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BS 2000-309:2015

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Diesel and domestic heating fuels — Determination of cold filter plugging point — Stepwise cooling bath method

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National foreword

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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 secretary.

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Diesel and domestic heating fuels - Determination of cold filter plugging point - Stepwise cooling bath method

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

Dieselkraftstoffe und Haushaltsheizöle - Bestimmung des Temperaturgrenzwertes der Filtrierbarkeit durch stufenweise Abkühlung

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European foreword

This document (EN 116:2015) 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of an identical text or by endorsement, at the latest by February 2016, and conflicting national standards shall be withdrawn at the latest by February 2016.

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 116:1997.

Significant technical differences between this European Standard and the previous edition of EN 116 are:

— that an automated method is described in detail beside the former manual procedure;

— the scope and the precision statement have been changed based on a European Round Robin study including current available fuels for use in diesel engines. In addition, Fatty Acid Methyl Esters (FAME) and FAME blends in diesel have been checked.

An alternative technique, i.e. using a linear cooling bath, has been developed as in EN 16329 [1].

Round Robin studies conducted for this revision showed different precision values compared to the previous edition. The studies covered current situation of marketed fuels including non-fossil component or biofuels.

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 cold filter plugging point (CFPP) of diesel and domestic heating fuels (see 3.1) using automated test equipment. Manual test equipment may be used, but for referee purposes only automated test equipment is allowed.

This European Standard is applicable to fatty-acid methyl esters (FAME) and to distillate fuels as well as paraffinic diesel fuels, including those containing FAME, flow-improvers or other additives, 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 before the filtration may be used to investigate complaints of unsatisfactory performance under low temperature conditions.

WARNING — The use of this Standard can 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 users of this standard to take appropriate measures to ensure the safety and health of personnel prior to application of the standard, and fulfil statutory and regulatory requirements for this purpose.

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 ISO 3170, *Petroleum liquids — Manual sampling (ISO 3170)*

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

ISO 261, *ISO general purpose metric screw threads — General plan*

3 Terms and definitions

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

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 at intervals of 1 °C into a pipette under a controlled vacuum of 2 kPa 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 (CFPP).

5 Reagents and materials

5.1 Hydrocarbon solvents, technical grade, e.g. Heptane.

5.2 Light hydrocarbon solvent, technical grade, e.g. Isopentane.

5.3 Lintless filter paper, with particle retention of (5 ± 1) µm.

5.4 Certified reference materials.

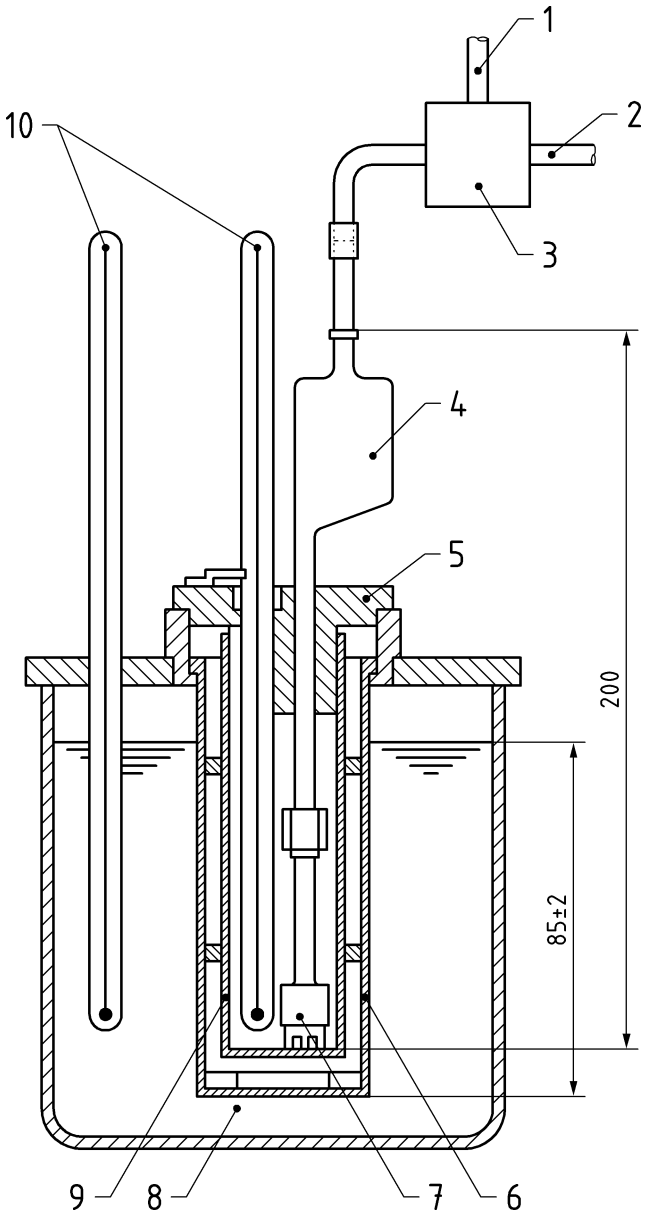
Certified reference materials may be obtained from the European Commission's Institute for Reference Materials and Measurements (IRMM), Retieseweg 111, B-2440 Brussels.

