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Dieselsko gorivo in kurilno olje za gospodinjstvo - Določevanje filtrirnosti

Diesel and domestic heating fuels - Determination of cold filter plugging point

Dieselkraftstoffe und Haushaltheizöle - Bestimmung des Temperaturgrenzwertes der Filtrierbarkeit

Combustibles pour moteurs diesel et pour installations de chauffage domestique - Détermination de la température limite de filtrabilité

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English version

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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 Standards supersedes EN 116:1981.

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of an identical text or by endorsement, at the latest by May 1998, and conflicting national standards shall be withdrawn at the latest by May 1998.

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.

Significant technical differences between this European Standard and the first edition of EN 116 are that an automated method is described in addition to, and fully equivalent with, the manual method, provisions are included for the use of certified reference materials for verification and calibration purposes and that the air flow rate in the vacuum regulator has been changed to 15 l/h from (3 to 4) l/h.

In this standard annex A is normative.

1 Scope

This European Standard specifies a method for the determination of the cold filter plugging point (CFPP) of diesel and domestic heating fuels (see 3.1) using either manual or automated test equipment. The manual test equipment and automated test equipment are both suitable for referee purposes.

This European Standard is applicable to distillate fuels, including those containing a flow-improving or other additive, intended for use in diesel engines and domestic heating installations.

The results obtained from the method specified in this European Standard are suitable for estimating the lowest temperature at which a fuel will give trouble-free flow in the fuel system.

> NOTE : In the case of diesel fuels the results are usually close to the temperature of failure in service except when the fuel system contains, for example, a paper filter installed in a location exposed to the weather or if the filter plugging temperature is more than 12 °C below the cloud point of the fuel. Domestic heating installations are usually less critical and often operate satisfactorily at temperatures somewhat lower than those indicated by the test results.

The difference in results obtained from the sample "as received" and after heat treatment at 45 °C for 30 min can be used to investigate complaints of unsatisfactory performance under low temperature conditions.

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 determine the applicability of regulatory limitations prior to use.

2 Normative references

This European Standard incorporates, by dated and undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and publications are listed 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 Petroleum liquids - Manual sampling

ISO 3171 Petroleum liquids - Automatic pipeline sampling

3 Definition

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

3.1 cold filter plugging point (CFPP): Highest temperature at which a given volume of fuel fails to pass through a standardized filtration device in a specified time, when cooled under standardized conditions.

4 Principle

A test portion of the fuel is cooled under the specified conditions and is drawn into a pipette under a controlled vacuum through a standardized wire mesh filter. The procedure is repeated, as the fuel continues to cool, for each 1 °C below the first test temperature. Testing is continued until the amount of wax crystals which have separated out of solution is sufficient to stop or slow down the flow so that the time taken to fill the pipette exceeds 60 s or the fuel fails to return completely to the test jar before the fuel has cooled by a further 1 °C.

The indicated temperature at which the last filtration was commenced is recorded as the cold filter plugging point.

5 Reagents and materials

5.1 Heptane, clean commercial or reagent grade.

5.2 Acetone

5.3 Lintless filter paper, (5 ± 1) μm retention[1].

5.4 Certified reference materials.

NOTE : Certified reference materials may be obtained from the Commission of the European Communities. Community Bureau of Reference, DG XII, Rue de la Loi 200, B-1049 Brussels, who can be contacted to obtain further information.

6 Apparatus

6.1 Manual equipment

6.1.1 General

The equipment, as detailed in 6.1.2 to 6.1.14, shall be arranged as shown in figure 1.

6.1.2 Test jar, cylindrical, of clear glass, flat bottomed, with an internal diameter of (31,5 ± 0,5) mm, a wall thickness of (1,25 ± 0,25) mm and a height of (120 ± 5) mm. The jar shall have a permanent mark at the 45 ml level.

NOTE : Test jars of the required dimensions may be obtained by selection from jars conforming to ISO 3016[2], which specifies wider tolerances.

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1) Durrieux 120 and Whatman No. 3 are examples of suitable products available commercially. This information is given for convenience of the users of this European Standard and does not constitute an endorsement by CEN of these products.

