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**Naftni proizvodi in derivati maščob ter olj - Določevanje fosforja v metilnih estrih maščobnih kislin (FAME) - Optična emisijska spektrometrija z induktivno sklopljeno plazmo (ICP OES)**

Petroleum products and fat and oil derivatives - Determination of phosphorus content in fatty acid methyl esters (FAME) - Optical emission spectral analysis with inductively coupled plasma (ICP OES)

Mineralölprodukte und Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Bestimmung des Gehalts an Phosphor in Fettsäuremethylestern (FAME) - Direkte Bestimmung durch optische Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP OES)

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Produits pétroliers et produits dérivés des corps gras - Détermination de la teneur en phosphore des esters méthyliques d'acides gras (EMAG) - Méthode par spectrométrie d'émission atomique à couplage inductif par plasma (ICP OES)

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Petroleum products and fat and oil derivatives - Determination of phosphorus content in fatty acid methyl esters (FAME) - Optical emission spectral analysis with inductively coupled plasma (ICP OES)

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This European Standard was approved by CEN on 13 October 2012.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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## Foreword

This document (EN 16294:2012) has been prepared by Technical Committee CEN/TC 19 "Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin", the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2013, and conflicting national standards shall be withdrawn at the latest by May 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document was first developed to present a determination of both low levels of sulfur and phosphorus in FAME at the same time. However, interlaboratory studies revealed that the precision for sulfur was not better than for other more common techniques for this element. It was thus decided to rewrite the method for phosphorus solely.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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**EN 16294:2012 (E)****1 Scope**

This European Standard specifies an inductively coupled plasma optical emission spectrometry (ICP OES) method for the determination of phosphorus content of Fatty Acid Methyl Esters (FAME) in the range of 2,5 mg/kg to 8,0 mg/kg.

**WARNING — The use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.**

**2 Normative references**

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 1042, *Laboratory glassware — One-mark volumetric flasks (ISO 1042)*

EN ISO 3170, *Petroleum liquids — Manual sampling (ISO 3170)*

EN ISO 3171, *Petroleum liquids — Automatic pipeline sampling (ISO 3171)*

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**3 Principle**

A weighed amount of FAME sample is diluted with kerosene. The solution is then introduced directly into an ICP OES spectrometer. Phosphorus content is determined by comparison with calibration solutions. An internal standard is employed to correct viscosity effects.

**4 Reagents**

If not specified otherwise, only chemicals of a known high degree of purity shall be used.

**4.1 Stock oil**, having a kinematic viscosity comparable to the test samples.

**4.2 Kerosene**, boiling range between 175 °C and 250 °C.

**NOTE** In case of unstable plasma or in case of special laboratory requirements, kerosene can be substituted by other suitable phosphorus free solvents.

**4.3 Phosphorus standard solution**, dissolved in oil, containing 1 000 mg/kg phosphorus.

Some commercial phosphorus element standard solutions are marketed with higher concentrations. Those solutions may be used instead of the required solutions, but an initial mass to mass dilution has to be done according to recommendations given in 7.1. in order to establish the given nominal concentrations as closely as possible.

**4.4 Phosphorus intermediate solution (25 mg/kg).**

Weigh 0,50 g ± 0,01 g of phosphorus standard solution (4.3) into a 50 ml bottle (5.2). Add kerosene (4.2) to 20,00 g ± 0,01 g. Each mass shall be weighed to the nearest 0,1 mg.

**4.5 Element standard solutions (cobalt, scandium, yttrium)**, dissolved in oil, for example with 1 000 mg/kg per element, available as single element standards.

If multi element standard solutions are used, they shall be phosphorus-free.

NOTE The element standard solutions are commonly available as single element standards with various element content.

**4.6 Argon**, with a purity  $\geq 99,996\%$ .

Small amounts of oxygen may be added to the argon gas stream using a metering valve (30 ml/min to 100 ml/min) to prevent carbon deposits in the area of the plasma torch.

## 5 Apparatus

**5.1 Volumetric flasks**, 20 ml and 500 ml, with taper sleeve and plug, according to EN ISO 1042.

**5.2 Bottles**, 30 ml, 50 ml and 100 ml, with screw caps, PE.

To avoid phosphorus contamination due to the phosphates contained in the detergents used for washing the glassware, rinse the latter at least twice with an approximate 5 mol/l solution of hydrochloric acid. Then rinse with distilled water and dry. To avoid contamination of the test samples, all solutions should be prepared in plastic bottles. Surfaces which can come in contact with the solutions should not be touched by hand.