6 Apparatus

The equipment, as detailed in 6.1 to 6.11, shall be arranged as shown in Figure 1. Unless otherwise stated, all dimensions in the figures are given in mm.

IMPORTANT — Parts of the apparatus made of copper, zinc or brass can interact with bio-components like fatty acid methyl ester (FAME). Care shall be taken and parts made from alternative materials are available.

Dimensions in millimetres



Key

1	atmosphere (6.7)	6	jacket (6.2)
2	vacuum regulator (6.7)	7	filter unit (6.5.2)
3	valve unit (6.7)	8	cooling bath (6.9)
4	pipette (6.5.1)	9	test jar (6.1)
5	stopper (6.4)	10	thermometers (6.8)

Figure 1 — General arrangement of apparatus

6.1 Test jar, cylindrical, of clear glass, flat bottomed, with an outside diameter of (34 ± 0,5) mm, a wall thickness of (1,25 ± 0,25) mm and a height of (120 ± 5) mm, having a permanent mark at the 45 ml level.

Test jars of the required dimensions may be obtained by selection from jars conforming to ISO 3016[1)] which specifies wider tolerances.

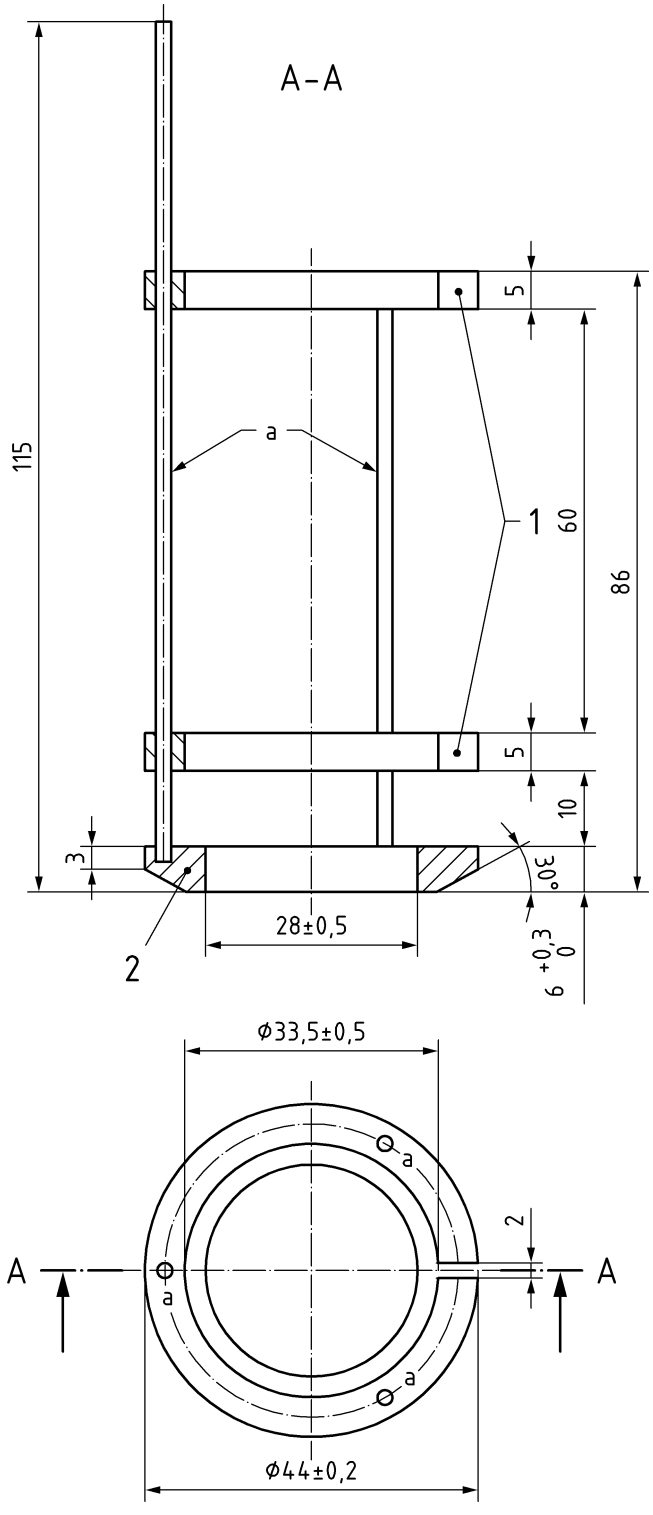
6.2 **Jacket**, watertight, cylindrical, flat bottomed, to be used as an air bath, having an inside diameter of (45 ± 0,25) mm, an outside diameter of (48 ± 0,25) mm and a height of (115 ± 3) mm.

6.3 **Spacer**, made from POM-C[2)], except for the stainless steel rods with 2 mm diameter, to be placed into the jacket (6.2), conforming to the dimensions as shown in Figure 2.

1) ISO 3016: Petroleum products – Determination of pour point

2) POM-C Polyoxymethylen Copolymer, Trade marks e.g. DELRIN.

Dimensions in millimetres



Key

a stainless steel wires ø 2 mm

1 spacer

2 insulating rings

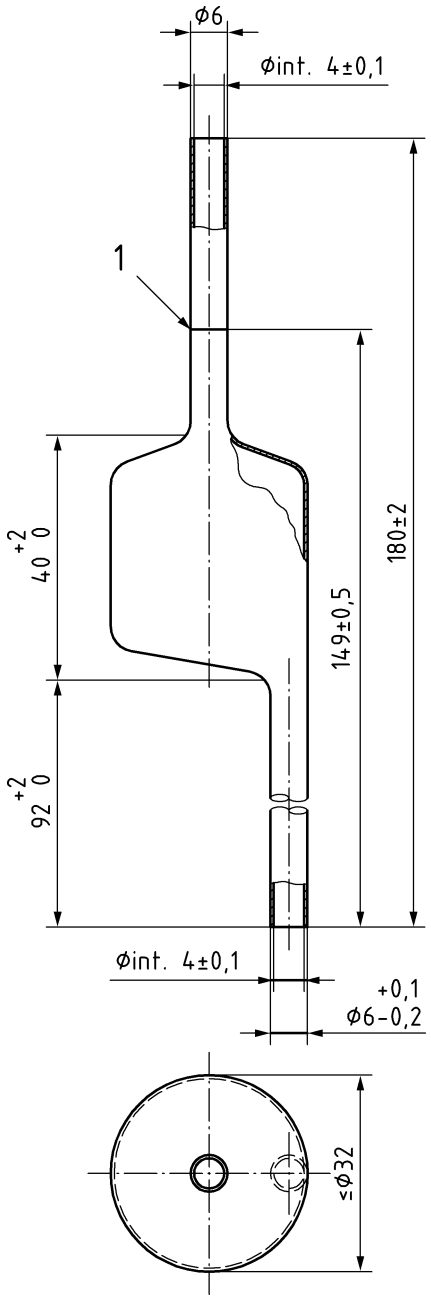
Figure 2 — Insulated ring (below) and spacer (above)

6.4 Stopper, of oil-resistant plastic and non-thermal conductive material, having three holes to accommodate the pipette (6.5), the thermometer (6.8) and to allow venting of the system and able to ensure that pipette and thermometer are safely positioned in the test jar.

6.5 Pipette with filter unit:

6.5.1 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 3). It shall be connected to the filter unit (6.5.2).

Dimensions in millimetres



Key

1 mark

Figure 3 — Pipette

6.5.2 A **filter unit** as in Figure 4, with ISO general purpose metric screw threads according to ISO 261, further consisting of a filter holder as in Figure 5 and a disc, 15 mm diameter, stainless steel wire mesh gauze with a nominal aperture size of 45 µm in basket weave (not twill). The nominal diameter of the wire shall be 32 µm and the tolerance for the size of an individual aperture shall be as follows:

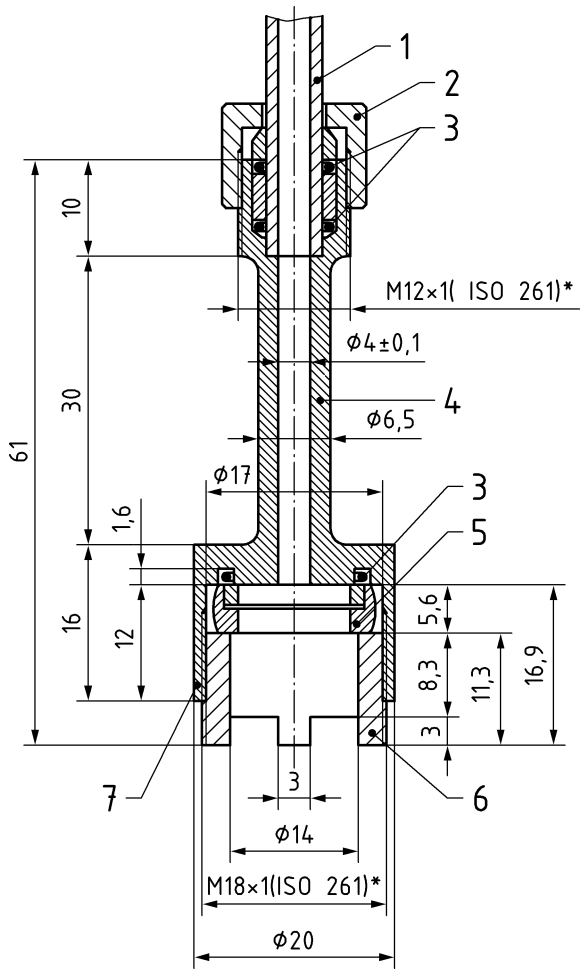
1) no aperture size shall exceed the nominal size by more than 22 µm;

2) the average aperture size shall be within ± 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.

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.

Dimensions in millimetres

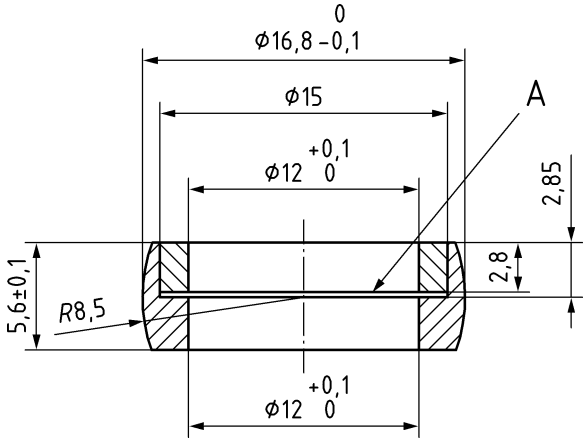


Key

1	pipette tube (6.5.1)	5	filter holder
2	brass screw cap	6	threaded brass cylinder
3	oil-resistant plastics O-ring	7	knurl
4	brass body		

