SLOVENSKI STANDARD

SIST EN 15837:2010

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Etanol kot komponenta za dodajanje motornemu bencinu - Določevanje fosforja, bakra in žvepla - Metoda z direktno spektrometrijo optične emisije z induktivno sklopljeno plazmo

Ethanol as a blending component for petrol - Determination of phosphorus, copper and sulfur content - Inductively coupled plasma optical emission spectrometric direct method

Ethanol zum Blendung in Benzin - Bestimmung des Gehalts von Fosfor, Kupfer und Schwefel - Induktiv gekoppeltes Plasma Optisch-Emissionspektrometrisches direktes Prüfverfahren

Ethanol comme constituant d'essence - Détermination de la teneur de fosfore, coupre et sulphur - Méthode spectrometrique optique directe par inductiv-couple plasme

Ta slovenski standard je istoveten z: EN 15837:2009

ICS:

71.080.60	Alkoholi. Etri	Alcohols. Ethers
75.160.20	Tekoča goriva	Liquid fuels

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15837

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ICS 75.160.20

English Version

Ethanol as a blending component for petrol - Determination of phosphorus, copper and sulfur content - Direct method by inductively coupled plasma optical emission spectrometry (ICP OES)

Ethanol comme base de mélange à l'essence - Détermination de la teneur en phosphore, en cuivre et en soufre - Méthode directe par spectrométrie d'émission atomique par plasma à couplage inductif (ICP OES)

Ethanol zur Verwendung als Blendkomponente in Ottokraftstoff - Bestimmung des Gehalts an Phosphor, Kupfer und Schwefel - Direktes Verfahren durch optische Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP OES)

This European Standard was approved by CEN on 7 November 2009.

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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 Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 15837:2009) 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 June 2010 and conflicting national standards shall be withdrawn at the latest by June 2010.

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.

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

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EN 15837:2009 (E)

1 Scope

This European Standard specifies an inductively coupled plasma optical emission spectrometry (ICP OES) method for the direct determination of elements content in ethanol, namely phosphorus in the range (0,13 to 1,90) mg/kg, copper in the range (0,050 to 0,300) mg/kg, and sulfur in the range (2,0 to 15,0) mg/kg.

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

NOTE   For the purposes of this document, the terms "% (*m/m*)" and "% (*V/V*)" are used to represent respectively the mass fraction (*w*) and volume fraction (φ).

2 Normative references

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

EN ISO 3170, *Petroleum liquids — Manual sampling (ISO 3170:2004)*

EN ISO 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method (ISO 3675:1998)*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

EN ISO 12185, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method (ISO 12185:1996)*

3 Principle

A portion of a sample is directly injected into the spray-chamber of an inductively coupled plasma emission spectrometer. The element content is determined by comparing the emission of the element in the test portion with the emission of the calibration solutions at the same wavelength.

The calibration solutions are prepared from suitable compounds dissolved in ethanol.

4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

4.1   Phosphorus standard solution, ready-made commercially available phosphorus aqueous standard solution, 1 000 mg/l.

4.2   Copper standard solution, ready-made commercially available copper aqueous standard solution, 1 000 mg/l.

4.3   Sulfur standard solution, which shall be either 4.3.1 or 4.3.2.

4.3.1 Sulfur standard solution, ready-made commercially available sulfur aqueous standard solution, 1 000 mg/l, or

4.3.2 Tetrabutylammonium hydrogen -sulfate $(CH_3CH_2CH_2CH_2)_4N(HSO_4)$, (MW 339,53 g/mol).

If tetrabutylammonium hydrogen sulfate is used, weigh 264,5 mg in a 25 ml volumetric flask (5.3). Bring to the mark with ethanol (4.5) and homogenise. The actual concentration of sulfur shall be calculated, as 1 000 mg/l only represents a target concentration.

NOTE   If a ready-made sulfur aqueous standard solution is employed, the solubility in ethanol of the sulfur compound should be checked. For instance, ammonium sulphate, used in most commercially available solutions, is insoluble in ethanol.

4.4   Water, for analytical laboratory use, conforming to grade 3 of EN ISO 3696.

4.5   Ethanol, 99 % minimum purity.

4.6   Argon, regulated compressed gas of 99,996 % minimum purity for the ICP spectrometer.

5   Apparatus

5.1   Inductively coupled plasma optical emission spectrometer, capable of monitoring one of the following wavelengths of each element, according to Table 1.

NOTE 1   Wavelengths are expressed as vacuum lines or as air lines (in brackets) according to the expression of the different manufacturers of ICP spectrometers.

NOTE 2   The copper line at 213,598 nm can interfere with the phosphorus line at 213,618 nm, but the low concentration range of copper does not show appreciable interference in phosphorus measurements.

A cooled spray-chamber set at 2 °C may be used, provided that the temperature is controlled (± 1 °C), if the plasma is not stable with ethanol.

