

**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)**

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

See Eesti standard EVS-EN 16294:2012 sisaldab Euroopa standardi EN 16294:2012 ingliskeelset teksti.	This Estonian standard EVS-EN 16294:2012 consists of the English text of the European standard EN 16294:2012.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 28.11.2012.	Date of Availability of the European standard is 28.11.2012.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

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ICS 75.160.20

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English Version

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)

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 par plasma à couplage  
inductif (ICP OES)

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

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

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

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.

## 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)*

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