2) ISO 3016: Petroleum products - Determination of pour point.

6.1.3 Jacket, brass, watertight, cylindrical, flat bottomed, to be used as an air bath. It shall have an inside diameter of (45 ± 0,25) mm, an outside diameter of (48 ± 0,25) mm and a height of (115 ± 3) mm (see figure 2).

6.1.4 Insulating ring, made from oil-resistant plastics or other suitable material, to be placed in the bottom of the jacket (6.1.3) to provide insulation for the bottom of the test jar. It shall fit closely inside the jacket and have a thickness of $(6^{+0,3}_{0})$ mm (see figure 3).

6.1.5 Spacers (two), approximately 5 mm thick, made from oil-resistant plastics or other suitable material, to be placed as shown in figure 1 around the test jar (6.1.2) to provide insulation for the test jar from the sides of the jacket. The spacers shall fit closely to the test jar and closely inside the jacket. The use of incomplete rings each with a 2 mm circumferential gap will accommodate variations in test jar diameter. The spacers and insulating ring may be made as a single part as shown in figure 3.

6.1.6 Supporting ring, of oil-resistant plastics or other suitable non-metallic, non-absorbent, oil-resistant material, used to suspend the jacket (6.1.3) in a stable and upright position in the cooling bath and to provide a concentric location for the stopper (6.1.7). A design is shown in figure 4 for guidance but this may be modified to suit the cooling bath.

6.1.7 Stopper, of oil-resistant plastics or other suitable non-metallic, non-absorbent, oil-resistant material, to fit the test jar and the support ring is shown in figure 5. It shall have three holes to accommodate the pipette (6.1.8), the thermometer (6.1.9) and to allow venting of the system. If necessary, when using the high-range thermometer, the upper part of the stopper shall be locally grooved to permit the thermometer [6.1.9 a)] to be read down to a temperature of -30 °C.

A pointer shall be fitted to the upper surface of the stopper to facilitate location of the thermometer in relation to the bottom of the test jar. A spring wire clip shall be used to retain the thermometer in the correct position.

6.1.8 Pipette with filter unit

6.1.8.1 A pipette of clear glass with a calibration mark corresponding to a contained volume of (20 ± 0,2) ml at a point (149 ± 0,5) mm from the bottom of the pipette (see figure 6). It shall be connected to the filter unit (6.1.8.2).

6.1.8.2 A filter unit (see figure 7) containing the following elements.

a) a brass body, with a threaded cavity that houses the wire mesh holder. The cavity shall be fitted with an O-ring of oil-resistant plastics. The internal diameter of the central tube shall be (4 ± 0,1) mm;

b) a brass screw cap to connect the upper part of the body of the filter unit (6.1.8.2) to the lower part of the pipette (6.1.8.1) to ensure a leak-free joint. An example of satisfactory connection is shown in figure 7;

c) a disc, 15 mm diameter, of plain weave stainless steel wire mesh gauze with a nominal aperture size of 45 μm. The nominal diameter of the wire shall be 32 μm and

the tolerance for the size of an individual aperture shall be as follows:

1) no aperture size shall exceed the nominal size by more than 22 μm;

2) the average aperture size shall be within $\pm$ 3,1 μm of the nominal size;

3) not more than 6 % of the apertures shall be above the nominal size by more than 13 μm.

NOTE : The requirements for the wire mesh are taken from ISO 3310-1[3], to which reference may be made for methods for testing the gauze.

d) a filter holder of brass, in which the disc of wire mesh gauze [6.1.8.2 c)] is firmly clamped by a retaining ring pressed into the filter holder. The diameter of the exposed part of the gauze shall be $(12^{+0,1}_{0})$ mm (see figure 8);

e) a brass cylinder, threaded on the outside, that can be screwed into the cavity of the body [6.1.8.2 a)] to clamp the filter holder [6.1.8.2 d)] against the O-ring [6.1.8.2 a)]. The lower end shall have four slots to allow the test portion to flow into the filter unit.

