APPLICATION NOTE

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Analysis of trace elements in pentanol using the Thermo Scientific iCAP 7000 Series ICP-OES

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Key Words

Drop-in Fuel, Organic Solvent, Pentanol, Trace Elements

Goal

This application note describes how the routine analysis of organic solvents like pentanol can be performed with the Thermo Scientific iCAP 7600 ICP-OES Duo.

Introduction

Pentanol is used in many fields of applications, for example the manufacture of corrosion inhibitors, fuel and lubricating oils additives, cosmetics and pharmaceuticals.

Besides its current use as a fuel additive, pentanol is also a promising drop-in fuel. It could be used as a direct replacement for gasoline, without the need for major changes to infrastructure, such as pumps and pipelines.

Currently, the main source of pentanol is synthetic hydrolysis of halogenated pentanes. However, researchers are exploring a variety of new technology pathways to produce hydrocarbons, including the use of algal and bacterial mass cultures. These are able to produce C4 and C5 alcohols, which have energy contents similar to that of gasoline. Unlike other biofuels produced from rape seed or wheat, the production does not compete with food crops and therefore it offers an alternative fuel for future purposes. As the fuels are derived from biomass they are also approaching carbon neutrality.

All of these applications require that manufacturers comply with the according regulations, to ensure that certain elements do not exceed the corresponding limit concentrations of trace elements.

Instrumentation

For this analysis the Thermo Scientific™ iCAP™ 7600 ICP-OES Duo was used with the organics sample introduction kit, consisting of a V-groove nebulizer, baffled spray chamber, 1 mm center tube and organics pump tubing. The iCAP 7600 ICP-OES Duo was chosen due to its low detection limits for elements of interest and to demonstrate its capability to handle complex sample matrices.

Sample preparation

Calibration standards were prepared by diluting the oil-based standards S-21+K 100 mg kg-1 (elements contained within the standard are listed in Table 3), As 100 mg kg-1 and Hg 100 mg kg-1, from Conostan, SCP SCIENCE on weight basis in 1-pentanol, from Fisher Scientific™ to produce two calibration standards (Table 1). In addition, a calibration blank was prepared from 1-pentanol and a further blank was spiked using all above mentioned element standards (Table 1).

Table 1. Concentrations of calibration standards and spiked blank.

Solution	Concentration (mg kg^{-1})		
	S-21+K	As	Hg
Low Standard	1.04	1.03	1.02
High Standard	4.93	4.91	4.92
Spike	2.45	2.49	2.48

Method development

Pentanol was aspirated and the plasma was observed. The nebulizer gas flow was adjusted so that the green sample channel was roughly reaching a height of 3 mm above the coil (Figure 1). The method parameters are shown in Table 2.

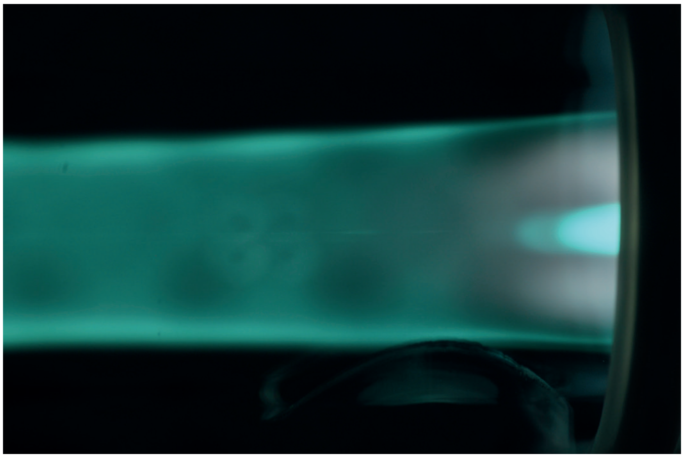


Figure 1. Aspirating 1-pentanol into the plasma after the nebulizer gas flow has been adjusted.