Figure 4 — Filter unit

Dimensions in millimetres



Key

A stainless steel plain weave wire gauze 45 µm

Figure 5 — Filter holder

6.6 An **automated detection system** comprising one sensor to record the filling of the pipette, prior to the subsequent aspiration cycle at a volume of (20 ± 0,2) ml, called the 20 ml mark, and a second sensor positioned (85 ± 4) mm from the bottom of the pipette, called bottom filling mark.

6.7 A **valve unit** that connects the pipette either to the vacuum regulator to allow sample sucking into the pipette, or to the atmosphere to allow backflow of the sample to the test jar. The connecting line between the valve unit and the pipette should have a maximum length of 2 m, and an internal diameter comprised between 4 mm and 6 mm.

6.8 The following **thermometers,** conforming to the essential dimensions set out in Annex A, shall be used:

6.8.1 A **platinum** resistance thermometer with digital display shall be used for measuring the sample temperature with a resolution of 0,1 °C and an accuracy of 0,5 °C (see Figure 6 and 10.4 for the positioning).

6.8.2 A **platinum** resistance thermometer with digital display shall be used for measuring the cooling bath temperature.

Dimensions in millimetres

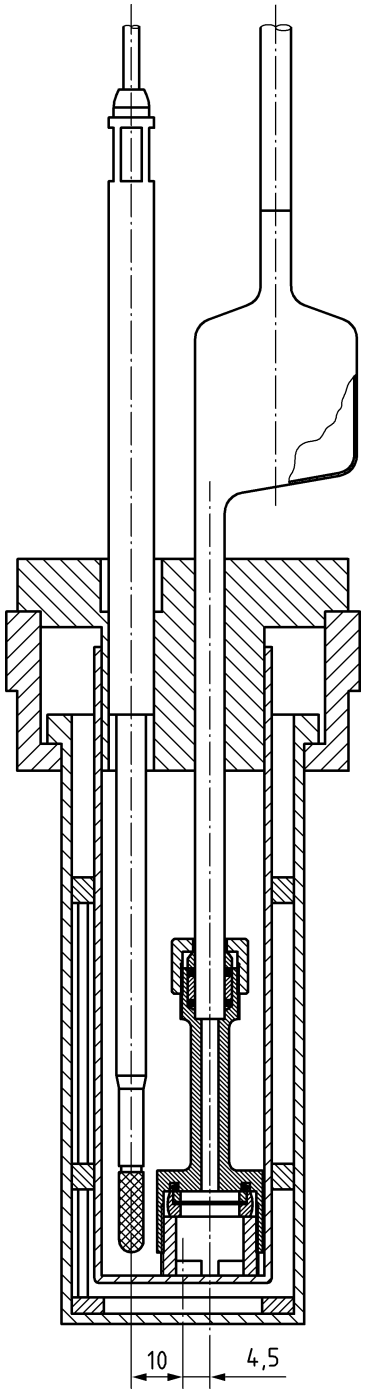


Figure 6— Filter unit and thermometer position

6.9 Cooling bath.

It shall be of a shape and size suitable for containing the jacket (6.2) in a stable and upright position at the required depth.

The jacket (6.2) 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 for liquid baths.

The bath temperature given in Table 1 shall be used, for different levels of cold filter plugging point. These may be obtained 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

Temperature of the test portion °C	**Required cooling bath temperature** °C
Above −20	(−34,0 ± 0,5)
Between −20 and −35	(−51,0 ± 1,0)
Below −35	(−67 ± 2)

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

6.10 Vacuum source, vacuum pump or water pump powerful enough to ensure an air flow rate in the vacuum regulator of (15 ± 1) l/h during the entire aspiration cycle.

