SIST

SLOVENSKI STANDARD
SIST EN 16576:2015

01-januar-2015

Goriva za motorna vozila - Določevanje mangana in železa v srednjih destilatih - Metoda z optično emisijsko spektrometrijo z induktivno sklopljeno plazmo (ICP OES)

Automotive fuels - Determination of manganese and iron content in middle distillate fuels - Inductively coupled plasma optical emission spectrometry (ICP OES) method

Kraftstoffe für Kraftfahrzeuge - Bestimmung des Gehaltes an Mangan und Eisen in Kraftstoffen aus Mitteldestillaten - Optische Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP OES)

Carburants pour automobiles - Détermination de la teneur en manganèse et fer dans des distillatsmoyens - Méthode d'émission atomique à couplage inductif par plasma (ICP OES)

Ta slovenski standard je istoveten z: **EN 16576:2014**

ICS:

75.160.20	Tekoča goriva	Liquid fuels

SIST EN 16576:2015 **en,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16576

November 2014

ICS 75.160.20

English Version

Automotive fuels - Determination of manganese and iron content in diesel - Inductively coupled plasma optical emission spectrometry (ICP OES) method

Carburants pour automobiles - Détermination des teneurs en manganèse et en fer dans carburants diesel - Méthode spectrométrique optique par plasma à couplage inductif (ICP OES)

Kraftstoffe für Kraftfahrzeuge - Bestimmung des Gehaltes an Mangan und Eisen in Dieselkraftstoff - Optische Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP OES)

This European Standard was approved by CEN on 20 September 2014.

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.

CEN members are the national standards bodies of 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 United Kingdom.

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 16576:2014 E

Contents

Page

Foreword

This document (EN 16576:2014) 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 2015 and conflicting national standards shall be withdrawn at the latest by May 2015.

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 answers requirements originating from the amended Fuels Quality Directive (FQD, [1]).

A similar technique for unleaded petrol is described in EN 16136 [2].

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, 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 a method based on inductively coupled plasma optical emission spectrometry (ICP OES) for the determination of manganese content and of iron content, each from about 0,5 mg/l to about 7,0 mg/l in diesel fuels including those containing up to about 10 % (*V*/*V*) fatty acid methylester (FAME).

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

NOTE 1 Manganese and iron contents higher than 7,0 mg/l can be measured after preliminary dilution of the sample with a suitable solvent. However, the precision has not been established for such a procedure.

NOTE 2 For the purposes of this European Standard, the term "% (*V*/*V*)" is used to represent the volume fraction (φ) of a material.

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)*

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

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

3 Principle

A diesel fuel sample is diluted with a hydrocarbon solvent. The solution is introduced directly into the plasma of an ICP OES spectrometer. Iron and manganese contents are calculated by comparison with calibration solutions prepared from suitable iron and manganese compounds. An internal standard is employed to correct viscosity effects.

4 Reagents

Unless specified otherwise, only chemicals which are known to have a high degree of purity shall be used.

4.1 Kerosene, boiling range between 150 °C and 250 °C, analytical reagent grade.

Other grades of kerosene with analyte concentrations below the detection limits for the two elements under investigation may be used. In this case, perform a wavelength check for absence of signals from the corresponding elements as well as for absence of spectral interference.

4.2 Manganese standard solution, dissolved in oil, μ(Mn) = 100 mg/kg.

A multi-element standard solution may also be used instead of the single element standard solution.

Some element standard solutions are supplied with different element content on the market. These solutions may be used instead of the required solutions, but an initial mass to mass dilution has to be done.

4.3 Iron standard solution, dissolved in oil, μ(Fe) = 100 mg/kg.

A multi-element standard solution may also be used instead of the single element standard solution.

Some element standard solutions are supplied with different element content on the market. These solutions may be used instead of the required solutions, but an initial mass to mass dilution has to be done.

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

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

4.5 Argon, with a purity of ≥ 99,995 % (*V*/*V*).

Small amounts of oxygen (purity O_2 ≥ 99,995 % (*V*/*V*)) may be added, for instance in accordance with the operating instructions of the equipment manufacturer, 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 Laboratory equipment

5.1.1 General

All glassware shall be cleaned carefully before use.

