

**Profile
ICP Series**

Determination of Trace Elements in Edible Oils

Application Note 1024

Solutions for Elemental Analysis

**LEEMAN
LABS, INC**

The Determination of Trace Elements in Edible Oil using Inductively Coupled Plasma Optical Emission Spectrometry

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Introduction

Edible oils are extracted and processed world-wide and hence are important domestic and international commodities. These oils are used in many aspects of food production such as baking and frying and for the manufacture of margarine, snack foods and salad oil products.

Vegetable oils have naturally occurring high contents of phosphorus (in the form of phospholipids), calcium and magnesium and contain traces of other elements like iron and copper. Significant concentrations of these substances in the final product can affect flavor, color and stability. To improve product quality, these substances are removed during the refining process.

Hydrogenation is a chemical process that changes the property of an edible oil to improve its stability and physical characteristics. This process employs a nickel catalyst. Complete removal of nickel in the final product is a necessity since this element promotes oxidation of the final product. Trace levels of nickel in the final product will increase the rate of oxidation causing the oil to turn rancid resulting in a shorter shelf-life.

Accurate elemental analysis of edible oils requires analytical methodology that is both sensitive and selective. Inductively-Coupled Plasma Optical Emission Spectrometry (ICP-OES) meets these requirements and has the capability of determining up to 70 different elements in a sample. ICP-OES permits close monitoring of elemental content throughout processing; starting with the raw oil and continuing through to the finished products. Hence, trace

metal analysis is an important part of quality control as well as final quality checks.

This application note describes the analysis of edible oils using axial and radial view ICP-OES. Information is provided regarding the most suitable wavelengths, background correction and integration times. Results of a detection limit study for both axial and radial plasmas are presented to demonstrate the improved performance of axial viewing. The accuracy of the analytical method is validated using soybean, olive and corn oil matrices for both radial and axial viewing.

Instrument and Method

This study was performed using a Leeman Labs Profile ICP configured for axial or radial viewing. The conditions used in this study are given for both axial and radial configurations in Table I. These conditions were found to be optimum for this application. However, these would not be expected to change significantly even for very different oil matrices.

Table I: Instrument operating conditions for axial and radial plasmas for oil analysis

PARAMETER	RADIAL	AXIAL
Plasma Power (kW)	1.2	1.2
Coolant Flow (L/min)	14	18
Auxiliary Flow (L/min)	1.0	1.2
Nebulizer Type	v-groove	v-groove
Nebulizer Pressure (PSI)	40	40
Uptake Rate (mL/min)	1.0	1.0
Spray Chamber Type	Double-Pass	Double-Pass

To prevent matrix effects in the sample introduction system, the viscosities of samples and standards were matched as closely as possible. The viscosities of all oils tested were approximately the same. Under these conditions, an internal standard to correct for differences in aerosol transport efficiencies

is unnecessary. High-purity kerosene was used as a solvent for all samples and standards and all dilutions were performed on a weight-to-weight basis. The edible oil matrices were prepared by diluting 1:10. The calibration standards for the detection limit and spike recovery studies were prepared by diluting Conostan® metallo-organic standards using a dilution ratio of 1:10. Standards were prepared at the 0.00 ppm, 0.50 ppm and 1.00 ppm levels for axial and 0.00 ppm, 1.00 ppm and 10.00 ppm for the radial-view analysis. For matrix-matching purposes, the 0.00 ppm standard was prepared by diluting Conostan® base oil 75 in a 1:10 ratio. A matrix blank for the detection limit study was prepared by diluting a high-purity soybean oil sample in a ratio of 1:10 with kerosene.

The wavelengths and background correction points used for the detection limit study are given in Table II. A subset of this group was used for the spike recovery analysis from the edible oil matrices. The peak wavelengths are the same as those commonly used in aqueous analysis. The background structure resulting from the organic matrix can differ slightly in comparison to an aqueous matrix. Therefore, the wavelengths used for background correction may differ from those used in an aqueous matrix.

Results and Discussions

An axially viewed plasma offers significant sensitivity advantages over radially viewed plasmas for the analysis of edible oils. This is demonstrated qualitatively using spectral scan data in Figure 1 for 5 ppm phosphorus in kerosene. The emission line viewed radially is barely noticeable whereas the axially measured spectral scan can be easily characterized. The 178.223 nm line for

Table II: Data collection conditions

Element	Peak Wavelength(nm)	Off Peak Background Correction (nm)	Integration Time (sec)
Ag	328.068	328.030	1
Al	308.215	308.180	3
Ba	455.403	455.459	1
Ca	317.933	317.975	1
Cd	214.438	214.413	1
Cr	267.716	267.685	1
Cu	324.754	324.717	1
Fe	259.940	259.910	1
Mg	279.553	279.521	1
Mn	257.610	257.580	1
Mo	202.030	202.007	1
Na	588.995	589.063	5
Ni	221.647	221.621	1
P	213.618	213.644	5
P	178.223	178.202	5
Pb	220.353	220.326	3
Si	251.611	215.642	3
Sn	189.926	189.948	3
Ti	334.941	334.980	1
V	310.230	310.266	1
Zn	213.856	213.831	1

phosphorus can be a useful alternative to the 213.618 nm line, since high levels of copper can cause an interference on the 213.618 nm line. The analyst should consider using the 178.223 nm line when determining low concentrations of phosphorus using an axial-viewed plasma.

The greater sensitivity and improved detection limits of axial viewing can be demonstrated quantitatively by comparing detection limits for each viewing mode in Table III. The detection limits for both viewing methods can be considered excellent. However, a significant improvement in detection limit is obtained in going from radial to axial viewing. Detection limits improve by approximately a factor of 2 to 10. Axial-view detection limit data for the phosphorus 178.223 nm line is included in Table III to give an indication of the achievable sensitivity for this wavelength.

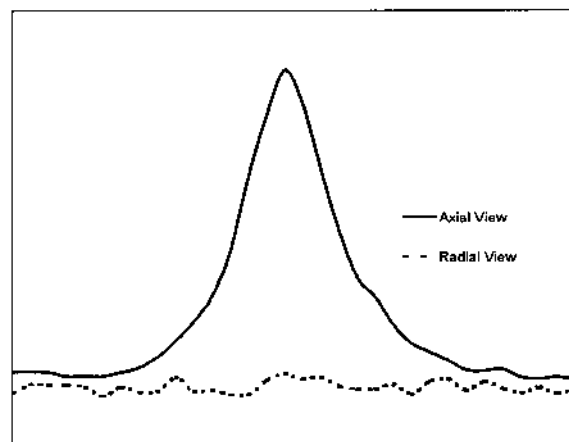


Figure 1: Spectral scan of a 5 ppm phosphorous solution at the 178-223 nm line using axial and radial viewing

Spike recovery analysis was performed on the oil matrices to test the validity of the method for both the radial and axial view plasma and to determine if matrix interferences would be problematic. Each matrix was spiked to yield a solution concentration of each element of 2.000 ppm for the radial-view test and 0.250 ppm for the axial-view test. The results are included in Table IV. Inspection of this data indicates that excellent recoveries were achieved for both viewing modes and for all elements.

Conclusions

Edible oils are easily analyzed using the Leeman Labs Profile ICP. This ICP uses a plasma generated with a water-cooled, free-running oscillator. This produces a robust plasma capable of tolerating the direct introduction of low-volatility organic solvents in either the axial or radial viewing mode. Marked improvement in detection limits is obtained in going from radial to axial viewing. The use of viscosity-matched standards eliminates the need for internal standardization.

Samples are easily prepared by dilution with a suitable solvent. Excellent recoveries were obtained from spiked oil samples. This indicates that the method is suitable for the analysis of edible oils by axial or radial-viewed plasmas and that matrix interferences are not a problem.

Table III: Detection limits for axial and radial plasmas in a 1:10 diluted soybean oil matrix

Element	Wavelength(nm)	Radial LOD (ppb)	Axial LOD (ppb)
Ag	328.068	8.6	2.9
Al	308.215	42.2	7.8
Ba	455.403	1.0	0.2
Ca	317.933	10.1	5.3
Cd	214.438	1.7	0.8
Cr	267.716	5.3	1.0
Cu	324.754	6.3	2.2
Fe	259.940	5.6	2.0
Mg	279.553	0.2	0.1
Mn	257.610	1.1	0.5
Mo	202.030	16.4	5.0
Na	588.995	94.5	19.7
Ni	221.647	12.8	2.2
P	213.618	44.0	19.1
P	178.223	N/A	26.3
Pb	220.353	26.2	7.1
Si	251.611	24.3	2.7
Sn	189.926	42.2	6.7
Ti	334.941	3.7	0.8
V	310.230	10.0	2.1
Zn	213.856	5.8	1.0

Table IV: Recovery data for radial and axial view for method validation

Element	Soybean Oil				Olive Oil				Corn Oil			
	Radial %Rec	Radial S.D. (%)	Axial %Rec	Axial S.D. (%)	Radial %Rec	Radial S.D. (%)	Axial %Rec	Axial S.D. (%)	Radial %Rec	Radial S.D. (%)	Axial %Rec	Axial S.D. (%)
Na	100.5	1.9	105.7	7.0	100.9	1.8	98.6	1.1	101.8	2.2	103.7	3.1
Al	99.4	0.4	97.0	1.0	98.9	1.9	95.9	1.5	98.7	1.0	96.8	1.9
Cd	98.9	0.7	100.0	1.0	99.7	0.7	99.0	1.6	98.1	1.2	99.6	1.4
Cr	100.3	0.1	98.7	0.9	100.0	1.0	102.5	1.0	100.0	1.7	100.2	2.2
Cu	97.7	1.0	100.4	2.7	98.6	1.9	98.6	2.5	98.2	1.0	101.5	2.1
Fe	101.4	0.8	100.7	1.5	101.1	1.5	98.5	2.4	101.2	0.6	98.7	0.6
Mg	101.0	0.8	99.0	0.4	100.6	0.5	96.8	2.8	100.1	1.6	98.6	1.1
P	93.0	1.5	100.4	1.6	95.0	1.0	99.6	1.5	96.1	1.6	98.5	1.0
Pb	100.1	1.6	99.4	3.2	100.3	1.8	100.2	1.4	99.7	1.5	98.3	1.6
V	93.6	1.3	97.0	0.8	97.9	2.6	95.8	1.7	97.0	0.6	98.7	1.0
Zn	99.1	1.8	95.9	2.2	98.8	1.0	95.9	3.1	99.2	0.8	97.5	0.4
Ni	100.0	0.5	99.0	2.4	100.6	1.1	100.0	1.9	100.7	1.0	98.4	1.0
Ca	99.8	0.9	98.9	1.3	99.5	0.8	100.4	2.2	99.1	1.8	99.5	0.8