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Liquid petroleum products — Determination of total contamination

Part 2: Fatty acid methyl esters

National foreword

This British Standard is the UK implementation of EN 12662-2:2024. Together with BS EN 12662-1:2024, it supersedes BS EN 12662-2:2014, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum Testing and Terminology.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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Flüssige Mineralölerzeugnisse - Bestimmung der Gesamtverschmutzung - Teil 2: Fettsäure-Methylester

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European foreword

This document (EN 12662-2:2024) 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of an identical text or by endorsement, at the latest by December 2024, and conflicting national standards shall be withdrawn at the latest by December 2024.

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

This document supersedes EN 12662-2:2014.

In comparison with the previous edition, the following technical modifications have been made:

- split of the scope of the previous edition in two parts, with Part 2 covering the neat FAME in this document and with Part 1 covering the middle distillates and the diesel fuels containing up to 30 % (V/V) of fatty acid methyl ester (FAME) in a separate document.
- update of the precision data following the statistical analysis [4] of the interlaboratory tests data according to EN ISO 4259-1:2017 [1].

Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

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Introduction

Excessive contamination in a fuel system can give rise to premature blocking of filters and/or hardware failure, and is therefore undesirable. The determination of the content of undissolved substances, referred to as total contamination, is a way to control this issue.

In the previous version of this method, the scope was covering middle distillates, diesel fuels containing up to 30 % (V/V) of FAME and neat FAME. It was found that the improvement sought in 2014, give problems in the lab in testing FAME and correlate the results to those obtained with the previous version of the method. A solution has been found, which resulted in splitting the methodology in two parts: to include the previous version as Part 1 and to develop a separate standard for neat FAME as Part 2.

An interlaboratory study was conducted to determine the valid precision of the method for determining total contamination in neat FAME according to this document.

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1 Scope

This document specifies a method for the determination of the content of undissolved substances, referred to as total contamination, in neat fatty acid methyl esters (FAME). The working range is from 5 mg/kg to 27 mg/kg and it was established in an interlaboratory study by applying EN ISO 4259-1 [1].

This document in general is applicable to FAME having a kinematic viscosity not exceeding 8 mm²/s at 20 °C, or 5 mm²/s at 40 °C, e.g. as specified in EN 14214 [2].

This test method can be used for FAME having a kinematic viscosity exceeding 8 mm²/s at 20 °C, or 5 mm²/s at 40 °C, however in such cases the precision of the test method has not been determined.

NOTE For the purposes of this document, the term “ φ (V/V)” is used to represent the volume fraction, φ , of a material.

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3170, *Petroleum liquids — Manual sampling (ISO 3170)*

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

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org>

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. 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 membrane filter (6.17).

NOTE Heptane used as a reference fuel in EN ISO 5164 [3] 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 Equipment

All glassware and sample containers shall 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. A different filtration apparatus may be used if it is suitable to take the filters given in 6.2.

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 have been found suitable for total contamination measurements. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product.

6.3 Beakers, tall form 0,5 l and 1 l.

6.4 Cylinders, 500 ml and 1 000 ml graduated cylinders.

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

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

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

6.8 Glass Petri dishes with covers, greater than 50 mm in diameter **or an equivalent alternative** to handling the filter, which is FAME and temperature resistant, for example an aluminium bowl.

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

6.10 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.

6.11 Water bath or oven, capable of maintaining the following temperatures: (40 ± 1) °C and (60 ± 1) °C.

6.12 Wash bottle, fitted with spray nozzle, suitable for use with heptane (5.1).

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

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

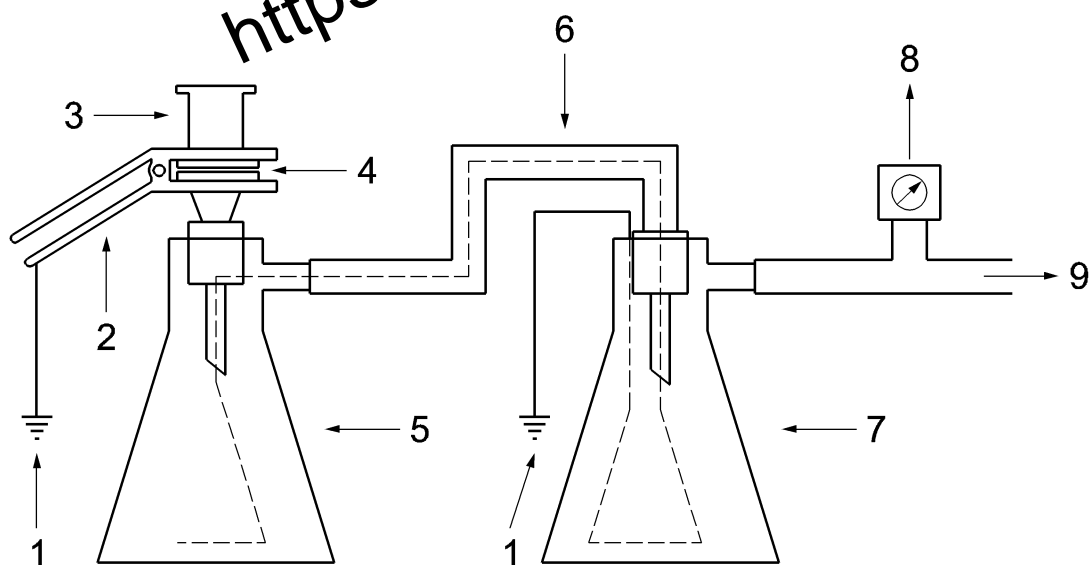
NOTE The vacuum range excludes the use of a water vacuum pump.

