Application Example –
PlasmaQuant® PQ 9000 Elite

Elemental Analysis of Tomatoes by HR ICP-OES

Abstract

Growing interest in safety, nutritious value and authenticity of food has spurred a lot of interest among food quality control (QC) labs in ICP techniques. While the emphasis in this industry clearly is on sample management and cost, enhancing analytical performance parameters such as sensitivity and precision is highly desirable, too.

Aiming for lower detection limits of heavy metal impurities including Cobalt, Chromium, Molybdenum, Nickel and Vanadium a microwave-digested tomato sample was analyzed on PlasmaQuant® PQ 9000 Elite equipped with Standard Kit.

It was further tested if matrix-elements like Calcium, Magnesium, Sodium and Potassium, elemental impurities as well as Iodine can be determined from the same sample with excellent precision using Dual View PLUS. It's worth noticing that Iodine detection in food samples by ICP-OES is often falsified by Phosphorous interferences; however, the latter can be resolved on by High-Resolution Array ICP-OES.

Excellent agreement of results, RSD values below 1 % and good recovery rates for an elaborate spike recovery test highlight the extra potential of the PlasmaQuant® PQ 9000 Elite for routine QC labs dealing with food samples. Matrix-specific detection limits well below 1 µg/L (ppb) for all metals as well as 5 ppb for Iodine were achieved.

Challenge

Reduction of detection limits for heavy metals and integration of Iodine analysis in Dual View PLUS routine

Matrix

Aqueous digest of vegetables (tomatoes)

Purpose

Proof of principal for food QC lab

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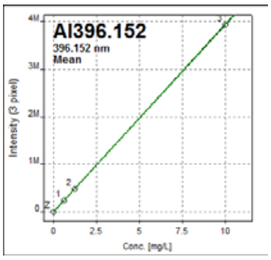
Materials and Methods

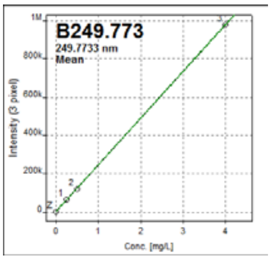
Calibration

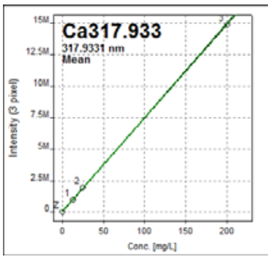
Table 1: Concentration of calibration standards

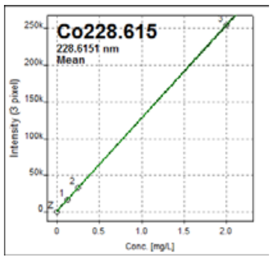
Element	Unit	Cal. 0	Cal.1	Cal.2	Cal.3	Cal.4
Al	mg/L	0	0.625	1.25	10	-
B	mg/L	0	0.25	0.5	4	-
Ca, K	mg/L	0	12.5	25	200	-
Co, Cr, Mo, Ni, V	mg/L	0	0.125	0.25	2	-
Cu, Zn	mg/L	0	0.1875	0.375	3	8
Fe	mg/L	0	0.5625	1.125	9	-
I	µg/L	0	0.2	-	-	-
Mg	mg/L	0	2.5	5	40	-
Mn	mg/L	0	0.1875	0.375	3	5
Na	mg/L	0	5	10	80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

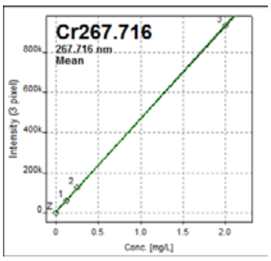
Calibration curves

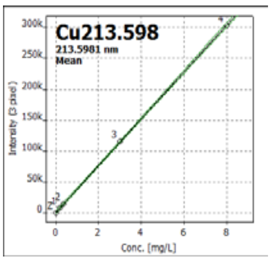


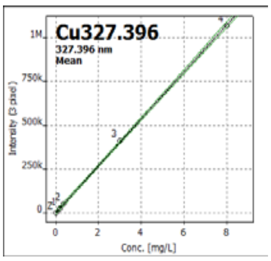


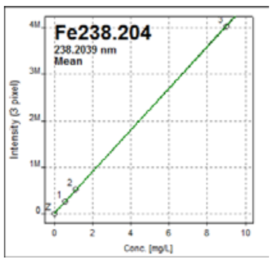












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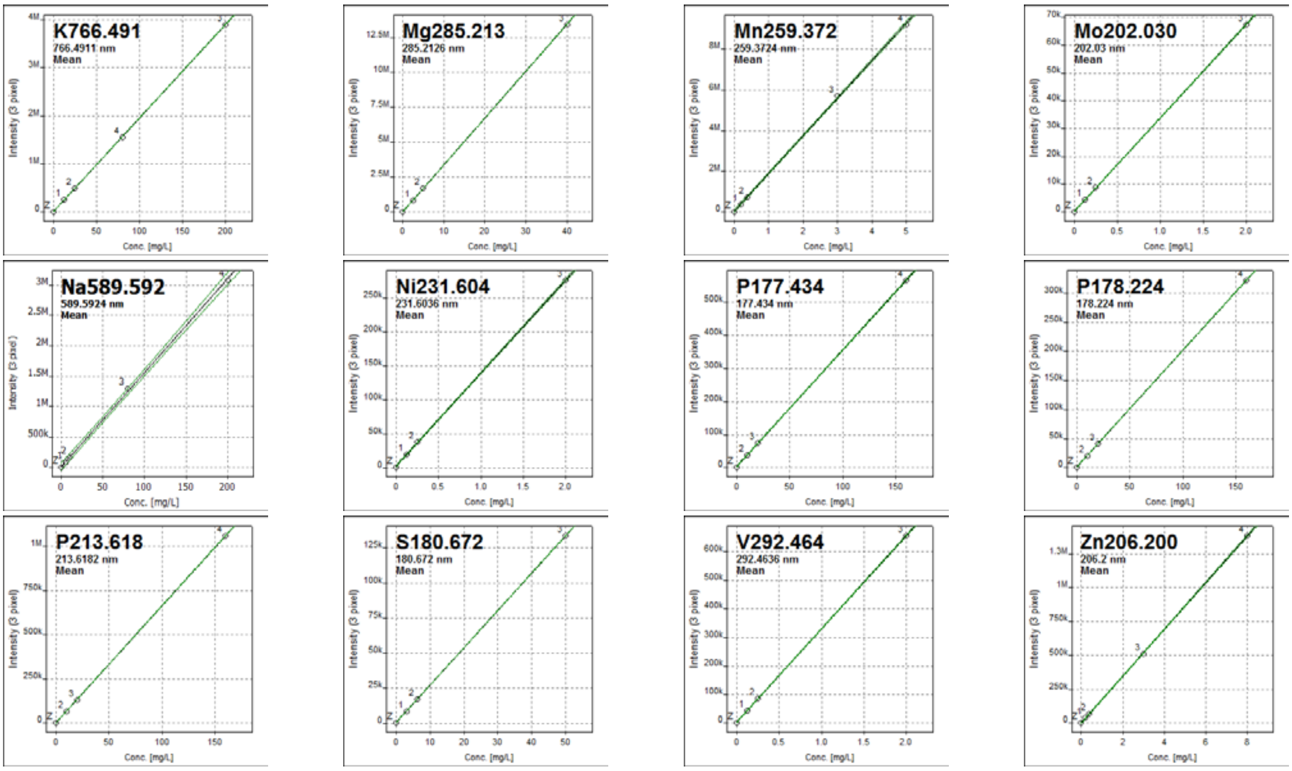

Method Parameters

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	-
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[3]
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	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	180.672	axial	spectrum	3	3	ABC	auto	-
	181.975	axial	spectrum	3	3	ABC	auto	-
	182.565	axial	spectrum	3	3	ABC	auto	-
V	292.464	axial	spectrum	3	3	ABC	auto	-
Zn	206.200	axial	spectrum	3	3	ABC	auto	-

1 ... automatic baseline correction (ABC)
2 ... internal standard (IS) and/or mathematical correction of spectral interferences by CSI-Tool
3 ... correction of phosphorous interference by CSI-Tool

Results and Discussion

Table 4: Overview of results for tomato sample

Element	Line [nm]	Sample concentration in mg/L		RSD[1]	Spike recovery test		DL[2]
		expected	found	%	mg/L	%	µg/L
Al	396.152	1.71	1.70±0.0848	0.54	4.0	97.7	0.42
B	249.773	0.14	0.15±0.019	0.44	1.6	99.1	0.46
Ca	317.933	227.5	240.0)[3]±4.596	0.1	80	99.4	-[4]
Co	228.615	< 0.01	< DL[5]	-	0.8	97.3	0.39
Cr	267.716	< 0.02	0.003±0.0315	3.83	0.8	98.6	0.11
Cu	213.598	-[4]	0.52±0.0692	0.57	3.2	98.4	1.54
	327.396	0.51	0.53±0.0851	0.55	3.2	97.6	0.52
Fe	238.204	2.27	2.26±0.0719	0.5	3.6	97.7	0.13
I	178.215	-[4]	< DL	-	8.0	98.8	4.97
K	766.491	128.7	131.0±0.6364	0.74	80	96.9	-[4]
Mg	285.213	44.0	45.63±0.2126	0.44	16	98.7	0.16
Mn	259.372	1.17	1.22		0.0922	0.24	2.0
Mo	202.030	< 0.005	< DL		0.8	94.5	0.76
Na	589.592	6.59	6.4±4.78	0.85	80	103	-4
Ni	231.604	< 0.005	< DL	-	0.8	96.7	0.35
P	177.434	-[4]	19.5±0.8421	1.43	-[4]	-[4]	-[4]
	178.224	18.6	19.6±0.9828	0.37	64	102	11.0
	213.618	-[4]	18.1±0.7264	0.67	64	96	10.5
S	180.672	-[4]	50.0±0.216	0.49	20	101	5.2
	181.975	47.0	50.3±0.3073	0.85	20	91.4	-[4]
	182.565	-[4]	49.9±0.2093	0.69	20	101	-[4]
V	292.464	< 0.005	< DL	-	0.8	102	0.2
Zn	206.200	0.66	0.65±0.0337	0.2	3.2	102	0.19

1 ... RSD from 3 replicate measurements per sample; not given when sample content lower than detection limits
2 ... method-specific detection limit obtained from 3σ of SD on QC Blank (11 repetitive runs)
3 ... value exceeding the calibration range
4 ... value not obtained
5 ... value below detection limit

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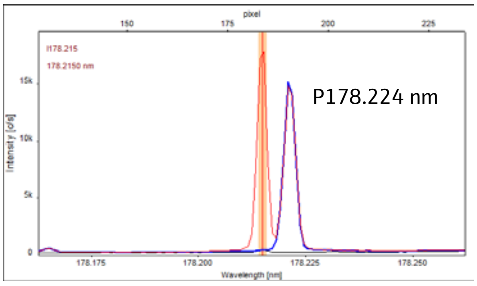
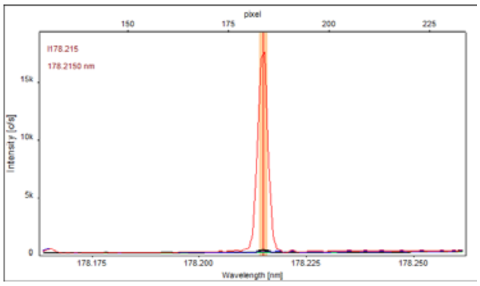
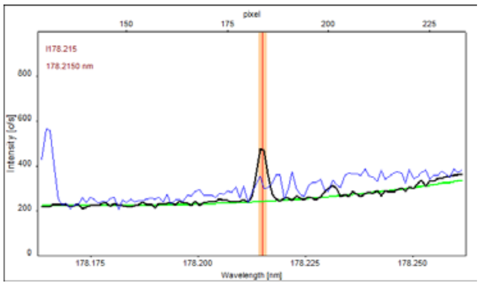
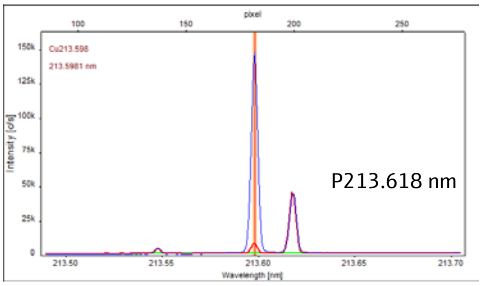
Application advantages

Table 5: High-Resolution spectra of tomato sample (blue), spiked tomato sample (red), calibration standard (black) and ABC (green) showing the applicability of the CSI Software Tool

	as-received spectrum	after interference removal (by CSI)
I 178.215 nm - P178 line is in close proximity of Iodine line and since Phosphorous is a matrix-element it does lead to a false positive result for iodine (blue, panel A) - when a Phosphorous spectrum is used for correction by CSI-Tool an interference-free iodine spectrum can be obtained (red, panel B) - the corrected spectrum of the original sample shows that the iodine levels were below detection limit as only noise was detected (blue, panel C)	 A	 B  C
Cu213.598 nm - line is interference-free, i.e. P213 line is well-separated thanks to the exceptional resolving power of the PlasmaQuant® PQ 9000 Elite - this finding is confirmed by good agreement of values for Cu213 and Cu327 (see Table 4)	 	

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