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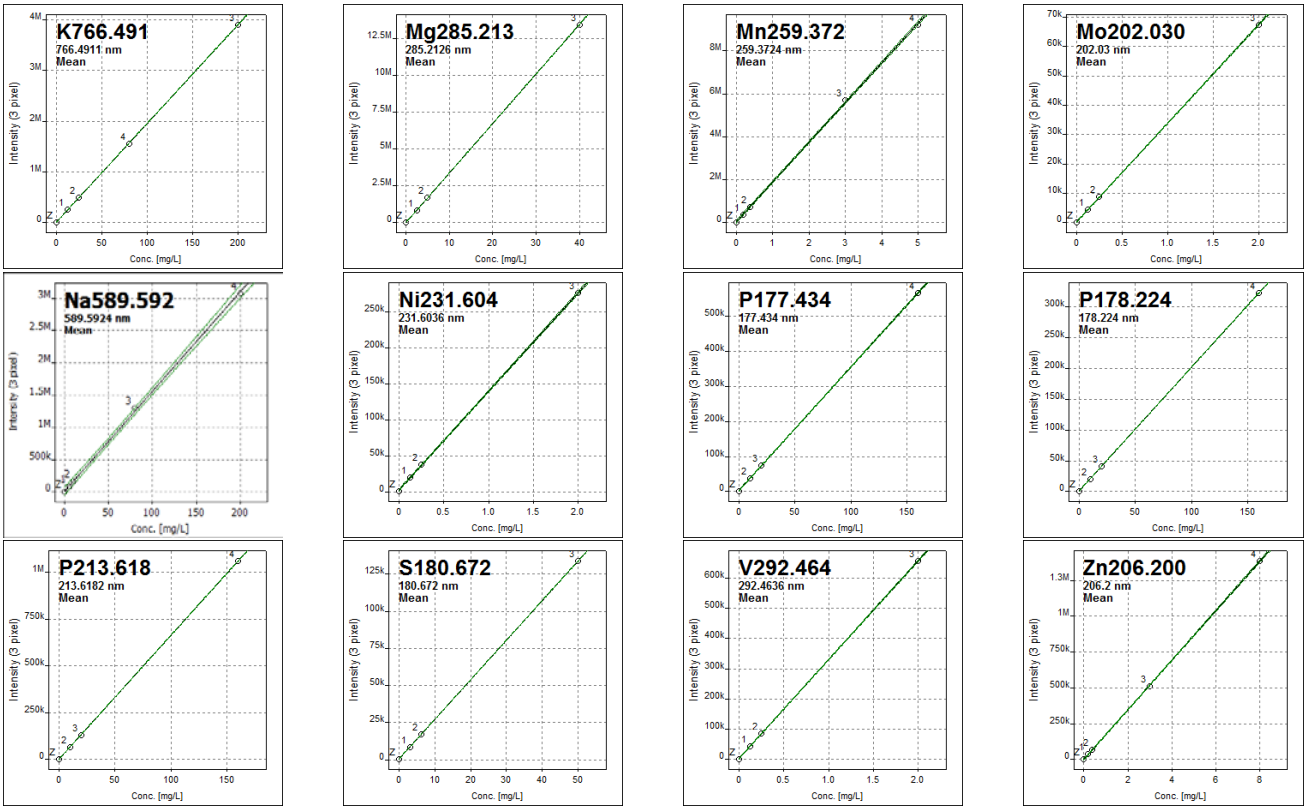

Method Parameters

Table 2: Plasma configurations and set-up of the sample introduction system

Parameter	Specification
Power	1200 W
Plasma gas flow	12 L/min
Auxillary gas flow	0.5 L/min
Nebulizer gas flow	0.6 L/min
Nebulizer	Concentric nebulizer, 1.0 mL/min, borosilicate
Spray chamber	Cyclonic Spray Chamber, 50 mL, borosilicate
Outer tube/inner tube	Quartz / Quartz
Injector	Quartz, 2 mm
Pump tubing	PVC
Sample pump rate	1.0 mL/min
Rinse/ Read delay	45 s
Pump fast run time	15 s
Autosampler	yes

