SLOVENSKI STANDARD
SIST EN ISO 20846:2004

01-september-2004

Naftni proizvodi - Določevanje žvepla v gorivih za motorna vozila - Ultravijolična fluorescenčna metoda (ISO 20846:2004)

Petroleum products - Determination of sulfur content of automotive fuels - Ultraviolet fluorescence method (ISO 20846:2004)

Mineralölerzeugnisse - Bestimmung des Schwefelgehaltes von Kraftstoffen für Kraftfahrzeuge - Ultraviolettfluoreszenz-Verfahren (ISO 20846:2004)

Produits pétroliers - Détermination de la teneur en soufre des carburants pour automobiles - Méthode par fluorescence ultraviolette (ISO 20846:2004)

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ICS:

75.160.20	Tekoča goriva	Liquid fuels

SIST EN ISO 20846:2004 **en**

EUROPEAN STANDARD

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EUROPÄISCHE NORM

EN ISO 20846

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ICS 75.080; 75.160.30

English version

Petroleum products - Determination of sulfur content of automotive fuels - Ultraviolet fluorescence method (ISO 20846:2004)

Produits pétroliers - Détermination de la teneur en soufre des carburants pour automobiles - Méthode par fluorescence ultraviolette (ISO 20846:2004)

Mineralölerzeugnisse - Bestimmung des Gesamtschwefelgehaltes von flüssigen Mineralölerzeugnissen - Ultraviolettfluoreszenz-Verfahren (ISO 20846:2004)

This European Standard was approved by CEN on 1 March 2004.

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

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Foreword

This document (EN ISO 20846:2004) 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 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of an identical text or by endorsement, at the latest by September 2004, and conflicting national standards shall be withdrawn at the latest by September 2004.

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

Endorsement notice

The text of ISO 20846:2004 has been approved by CEN as EN ISO 20846:2004 without any modifications.

INTERNATIONAL STANDARD

ISO 20846

First edition
2004-03-15

Petroleum products — Determination of sulfur content of automotive fuels — Ultraviolet fluorescence method

Produits pétroliers — Détermination de la teneur en soufre des carburants pour automobiles — Méthode par fluorescence ultraviolette

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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

ISO 20846 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

Petroleum products — Determination of sulfur content of automotive fuels — Ultraviolet fluorescence method

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 an ultraviolet (UV) fluorescence test method for the determination of the sulfur content of motor gasolines, including those containing up to 2,7 % (*m/m*) oxygen, and of diesel fuels, including those containing up to 5 % (*V/V*) fatty acid methyl ester (FAME), having sulfur contents in the range 3 mg/kg to 500 mg/kg. Other products may be analysed and other sulfur contents may be determined according to this test method; however, no precision data for products other than automotive fuels and for results outside the specified range have been established for this International Standard. Halogens interfere with this detection technique at concentrations above approximately 3 500 mg/kg.

NOTE 1 Some process catalysts used in petroleum and chemical refining might be polluted when trace amounts of sulfur-bearing materials are contained in the feedstocks.

NOTE 2 This test method may be used to determine sulfur in process feeds and may also be used to control sulfur in effluents.

NOTE 3 For the purposes of this International Standard, the terms "% (*m/m*)" and "% (*V/V*)" are used to represent the mass fraction and the volume fraction of a material respectively.

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.

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 3170:2004, *Petroleum liquids — Manual sampling*

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

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

ISO 12185:1996, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method* (including Technical Corrigendum 1:2001)

3 Principle

A hydrocarbon sample is directly injected into a UV fluorescence detector. The sample enters into a high-temperature combustion tube (1 000 °C to 1 100 °C), where the sulfur is oxidized to sulfur dioxide (SO_2) in an oxygen-rich atmosphere. Water produced during the sample combustion is removed and the sample combustion gases are exposed to ultraviolet (UV) light. The SO_2 absorbs the energy from the UV light and is converted to excited sulfur dioxide (SO_2*). The fluorescence emitted from the excited SO_2* as it returns to a stable state SO_2 is detected by a photomultiplier tube and the resulting signal is a measure of the sulfur contained in the sample.

4 Reagents and materials

4.1 Inert gas, argon or helium, of high purity grade with a minimum purity of 99,998 % (*V/V*).

4.2 Oxygen, of high purity grade with a minimum purity of 99,75 % (*V/V*).

CAUTION — Vigorously accelerates combustion.