**5.3 Analytical balance**, capable of weighing to the nearest 0,1 mg.

**5.4 ICP OES-spectrometer**, as follows:

### 5.4.1 General

Simultaneous and sequential ICP OES spectrometers equipped for the analysis of organic liquids, with a high-frequency generator and a nebuliser suitable for organic solvents. The use of a feed pump for sample introduction into the nebuliser is required. Pump tubing shall be suitable for organic use. Both setup and operation of the ICP OES spectrometer shall be done in accordance with operating instructions of the manufacturer.

NOTE The use of a radial ICP-OES is advisable to avoid carbon residue formation and minimise matrix interference due to the presence of carbon in the plasma.

### 5.4.2 Recommended wavelengths

The recommended wavelengths of phosphorus are given in Table 1. Wavelengths are expressed as vacuum lines or as air lines (in brackets) according to the expression of the different manufacturers of ICP spectrometers.

Table 1 — Recommended wavelengths

Element	Wavelength nm			
	Phosphorus	177,499 (177,434)	178,287 (178,222)	213,618
Cobalt	238,892			
Scandium	361,383			
Yttrium	224,306	371,029	360,073	

As the magnitude of the background signal highly depends on spectral structures caused by the sample nature and origin, only net intensities have to be recorded. To keep background noise as low as possible, signal and background shall be measured simultaneously.

## 6 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in EN ISO 3170 or EN ISO 3171 and/or in accordance with the requirements of national standards or regulations for the sampling of the product under test.

New or thoroughly cleaned sample bottles made of plastic material (HDPE (high-density polyethylene), FEP (fluorinated ethylene propylene), or PFA (perfluoroalkoxy)) are recommended.

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## 7 Preparation of calibration solutions

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### 7.1 General

In order to avoid inhomogeneity, the standard solutions (4.3 and 4.5) shall be thoroughly shaken before use.

The masses given correspond to a nominal element content of 25 mg/kg in the phosphorus intermediate solution (4.4).

It is strongly advised to use freshly prepared calibration solutions.

### 7.2 Internal standard solution

Weigh approximately 5 g of cobalt, scandium or yttrium element standard solution (4.5) in a 500 ml volumetric flask (5.1). Fill with kerosene (4.2) to the mark.

The solution shall be homogenised by vigorous shaking.

The same standard batch shall be used for all samples and calibration standards.

### 7.3 Calibration solutions

For each calibration solution, weigh the mass of phosphorus intermediate solution (4.4) as indicated in Table 2. The mass shall be weighed to the nearest 0,1 mg in a 20 ml volumetric flask (5.1).



Table 2 — Phosphorus calibration solutions

Calibration solution	Phosphorus content mg/l	Phosphorus Intermediate solution g
1 (blank)	0	-
2	0,25	0,2
3	0,50	0,4
4	1,00	0,8
5	2,00	1,6

Add 5,00 g of the stock oil (4.1) and 5,00 g of the internal standard solution (7.2).

Fill with kerosene (4.2) to the mark.

All prepared solutions shall be homogenised by vigorous shaking.

The exact concentration of the calibration solution shall be calculated considering the exact weighed portion.

## 8 Calibration

### 8.1 General

The ICP OES is started up and checked according to the manufacturer's recommendations and user manuals, including functional performance tests.

The choice of the instrumental parameters is made to obtain the best signal/background ratio for all elements.

Net intensity of analytical lines shall be calculated by subtracting the intensity measured at appropriate background wavelengths. Some instruments are equipped with software which allows the automatic correction of the background.

### 8.2 Calibration of the ICP OES spectrometer

The calibration of the ICP OES spectrometer shall be done by the measurement of the blank solution and of the calibration solutions (7.3) using three replicates. For the determination of the elements, the wavelengths recommended in Table 1 shall be used. The background subtraction shall be performed at wavelengths not affected by other lines.

**IMPORTANT — Ensure that the wavelengths used in calibration also match exactly the ones used in the sample measurement.**

Depending on the spectrometer (software), follow either procedure A or B.

### 8.3 Procedure A

Conduct the aspiration of the calibration solutions (7.3).

For each calibration solutions, measure the net emission intensity of phosphorus,  $I_P$ , and the net emission intensity of the internal standard,  $I_{IS}$ , at the chosen wavelengths.

Calculate the intensity ratio of phosphorus,  $R_P$ , of each calibration solutions using the following formula: