Application Example –
PlasmaQuant® PQ 9000 Elite

Analysis of Phosphorus in Sunflower Oil by HR ICP-OES

Abstract

Phosphorus is monitored in the refining of edible oils as phosphorous compounds affect the quality of the final oil. That is, for example in refining processes turbidity is reduced, taste characteristics and the stability towards heating, e.g. deep-frying are improved.

While phosphorus can be determined using various techniques, this work describes a straightforward method for ICP-OES on the PlasmaQuant® PQ 9000 Elite, which allows the analysis of phosphorus among other elements in one run with exceptionally low detection limits.

Given the high viscosity of edible oils, kerosene is used to allow for homogeneous pumping and aspiration of such samples. While sample dilution strategies can elevate the effective detection limits for phosphorus, it is the prominent spectral signature of kerosene that can obscure trace signals of phosphorus. Here, the unique sensitivity, the high spectral resolution and the availability of the CSI software tool of the PlasmaQuant® PQ 9000 Elite aid the reduction of effective detection limits for phosphorus in edible oils.

Challenge

Interference-free analysis of Phosphorus

Matrix

- Sunflower oil
- Kerosene

Purpose

Method development for process control lab

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Materials and Methods

Samples and Reagents

- Sunflower oil
- Kerosene
- Blank oil (75 cSt)
- Standards: organometallic S-21+K and organometallic yttrium (1,000 ppm; CONOSTAN)

Sample Preparation

Five process control samples from a sunflower oil production plant were homogenized in ultrasonic bath for 10 min and subsequently diluted with kerosene by factor 5. Calibration standards were prepared accordingly using organometallic S-21+K (1,000 ppm; CONOSTAN) and blank oil (75 cSt) in kerosene. Organometallic Yttrium (1,000 ppm; CONOSTAN) was used as internal standard (IS).

Calibration

Table 1: Concentration of calibration standards

Element	Unit	Cal.0	Cal.1	Cal.2	Cal.3	Cal.4	Cal.5	Cal.6	Cal.7	Cal.8
P	mg/kg						0.5013	0.9940	1.9909	4.9563
Y[1]	mg/kg	1	1	1	1	1	1	1	1	1

1 .... internal standard

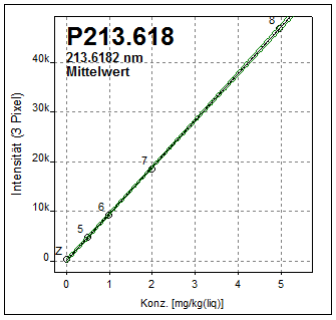

Fig. 1: P calibration curve

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Instrumentation

The analysis was done using the High-Resolution Array ICP-OES PlasmaQuant® PQ 9000 Elite equipped with temperature cooled organic kit.

Instrument Settings and Method Parameters

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

Parameter	Specification
Power	1300 W
Plasma gas flow	15 L/min
Auxillary gas flow	0.5 L/min
Nebulizer gas flow	0.4 L/min
Nebulizer	Borosilicate concentric nebulizer (0.4 mL/min)
Spray chamber	IsoMist cyclonic spray chamber with dip tube, 50 mL, Peltier cooled to 20°C
Injector	1 mm, Quartz
Outer Tube/ Inner Tube	Quartz / Quartz
Pump tubing	PVC (0.76 mm ID) black/black
Sample pump rate	0.4 mL/min
Rinse/ Read delay	80 s
Auto sampler	no

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
P	213.618	axial	peak	3	3	ABC	auto	CSI

1 ABC – automatic baseline fitting

2 CSI – software tool for the correction of spectral interferences originating from the fuel oil matrix

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Results and Discussion

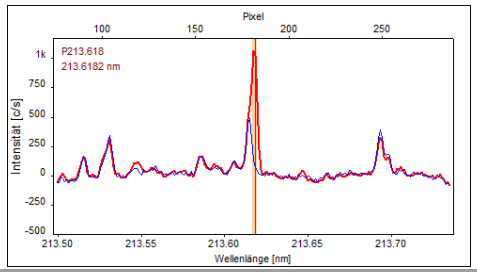
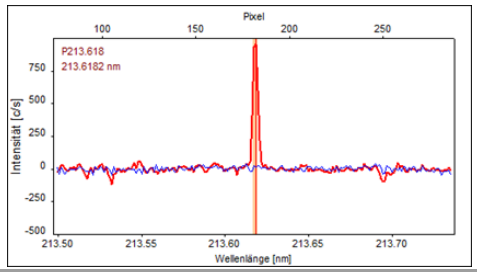
Table 4: Overview of results for P analysis (for samples A, B, C) and method robustness test (spike recovery)

	Conc. [mg/kg]	RSD [%]	DL[a] [µg/kg]	QC Cal.4 recovery [%]
Samples bleached	48[c]	0.97	4.81	98.6
Samples washed	2.84	1.8		
Samples pre-dewaxed	0.56	4.5		
Samples de-gummed	0.29	0.5		
Samples prior refining	0.03	1.5		

a … detection limits obtained from 3σ of SD on QC Blank (11 repetitive runs)

Application Advantages

Table 5: Collection of High-Resolution spectral data and applicability of the CSI Software Tool

Element	as-received spectrum	spectrum corrected by CSI[1]
P 213.618 nm red … sunflower oil blue … kerosene		

1 … mathematical correction of spectral interferences (CSI)

Conclusion/Comments

Thanks to the high spectral resolution of the PlasmaQuant® PQ 9000 Elite and its CSI Software tool undesired spectral interferences arising from the complex spectral signature of kerosene matrix were removed successfully, see table 5. Besides, the routine analysis of sunflower oil products largely benefit from the quality and the ease of the baseline fit (ABC), which improve precision and detection limits.

Reference: ICP_OES_54a_15_en.docx

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