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APPLICATION NOTE 43360

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

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Goal

This application note describes how the routine analysis of organic solvents like kerosene can be performed with the Thermo Scientific iCAP 7400 ICP-OES Radial.

Introduction

Kerosene is a middle distillate fuel, which is a mixture of compounds produced from the distillation of crude oil. Kerosene has many uses, some of which include: a major component of aviation fuel (typically present at concentrations greater than 60%), a house hold fuel for heating and a cleaning agent. During the production of kerosene multiple steps of purification are executed to remove contaminants which could affect the combustion properties of any fuel which contains kerosene as a component. Contamination of kerosene may not only occur during production but also during transportation and storage, for example, water, rust and/or scale accumulation could occur from pipelines or tanks. The presence of trace elements in middle distillate fuels may not only affect combustion but also could cause corrosion especially in fuels used in turbine engines, such as aviation fuel. One example would be the presence of copper which would lead to oxidation and the production of deposits in the engine.

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To ensure the quality of kerosene, manufacturers and distributors of kerosene carry out routine analysis of their products to ensure that certain elements do not exceed set limits for specific trace elements.

This application note will focus on this analysis using the ASTM test method ASTM D7111 'Standard Test Method for Determination of Trace Elements in Middle Distillate Fuels by Inductively Coupled Plasma Atomic Emission Spectrometry' (ICP-AES, also known as ICP-OES).

Instrumentation

For this analysis the Thermo Scientific™ iCAP™ 7400 ICP-OES Radial was used with the organics sample introduction kit, consisting of the components listed in Table 2. The radial system was chosen because of its high matrix tolerance and its ability to optimize the radial viewing height. The optimization of the radial viewing height will reduce interferences. This is critical as interferences from carbon based emissions can be reduced especially in the visible region of the spectrum.

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) on weight basis in PremiSolv™. In addition, a calibration blank was prepared and further, a sample from kerosene was spiked using all above mentioned element standards (Table 3). All solutions were spiked with an yttrium internal standard. Table 1 shows the concentrations of calibration standards and spiked kerosene sample.

Table 1. Concentrations of calibration standards and spiked kerosene sample.

Solution	Concentration (mg·kg^{-1})	
S-21+K		Y
Low standard	1.04	2
High standard	2.04	2
Spiked kerosene	0.1	2

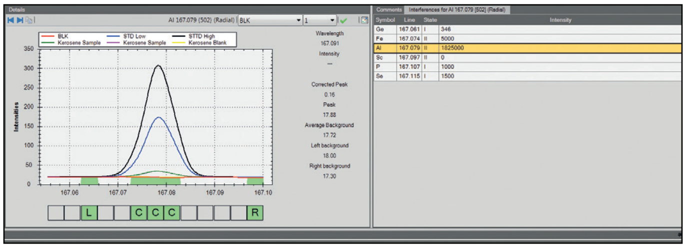
Method development and analysis

Kerosene was aspirated and the plasma was observed. The nebulizer gas flow was adjusted such that the green sample channel reached a height of approximately 3 mm above the coil. The complete method parameters are shown in Table 2.

Table 2. Instrument parameters.

Parameter	Setting
Pump Tubing (Standard Pump)	Sample Solvent Flex orange/white Drain Solvent Flex white/white
Pump Speed	25 rpm
Nebulizer	V-groove
Nebulizer Gas Flow	0.45 L·min^{-1}
Spray Chamber	Baffled cyclonic
Auxiliary Gas Flow	1.5 L·min^{-1}
Coolant Gas Flow	14 L·min^{-1}
RF Power	1350 W
Radial Viewing Height	10 mm
Exposure Time	UV 15, Vis 5 s

The instrument was calibrated and 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. For each element, wavelengths were selected using the intuitive wavelength selection tool of the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software. To ensure freedom from interferences, the subarray plots were examined and background correction points were set appropriately. Figure 1 shows the subarray window for Al 167.079 nm, indicating the peak center and background correction points. The analyzed wavelengths can be found in Table 3.



Wavelength
167.091
Intensity

Corrected Peak
0.16
Peak
17.88
Average Background
17.72
Left background
18.00
Right background
17.30

Interferences for Al 167.079 {502} (Radial)

Symbol	Line	State	Intensity
Ge	167.061	I	346
Fe	167.074	II	5000
Al	167.079	II	1825000
Sc	167.097	II	0
P	167.107	I	1000
Se	167.115	I	1500

Figure 1. Subarray plot for Al 167.079 nm, indicating the peak center and background correction points.

Results

The results (Table 3) show that the vast majority of element spike recoveries fall within acceptable limits of ±10% of the spiked values. For the majority of analyzed elements, the method detection limits (MDL) are within the single digit µg·kg^{-1} range or lower. The concentration values of the unspiked kerosene sample were for the majority of analyzed elements below the detection limit.

Table 3. Results of the analysis.

Element and wavelength (nm)	Spike concentration (µg·kg^{-1})	Measured spike concentration (µg·kg^{-1})	Spike recovery (%)	Unspiked kerosene concentration (µg·kg^{-1})	MDL (µg·kg^{-1})
Al 167.079	100	110	110	<DL	7.4
Ba 455.403	100	110	110	2.8	0.8
Ca 393.366	100	112	112	<DL	0.1
Cr 284.325	100	108	108	<DL	8.8
Cu 324.754	100	105	105	<DL	7.2
Fe 259.837	100	104	104	<DL	17.8
Pb 220.353	100	115	115	<DL	37.1
Mg 279.553	100	110	110	0.4	0.1
Mn 257.610	100	112	112	<DL	1.2
Mo 202.030	100	108	108	<DL	5.1
Ni 221.647	100	110	110	<DL	6.7
K 766.490	500	600	120	<DL	146.1
Na 588.995	500	588	117	<DL	58.0
Si 212.412	100	92	92	<DL	17.5
Ag 328.068	100	92	92	<DL	4.3
Ti 334.941	100	109	109	<DL	1.7
V 309.311	100	109	109	<DL	3.7
Zn 213.856	100	107	107	<DL	1.5

* <DL: measured concentration below detection limit.

Conclusion

The Thermo Scientific iCAP 7400 ICP-OES Radial used in conjunction with organics sample introduction kit allows for simplified analysis of kerosene and similar organic solvents. Sub single figure µg·kg^{-1} concentrations of various elements can be detected within this complex and challenging matrix.

In case that lower detection limits for elements in the high visible wavelength range are required, an additional mass flow controller for the addition of air to the plasma can be used. This helps to reduce interferences in the high region of the spectrum and improves detection capabilities.

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

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