



SLOVENSKI STANDARD
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Tekoči naftni proizvodi - Določevanje nečistoč v srednjih destilatih, dizelskem gorivu in v metilnih estrih maščobnih kislin

Liquid petroleum products - Determination of total contamination in middle distillates, diesel fuels and fatty acid methyl esters

Flüssige Mineralölerzeugnisse - Bestimmung der Gesamtverschmutzung in Mitteldestillaten, Dieselkraftstoff und Fettsäure-Methylestern

Produits pétroliers liquides - Détermination de la contamination des distillats moyens, gazoles et methylesters gras

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Liquid petroleum products - Determination of total contamination in middle distillates, diesel fuels and fatty acid methyl esters

Produits pétroliers liquides - Détermination de la
contamination des distillats moyens, gazoles et
methylesters gras

Flüssige Mineralölerzeugnisse - Bestimmung der
Gesamtverschmutzung in Mitteldestillaten, Dieselkraftstoff
und Fettsäure-Methylestern

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Foreword

This document (prEN 12662:2012) 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 document is currently submitted to the CEN Enquiry.

This document will supersede EN 12662:2008.

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SIST EN 12662:2014

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1 Scope

This European Standard specifies a method for the determination of total contamination as the content of undissolved substances in middle distillates, in diesel fuels containing up to 30 % (V/V) fatty acid methyl esters (FAME), and in pure FAME. This method is applicable for contaminant contents from about 12 mg/kg to about 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 in general applies to products having a kinematic viscosity not exceeding 8 mm²/s at 20 °C, or 5 mm²/s at 40 °C, e.g. diesel fuel as specified in EN 590 [1].

Although the test method precision has not been defined, the method described may also be used for blends containing more than 30 % (V/V) FAME and for petroleum products having a viscosity exceeding the above.

For liquid petroleum products having a kinematic viscosity exceeding 8 mm²/s at 20 °C or 5 mm²/s at 40 °C a separate procedure is in 11.2.

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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)*

EN ISO 3171, *Petroleum liquids — Automatic pipeline sampling (ISO 3171)*

ISO 3819, *Laboratory glassware – Beakers*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply:

3.1

total contamination

undissolved substances retained on a filter after filtration under test conditions

3.2

absolute pressure

pressure measured relative to zero pressure or a total vacuum

4 Principle

A sample portion is weighed and filtered under vacuum through a pre-weighed filter. In the case of pure FAME, the weighed sample portion is diluted with a solvent before filtration. 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 [2] is suitable.

5.2 Xylene, analytical grade, filtered using a filter (membrane) with a mean pore size of 0,45 µm.

5.3 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.

5.4 Solvent, add 750 ml heptane (5.1) and 250 ml xylene (5.2) to a 1 l glass bottle and mix thoroughly.

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

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

NOTE Glass fibre filters Whatman GF-F type are suitable for total contamination measurements.

6.3 Beakers, tall form 0,5 l and 1 l, conforming with ISO 3819 or an equivalent national standard, and 500 ml and 1 000 ml graduated cylinder.

6.4 Glass bottles, 0,5 l and 1 l, with screw caps.

6.5 Oven, of the static type (without fan assisted circulation), explosion-proof, capable of heating to $(110 \pm 5) ^\circ\text{C}$.

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

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

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

6.9 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.

6.10 Water bath or oven, capable of maintaining the following temperatures: $40 ^\circ\text{C} \pm 1 ^\circ\text{C}$ and $60 ^\circ\text{C} \pm 1 ^\circ\text{C}$.

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

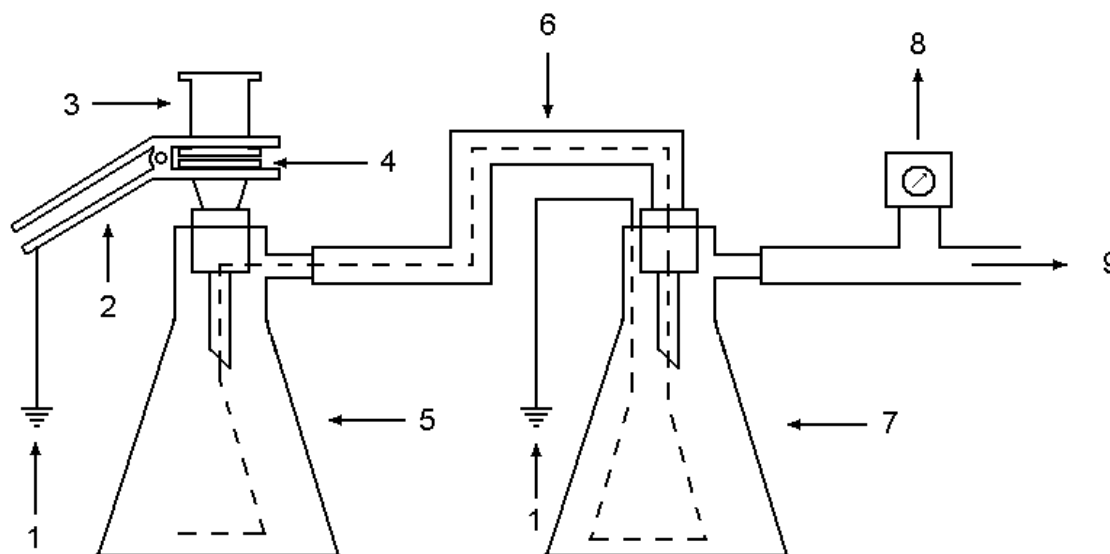
6.12 Top load balance, capable of weighing 1 500 g to the nearest 0,1 g.