6.1.9 Thermometers

The following thermometers are required:

a) high-range thermometer, range -38 °C to +50 °C, for measuring cold filter plugging point down to -30 °C;

b) low-range thermometer, range -80 °C to +20 °C, for measuring cold filter plugging point below -30 °C;

c) cooling bath thermometer, range -80 °C to +20 °C.

Thermometers conforming to the essential requirements set out in annex A to this standard shall be used.

6.1.10 Cooling bath

The type of cooling bath is optional, but it shall be of a shape and size suitable for containing the jacket (6.1.3) in a stable and upright position at the required depth.

The bath shall be fitted with a cover with one or more holes in it to accommodate the supporting ring (6.1.6). The jacket (6.1.3) may be fixed permanently in the lid.

The bath temperature shall be maintained at the required value by a refrigeration unit or by the use of suitable freezing mixtures, ensuring a homogenous temperature in the bath by stirring or other means of agitation.

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3) ISO 3310-1: Test sieves - Technical requirements and testing - Part 1: Metal cloth

The bath temperatures given in table 1 shall be used, for different levels of cold filter plugging point. These may be obtained either by separate cooling baths or by adjusting the refrigeration unit. If a refrigeration unit is used it shall be capable of changing the bath temperature within 2 min 30 s.

Table 1 : Cooling bath temperature

Expected cold filter plugging point	Required cooling bath temperature(s)
Above -20 °C	(-34 ± 0,5) °C
Between -20 °C and -35 °C	(-34 ± 0,5) °C, then (-51 ± 1,0) °C
Below -35 °C	(-34 ± 0,5) °C, then (-51 ± 1,0) °C, then (-67 ± 2) °C

If several testing units are placed in one large cooling bath, the distance between the jacket wells shall be at least 50 mm.

6.1.11 Stopcock, glass, with double oblique bore of 3 mm bore diameter.

6.1.12 Vacuum source, vacuum pump or water pump powerful enough to ensure an air flow rate in the vacuum regulator (6.1.13) of (15 ± 1) l/h for the duration of the test.

6.1.13 Vacuum regulator, consisting of a glass bottle, at least 350 mm high, not less than 5 l capacity, partially filled with water. It shall be closed by a stopper with three holes of convenient diameters for glass tubes. Two tubes shall be short and shall not go below the water level. The third tube, with an internal diameter of (10 ± 1) mm, shall be long enough for one end to be approximately 200 mm beneath the surface of the water while the other end reaches a few centimetres above the stopper. The depth of the immersed part shall then be adjusted to obtain a depression of (200 ± 1) mm ($2^{0}_{-0,05}$) kPa on the manometer which shall contain water.

A second empty 5 l bottle shall be fitted in the line, to serve as vacuum reservoir to ensure a constant depression. The arrangement is shown in figure 1.

6.1.14 Stopwatch, with a graduation or reading of 0,2 s or lower, with an accuracy of 0,1 % over a period of 10 min.

6.2 Automated equipment

6.2.1 General

The automated equipment shall include elements conforming to 6.1.2 to 6.1.8, platinum resistance thermometers, cooling bath(s), vacuum pump and suitable electronic control devices.

6.2.2 Cooling bath

The refrigeration unit used shall be capable of maintaining the cooling bath at the required temperature (see table 1) and also of automatically changing the bath temperature within 2 min 30 s at the appropriate stage, as given in table 1.

6.2.3 Vacuum source

Vacuum pump powerful enough to ensure an air flow rate in the vacuum regulator of a minimum of 15 l/h, and to maintain a constant vacuum of (200 ± 1) mm ($2^{0}_{-0,05}$) kPa for the duration of the test. For multi-position testers using the same vacuum source, the flow rate shall be checked when several positions are operating simultaneously.

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 the test sample

Filter approximately 50 ml of the sample at laboratory ambient temperature, but in any case not less than 15 °C, through dry filter paper (5.3).

