SLOVENSKI STANDARD

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Tekoļ i naftni proizvodi - Doloļ evanje neļ istoļ v srednjih destilatih

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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EUROPEAN STANDARD

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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 24 February 2008.

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Foreword

This document (EN 12662:2008) 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of an identical text or by endorsement, at the latest by September 2008, and conflicting national standards shall be withdrawn at the latest by September 2008.

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 supersedes EN 12662:1998.

The major update of this document in comparison with the former edition is that filtration procedure has been updated in order to improve precision and scope. At this stage an interlaboratory study with field samples, following a study with artificial samples, is pending and therefore the repeatability and reproducibility have not yet been fully established, also because of some indications towards problems when the test method is applied to 100 % (V/V) FAME. CEN intends to revise this method when the results of the work on FAME (also at levels of 7 % and 10 %) will be known.

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 the United Kingdom.

1 Scope

This European Standard specifies a method for determining contamination as the content of undissolved substances in middle distillates containing up to 5 % (*V/V*) fatty acid methyl esters (FAME) and in 100 % (*V/V*) FAME. This method can be applied for contaminant content from 6 mg/kg to 30 mg/kg.

NOTE 1 Excessive contamination in a fuel system can give rise to premature blocking of filters and / or hardware failure, and 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.

Although the test method precision has not been defined, the method described may also be used for blends containing more than 5% (*V/V*) FAME and for petroleum products having a viscosity exceeding the above.

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

WARNING — 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 14275, *Automotive fuels — Assessment of petrol and diesel fuel quality - Sampling from retail site pumps and commercial site fuel dispensers*

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

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling (ISO 3171:1988)*

ISO 3819 *Laboratory glassware - Beakers*

3 Terms and definitions

For the purposes of this standard, the following term and definition applies.

contamination
undissolved substances retained on a filter after filtration under test conditions

4 Principle

A sample portion of 800 ml ± 25 ml is weighed and filtered under vacuum through a pre-weighed filter. The filter with the residue is washed, dried and weighed. Contamination is calculated from the difference in mass of the filter and expressed relative to the sample mass as mg/kg.

5 Reagents and materials

5.1 Heptane, with a purity no less than 99,0 % (*V/V*), filtered using a filter (membrane) with a mean pore size of 0,45 µm.

NOTE Heptane used as a reference fuel in EN ISO 5164 is suitable.

5.2 Propan-2-ol, with a purity no less than 99,0 % (*V/V*).

NOTE Propan-2-ol is used to dry glassware and the sample container after rinsing with water.

6 Apparatus

All glassware and sampling vessels to be carefully cleaned as described in Clause 7.

Usual laboratory apparatus and glassware, together with the following:

6.1 Filter apparatus, suitable for a filter (6.2), as shown in Figure 1.

6.2 Filters, of high retention glass fibre type, 47 mm or 50 mm in diameter and with a 0,7 µm mean pore size.

6.3 Beakers, tall form 1 l, conforming with ISO 3819 or an equivalent national standard.

6.4 Oven, of the static type (without fan assisted circulation), explosion-proof, capable of heating to 110 °C ± 5 °C.

6.5 Desiccator, containing freshly activated silica gel (or equivalent desiccant) with a moisture content indicator.

6.6 Glass Petri dishes with covers, greater than 50 mm in diameter.

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

6.8 Forceps, with round shaped tips for transferring the filter from the filter holder to the Petri dish and from the latter on to the dish of the analytical balance.

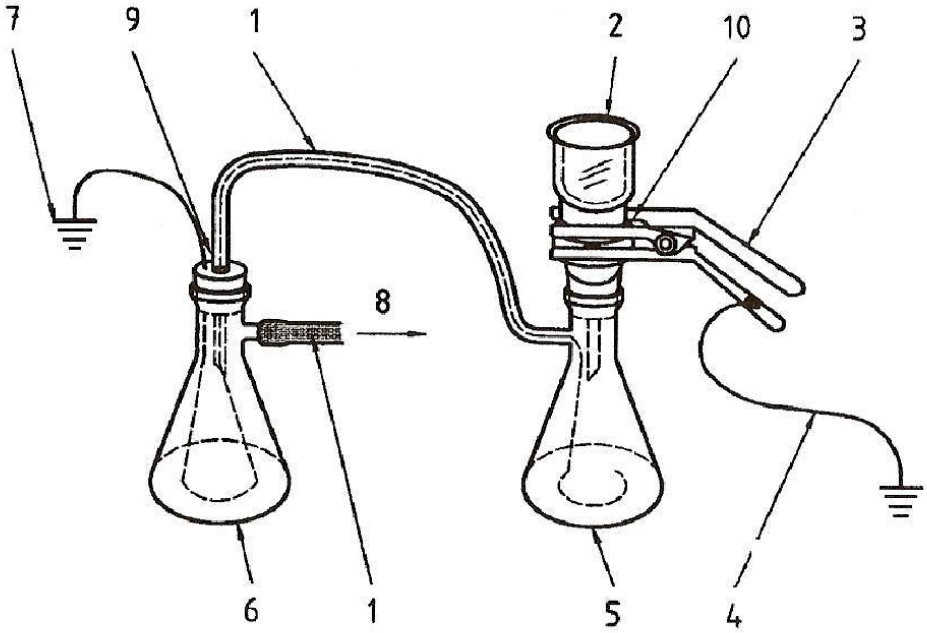
6.9 Water bath or oven, capable of maintaining a temperature of 40 °C ± 1 °C.