6.13 Vacuum source, capable of maintaining an absolute pressure of 2 kPa to 5 kPa inside the filtration apparatus (see Figure 1).

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6.14 Suitable clean sample containers and sampling vessels.

6.15 Stopwatch, capable of measuring 30 min \pm 1 min.



Key

1	wire to ground	4	filter holder	7	safety flask
2	clamp	5	receiving flask	8	vacuometer
3	funnel	6	vacuum tubing	9	to vacuum pump

Figure 1 — Apparatus for determining contamination

7 Preparation of sample containers and apparatus

IMPORTANT — 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:

- likely to come into contact with the sample or flushing fluid, or
- 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 (5.3).

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 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 test portion

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 content in a water bath or oven (6.10) to ensure that any components that have separated out have dissolved again.

9.2.1 Middle distillates and diesel fuels at 40 °C for (30 to 60) min.

9.2.2 Pure FAME at 60 °C for 2 h.

9.3 Remove the sample container from the water bath or oven 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 Middle distillates and diesel fuels are directly filtered (9.5). Pure FAME is filtered after dilution (9.6).

9.5 Direct filtration

9.5.1 Place the beaker (6.3) onto the balance (6.12) and tare.

9.5.2 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.

9.5.3 Weigh into the beaker a test portion equivalent to approximately 300 ml. The sample shall be poured very quickly into the beaker, without trying to get a volume as close as possible to 300 ml.

Record the mass of the test portion m_E .

9.6 Filtration after dilution for pure FAME

9.6.1 Place the bottle (6.4) onto the balance (6.12) and tare.

9.6.2 Shake the container for at least 10 s, one-to-two strokes per second, using 10 cm to 25 cm strokes.

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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.

9.6.3 Weigh into the bottle (6.4) a test portion equivalent to approximately 300 ml. The sample shall be poured very quickly into the beaker, without trying to get a volume as close as possible to 300 ml.

Record the mass of the test portion m_E .

Add 300 ml of the solvent (5.4) by means of a 500 ml graduated cylinder. Mix thoroughly and leave at ambient temperature for 2 h before filtration.

10 Preparation of the equipment

10.1 Preparation of the filtration apparatus

10.1.1 Visually check that the filtration apparatus (6.1) is clean both internally and externally. If not clean, repeat as per Clause 7.

10.1.2 Follow all existing safety precautions and earth the apparatus to avoid electrostatic build-up and discharge.

10.1.3 Assemble the filtration apparatus without the filter and wash the inside with heptane (5.1). Ensure seal between filter holder and receiving flask, and between tube, hose and wire and safety flask with appropriate sealant.

10.2 Preparation of the filter

10.2.1 For all operations handle the filter (6.2) by the edge using forceps (6.9).

10.2.2 Place the filter properly centred on to the filter holder of the pre-cleaned apparatus. Soak the filter with heptane and apply vacuum. Release the vacuum slowly and then remove carefully the filter from the filter holder by means of the forceps (6.9), place it on the Petri dish (6.7) and place in the oven (6.5) at $110\text{ °C} \pm 5\text{ °C}$ for at least 45 min. Use the cover during transport to the oven, remove the cover when the Petri dish is placed in the oven.

Check carefully that the filter is centred on the filter holder. The filter shall not be damaged by the filter holder equipment. Damaged filters affect the weight of the filter and lead to erroneous results.

10.2.3 Remove the Petri dish and filter from the oven, apply the cover and cool in the desiccator (6.6), located near to the analytical balance, for approximately 45 min.

10.2.4 Immediately before the determination, remove the filter from the Petri dish and using the analytical balance (6.8) weigh the filter (6.2) to the nearest 0,1 mg. Record this weight m_1 .

10.2.5 Place the filter directly on to the filter holder of the pre-cleaned apparatus and fix the funnel with the clamp. Soak the filter with heptane. Ensure that the filter is free from bubbles and is firmly fixed between the round surfaces of the filter apparatus.

11 Procedure

WARNING — Electrostatic charges can be generated during the filtration of petroleum products; therefore the filter apparatus shall be earthed.

11.1 Products with a general viscosity

11.1.1 This procedure applies to liquid petroleum products with a kinematic viscosity not exceeding $8\text{ mm}^2/\text{s}$ at 20 °C , or not exceeding $5\text{ mm}^2/\text{s}$ at 40 °C .