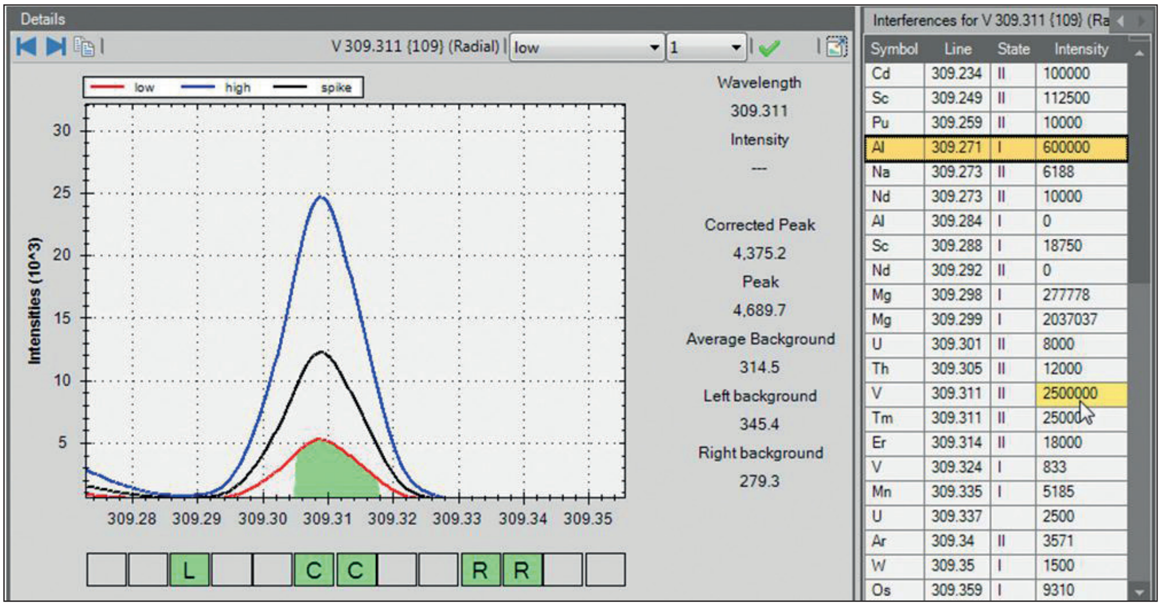
Table 2. Method parameters.

Parameter	Setting
Pump Tubing	Sample solvent flex orange/white Drain solvent flex white/white
Pump Speed	40 rpm
Spray Chamber	Baffled cyclonic
Nebulizer	V-groove
Nebulizer Gas Flow	0.35 L min^{-1}
Auxiliary Gas Flow	1.5 L min^{-1}
Coolant Gas Flow	12 L min^{-1}
Center Tube	1 mm
RF Power	1150 W
Plasma View	Radial/axial
Exposure Time	UV 15 s, Vis 5 s for both axial and radial view

Analysis

For each element, wavelengths were selected using the intuitive wavelength selection tool of the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS). Viewing mode of the individual element wavelength (either axial or radial) is displayed in Table 3. To ensure freedom from interferences, the subarray plots were examined carefully and background correction points were set appropriately (Figure 2). The analyzed wavelengths are listed in Table 3.

The instrument was calibrated and the spiked blank was analyzed and the recovery calculated. A detection limit study was carried out by analyzing the calibration blank with ten replicates and multiplying the standard deviation of this analysis by three. This was repeated three times and the average values for detection limits were calculated.



Wavelength
309.311
Intensity

Corrected Peak
4,375.2
Peak
4,689.7
Average Background
314.5
Left background
345.4
Right background
279.3

Interferences for V 309.311 {109} (Ra

Symbol	Line	State	Intensity
Cd	309.234	II	100000
Sc	309.249	II	112500
Pu	309.259	II	10000
Al	309.271	I	600000
Na	309.273	II	6188
Nd	309.273	II	10000
Al	309.284	I	0
Sc	309.288	I	18750
Nd	309.292	II	0
Mg	309.298	I	277778
Mg	309.299	I	2037037
U	309.301	II	8000
Th	309.305	II	12000
V	309.311	II	2500000
Tm	309.311	II	25000
Er	309.314	II	18000
V	309.324	I	833
Mn	309.335	I	5185
U	309.337		2500
Ar	309.34	II	3571
W	309.35	I	1500
Os	309.359	I	9310

Figure 2. Subarray plot for V 309.311 nm, indicating the peak center, background correction points and a possible interference from aluminum on the left side of the array.