For Vacuum control either follow 6.10.1 or 6.10.2.

6.10.1 Vacuum regulator with glass bottles, comprising of:

A **vacuum pump or water pump** with a suction performance of 60 l/h to 100 l/h. A performance of less than 60 l/h will not be sufficient to ensure the required vacuum if several test units are operated simultaneously. A performance higher than 100 l/h can lead to turbulence in the water filled bottle and negatively influence the constancy of the vacuum.

A **glass bottle**, at least 350 mm high, with a capacity of not less than 5 l, is partially filled with water. It shall be closed by a stopper with three holes of convenient diameter for glass or stainless steel tubes. Two tubes shall be short and shall not go below the water level. The third tube 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 the required depression 2 kPa ± 0,05 kPa on the manometer.

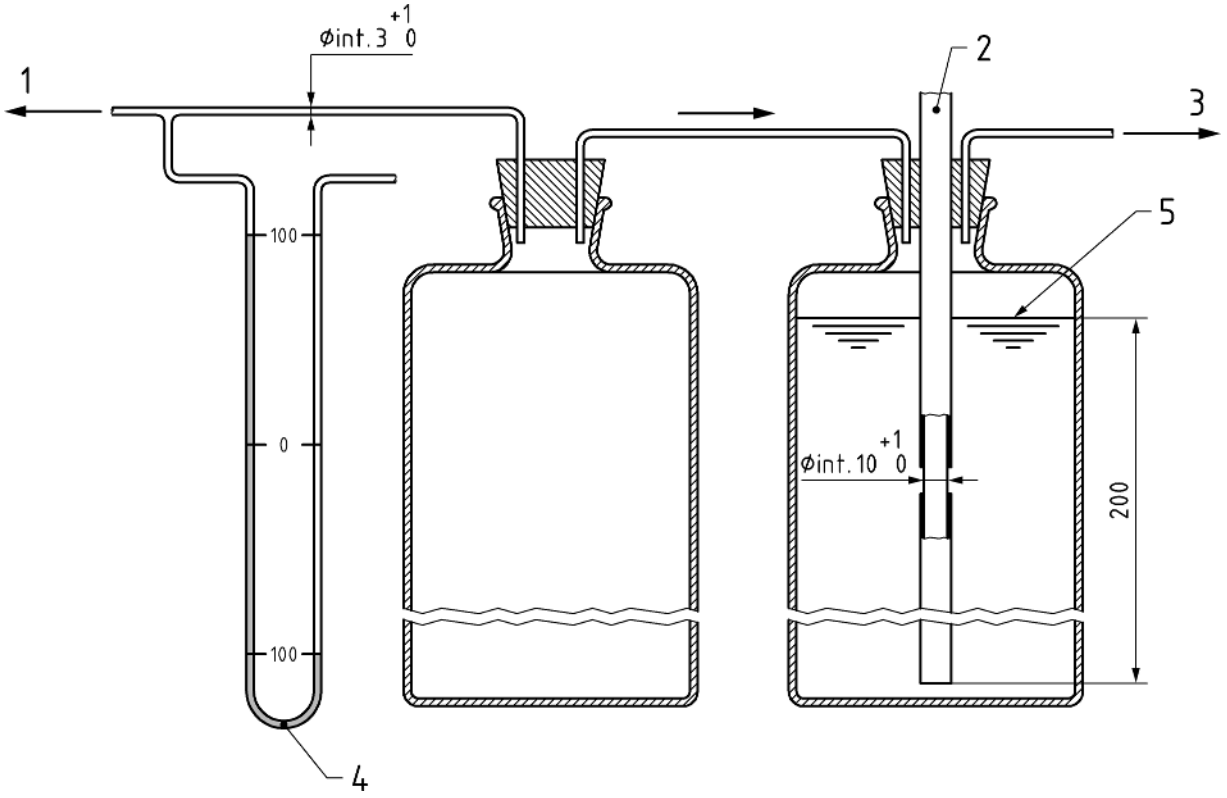
A second empty **5 l bottle** shall be fitted in the line to serve as vacuum reservoir in order to ensure a constant depression. The arrangement is shown in Figure 7.

