SLOVENSKI STANDARD
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Dieselsko gorivo - Določ evanje filtrirnosti - Metoda SFPP

Automotive diesel fuels - Determination of filtrability - SFPP method

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Automotive diesel fuels - Determination of filtrability - SFPP method

Combustibles pour moteurs diesel (gazole) - Détermination de la filtrabilité - Méthode SFPP

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Foreword

This CEN Report has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products" the Secretariat of which is held by the Netherlands Standardization Institute (NNI)).

1. Scope

This CEN Report describes a method of test which can be used to determine the simulated filter plugging point (SFPP) of automotive diesel fuels.

The method descibed is applicable to distillate fuels, including those containing flow-improving or other additives, intended for use in automotive diesel engines and may be used to estimate the lowest temperature at which a fuel will give trouble-free operation in a diesel-engined vehicle.

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

2. Normative references

This CEN Report incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the 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 of revision. For undated references the latest edition of the publication referred to applies.

EN 116, *Diesel and domestic heating fuels - Determination of cold filter plugging point.*

EN ISO 3170, *Petroleum liquids — Manual sampling.*

EN ISO 3171, *Petroleum liquids — Automatic pipeline sampling.*

ISO 3015, *Petroleum products — Determination of cloud point.*

3. Definition

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

3.1
simulated filter plugging point (SFPP)
lowest temperature at which a given volume of fuel, cooled under standardized conditions, will pass through a standardized filtration device in a specified time

4. Principle

A test portion of fuel is cooled under controlled conditions and is drawn into a pipette, under vacuum, through a standard wire mesh filter. As the fuel follows a prescribed cooling regime, the procedure is repeated at each 1 °C below the first test temperature until the first (highest) temperature at which the pipette cannot be filled within 60 s, or at which the pipette cannot be emptied within 40 s, is reached. The temperature at which the last successful filtration occurs is recorded as the simulated filter plugging point.

5. Reagents and materials

5.1 Cleaning solvent, capable of dissolving fuel and wax at ambient laboratory temperature and capable of being completely evaporated during the drying procedure (see 9.2).

NOTE: Solvents such as heptane or similar commercial cleaning solvents have been found to be suitable.

5.2 Lintless filter paper, between 20 µm and 30 µm retention [1].

5.3 Supply of dry compressed air.

6. Apparatus

Usual laboratory apparatus and glassware, together with the following:

6.1 Automatic test unit, consisting of the components detailed in 6.1.1 to 6.1.6.

NOTE: A general arrangement of the test unit is shown in figure 1.

6.1.1 Brass jacket, watertight, cylindrical and flat-bottomed, conforming to figure 2, with an internal diameter of (45 ± 0,25) mm and an external diameter of (48 ± 0,25) mm, immersed to a depth of not less than 85 mm in the liquid of the cooling bath (6.1.3).

6.1.2 Stopper, conforming to figure 3, made from plastics of other non-metallic, non-absorbent material, resistant to both the test sample and the cleaning solvent (5.1).

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[1] Durieux 120 and Whatman No. 113 V are examples of suitable products available commercially. This information is given for the convenience of users of this CEN Report and does not constitute an endorsement by CEN of these products.

6.1.3 Cooling bath, with refrigeration unit capable of maintaining the bath at a temperature of (-34 ± 0,5) °C until the test portion (8.2) has cooled to -20 °C. If the failure temperature (10.9) has not been reached at -20 °C, the bath shall then change to a temperature of (-51 ± 1,0) °C. When the test portion (8.2) has further cooled to -35 °C and the failure temperature (10.9) has still not been reached, the bath shall change to a temperature of (-67 ± 2) °C. The unit shall be capable of changing the bath temperature within 2 min 30 s, at the appropriate stages.

6.1.4 Platinum resistance thermometer (PRT), with a resistance of 100 Ω at 0 °C and a precision of ± 0,5 °C.

6.1.5 Light sensor assembly, capable of registering the filling and emptying of the pipette (6.7.1).

6.1.6 Electronic timing system, capable of recording the time and stopping the test when failure occurs (see 10.9).

6.2 Clamp, for the PRT (6.1.4), allowing adjustment of the vertical position of the PRT relative to the pipette/filter unit/light sensor assembly.

6.3 Temperature-controlling sheath, containing the components detailed in 6.3.1 to 6.3.7 and conforming to the arrangement shown in figure 4.