Table 1 — ICP-OES wavelength capability

Element	Wavelength [nm]		
Phosphorus	177,499 (177,434)	(213,618)	178,287 (178,222)
Copper	324,754	327,395	
Sulfur	180,731 (180,669)	182,034 (181,972)	182,624 (182,562)

5.2   Balance, capable of weighing to the nearest 0,1 mg.

5.3   Glassware, 25 ml, 50 ml and 100 ml volumetric flasks. In order to avoid contamination due to phosphates contained in the detergents used for glassware cleaning, wash the flasks at least twice with an approximate 5 mol/l solution of nitric acid. Rinse with water (4.4) and dry.

5.4   Graduated pipettes or variable volume automatic pipettes, fitted with disposable polypropylene tips.

5.5   Ultrasonic bath, able to contain 100 ml flasks.

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6 Sampling

Unless otherwise specified, obtain samples in accordance with the procedures given in EN ISO 3170 and/or in accordance with the requirements of national standards or regulations for the sampling of the product under test.

High density polyethylene containers (HDPE) shall be used. The containers shall be carefully cleaned and rinsed with water (4.4) to avoid contamination, and then dried.

Thoroughly mix the samples in their containers immediately prior to withdrawal of the test portions.

7 Preparation of calibration solutions

Dilute the standard solution (4.1 to 4.3) with ethanol (4.5) in five 100 ml volumetric flasks to obtain calibration solutions having the element concentrations as specified in Table 2.

Table 2 — Concentrations for calibration solutions

Solution	Phosphorus mg/l	Copper mg/l	Sulfur mg/l
1 (blank)	0	0	0
2	0,1	0,05	1,0
3	0,5	0,10	5,0
4	1,0	0,20	10,0
5	1,5	0,40	20,0

The solutions shall be freshly prepared for each series of analyses.

8 Calibration

8.1 Preparation of the instrumentation

Since instrumentations stemming from diverse manufacturers have different configurations and settings, it is difficult to specify an exact procedure. Follow the manufacturer's instructions for setting up the instrument with organic solutions.

The choice of the instrumental parameters shall be determined so as to obtain the best signal/background ratio for all elements.

The analytical lines recommended in Table 1 shall be used. A background correction shall be performed using wavelengths close to each analytical line, but not affected by other lines.

It is important to ensure that analytical lines and background wavelengths used in calibration also match exactly the ones used in the check of calibration (8.3) and samples analysis (9).

8.2 Execution of the calibration

Conduct the aspiration of the calibration solutions 1 to 5 (see Table 2).

Read out the intensity of the analytical lines and the corresponding background wavelengths chosen for copper, phosphorus and sulfur of each solution.

Calculate the net intensities of the analytical lines by subtracting the intensities of the corresponding background wavelengths. Some instruments are equipped with software which allows the automatic correction for the background.

Carry out three measurements (x) for each solution.

Calculate the arithmetical mean of the three measurements, $\overline{x}$.

Calculate the relative standard deviation (RSD) in percent of each mean according to:

$$RSD = \frac{100 * \sqrt{\sum (x - \overline{x})^2 / (n - 1)}}{\overline{x}}$$
(1)

where

n is the number of measurements (i.e. n = 3).

The *RSD* of calibration solutions 2 to 5 (Table 2) for all elements shall be lower than 5 %. If this is not the case, refer to manufacturers' instructions for better instrumental parameters.

Construct the calibration curve of each element from these means using linear regression, by plotting the emission intensity values versus the concentrations of the element.

The regression coefficient of the linear regression shall be at least 0,995. If not, the procedure should be corrected in case of errors and, if necessary, the calibration procedure should be repeated from Clause 7 onward.

8.3 Check of calibration

A Quality Control (QC) sample shall be freshly prepared for each series of analyses.

The concentration of the QC sample is set in accordance with the specification level to be checked. The QC sample shall be prepared from certified reference materials or other sources of certified stock solutions in order to verify sensitivity and accuracy of the calibration curve.

The QC sample concentration shall be measured after the calibration has been established. If the QC check differs from the reference values for copper, phosphorus or sulfur by more than *R*/1,41 (reproducibility divided by 1,41), verify that the concentration is correct by preparing a new QC sample. If the new QC sample differs from the given values, a new calibration shall be established.

The values obtained with the QC sample shall be measured with a *RSD* (8.2) lower than 5 %.

9   Sample analysis

Shake vigorously the sample container and homogenise using the ultrasonic bath (5.5) for 1 minute prior to withdrawal of the test portions.

Conduct the aspiration of the sample solution. Carry out three measurements in the same way as for the calibration solutions (8.2). Calculate the arithmetical mean of the three measurements.

The drift of the spectrometric system shall be checked before and at the end of the ethanol sample series or at least between every 10 samples with the QC sample (8.3). If the QC check differs from the reference values for copper, phosphorus, or sulfur by more than *R*/1,41, proceed as indicated in 8.3.

Determine, according to the calibration curve of each element, the phosphorus, copper and sulfur content of the sample solution in mg/l.