9 Preparation of apparatus

NOTE : The correct functioning of manual and automated apparatus should be verified preferably at least two times a year and, where possible, using certified reference materials. The apparatus should be checked more frequently (e.g. weekly) using an in-house secondary reference material, such as fuel of known CFPP value. Deviations greater than the test repeatability, or an unacceptable statistical quality control bias, should be investigated and resolved. The manufacturer's instruction manual should provide guidance on ensuring that the equipment is correctly set up and calibrated.

9.1 Manual equipment

Before each test, dismantle the filter unit (6.1.8.2) and wash the pieces and the test jar (6.1.2), the pipette (6.1.8.1) and the thermometer (6.1.9) with heptane (5.1), then rinse with acetone (5.2) and dry in a stream of filtered air. Check the cleanliness and dryness of all elements, including the jacket. Examine the wire mesh [6.1.8.2 c)] and the joints [6.1.8.2 a) and 6.1.8.2 b)] for damage; if necessary renew them. Check the calibration of the thermometers.

Assemble the apparatus as shown in figure 1. Check that the screw cap [6.1.8.2 b)] is tight enough to prevent leakage.

9.2 Automated equipment

Prepare the automated equipment in accordance with the manufacturers instructions. Before each test, wash, dry and examine the pieces as described in 9.1.

NOTE : The calibration of the temperature indicators and platinum resistance thermometers should be checked periodically (i.e. at least once per year).

10 Procedure

10.1 Using the manual equipment

10.1.1 Place the insulating ring (6.1.4) on the bottom of the jacket (6.1.3).

10.1.2 If the spacers (6.1.5) are not mounted on the insulating ring (6.1.4), position them approximately 15 mm and 75 mm above the bottom of the test jar (6.1.2).

10.1.3 Pour the filtered test sample (clause 7) into the test jar to the mark (45 ml).

10.1.4 Close the test jar with the stopper (6.1.7) carrying the pipette with filter unit (6.1.8), and the appropriate thermometer (6.1.9). Use a low-range thermometer if the expected cold filter plugging point is below -30 °C. Thermometers shall not be changed during the test. Adjust the apparatus in such a way that the bottom of the filter unit [6.1.8.2 e)] rests on the bottom of the test jar and position the thermometer so that its lower end is (1,5 ± 0,2) mm above the bottom of the test jar. Take care to ensure that the bulb of this thermometer is not in contact with the side of the test jar or the filter body.

10.1.5 If the jacket is not an integral part of the cooling bath, place the jacket vertically to a depth of (85 ± 2) mm in the cooling bath (6.1.10) which is maintained at the temperature of (-34 ± 0,5) °C.

10.1.6 Insert the test jar assembly in a stable vertical position into the jacket.

10.1.7 With the stopcock (6.1.11) open to atmosphere, connect the pipette to the vacuum system (6.1.12 and 6.1.13) by means of flexible tubing attached to the stopcock (see figure 1). Switch on the vacuum source and regulate to ensure an air flow rate of 15 l/h in the vacuum regulator (6.1.13). Before starting a test, check that the U-tube manometer indicates a (200 ± 1) mm ($2^{0}_{-0,05}$) kPa depression.

10.1.8 Start the test immediately after inserting the test jar assembly into the jacket, but if the cloud point of the sample is known it is permitted to wait until the test portion has cooled to a temperature of not less than 5 °C above its cloud point. Always use the bath maintained at (-34 ± 0,5) °C for the first cooling stage (see 10.1.10).

When the temperature of the test portion reaches a suitable integral value turn the stopcock (6.1.11) so that the filter assembly is connected to the vacuum source, causing the test portion to be drawn through the wire mesh into the pipette; simultaneously start the stopwatch.

When the test portion reaches the mark on the pipette stop the stopwatch and turn the stopcock to its initial position to vent the pipette and so allow the test portion to return to the test jar.

If the time taken to reach the mark exceeds 60 s abandon the test and repeat it on a fresh portion starting at a higher temperature.

10.1.9 Repeat the operation (10.1.8) for each 1 °C decrease of the test portion temperature until the temperature is reached at which the pipette is not filled to the etched mark within 60 s. Record the temperature at which this last filtration was commenced as CFPP (see clause 11).