6.3.1 Copper inner cylinder, having an internal diameter of (34,3 ± 0,05) mm and conforming to the shape and dimensions given in figure 5.

6.3.2 Heating membrane[2]**,** resistant to water, the test sample and the cleaning solvent, with a width of (101,6 ± 1) mm and a height of (76,2 ± 1) mm, incorporating a temperature sensor and electrical leads positioned in accordance with the dimensions given in figure 6. It shall have an operating range of -100 °C to 150 °C and shall provide evenly distributed heating sufficient to give accurate control of the cooling rate of the sheath to (6 ± 0,5) °C/h. It shall be attached to the inner cylinder in accordance with the manufacturers instructions (for example, by means of a shrink-fit band).

6.3.3 Copper cooling block, conforming to the dimensions given in figure 7.

6.3.4 Thermal insulation tape, self-adhesive and manufactured from closed-cell elastomeric material having a water vapour permeability not greater than 1,0 mg/(Nh) and a thermal conductivity not greater than 0,041 W/m °C at 20 °C.

6.3.5 Securing pieces, nylon, conforming to the shape and dimensions given in figures 8 and 9.

6.3.6 Heat transfer paste, with a thermal conductivity of 0,9 W/(m x K) and an operating temperature range from -100 °C to not less than 150 °C. The paste is applied only to the inner and outer surfaces of the cooling block.

6.3.7 Outer sleeve, metal, thermal conductivity at 0 °C of at least 90 W/(m x K), with an outside diameter of (44,5 + 0,5) mm as shown in figure 10. The assembly is sealed with silicone sealant resistant to water, the test sample and the cleaning solvent.

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[2] Model HSK 9028 by Minco Products Inc., Minneapolis, Ma, USA, is an example of a product available commercially. This information is given for the convenience of users of this CEN Report and does not constitute an endorsement by CEN of this product.

6.4 Synthetic foam sealing ring for the sheath (6.3).

6.5 Control unit, capable of performing the functions described in 6.5.1 to 6.5.3.

6.5.1 Providing the following temperature profile:

a) a set point at the start of 25 °C;

b) step down to a temperature at least 10 °C above the approximate cloud point of the test sample;

c) setting a smooth and continuous cooling rate of 6 °C/h after 6 min ± 30 s of operation.

6.5.2 Application of vacuum at each 1 °C below the first test temperature, ensuring a smooth and continuous increase in vacuum at the filter unit (6.7.3), following a profile falling within the limits shown in table 1.

Table 1 - Vacuum profile

Time from start of suction (s)	Vacuum (k Pa)
10 - 11	9
24 - 28	13,5
60	14,5 to 16

6.5.3 Recording the time taken for the pipette to empty after the vacuum is switched off and stopping the test if this time exceeds 40 s.

6.6 Test jar, cylindrical, of clear glass, flat bottomed, with internal diameter (31,5 ± 0,5) mm, external diameter (34,1 ± 0,1) mm and height (120 ± 5) mm.

6.7 Pipette with filter unit

6.7.1 Pipette, clear glass with a calibration mark corresponding to a contained volume of (5 ± 0,2) ml at a point (150 ± 10) mm from the bottom of the pipette and conforming to the shape and dimensions shown in figure 11.

6.7.2 Flexible tubing, resistant to both the test sample and the cleaning solvent, to connect the pipette (6.7.1) to the control unit (6.5).

6.7.3 Filter unit, as shown in figure 12, containing the elements described in 6.7.3.1 to 6.7.3.4 and having the following flow characteristics when tested at a temperature of between 22 °C and 25 °C using a diesel fuel with a viscosity of (3,8 ± 0,4) $mm^2 s^{-1}$ at 20 °C, with the filter unit immersed in a reservoir of the fuel to a depth of (68 ± 2) mm:

a) 20 ml, held in a pipette connected to the filter unit such that the liquid level is (203 ± 5) mm above the base of the brass body, shall discharge in (18 ± 3) s;

b) with a solid metal disc in place of the filter holder 20 ml shall discharge from the unit in (25 ± 3) s;

c) with a solid metal disc in place of the filter holder, and with vacuum applied to the pipette as given in 6.5.2, leakage through the vent pipe and the ball valve shall be less than 2 ml in 2 min.