5.1.2 Glassware, usual laboratory glassware, together with the following:

5.1.2.1 Beakers, 50 ml.

5.1.2.2 Volumetric flasks, 20 ml, 50 ml and 500 ml, according to EN ISO 1042, with taper sleeve and plug.

5.1.3 Bottles, 50 ml and 500 ml, with screw caps, high-density polyethylene (HDPE).

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

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

5.3 ICP OES spectrometer

5.3.1 General

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

5.3.2 Wavelengths

Table 1 gives the recommended wavelengths. As the magnitude of the background signal highly depends on spectral structures caused by the sample’s nature and origin, only net intensities are to be used for calculations.

Table 1 – Recommended wavelengths

Element	Wavelength nm
Manganese	257,610
	259,372
	260,569
	279,482
	279,827
	293,931
Iron	234,350
	238,204
	240,488
	259,940
	261,187
Cobalt	238,892
Scandium	361,383
Yttrium	224,306
	360,073
	371,029

6 Sampling

IMPORTANT — The laboratory shall receive a sample which is truly representative and was not damaged or altered during transport or storage.

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 regulations for the sampling of the product under test.

The samples shall be filled into clean containers.

7 Preparation of solutions

7.1 General

In order to avoid inhomogeneity, the element standard solutions (4.2, 4.3 and 4.4) shall be shaken vigorously before use. It is strongly advised to use freshly prepared calibration solutions.

7.2 Preparation of the Internal standard solution for dilution

Weigh 5 g of cobalt, scandium or yttrium stock solution (4.4) with a precision of 0,1 g in a 500 ml volumetric flask (5.1.2.2).

Fill up to 500 ml with kerosene solvent (4.1).

The prepared solution shall be homogenized by vigorous shaking.

NOTE Experience from daily practice with yttrium used as internal standard has shown that internal standard solutions can be used for about two weeks.

Internal standard solution with different element content may be used instead of the 1 000 mg/kg one. In that case, the mass has to be adjusted in order to get a minimum content of 10 mg/l in the internal standard solution for dilution.

7.3 Preparation of the manganese intermediate solution

Weigh 3,00 g ± 0,01 g of manganese standard solution (4.2) into a 50 ml HDPE bottle (5.1.3). Add kerosene solvent (4.1) to 15,00 g ± 0,01 g. In case manganese standard solutions (4.2) with different manganese content are used, the mass of standard solution shall be adjusted accordingly to achieve 20 mg/kg manganese content.

7.4 Preparation of the iron intermediate solution

Weigh 3,00 g ± 0,01 g of iron standard solution (4.3) into a 50 ml HDPE bottle (5.1.3). Add kerosene solvent (4.1) to 15,00 g ± 0,01 g. In case iron standard solutions (4.3) with different iron content are used, the mass of standard solution shall be adjusted accordingly to achieve 20 mg/kg iron content.

7.5 Preparation of the calibration solutions

The calibration solutions shall be prepared as indicated in Table 2. Each mass of manganese intermediate solution (7.3) and iron intermediate solution (7.4) shall be weighed to the nearest 0,1 mg into a 20 ml volumetric flask (5.1.2.2). Fill with kerosene (4.1) to the mark.

All solutions thus prepared shall be homogenized by shaking.

Table 2 — Concentration of iron and manganese in the calibration solutions

Calibration solution	**Manganese intermediate solution (7.3)** g	**Iron intermediate solution (7.4)** g	**Internal standard solution (7.2)** ml	**Manganese concentration** mg/l	**Iron concentration** mg/l
Blank	0,00	0,00	10	0,00	0,00
1	0,50	0,50	10	0,50	0,50
2	2,00	2,00	10	2,00	2,00
3	3,50	3,50	10	3,50	3,50

7.6 Preparation of manganese and iron quality control solution

A 1,0 mg/l quality control (QC) solution shall be prepared using independent manganese and iron standard solutions. The mass given is based on an element (Fe, Mn) content of 100 mg/kg. In case a standard solution with different element content is used, the mass shall be adjusted accordingly to obtain the specified element content.

Weigh 0,5 g of manganese standard solution (4.2) and 0,5 g of iron standard solution (4.3) to the nearest 0,1 mg into a 50 ml volumetric flask (5.1.2.2), add 25 ml of the internal standard solution (7.2), and fill with kerosene (4.1) to the mark.

All solutions thus prepared shall be homogenized by shaking.