6.15 Suitable clean sample containers.

6.16 Clean plastic film or aluminium foil.

6.17 Membrane filter, with a mean pore size of 0,45 μm .

6.18 Temperature measuring device, calibrated in the range [20 °C, 80 °C] with a maximum deviation after calibration of 1 °C to a reference value.



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 — Filtration apparatus for determining contamination

7 Cleansing of sample containers and filtration 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 minimize 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 and parts of the apparatus that are:

- a) likely to come into contact with the sample or heptane (5.1), or
- 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 Handling container caps externally only with clean laboratory tongs or gloves during rinsing with water 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 (6.1) with clean plastic film or aluminium foil (6.16) 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 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 (6.15).

8.4 Where it is only possible to obtain samples 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 homogenization 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.

8.7 The collected sample shall be homogenized according to the procedure given in 9.2.4 before any other analytical measurements are carried out, to avoid non-representative sampling when this method is performed.

9 Preparation of the test portion

9.1 General

Make sure that the sample container (6.15) is free of adhering particles which can distort the analysis. In case of doubt, 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 Neat FAME

9.2.1 Loosen the sample container closure and place the container and its content in a water bath or oven (6.11). Heat the sample to 60 °C, then maintain at this temperature for at least 2 h to ensure that any components that have separated out have dissolved again.

9.2.2 Remove the sample container from the water bath or oven and tighten the container closure. Wash the outside of the container with propan-2-ol (5.2).

9.2.3 Place the beaker (6.3) onto the balance (6.13) and tare.

9.2.4 Shake the sample 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 container walls, repeat shaking procedure.

The use of a mixer is not allowed.

9.2.5 Weigh into the beaker a test portion equivalent to approximately 500 ml. The sample shall be poured very quickly into the beaker, without trying to get a volume as close as possible to 500 ml. Record the mass of the test portion m_E to the nearest 0,1 g.

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 in accordance with 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 (6.1) without the filter (6.2) 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 (6.10).

10.2.2 Place the filter (6.2) properly centred on to the filter holder of the pre-cleaned apparatus. Rinse the filter (6.2) with heptane (5.1) and apply vacuum. Release the vacuum slowly and then carefully remove the filter from the filter holder by means of the forceps (6.10), place it on the Petri dish or equivalent (6.8) and place in the oven (6.6) at (110 ± 5) °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 mass of the filter and lead to erroneous results.

10.2.3 Remove the Petri dish or equivalent (6.8) and filter from the oven (6.6), apply the cover and cool in the desiccator (6.7), located near to the analytical balance (6.9), for at least 45 min.

10.2.4 Immediately before the determination, either remove the filter (6.2) from the Petri dish or equivalent (6.8) and using the analytical balance (6.9) weigh the filter to the nearest 0,1 mg **or** weigh the filter together with the Petri dish or the equivalent (6.8) with the analytical balance (6.9) to the nearest 0,1 mg. Record this mass m_1 .

10.2.5 Place the filter (6.2) directly on to the filter holder of the pre-cleaned apparatus and fix the funnel with the clamp. Rinse the filter with heptane (5.1). 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 Place the weighed test portion (m_E , 9.2.5) in a water bath (6.11) at 40 °C for at least 30 min.

11.2 Filter the test sample (9.2.5) using the filter after its preparation (10.2) applying suction or vacuum as necessary to reach an absolute pressure of 2 kPa to 5 kPa (6.14) inside. The sample shall be transferred into the filtration apparatus (6.1) in small portions. Be careful not to let the filter assembly dry during this transfer.

If foaming is observed in the receiving flask, check the vacuum pressure. A pressure lower than that mentioned above can lead to foaming.

The beaker containing the sample portion should be maintained at approximately 40 °C throughout the filtration, to avoid the cooling of the sample to room temperature.

