SLOVENSKI STANDARD

SIST EN ISO 14596:1999

01-november-1999

Naftni proizvodi ï Dolo evanje ǵvepla ï Valovno disperzivna rentgenska fluorescen na spektrometrija (ISO 14596:1998)

Petroleum products - Determination of sulfur content - Wavelength-dispersive X-ray fluorescence spectrometry (ISO 14596:1998)

Mineralölerzeugnisse - Bestimmung des Schwefelgehaltes - Wellenlängendispersive Röntgenfluoreßenz-Analyse (ISO 14596:1998)

Produits pétroliers - Dosage du soufre - Spectrométrie de fluorescence X dispersive en longueur d'onde (ISO 14596:1998)

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ICS:

75.080	Naftni proizvodi na splošno	Petroleum products in general

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English version

Petroleum products - Determination of sulfur content - Wavelength-dispersive X-ray fluorescence spectrometry (ISO 14596:1998)

Produits pétroliers - Dosage du soufre - Spectrométrie de fluorescence X dispersive en longueur d'onde (ISO 14596:1998)

Mineralölerzeugnisse - Bestimmung des Schwefelgehaltes - Wellenlängendispersive Röntgenfluoreszenz-Analyse (ISO 14596:1998)

This European Standard was approved by CEN on 13 May 1998.

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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 Central Secretariat has the same status as the official versions.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

The text of the International Standard ISO 14596:1998 has been prepared by Technical Committee ISO/TC 28 "Petroleum products and lubricants" in collaboration with Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NNI.

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of an identical text or by endorsement, at the latest by December 1998, and conflicting national standards shall be withdrawn at the latest by December 1998.

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

Endorsement notice

The text of the International Standard ISO 14596:1998 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 14596

First edition
1998-06-15

Petroleum products — Determination of sulfur content — Wavelength-dispersive X-ray fluorescence spectrometry

Produits pétroliers — Dosage du soufre — Spectrométrie de fluorescence X dispersive en longueur d'onde

Reference number
ISO 14596:1998(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 14596 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants.*

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Petroleum products — Determination of sulfur content — Wavelength-dispersive X-ray fluorescence spectrometry

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

1 Scope

This International Standard specifies a method for the determination of the sulfur content of liquid petroleum products, additives for petroleum products, and semi-solid and solid petroleum products that are either liquefied by moderate heating or soluble in organic solvents (see 4.1) of negligible or accurately known sulfur content. The method is applicable to products or additives having sulfur contents in the range 0,001 % (*m/m*) to 2,50 % (*m/m*); higher contents may be determined by appropriate dilution. Other elements do not interfere at concentrations anticipated in the materials subject to this analysis.

NOTE 1 For the purposes of this International Standard, the term "% (*m/m*)" is used to represent the mass fraction.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3170:1988, *Petroleum liquids — Manual sampling.*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling.*

3 Principle

The test portion and a zirconium solution as internal standard are mixed in a given mass ratio and exposed, in a sample cell, to the primary radiation of an X-ray tube.

The count rates of the S-K_{α} at 0,537 3 nm and Zr-$L_{\alpha 1}$ at 0,607 0 nm fluorescence thus excited and the count rate of the background radiation at 0,545 nm are measured, and the ratio of these net count rates calculated. The sulfur content of the sample is determined from a calibration curve prepared on the basis of sulfur calibration standards.

4 Reagents and materials

4.1 White oil (light paraffin oil), high purity grade, sulfur content 1 mg/kg maximum.

4.2 Sulfur compounds, of sulfur content accurately known to the nearest 0,01 % (*m/m*), used for the preparation of the primary standards.

NOTE 2 The compounds given in 4.2.1 to 4.2.3 are suitable, and their nominal sulfur contents are given. Where the purity of these compounds is less than 99 %, certified materials are required, or the nature of all impurities and their concentrations should be accurately known to the nearest 0,01 % (*m/m*).

4.2.1 Dibenzothiophene (DBT), nominal sulfur content 17,399 % (*m/m*).

4.2.2 Dibutyl sulfide (DBS), nominal sulfur content 21,915 % (*m/m*).

4.2.3 Thionaphthene (Benzothiophene)(TNA), nominal sulfur content 23,890 % (*m/m*).

4.3 Certified sulfur reference materials

Use materials from a national standards body or accredited suppliers, if available.