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Evaluation Parameters

Table 3: Overview of method-specific evaluation parameters

Element	Line [nm]	Plasma-view	Integration-Mode	Read time [s]	Evaluation			
					No. of pixel	Baseline fit	Polynomial degree	Correction[2]
Al	396.152	axial	spectrum	3	3	ABC[1]	auto	-
B	249.773	axial	spectrum	3	3	ABC	auto	CSI[3]
Ca	317.933	radial	spectrum	3	3	ABC	auto	-
Co	228.615	axial	spectrum	3	3	ABC	auto	-
Cr	267.716	axial	spectrum	3	3	ABC	auto	-
Cu	213.598	axial	spectrum	3	3	ABC	auto	-
	327.396	axial	spectrum	3	3	ABC	auto	-
Fe	238.204	axial	spectrum	3	3	ABC	auto	-
I	178.215	axial	spectrum	3	3	ABC	auto	CSI, IEC[4]
K	766.491	radial	spectrum	3	3	ABC	auto	-
Mg	285.213	axial PLUS	spectrum	3	3	ABC	auto	-
Mn	259.372	axial	spectrum	3	3	ABC	auto	-
Mo	202.030	axial	spectrum	3	3	ABC	auto	-
Na	589.592	radial PLUS	peak	3	3	ABC	auto	-
Ni	221.648	axial	spectrum	3	3	ABC	auto	-
	231.604	axial	spectrum	3	3	ABC	auto	-
P	177.434	axial	spectrum	3	3	ABC	auto	-
	178.224	axial	spectrum	3	3	ABC	auto	-
	213.618	axial	spectrum	3	3	ABC	auto	-
S	182.565	axial	spectrum	3	3	ABC	auto	-
V	292.401	axial	spectrum	3	3	ABC	auto	-
	292.464	axial	spectrum	3	3	ABC	auto	-
Zn	202.548	axial	spectrum	3	3	ABC	auto	-
	206.200	axial	spectrum	3	3	ABC	auto	-

1 … automatic baseline correction (ABC)
2 … internal standard (IS) and/or mathematical corrections of spectral interferences
3 … correction of iron interference by CSI-Tool recommended, but not used here
4 … correction of phosphorous interference by CSI-Tool or inter-element correction (IEC) were used

Results and Discussion

Table 4: Overview of results for animal feed sample

Element	Line [nm]	Potential interference[1]	Sample concentration in mg/L		RSD[2]	Spike recovery test		DL[3]
			expected	found	%	mg/L	%	µg/L
Al	396.152		1.05	1.006	0.26	4	97.7	0.42
B	249.773		0.060	0.058	0.84	1.6	99.1	0.46
Ca	317.933		60.7	61.05±2.32	0.49	80	99.4	-[4]
Co	228.615		< 0.01	< DL[5]	-	0.8	97.3	0.39
Cr[6]	267.716	P	0.016	0.004±0.0315	2.72	0.8	98.6	0.11
Cu[7]	327.396		1.01	1.083±0.0818	0.33	3.2	97.6	0.52
	213.598	P	-	1.099±0.0664	0.38	3.2	98.4	1.54
Fe	238.204		3.21	3.163±0.072	0.18	3.6	97.7	0.13
I	178.215	P	-	153.6[8]	0.27	8	98.8	4.97
K	766.491		56.2	55.1±0.5355	0.48	80	96.9	-[4]
Mg	285.213		19.97	19.93±0.1207	0.71	16	98.7	0.16
Mn	259.372		0.70	0.698±0.0966	0.62	2	98	0.02
Mo	202.030		< 0.01	0.0031±0.0186	12.29	0.8	94.5	0.76
Na	589.592		14.88	16.24±4.623	0.31	80	103	-[4]
Ni[9]	221.648		-	0.0035±0.0406	5.87	0.8	103	0.50
	231.604	OH	0.025	0.0032±0.0421	6.13	0.8	96.7	0.35
P[10]	177.434		-	52.44±0.7913	0.24	-[4]	-[4]	-[4]
	178.224	I	52.69	52.88±0.9349	0.15	64	102	11.0
	213.618	Cu	-	49.22±0.686	0.41	64	96	10.5
S	180.672		19.22	20.53±0.1324	0.47	20	101	5.2
V	292.401		< 0.005	< DL	-	0.8	102	0.23
	292.464		-	< DL	-	0.8	102	0.2
Zn	202.548		-	21.66[11]±0.1445	0.6	3.2	102	0.19
	206.200		22.9	21.58[11]±0.3608	0.36	3.2	95.7	0.25

1 … likely spectral interferences on conventional ICP-OES instruments due to line overlap that are well-resolved (not present) on the PlasmaQuant® PQ 9000 Elite

2 … RSD from 3 replicate measurements per sample; not given when sample content was lower than detection limit

3 … method-specific detection limit obtained from 3σ of SD on QC Blank (11 repetitive runs)

4 … value not obtained

5 … value below detection limit

6 … expected value too high probably due to interference from P 267.711 nm line (seems plausible as Phosphorous concentration in the sample is very high)

7 … excellent agreement of copper results for two lines shows that Cu 213.598 nm lines is not interfered by P 213.618 nm even for high Phosphorous contents

8 … no confidence range due to number of calibration points; CSI-corrected value given here that was confirmed by inter-element correction (IEC, using P 177 line)

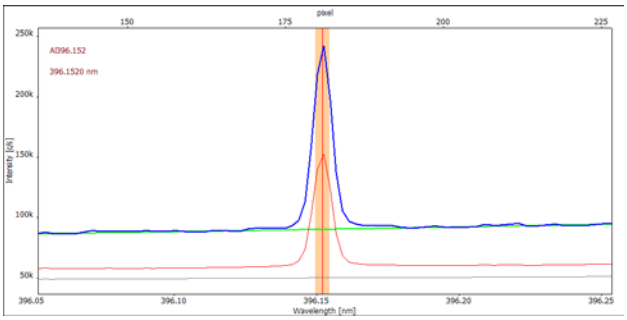
9 … expected value too high probably due to interference from OH line; see good agreement of results for Ni 221 and Ni 231 on PlasmaQuant® PQ 9000 Elite

10 … agreement of Phosphorous results for three different lines two of which typically are spectrally interfered on conventional ICP-OES instruments

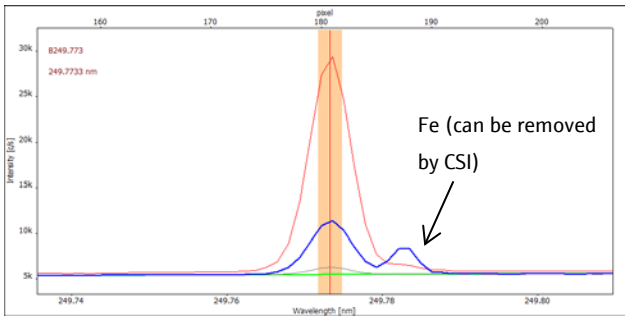
11 … value exceeding the calibration range

Application advantages

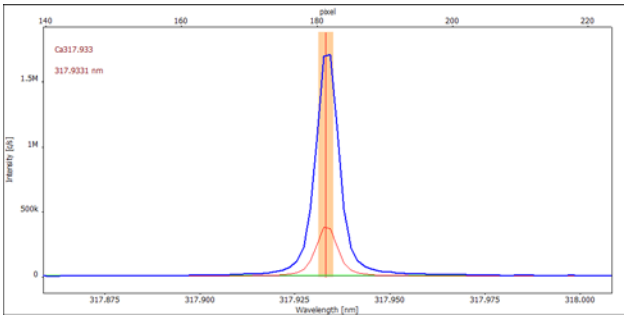
Selection of High-Resolution spectra for animal feed sample (blue), Cal.1 (red) and blank (grey); automatic baseline fit (ABC, green)



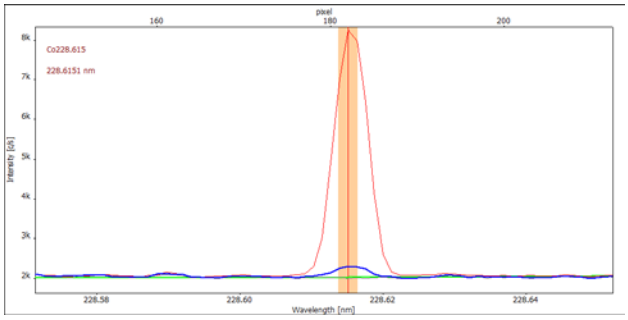
Al 396.152 nm



B 249.773 nm



Ca 317.933 nm



Co 228.615 nm

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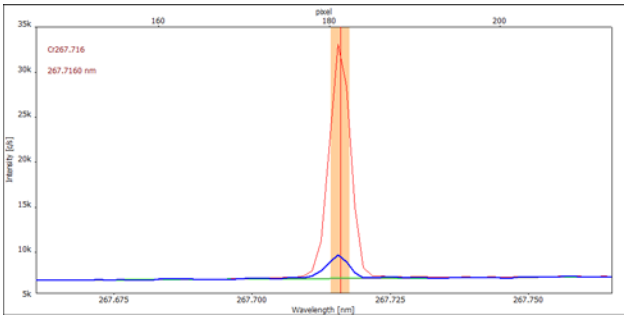
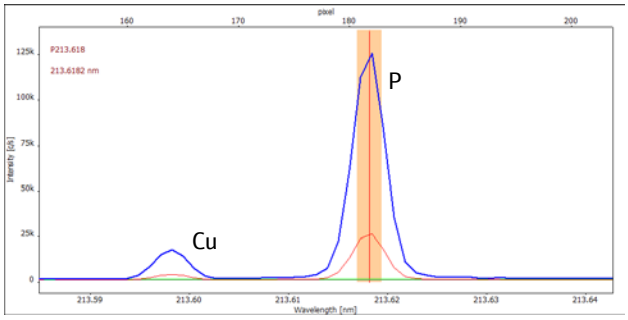
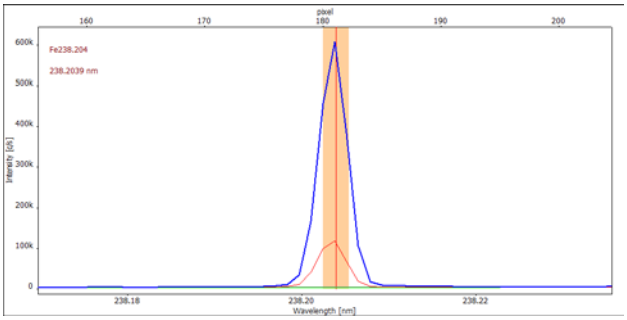