6.7.3.1 Brass body, with connecting tube and a threaded cavity housing a wire mesh holder and a one-way ball valve. The ball valve leads to a vent pipe, the internal diameter of which is (2,62 ± 0,04) mm. The cavity is fitted with an O-ring which is resistant to both the test sample and the cleaning solvent. The internal diameter of the connecting tube is (2 ± 0,1) mm.

6.7.3.2 Brass screw cap, to connect the upper part of the body of the filter unit (6.7.3) to the lower part of the pipette (6.7.1) to ensure a leak-free joint.

6.7.3.3. Disc, 10 mm diameter, of plain square weave stainless steel wire mesh gauze with a nominal aperture size of 25 µm. The nominal diameter of the wire is 25 mm and the tolerance for the size of an individual aperture shall be as follows:

a) no aperture size shall exceed the nominal size by more than 60 %;

b) the average aperture size shall be within ± 10 % of the nominal size;

c) not more than 6 % of the apertures shall exceed the nominal size by more than 40 %.

6.7.3.4 Threaded filter holder, brass, into which the disc of wire mesh gauze (6.7.3.3) is firmly clamped by means of a retaining ring pressed into the filter holder. The diameter of the exposed part of the gauze is (7 ± 0,1) mm. The filter holder screws into the filter unit body, sealing against the O-ring.

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 sample

If the storage history of the sample is unknown, or if it has been subjected to a temperature at or below its approximate cloud point, condition between 100 ml and 150 ml of the laboratory sample for 1 h at between 45 °C and 50 °C and allow the conditioned sample to cool to ambient laboratory temperature or to at least 10 °C above its approximate cloud point, if the laboratory temperature is below this.

Filter a test portion of at least 60 ml of the sample, at a temperature of at least 10 °C above its approximate cloud point, through dry filter paper (5.2) and store the filtered sample at a temperature of at least 10 °C above its approximate cloud point.

9. Preparation of apparatus

Prepare the test unit (6.1) in accordance with the manufacturer's instructions.

Clean the filter unit (6.7.3) and the pipette (6.7.1) by immersing the filter unit in the cleaning solvent and, using vacuum, flush the filter unit and pipette at least 3 times. Allow the filter unit and pipette to drain and, using compressed air (5.3), blow completely dry.

10. Procedure

10.1 Fit the foam sealing ring (6.4) around the sheath (6.3). Align the sheath, such that the connecting wires of the heating membrane and temperature sensor (6.3.2) protrude directly opposite to the position of the PRT in the arrangement, as shown in figure 1.

10.2 Check that the tip of the PRT (6.1.4) is 35 mm above the filter mesh by first lifting an empty fuel jar onto the sheath. Raise the whole unit carefully so as to retain the relative position of the PRT to the filter/pipette unit. Measure the distance between the PRT tip and the filter mesh and adjust, if necessary, by sliding the PRT up or down in the plastic clamp (6.2).

10.3 Turn on the main switch of the SFPP control unit (6.5). Set the parameters on the control unit to follow the cooling profile given in 6.5.1.

10.4 Set the test unit (6.1) to start testing at least 10 °C above the approximate cloud point of the fuel.

10.5 Pour 57 ml of the prepared test portion (clause 8) into the test jar (6.6).

10.6 Lift the test jar and contents carefully onto the filter unit (6.7.3), lower both into the sheath (6.3). Ensure that the filter unit rests on the bottom of the test jar and that the PRT clamp (6.2) is resting on the light sensor assembly.

10.7 Flush the pipette at least three times with the test portion.

10.8 Start the test unit (6.1) and the SFPP control unit (6.5).

NOTE: At the temperature indicated in 10.4, the apparatus will apply vacuum to the test portion. If the filter is not filled to the 5 ml mark within 60 s, or does not empty within 40 s, the apparatus will stop the test. Otherwise, cooling of the test portion will continue and vacuum will be applied at 1 °C intervals until one or other of these failure conditions occurs.

10.9 Record the failure temperature at which the pipette either has not been filled within 60 s, or has not been emptied within 40 s.

10.10 Stop the program on the control unit (6.5) and reset to the 25 °C set point. Wait at least 5 min, apply vacuum to flush the test portion into the pipette one or more times, raise the filter assembly from the test jar and allow the assembly to drain. Clean the filter unit and pipette in accordance with 9.2.

11. Expression of results

Report the simulated filter plugging point (SFPP) of the sample as 1 °C above the failure temperature (see 10.9).