4.3 Solvent

4.3.1 General

Use either the solvent specified in 4.3.2 or 4.3.3 or a solvent similar to that occurring in the sample under analysis. Correction for sulfur contribution from solvents used in standard preparation and sample dilution is required. Alternatively, use of a solvent with non-detectable sulfur contamination relative to the unknown sample makes the blank correction unnecessary.

4.3.2 Toluene, reagent grade.

4.3.3 Isooctane, reagent grade.

CAUTION — Flammable solvents.

4.4 Sulfur compounds

4.4.1 Compounds with a minimum purity of 99 % (*m/m*). Examples are given in 4.4.1.1 to 4.4.1.3. Where the purity of these compounds is less than 99 % (*m/m*), the concentrations and nature of all impurities are to be established.

NOTE A correction for chemical impurity may be applied when the sulfur content is known with accuracy.

Certified reference materials (CRMs) from accredited suppliers are suitable alternatives to the compounds listed in 4.4.1.1 to 4.4.1.3.

4.4.1.1 Dibenzothiophene (DBT), of molecular mass 184,26 with a nominal sulfur content of 17,399 % (*m/m*).

4.4.1.2 Dibutyl sulfide (DBS), of molecular mass 146,29 with a nominal sulfur content of 21,915 % (*m/m*).

4.4.1.3 Thionaphthene (benzothiophene) (TNA), of molecular mass 134,20 with a nominal sulfur content of 23,890 % (*m/m*).

4.5 Sulfur stock solution

Prepare a stock solution of sulfur content approximately 1 000 mg/l by accurately weighing the appropriate quantity of sulfur compound (4.4) in a volumetric flask (5.9). Ensure complete dissolution with solvent (4.3). Calculate the exact sulfur concentration of the stock solution to the nearest 1 mg/l. This stock solution is used for the preparation of calibration standards. As an alternative procedure, a sulfur stock solution of approximately 1 000 mg/kg can be prepared by accurately weighing the appropriate quantity of sulfur compound (4.4) in a volumetric flask (5.9) and reweighing the volumetric flask once it has been filled to the mark with the solvent (4.3). Take precautions to ensure that evaporation of the solvent and/or sulfur compounds is not causing weighing errors.

NOTE 1 The appropriate mass of sulfur compound described in 4.4.1.1 to 4.4.1.3 to add to the 100 ml flask is 0,574 8 g (DBT), 0,456 3 g (DBS) or 0,418 6 g (TNA).

NOTE 2 The shelf life of the stock solution is approximately three months when stored at low temperature, typically in a refrigerator.

4.6 Calibration standards

Prepare the calibration standards by dilution of the stock solution (4.5) with the selected solvent (4.3).

Calculate the exact sulfur content of each calibration standard.

Calibration standards with a known sulfur concentration, in milligrams per litre (or content in milligrams per kilogram), can be obtained with a volume/volume (or mass/mass, respectively) dilution of the stock solution at 1 000 mg/l (or milligrams per kilogram, respectively). Other practices are possible but those mentioned above avoid any density correction.

New calibration standards should be prepared on a regular basis depending upon the frequency of use and age. When stored at low temperature, typically in a refrigerator, the calibration standards, with a sulfur content above 30 mg/kg (or mg/l) have a useful life of at least one month. Below this sulfur content (30 mg/kg), the shelf life should be reduced.

4.7 Quality control samples

These are stable samples representative of the materials being analysed, that have a sulfur content that is known by this test method over a substantial period of time. Alternatively, there are standard materials with a certified value commercially available. Ensure before use that the material is within its shelf life.

4.8 Quartz wool

Follow the manufacturer's recommendations.

5 Apparatus

5.1 Furnace, comprising an electric device, capable of maintaining a temperature sufficient to pyrolyse all of the sample and oxidize all sulfur to sulfur dioxide (SO_2).

It can be set either in a horizontal or vertical position.

5.2 Combustion tube, of quartz, constructed to allow the direct injection of the sample into the heated oxidation zone of the furnace (5.1).

The combustion tube shall have side arms for the introduction of oxygen and carrier gas. The oxidation section shall be large enough to ensure complete combustion of the sample. It can be set either in a horizontal or vertical position.

5.3 Flow controllers, capable of maintaining a constant supply of oxygen and carrier gas.