SLOVENSKI STANDARD
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Liquid petroleum products - Determination of contamination in middle distillates

Flüssige Mineralölerzeugnisse - Bestimmung der Verschmutzung in Mitteldestillaten

Produits pétroliers liquides - Détermination de la contamination des distillats moyens

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English version

Liquid petroleum products - Determination of contamination in middle distillates

Produits pétroliers liquides - Détermination de la contamination des distillats moyens

Flüssige Mineralölerzeugnisse - Bestimmung der Verschmutzung in Mitteldestillaten

This European Standard was approved by CEN on 1 April 1998.

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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

This European Standard has been prepared by 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of an identical text or by endorsement, at the latest by January 1999, and conflicting national standards shall be withdrawn at the latest by January 1999.

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.

1 Scope

This European Standard specifies a method for determining contamination as the content of undissolved substances in middle distillates, expressed as a mass fraction in milligrams/kilogram.

> NOTE : Contaminants can give rise to faults, for example the blocking of filters, and their presence is therefore undesirable.

This standard applies to liquid petroleum products having a kinematic viscosity not exceeding 8 mm^2/s at 20 °C, or 5 mm^2/s at 40 °C, e.g. diesel fuel as specified in EN 590[1)] or light fuel oils.

The method described may also be used for petroleum products having a viscosity exceeding 8 mm^2/s at 20 °C, or 5 mm^2/s at 40 °C, although these need to be suitably diluted in order to obtain an acceptable filtration time.

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

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

This European Standard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are cited hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ISO 3170 + DAM 1:1997 Petroleum liquids - Manual sampling

ISO 3171 Petroleum liquids - Automatic pipeline sampling

ISO 3819 Laboratory glassware - Beakers

1) EN 590 Automotive fuels - Diesel -Requirements and methods of test

3 Definition

For the purposes of this standard, the following definition applies:

3.1 Contamination

undissolved substances retained on a membrane filter after filtration

NOTE : Typical contaminants include rust, sand and undissolved organic constituents.

4 Principle

A sample portion of between 250 g and 500 g is filtered at 40 °C through a pre-weighed membrane filter. The filter and the residue are washed, dried and weighed. The contamination is calculated from the difference in mass and expressed as milligrams/kilogram.

5 Material

5.1 Heptane, purity not less than 99,0 % (*V/V*), filtered using a membrane filter with a mean pore size of 0,10 µm.

NOTE : Heptane used as a reference fuel in EN 25164[2)] is suitable.

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Membrane filter apparatus, suitable for a membrane filter (6.2).

6.2 Membrane filter, of cellulose nitrate, containing no plasticizer fractions, 47 mm or 50 mm in diameter and 0,8 µm mean pore size.

6.3 Beakers, tall form, 250 ml, 600 ml and 1l, conforming either with ISO 3819 or with an equivalent national standard.

6.4 Oven, explosion proof, capable of heating to a temperature of at least 120 °C.

6.5 Desiccators, with drying agent.

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2) EN 25164 Motor fuels - Determination of knock characteristics - Research method (ISO 5164:1990)

6.6 Watch glass, greater than 50 mm diameter.

6.7 Analytical balance, capable of weighing to an accuracy of 0,1 mg.

7 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in ISO 3170 or ISO 3171, and/or in accordance with the requirements of national standards or regulations for the sampling of the product under test.

8 Preparation of sample and apparatus

8.1 Preparation of sample

8.1.1 If the capacity of the sample container is at least one-fifth larger than the volume of the laboratory sample, shake the laboratory sample vigorously at room temperature in the closed supply container until all precipitations are as uniformly distributed as possible in the laboratory sample. Weigh immediately a sample portion (m_E) of 250 g to 500 g into a beaker (6.3).

8.1.2 If the capacity of the sample container is less than one-fifth larger than the volume of the laboratory sample, operate as follows: pour approximately one-third of the sample into a closable, clean and dry vessel which is at least one-fifth larger than the volume of the laboratory sample supplied. Shake the remainder of the sample in the supply container in order to obtain a uniform distribution of all precipitations, transfer to the larger vessel and shake again. Half-fill the supply container with part of the uniform sample, shake and pour back the contents into the vessel. Weigh immediately a sample portion (m_E) of 250 g to 500 g into a beaker (6.3).

8.2 Preparation of membrane filter and filter apparatus

8.2.1 Dry the membrane filter (6.2) before use on a watch glass (6.6) in the oven (6.4) for approximately 45 min at (110 ± 5) °C and store in the desiccator (6.5) for approximately 45 min.

8.2.2 Immediately before the determination, weigh the watch glass (6.6) and membrane filter (6.2) to the nearest 0,1 mg (m_1). Soak the membrane filter with heptane (5.1) and place on the support screen of the filter apparatus (6.1). Ensure that the membrane filter is free from bubbles when in use and is firmly fixed between the ground surfaces of the filter apparatus.

9 Procedure

WARNING : Electrostatic charges may be generated during the filtration of petroleum products, therefore the filter apparatus is earthed.

9.1 Liquid petroleum products with a kinematic viscosity not exceeding 8 mm^2/s at 20 °C, or 5 mm^2/s at 40 °C

9.1.1 Heat the weighed sample portion (m_E) at (40±1) °C for approximately 30 min and filter using the membrane filter (6.2) in the filter apparatus (6.1) at a pressure of 2 kPa to 5 kPa. Carefully rinse the beaker with heptane (5.1) to wash, so far as is possible, all contaminants onto the filter.

> NOTE: The sample is heated to 40 °C before filtration since the presence of, e.g. fluidity improvers, may give rise to blocking of the filter and excessively high values.

9.1.2 Wash the inside wall of the funnel of the filter holder and the membrane filter containing the residue free of oil with heptane and dry under suction. Remove the filter holder and any particles adhering to its lower boundary zone and transfer onto the membrane filter. Wash off any oil residues in the boundary zones of the filter with heptane.

9.1.3 After disconnection of the vacuum, remove the membrane filter carefully from the filter apparatus and place on the watch glass, which has been weighed with the membrane filter (see 8.2.2). Place the watch glass, including the filter, into the oven and dry for approximately 45 min at (110 ± 5) °C. Allow to cool for approximately 45 min in the desiccator (6.5). Weigh the membrane filter with the watch glass to the nearest 0,1 mg (m_2).

9.2 Liquid petroleum products with a kinematic viscosity exceeding 8 mm^2/s at 20 °C, or 5 mm^2/s at 40 °C

Dilute the weighed sample portion (m_E) of 250 g to 500 g with heptane (5.1) to a kinematic viscosity not exceeding 8 mm^2/s at 20 °C, or 5 mm^2/s at 40 °C. Carry out the determination as described in 9.1.

10 Calculation

Calculate the contamination (c) as a mass fraction $W(c)$ in milligrams per kilogram, using the following equation:

$$W(c) = \frac{1000(m_2 - m_1)}{m_E}$$

where:

m_1 is the mass of the membrane filter and watch glass, in milligrams;

m_2 is the mass of the membrane filter with the residue and the watch glass, in milligrams;

m_E is the mass of the sample portion in grams.

11 Expression of results

Report the contamination (c) as a mass fraction $W_{(C)}$ in milligrams per kilogram.

12 Precision

12.1 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the mean value by 10 % (relative) only in one case in twenty.

12.2 Reproducibility

The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the mean value by 30 % (relative) only in one case in twenty.