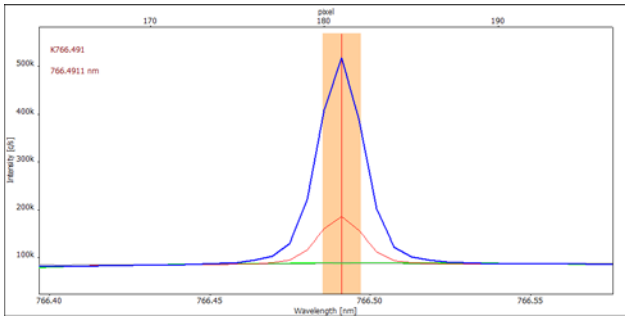
Cr 267.716 nm



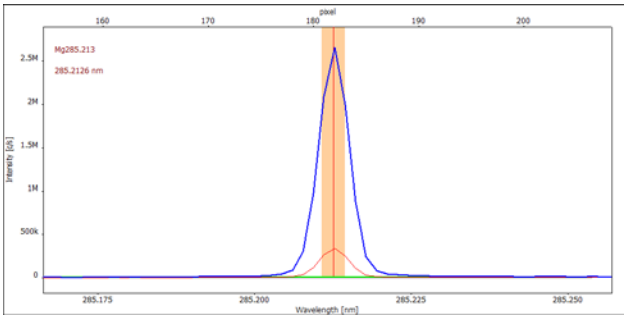
Cu 213.598 nm and P 213.618 nm



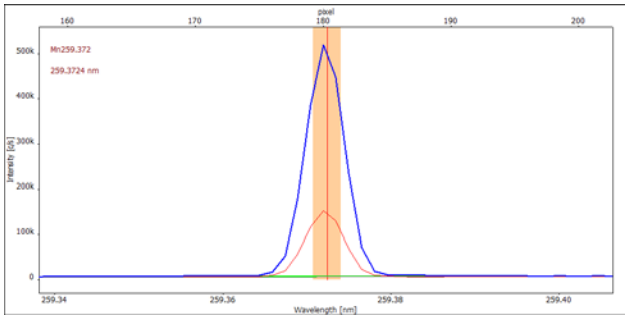
Fe 238.204 nm



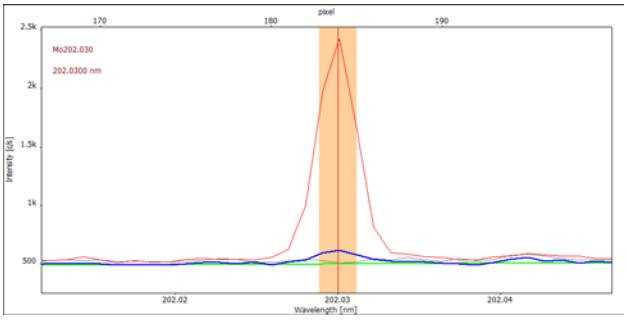
K 766.491 nm



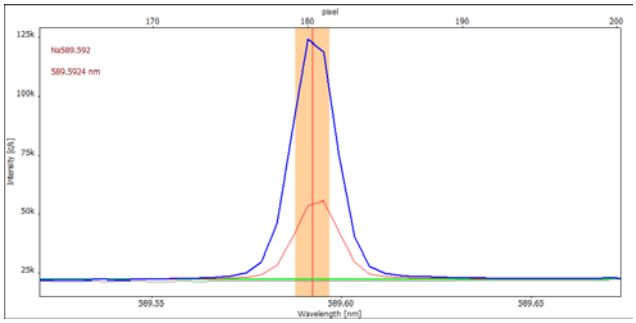
Mg 285.213 nm



Mn 259.372 nm



Mo 202.030 nm



Na 589.592 nm

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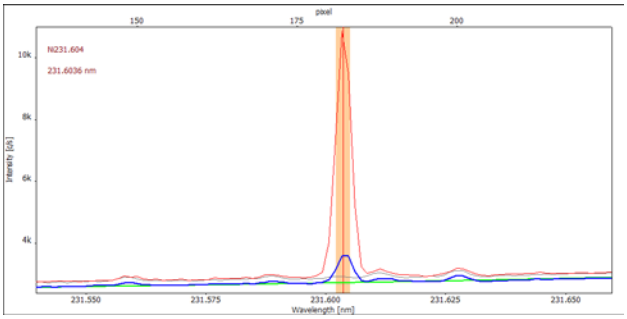
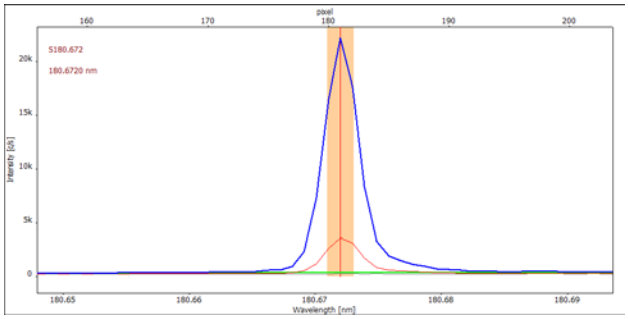
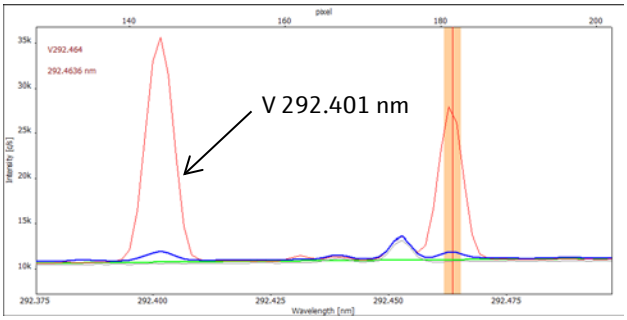