4.4 Zirconium solution A

Zirconium octoate solution with a zirconium content in the range of 12 % (*m/m*) to 18 % (*m/m*), or another oil-soluble zirconium compound dissolved in white oil (4.1) to provide a zirconium content in the range of 12 % (*m/m*) to 18 % (*m/m*).

4.5 Zirconium solution B

Dilute the zirconium solution A (4.4) with white oil (4.1) to provide a content of approximately 1 % (*m/m*) zirconium.

5 Apparatus

5.1 Wavelength-dispersive X-ray fluorescence spectrometer, use any suitable spectrometer, provided that the design incorporates the features given in table 1. It shall be set up according to the manufacturer's instructions.

Table 1 — General requirements of spectrometer

Component	Requirement
Anode	Rhodium, scandium or chromium target
Voltage [1)]	30 kV to 50 kV
Current [1)]	30 mA to 70 mA
Collimator	Coarse
Analysing crystal	Germanium, pentaerythritol, or graphite
Optical path	Helium
Cell window	Polyester or polypropylene film, sulfur-free, thickness 2 µm to 6 µm
Detector	Gas flow proportional counter with a pulse-height analyser
1) Lower power systems may be used, provided that they have been validated to meet the requirements specified in 8.3 and clause 12.	

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

5.3 Homogenizer, non-aerating, high speed shear type, or **heatable magnetic** or **ultrasonic stirrer**.

5.4 Flasks, of 50 ml capacity, narrow-necked, conical, made of borosilicate glass, and fitted with ground-glass stoppers. Use flasks of higher capacity for stock solutions (7.2).

6 Samples and sampling

6.1 Unless otherwise specified, samples shall be taken according to the procedure described in ISO 3170 or ISO 3171.

6.2 Test portions from the samples shall be drawn after thorough mixing and subdivision. Heat viscous samples to a temperature which renders the sample liquid, and homogenize, using the homogenizer (5.3) as necessary.

NOTE 3 For the purpose of this procedure, the term "sample" also includes solutions prepared from additives, semi-solid or solid petroleum products that have been appropriately pre-treated and/or diluted.

7 Calibration solutions

7.1 General

Use either certified reference materials (4.3) or primary standards prepared from sulfur compounds (4.2) dissolved in white oil (4.1) as a basis for the preparation of the appropriate range of sulfur stock solutions.

7.2 Preparation of stock solutions

Weigh, to the nearest 0,1 mg, a quantity of sulfur compound (4.2) or certified reference material (4.3) to prepare stock solutions of approximately 2,50 % (*m/m*) and 0,10 % (*m/m*) sulfur content, calculated to the nearest 0,001 % (*m/m*), and dissolve in white oil (4.1) at room temperature. Mix the contents thoroughly using a homogenizer (5.3).

NOTE 4 The approximate quantities of sulfur compounds (4.2) to be added to 100 g of white oil (4.1) to prepare the stock solutions are:

DBT (4.2.1) 16,75 g [2,5 % (*m/m*)] and 0,56 g [0,1 % (*m/m*)]

DBS (4.2.2) 12,85 g [2,5 % (*m/m*)] and 0,45 g [0,1 % (*m/m*)]

TNA (4.2.3) 11,65 g [2,5 % (*m/m*)] and 0,40 g [0,1 % (*m/m*)]

NOTE 5 It is recommended that a polytetrafluorethylene or glass-coated magnetic stirrer and stirring device are used to mix the contents of the flask.

Calculate the exact sulfur content, $w_{S,2}$, as a percentage by mass, to three decimal places, in each case from the amounts of white oil and sulfur compound used as follows:

$$w_{S,2} = \frac{m_c \times w_{S,1}}{m_c + m_o} \quad \ldots (1)$$

where

m_c is the mass, in grams, of sulfur compound;

$w_{S,1}$ is the sulfur content, as a percentage by mass, of the sulfur compound;

m_o is the mass, in grams, of white oil.