NOTE: The temperature recorded in 10.9 is likely to be negative and care should be taken to ensure that the reported result is 1 °C warmer than the recorded failure temperature.

12. Precision

12.1 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 1 °C only in one case in twenty.

12.2 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 3 °C only in one case in twenty.

13 Test report

The test report shall contain at least the following information:

a) the type and identification of the product under test;

b) a reference to this CEN Report;

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

d) the result of the test (see clause 11);

e) any deviation from the procedure described;

f) the date of the test.

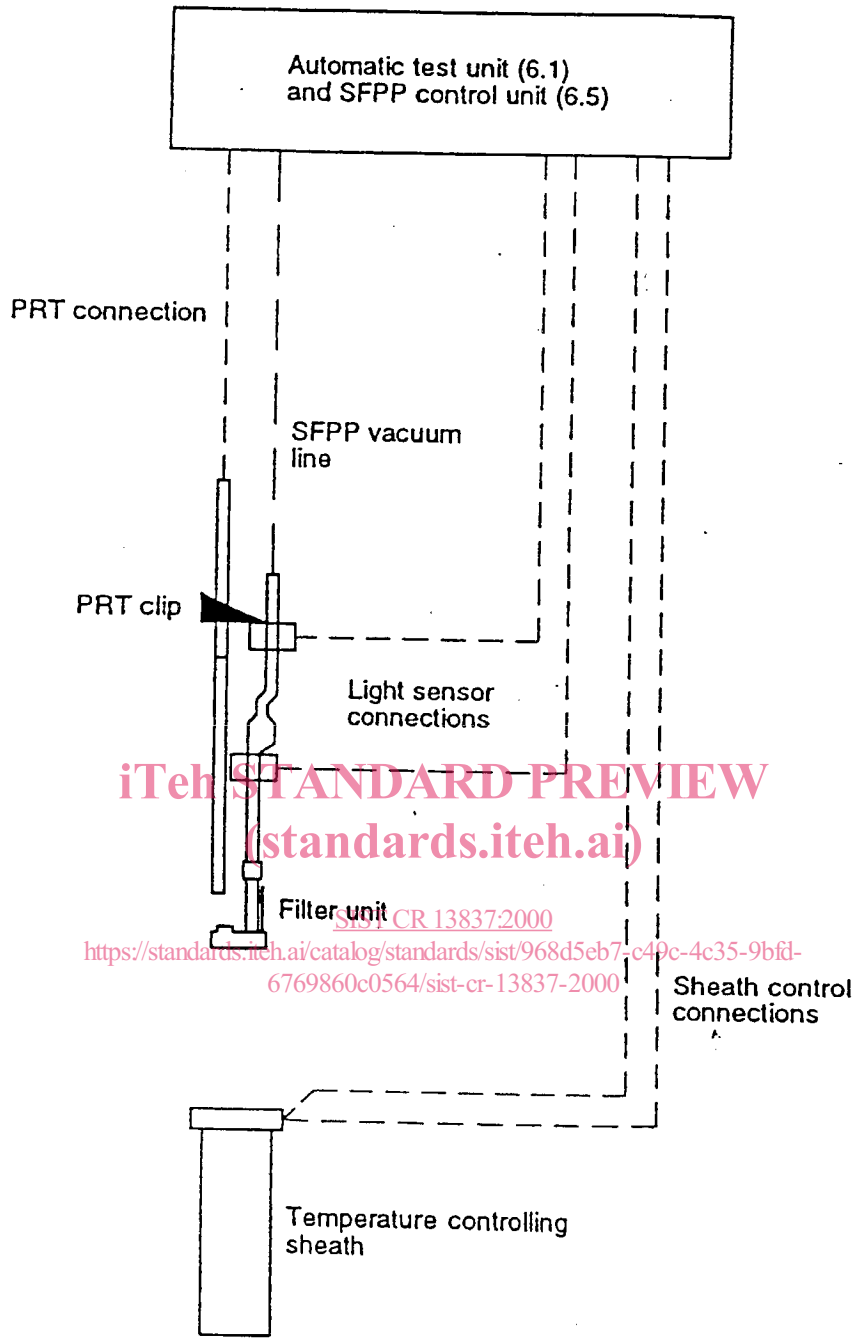

Figure 1 — General arrangement of the automatic test unit