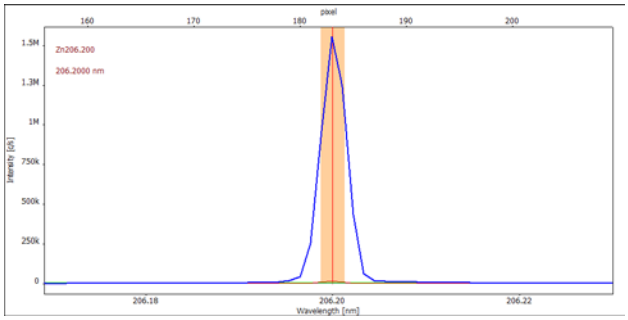
Ni 231.604 nm



S 180.672 nm



V 292.464 nm



Zn 206.200 nm

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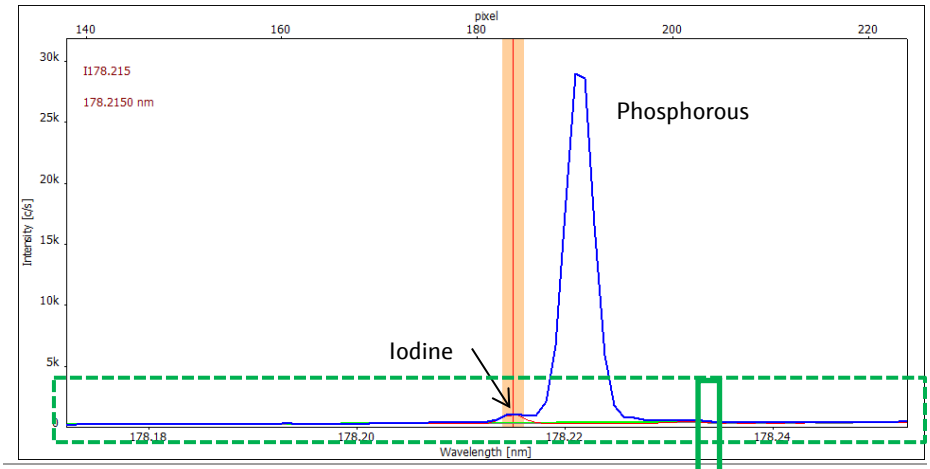
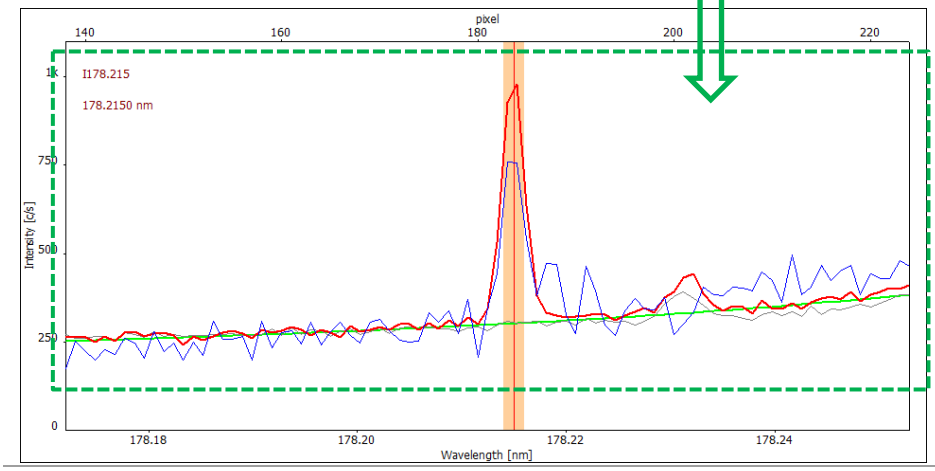
as-received HR ICP-OE spectrum



I 178.215 nm

- Phosphorous that is a matrix-element does affect the Iodine trace detection (prominent P178 partly overlaps I178)
- - false positive results for Iodine, if this spectral interference from Phosphorous is not removed

after interference removal by CSI-Tool



I 178.215 nm

- when a Phosphorous spectrum is subtracted a well-resolved Iodine signal with a FWHM of 2.4 pm is obtained

Reference: ICP_OES_24_16_en.docx

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