With this vacuum regulation up to 10 test units may be operated simultaneously. A prerequisite is that each test unit has a separate line from the valve unit to the vacuum regulator, and that a line length of 5 m is not exceeded. Commercially available silicone tubing with an internal diameter of 4 mm to 6 mm may be used for the supply lines.

6.10.2 Electronic vacuum regulator (for automatic method), able to guarantee the requested vacuum of 2 kPa ± 0,05 kPa over the whole aspiration cycle.

6.11 Time measuring device, having a resolution of 0,2 s and an accuracy of 0,1 % over a period of 10 min.

Dimensions in millimetres



Key

1 connection to the filter unit (6.5.2)

2 atmosphere (6.7)

3 connection to the pump

4 water

5 water level

Figure 7 — Vacuum regulator with glass bottles

7 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in EN ISO 3170 or EN 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

IMPORTANT — The correct functioning of the automated apparatus should be verified preferably at least twice 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.

Prepare the equipment according to the manufacturer's instructions. Before each test, dismantle the filter unit (6.5.2), wash the pieces, the test jar (6.1), the pipette (6.5.1) and the thermometer (6.8.1) with hydrocarbon solvent (5.1), then rinse with light hydrocarbon solvent (see 5.2) and dry in a stream of filtered air. Check the cleanliness and dryness of all elements, including the jacket (6.2). Examine the wire mesh and the joints for damage; if necessary renew them. Check that the screw cap is tight enough to prevent leakage.

It is strongly recommended to inspect the filter holder on a regular basis and clean it, as needed, depending on the type of samples.

10 Procedure

10.1 Position the spacer (6.3) in the jacket (6.2). The jacket shall be free from moisture/ice; this shall be checked immediately before the positioning.

10.2 Pour the filtered test sample (see Clause 8) into the test jar to the mark (45 ml).

10.3 Check that the cooling bath with the positioned jacket is at a temperature at (−34 ± 0,5) °C.

10.4 Place the test jar with its stopper (6.4), and the pipette with filter unit (6.5) and suitable thermometer (6.8.1) into the jacket as shown in Figure 6.

Ensure that the bottom of the filter unit rests on the bottom of the test jar. Check that the thermometer does neither touch the wall of the test jar nor the filter unit. The lower end of the thermometer is (1,5 +/- 0,2) mm above the bottom of the test jar.

10.5 Connect the pipette to the vacuum system.

10.6 Start the test now according to the manufacturer's instruction. The cooling bath shall be brought at the appropriate temperature as indicated in Table 1. Most apparatus will automatically carry out the test procedure.

10.7 When the temperature of the test portion reaches an integer value, the first aspiration cycle is started by connecting the pipette via the valve unit to the vacuum regulator, causing the test portion to be drawn into the pipette. When the pipette is filled to the 20 ml mark, it is connected via the valve unit to the atmosphere, thus allowing the test portion to return to the test jar.

10.8 The aspiration cycle is repeated for each 1° C decrease of the test portion's temperature until the temperature is reached at which the pipette is not filled to the 20 ml mark within 60 s. Record the temperature at which this last filtration was commenced as CFPP (see also Clause 12).

A small minority of samples may exhibit anomalous aspiration behaviour, which may be detected by examining the aspiration times recorded in the test print-out for signs of an unexpected reduction in the time to fill the pipette, after which the aspiration time again continues to increase progressively, until the failure limit of 60 s is reached.

10.9 If after cooling in accordance with 10.7, the test portion fills the pipette to the 20 ml mark in less than 60 s, but does not flow back to the bottom filling mark when the pipette is vented to atmosphere via the valve unit (see 6.7) before the start of the next aspiration, record the temperature at the commencement of the filtration as the CFPP (see also Clause 12).