6.10 Wash bottle fitted with spray nozzle, suitable for use with heptane.

6.11 Top load balance, capable of weighing 1 500 g to the nearest 0,1 g.

6.12 Vacuum source, capable of maintaining an absolute pressure of 2 kPa to 5 kPa.

6.13 Suitable clean sample containers and sampling vessels.



Key

1) Vacuum tubing
2) Funnel
3) Clamp
4) Wire to ground
5) Receiving flask
6) Safety flask
7) Laboratory ground (recommended)
8) To vacuum pump
9) Ensure seal between tube, hose and wire with appropriate sealant
10) File clamp jaws and handle where wire attaches to bare metal

Figure 1 — Apparatus for determining contamination

7 Preparation of sample containers and apparatus

Due to the extremely low levels of material being measured, it is essential that this testing is performed in a clean environment to minimise the possibility of contamination.

7.1 Clean strictly, in the manner described in 7.2 to 7.7, all the surfaces of all components of the sample containers (after removal of any labels, tags, etc.), sampling vessels, and parts of the apparatus that are:

a) likely to come into contact with the sample or flushing fluid;

b) capable of transferring extraneous matter to the filter.

7.2 Wash with warm tap water containing water soluble detergent.

7.3 Rinse thoroughly with warm tap water.

7.4 Rinse thoroughly with water, handling container caps externally only with clean laboratory tongs during this and subsequent washings.

7.5 Rinse thoroughly with propan-2-ol (5.2).

7.6 Rinse thoroughly with heptane (5.1).

7.7 Cover the top of the sample container and the funnel opening of the assembled filtration apparatus with clean plastic film or aluminium foil previously rinsed with heptane (5.1) and air dried.

8 Sampling

8.1 Unless otherwise specified, obtain samples in accordance with the requirements of EN ISO 3170, EN ISO 3171, EN 14275, or an equivalent national standard.

8.2 The preferred procedure is to take samples dynamically from a sampling loop in a distribution line or from the flushing line of an automatic pipeline sampling device in accordance with the principles specified in EN ISO 3171. Ensure that the line to sampler is flushed with fuel before taking the sample.

8.3 If samples are taken manually the samples shall be taken directly into the sample container.

8.4 Where it is only possible to obtain sample from static storage follow the procedures given in EN ISO 3170, ensuring that the final sample has not passed through intermediate containers prior to placement in the prepared container.

8.5 One litre glass containers shall be used to take and store the samples. These containers should be cleaned according to Clause 7. Glass is used in order to facilitate the visual surveillance of the sample homogenisation before subsequent analysis. Ensure that the samples receive the minimum exposure to light. Use either brown glass containers or shield the samples from light during transportation and storage. To facilitate sampling from refuelling nozzles, wide necked bottles should be used.

8.6 Fill the sample container to between 80 % and 85 % of its capacity.

9 Preparation of the sample

9.1 Before opening the sample container, rinse the outside of the container and its closure with water and propan-2-ol (5.2), as described in 7.2 to 7.5, to remove any adhering particles and avoid introducing undesirable contamination in the test sample.

9.2 Loosen the sample container closure and place the container and its contents in a water bath or oven (6.9) at 40 °C for (30 to 60) min. to ensure that any components that have separated out have dissolved again.

9.3 Remove the sample container from the water bath and tighten the container closure and wash the outside of the container with propan-2-ol. Let it cool down to room temperature.

9.4 Place the beaker (6.3) onto the balance (6.11) and tare.

9.5 Shake the container for at least 10 s, one–to-two strokes per second, using 10 cm to 25 cm strokes. Invert the container and continue to shake for at least a further 10 s, then re-invert and shake for at least a further 10 s. If there are any visible signs of contaminant adhering to the vessel walls, repeat shaking procedure.

9.6 Immediately weigh into the beaker a test portion equivalent to approximately 800 ml. Record the mass of the test portion m_E.