



SLOVENSKI STANDARD
SIST EN 15486:2007
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Ethanol as a blending component for petrol - Determination of sulfur content - Ultraviolet fluorescence method

Ethanol zur Verwendung als Blendkomponente in Ottokraftstoff - Bestimmung des Schwefelgehaltes - Ultraviolettfluoreszenz-Verfahren

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Ethanol comme base de mélange à l'essence - Dosage du soufre - Méthode par fluorescence ultraviolette

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ICS:

71.080.60	Alkoholi. Etri	Alcohols. Ethers
75.160.20	V^\[æ[iææ	Liquid fuels

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ICS 71.080.60

English Version

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This European Standard was approved by CEN on 30 June 2007.

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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 15486:2007) 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of an identical text or by endorsement, at the latest by February 2008, and conflicting national standards shall be withdrawn at the latest by February 2008.

The method described in this document is based on EN ISO 20846 [1].

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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1 Scope

This document specifies an ultraviolet (UV) fluorescence test method for the determination of the sulfur content of ethanol from 5 mg/kg to 20 mg/kg. Other products may be analysed and higher sulfur contents may be determined according to this test method, however, no precision data for products other than ethanol and for results outside the specified range have been established for this document. Halogens interfere with this detection technique at concentrations above approximately 3 500 mg/kg.

NOTE For the purposes of this document, the terms “% (m/m)” and “% (V/V)” are used to represent the mass fraction and the volume fraction of a material respectively.

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 to determine the applicability of regulatory limitations prior to use.

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 1042, *Laboratory glassware — one-mark volumetric flasks (ISO 1042:1998)*

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

3 Principle

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An ethanol 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 (SO₂) 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 SO₂ absorbs the energy from the UV light and is converted to excited sulfur dioxide (SO₂*). The fluorescence emitted from the excited SO₂* as it returns to a stable state SO₂, is detected by a photomultiplier tube and the resulting signal is a measure of the sulfur contained in the sample.

CAUTION — Exposure to excessive quantities of ultraviolet (UV) light is injurious to health. The operator must avoid exposing any part of his/her person, especially his/her eyes, to not only direct UV light but also to secondary or scattered radiation that may be present.

4 Reagents and materials

4.1 Inert gas

Argon or helium, high purity grade with a minimum purity of 99,998 % (V/V).

4.2 Oxygen

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 Ethanol, reagent grade.

4.3.3 Toluene, (optional), reagent grade.

CAUTION — Flammable solvents.

4.4 Sulfur compounds

4.4.1 General

Use compounds with a minimum purity of 99 % (*m/m*).

Certified reference materials (CRMs) from accredited suppliers are suitable alternatives to the compounds listed in 4.4.2 to 4.4.4.

4.4.2 Dibenzothiophene (DBT), MW 184,26 g/mol with a nominal sulfur content of 17,399 % (*m/m*).

4.4.3 Dibutylsulfide (DBS), MW 146,29 g/mol with a nominal sulfur content of 21,915 % (*m/m*).

4.4.4 Thionaphthene (Benzo thiophene) (TNA), MW 134,20 g/mol with a nominal sulfur content of 23,890 % (*m/m*).

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4.5 Sulfur stock solution

Prepare a stock solution of approximately 1 000 mg/l sulfur content 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 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 4.4.2 to 4.4.4 to add to the 100 ml flask is 0,574 8 g (DBT), 0,456 3 g (DBS) and 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 content in milligrams per litre (or 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 these ones above avoid any density correction.

NOTE 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 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 (SO₂). 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.

5.4 Vapour drier, capable of removing water vapour formed during combustion prior to measurement by the detector (5.5).

5.5 UV fluorescence detector, a selective and quantitative detector capable of measuring UV light emitted from the fluorescence of excited sulfur dioxide.