11 Calibration

11.1 General

The following components of the test equipment shall be calibrated at regular intervals. Unless otherwise stated, a calibration is recommended every half year.

11.2 Thermometers

The calibration of temperature measuring devices, including Pt 100 (6.8.1 and 6.8.2) shall be checked at regular intervals (i.e. at least once a year) by means of reference thermometers traceable to national or international SI standards.

11.3 Cooling unit

The individual cooling bath temperatures shall be checked with a certified thermometer.

11.4 Vacuum unit

The vacuum shall be checked by means of a manometer traceable to national or international SI standards.

12 Expression of results

Report the temperature read or indicated at the beginning of the last filtration to the nearest 1 °C (see 10.8 and 10.9) as the cold filter plugging point.

13 Precision

13.1 General

The statistical analysis was carried out using EN ISO 4259 [2].

The precision analysis is based on a study carried out in 2011. That study included FAME, heating oil and diesel fuels covering the range from 0 % to 30 % in volume of FAME, as well as paraffinic diesel fuels.

The range of CFPP values for the Round Robin was from −47 °C to 11 °C. Extrapolations to measurements more than a few degrees outside this range are unsupported by the data.

NOTE A technical report with further details on the analysis is under development within CEN.

13.2 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values calculated from the following formula in absolute value only in one case in 20:

$$r = 1,2 - 0,027X \quad (1)$$

where

X is the average of the two results being compared, in °C.

13.3 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values calculated from the following formula in absolute value only in one case in 20:

$$R = 3,0 - 0,060X \quad (2)$$

where

X is the average of the two results being compared, in °C.

14 Test report

The test report shall include at least the following information:

a) a reference to this European Standard, i.e. EN 116;

b) the type and complete identification of the product under test;

c) the sampling procedure used (see Clause 7);

d) the result of the test (see Clause 12);

e) any deviation from the procedure described (see 2nd paragraph in 10.8);

f) the date of the test.

Annex A
(normative)

Thermometer requirements

This annex describes the required dimensions of the thermometer (6.8.1 and 6.8.2) used in this document.

Table A.1 gives the dimensions for the platinum resistance thermometer (PRT) for each part as depictured and indicated as in Figure A.1.

For platinum resistance thermometer (PRT) made with a full metal shaft, the supplier shall confirm that the thermometer has been manufactured according to the dimensions.

Table A.1 — Thermometer dimensions

Indication	Description	Dimension mm
A	Probe diameter	4,5 ± 1,0
B	Distance from bottom to wire coil	3,0 ± 2,0
C	Length of wire coil including tip	12,0 ± 3,0
D	Stem outside diameter	6,5 ± 1,0

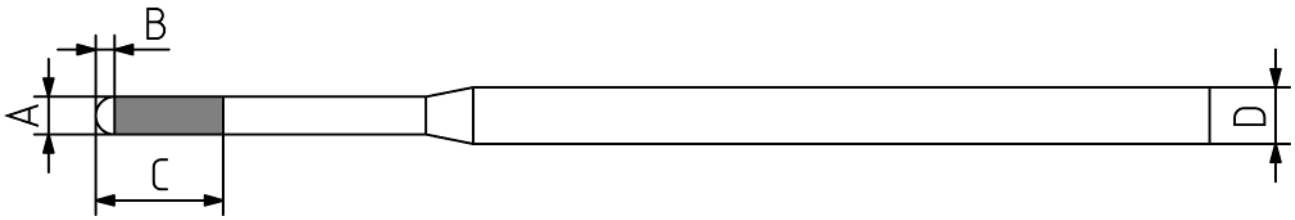


Figure A.1 — Thermometer description

Bibliography

[1] EN 16329, *Diesel and domestic heating fuels — Determination of cold filter plugging point — Linear cooling bath method*

[2] EN ISO 4259, *Petroleum products — Determination and application of precision data in relation to methods of test (ISO 4259)*

[3] ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

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