SLOVENSKI STANDARD
SIST EN 12662:2014

01-maj-2014

Nadomešča:
SIST EN 12662:2008

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

Ta slovenski standard je istoveten z: **EN 12662:2014**

ICS:

75.160.20	Tekoča goriva	Liquid fuels

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EUROPEAN STANDARD

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English Version

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

Produits pétroliers liquides - Détermination de la contamination totale des distillats moyens, des gazoles et des esters méthyliques d'acides gras

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

This European Standard was approved by CEN on 13 December 2013.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 12662:2014) 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of an identical text or by endorsement, at the latest by August 2014, and conflicting national standards shall be withdrawn at the latest by August 2014.

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 12662:2008.

The significant technical changes between this European Standard and the previous edition are:

— extension of the scope to middle distillates, diesel fuels containing up to 30 % (*V/V*) fatty acid methyl ester (FAME) and neat FAME;

— update of the working range and precision statement based on interlaboratory study with field samples carried out in 2011 within CEN/TC 19;

— inclusion of a dilution procedure for the determination of total contamination of neat FAME;

— improved description of the filtration procedure and equipment.

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, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of the content of undissolved substances, referred to as total contamination, in middle distillates, in diesel fuels containing up to 30 % (*V/V*) fatty acid methyl esters (FAME), and in neat FAME. The working range is from 12 mg/kg to 30 mg/kg and it was established in an interlaboratory study by applying EN ISO 4259 [1].

This European Standard in general applies to 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 [2] and FAME as in EN 14214 [3].

This test method may be used for diesel fuels containing more than 30 % (*V/V*) FAME and for petroleum products having a kinematic viscosity exceeding 8 mm^2/s at 20 °C, or 5 mm^2/s at 40 °C, however in such cases the precision of the test method has not been defined.

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

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

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 neat FAME and liquid petroleum products having a kinematic viscosity exceeding 8 mm^2/s at 20 °C, or 5 mm^2/s at 40 °C, 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 membrane filter (6.18).

NOTE Heptane used as a reference fuel in EN ISO 5164 [4] is suitable.

5.2 Xylene, analytical grade, filtered using a membrane filter (6.18).

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 (6.5) and mix thoroughly.

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 European Standard and does not constitute an endorsement by CEN of this product.

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

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.

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).

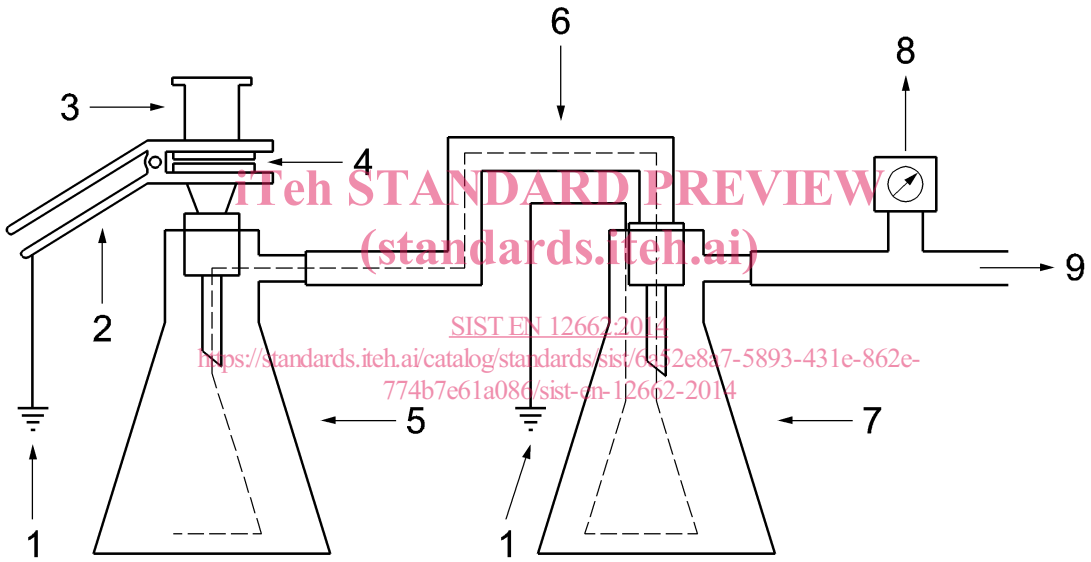
NOTE The vacuum range excludes the use of a water vacuum pump.

6.15 Suitable clean sample containers.

6.16 Stopwatch, capable of measuring 30 min ± 1 min.

6.17 Clean plastic film or aluminium foil.

6.18 Filter, membrane with a mean pore size of 0,45 µm.



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 Rinse thoroughly with water, handling container caps externally only with clean laboratory tongs or gloves 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 (6.1) with clean plastic film or aluminium foil (6.17) 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 (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 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.

8.7 The collected sample shall be homogenised 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 (5.3), as described in 7.2 to 7.5, to remove any adhering particles and avoid introducing undesirable contamination in the test sample.

9.2 Middle distillates and diesel fuels

9.2.1 Loosen the sample container closure and place the container and its content in a water bath or oven (6.11) at 40 °C for 30 min to 60 min 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. Let it cool down to room temperature. Wash the outside of the container with propan-2-ol.