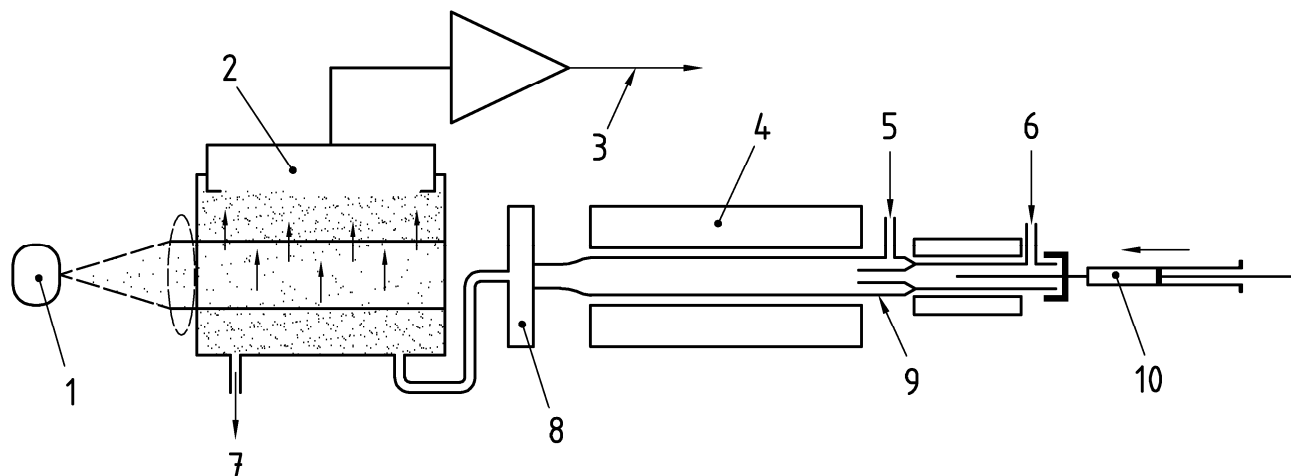
5.6 Microlitre syringe, capable of accurately delivering between 5 µl to 50 µl quantities. Follow the manufacturer's instructions for determining the length of the needle required. For vertical injection, syringes with a polytetrafluoroethylene (PTFE) plunger are recommended.

5.7 Sample inlet system, either positioned vertically or horizontally. It shall consist of a direct injection inlet system capable of allowing the quantitative delivery of the material to be analysed into an inlet carrier stream which directs the sample into the oxidation zone at a controlled and repeatable rate. A syringe drive mechanism, which discharges the sample from the microlitre syringe at a constant rate of approximately 1 µl/s maximum, is required.

NOTE Boat injection systems may be used if they meet the performance requirements of clause 12.

5.8 Balance, capable of weighing with an accuracy of at least 0,1 mg.

5.9 Volumetric flasks, Class A one-mark volumetric flasks, conforming to EN ISO 1042, of appropriate capacities, including 100 ml, for the preparation of sulfur stock solution (4.5) and calibration standards (4.6).



Key

1	UV source	6	inert gas input
2	photomultiplier	7	gases output
3	output signal	8	drier
4	1 000 °C to 1 100 °C	9	quartz tube
5	oxygen input	10	microlitre syringe

Figure 1 — Synopsis of the apparatus
(standards.iteh.ai)

6 Sampling

Unless otherwise specified, laboratory samples shall be obtained by the procedures described in EN ISO 3170. High density polyethylene containers shall be used. The containers should be carefully cleaned and rinsed with pure water to avoid contamination.

Samples should be analysed as soon as possible after removal from bulk supplies, to prevent loss of sulfur.

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

CAUTION — Samples that are collected at temperatures below room temperature can undergo expansion and rupture the container. For such samples, do not fill the container to the top; leave sufficient air space above the sample to allow room for expansion.

7 Apparatus preparation

7.1 Assemble the apparatus and check for leaks according to the manufacturer's instructions.

7.2 Adjust the inlet pressure and flow rate of each gas according to the manufacturer's instructions.

7.3 Refer to the manufacturer's instructions to set the temperature of the furnace (5.1) high enough to ensure all sulfur pyrolysis and oxidation, typically 1 100 °C in the case of a one temperature zone furnace or 750 °C for pyrolysis, and 1 000 °C to 1 100 °C for oxidation in the case of a two zone temperature furnace.

7.4 Adjust the instrument sensitivity and baseline stability, and perform instrument blanking procedures following the manufacturer's guidelines. Ensure that the UV light is stable before measurement.

NOTE For the UV light, a warm up time of at least 30 min is usually required.