Table 3. Results of the analysis.

Element and Wavelength (nm)	View	Spike Concentration (mg kg^{-1})	Measured Spike Concentration (mg kg^{-1})	Spike Recovery (%)	RSD on three Replicates of the Spike (%)	MDL (µg kg^{-1})
Ag 328.068	Radial	2.47	2.45	99.2	1.6	3.5
Al 167.079	Axial	2.47	2.82	114.2	0.2	1.4
As 189.042	Axial	2.49	2.58	103.6	0.2	7.6
Ba 455.403	Radial	2.47	2.53	102.4	1.8	0.3
Ca 393.366	Radial	2.47	2.46	99.6	1.9	0.2
Cd 214.438	Axial	2.47	2.69	108.9	0.3	0.3
Cr 267.716	Radial	2.47	2.48	100.4	1.6	4.8
Cu 324.754	Radial	2.47	2.41	97.6	2.0	2.4
Fe 238.204	Radial	2.47	2.50	101.2	1.6	4.8
Hg 184.950	Axial	2.48	2.59	104.4	0.6	2.6
K 766.490	Radial	2.47	2.41	97.6	2.4	55
Mg 279.553	Radial	2.47	2.49	100.8	1.7	0.1
Mn 257.610	Radial	2.47	2.46	99.6	1.7	0.6
Mo 202.030	Axial	2.47	2.62	106.1	0.4	0.9
Na 588.995	Radial	2.47	2.45	99.2	2.2	22
Ni 221.647	Axial	2.47	2.65	107.3	0.3	1.3
P 177.495	Axial	2.47	2.61	105.7	0.2	7.7
Pb 220.353	Axial	2.47	2.67	108.1	0.5	6.9
Si 212.412	Axial	2.47	2.57	104.0	0.6	3.3
Sn 189.989	Axial	2.47	2.68	108.5	0.3	7.4
Ti 334.941	Radial	2.47	2.45	99.2	1.6	1.4
V 309.311	Radial	2.47	2.46	99.6	1.7	2.1
Zn 213.856	Axial	2.47	2.62	106.1	0.3	0.5

Results

In Table 3, the elements and their respectively measured wavelengths, as well as the calculated spike recovery and method detection limits (MDL) are listed.

The results (Table 3) show that all element recoveries fall within the acceptable range of ± 10% of the prepared values, except for Al which is slightly above 110%. The %RSD of the three replicates of the spiked blank is 2% or less for all examined elements, except for K (2.4%) and Na (2.2%). The detection limit study reports values in the single digit µg kg^{-1} range or lower. Exceptions are K with 55 µg kg^{-1} and Na with 22 µg kg^{-1}. These elements emit light in the high visible range and suffer from interferences of carbon emission. To improve detection limits of K and Na, compressed air can be added to the auxiliary gas. By adding oxygen (from the compressed air) to the plasma, carbon species that originally emit light in the high visible range are converted into species that have a different emission spectrum. In this way the carbon background in the visible region is reduced.

Conclusion

The Thermo Scientific iCAP 7600 ICP-OES Duo used in conjunction with organics sample introduction kit allows for simplified analysis of pentanol and similar organic solvents.

Usually, radial instruments are used for the analysis of organic samples because of their higher matrix tolerance and reduced interferences. However, this study demonstrates that also the iCAP 7600 ICP-OES Duo delivers robust performance when analyzing organic solvents. Although a viewing height for the radial measurement cannot be selected, careful selection of interference-free wavelengths allows you to determine very low detection limits (single ppb) and excellent accuracy, proving that the iCAP 7600 ICP-OES Duo is an ideal choice for your analysis of organic samples.

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

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