Filter the whole sample.

11.3 Using the wash bottle (6.12) wash the sediment from the beaker (6.3) or the glass bottle (6.5) onto the filter (6.2) with heptane (5.1). Carefully rinse the inside wall and base of the beaker (6.3) and filter the rinsings. Repeat the washing operation twice more.

11.4 Using the wash bottle (6.12) wash the inside wall of the funnel of the filtration apparatus (6.1) and filter (6.2) with heptane (5.1) and dry under suction. The funnel should be washed with a gentle stream and circumferential movement. Repeat the washing operation two more times.

11.5 Carefully remove the funnel and with the vacuum applied, wash the filter (6.2) from the periphery inwards by directing a gentle stream of heptane (5.1). Take care not to wash any of the particulate from the surface of the filter (6.2). Maintain the vacuum after the final washing for approximately 10 s to 15 s or until all excess heptane is removed from the filter.

11.6 Release the vacuum slowly and then carefully remove the filter (6.2) from the filter holder by means of the forceps (6.10), place it on the Petri dish or equivalent (6.8) (see 10.2) and apply cover. Place the Petri dish or equivalent (6.8), including the filter (6.2), into the oven (6.6), remove the cover and dry at (110 ± 5) °C for 45 min. Allow to cool down to room temperature for approximately 45 min in the desiccator (6.7) located close to the analytical balance using the cover.

11.7 Using the analytical balance (6.9), weigh the filter (6.2) either without the Petri dish (6.8) to the nearest 0,1 mg **or** together with the Petri dish or equivalent (6.8) to the nearest 0,1 mg. Record this mass m_2 . For the determination of the mass, the same procedure as in 10.2.4 shall be followed.

The filter shall be weighed to a constant mass. Temperature and time given in 11.6 are adequate to remove the washing medium. However, it is necessary to verify that the filter has reached a constant mass. If not, the filter is further dried according to 11.6.

The expression “constant mass” means that the drying process should be repeated until the results of two consecutive weighings do not differ by more than 0,2 mg.

12 Calculation

Calculate the total contamination as a mass fraction w in mg/kg, using Formula (1):

$$w = \frac{1\,000(m_2 - m_1)}{m_E} \quad (1)$$

where

- m_1 is the mass of the filter, in milligrams (see 11.2.4);
 m_2 is the mass of the filter with the contaminant in milligrams (see 11.7);
 m_E is the mass of the sample test portion in grams (9.2.5).

13 Expression of results

Express the total contamination as a mass fraction, w , rounded to the nearest 0,5 mg/kg.

14 Precision

14.1 General

The precision given in 14.2 and 14.3 was determined by statistical examination of interlaboratory test results [4] in accordance with EN ISO 4259-1:2017 [1].

14.2 Repeatability

The difference between two independent results obtained using this method for test material considered to be the same in the same laboratory, by the same operator using the same equipment within short intervals of time, in the normal and correct operation of the method that is expected to be exceeded with an approximate probability of 5 % due to random variation, can be calculated using Formula (2):

$$r = 0,2650 * (X + 1,7330) \quad (2)$$

where

- X is the average of the two results being compared, in milligrams per kilogram (mg/kg).

14.3 Reproducibility

The difference between two independent results obtained using this method for test material considered to be the same in different laboratories, where different laboratory means a different operator, different equipment, different geographic location, and under different supervisory control, in the normal and correct operation of the method that is expected to be exceeded with an approximate probability of 5 % due to random variation, can be calculated using Formula (3):

$$R = 0,4187 * (X + 1,7330) \quad (3)$$

where

- X is the average of the two results being compared, in milligrams per kilogram (mg/kg).

15 Test report

The test report shall contain at least the following information:

- a) type and identification of the product under test;
- b) reference to and year of publication of this document, i.e. EN 12662-2:2024;
- c) sampling procedure used (see Clause 8);
- d) result of the test (see Clause 13);
- e) any deviation, by agreement or otherwise, from the procedure described;
- f) any unusual features observed;
- g) date of the test.

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Bibliography

- [1] EN ISO 4259-1:2017, *Petroleum and related products — Precision of measurement methods and results — Part 1: Determination of precision data in relation to methods of test (ISO 4259-1:2017)*
- [2] EN 14214, *Liquid petroleum products — Fatty acid methyl esters (FAME) for use in diesel engines and heating applications — Requirements and test methods*
- [3] EN ISO 5164, *Petroleum products — Determination of knock characteristics of motor fuels — Research method (ISO 5164)*
- [4] CEN/TC 19/WG 31 N96, *II S report for the determination of total contamination of neat fatty acid methyl esters (FAME)*

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BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK