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Elemental analysis of canola oil using the Thermo Scientific iCAP 7400 ICP-OES

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Goal

This note demonstrates the ability of the Thermo Scientific iCAP 7000 Plus Series ICP-OES to handle difficult organic matrices like oils with a minimum effort for sample preparation. For increased ease of use, the automated Plasma Optimization is used.

Introduction

Canola (CANadian Oil, Low Acid) is a type of rapeseed oil, developed in Canada in the 1960s and 70s from rapeseed species. Canola has a number of different uses from use as a food to fuel. Canola oil is extracted from the seeds of the Canola plant by crushing them and it is then refined. As foodstuff Canola oil is added to food and also used to cook with. It contains one of the lowest amounts of saturated fats of any edible oil, essential fatty acids such as omega-3 fatty acids, and is also a good source of vitamins such as E and K. For these reasons it is also marketed as a health food and there is some evidence that it can help to reduce the risk of heart diseases. One of the principal emerging uses of Canola oil is as a raw product for biodiesel. The Canola oil undergoes transesterification with an alcohol such as ethanol or methanol in the presence of a catalyst (sodium or potassium hydroxide) to produce Fatty Acid Methyl Ester (FAME) or biodiesel. There is a growing demand for biodiesel worldwide as it offers significant environmental and economic benefits over fossil fuels, such as reduced emission of polyaromatic hydrocarbons, carbon monoxide and particulate matter.

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The analysis of Canola oil for elemental content is important to determine its use for either foodstuffs or fuel. The maximum concentration of metals allowed in Canola oil as a foodstuff are much lower than those for it to be used as raw product for a fuel. This is not only to protect the consumer from harmful effects of toxic elements such as cadmium and lead, but also to prevent degradation in taste from other elements such as iron. When used as a fuel, the concentration of elements is of equal importance but for different reasons. The elements present affect the properties of the fuel such as combustion and there is also environmental legislation to take in to account, with most countries now limiting the sulfur content in fuels for example. As well as the above factors, the shelf life of the oil is also affected by the presence of trace elements. These levels must be carefully monitored during the refining process to ensure removal of the elements that cause degradation, whilst checking for contamination from the refining process. The concentration of some elements (such as phosphorus) can range from 600 mg·L^{-1} in raw Canola oil to sub mg·L^{-1} levels in the refined product.

Instrumentation

The Thermo Scientific™ iCAP™ 7400 ICP-OES Radial was chosen for this analysis. This model features full mass flow control of all gases for enhanced stability and advanced software features including Plasma Optimization, in addition to the inherent quality of excellent matrix tolerance. The organics sample handling kit was used for this application and the parameters in Table 1 were employed during analysis.

Table 1. Instrument parameters.

Parameter	Setting
Pump Tubing (Standard Pump)	Sample Solvent Flex orange/white Drain Solvent Flex white/white
Pump Speed	45 rpm
Spray Chamber	Baffled cyclonic
Nebulizer	V-groove
Nebulizer Gas Flow	0.4 L·min^{-1}
Coolant Gas Flow	14 L·min^{-1}
Auxiliary Gas Flow	1.5 L·min^{-1}
Center Tube	1 mm
RF Power	1150 W
Viewing Height	14 mm
Exposure Time	UV 15 s, Vis 5 s

Sample and standard preparation

The following samples were analyzed:

- Raw Canola oil
- Refined Canola oil
- Bleached Canola oil
- Deodorized Canola oil

Before analysis these samples were diluted by a factor of ten with PremiSolv™ (Conostan, Canada), based on weight. To prepare calibration solutions, 1000 mg·kg^{-1} S21 multi-element standard and 1000 mg·kg^{-1} S standard (both Conostan, Canada) were diluted in PremiSolv to the desired concentration. Blank oil was added to the standards prior to being made to volume, making the total oil concentration 10% by weight in the final standards. This was to ensure that the samples and standards had similar physical properties in order to remove any sample introduction effects (for example, nebulization efficiency, or transport to the plasma) which may affect the results.

Method development and analysis

Wavelengths were selected for each element which were interference-free in this matrix. The plasma and sample introduction parameters were then optimized to provide the best signal-to-background ratio using the Plasma Optimization function of the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software. The standards and samples were analyzed in a single run and the peak position and backgrounds were examined for each wavelength in the subarray, which facilitates changes to peak center and background point settings for method development. Some overlaid subarrays for P 177.497 nm are shown in Figure 1.

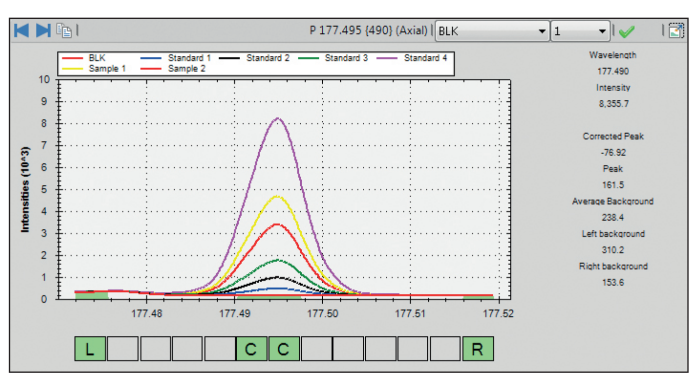

Figure 1. Subarray overlay for P 177.497 nm.

Results

The results of the analysis are as expected in that the concentration of the elements decreases as the Canola has a higher degree of processing (see Table 2). The concentration of phosphorus and sulfur are low in the deodorized Canola making it a good candidate material for processing into biodiesel (these elements are required to be at low concentrations in biodiesel by environment legislation). The detection limits achieved are based on 3 times the standard deviation of a ten replicate blank. They are sufficiently low to quantify the elements of interest at concentrations that would be classed as contamination of the Canola oil.

Table 2. Elemental concentration in mg·kg^{-1} in different processed Canola oils and detection limits (DL).

Element and wavelength (nm)	Raw	Refined	Bleached	Deodorized	Detection limit (µg·kg^{-1})
Ca 317.933	162.1	1.6	0.22	0.05	1.9
Cu 324.754	0.036	<DL	<DL	<DL	1.1
Fe 259.940	1.17	<DL	<DL	<DL	0.92
Mg 280.270	61.57	0.611	0.07	0.006	0.25
Na 589.592	0.122	1.145	0.28	<DL	11
Ni 231.604	<DL	<DL	<DL	<DL	1.3
P 177.495	282.109	3.018	1.213	0.579	7.2
Pb 220.353	<DL	<DL	<DL	<DL	10
S 180.731	7.031	2.93	1.484	3.495	58
Sn 189.989	0.112	0.075	0.089	0.01	3.7

<DL: measured concentrations below detection limit.

Conclusion

The analysis of Canola oil at all stages of the refining process is possible with one simple ICP-OES method. By preparing the samples and standards in the same manner, potential sample transport errors are minimized and the need for internal standardization is negated. The Thermo Scientific iCAP 7400 ICP-OES Radial proved ideal for this sample matrix due to its high matrix tolerance and excellent detection limit capabilities.

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