Standard Test Method for Cold Filter Plugging Point of Diesel and Heating Fuels

This standard is issued under the fixed designation D 6371; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the cold filter plugging point (CFPP) temperature of diesel and domestic heating fuels using either manual or automated apparatus.

NOTE 1—This test method is technically equivalent to test methods IP 309 and EN 116.

1.2 The manual apparatus and automated apparatus are both suitable for referee purposes.

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

1.4 This standard does not purport to address all of the safety concerns, if any, 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. For specific hazard statements, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:
D 2500 Test Method for Cloud Point of Petroleum Oils
D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D 4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D 5771 Test Method for Cloud Point of Petroleum Products (Optical Detection Stepped Cooling Method)
D 5772 Test Method for Cloud Point of Petroleum Products (Linear Cooling Rate Method)
D 5773 Test Method for Cloud Point of Petroleum Products (Constant Cooling Rate Method)
E 1 Specification for ASTM Thermometers

2.2 IP Standards:
IP 309 Diesel and domestic heating fuels - Determination of cold filter plugging point
Specifications for IP Standard Thermometers
IP 3310 Test sieves - Technical requirements and testing - Part 1: Metal cloth

2.3 ISO Standards:
ISO 3155 Diesel and heating fuels - Determination of cold filter plugging point

2.4 European Standards:
EN 116 Diesel and domestic heating fuels - Determination of cold filter plugging point

3. Terminology

3.1 Definitions of Terms Specific to This Standard:
3.1.1 cold filter plugging point, n—highest temperature, expressed in multiples of 1°C, at which a given volume of fuel fails to pass through a standardized filtration device in a specified time when cooled under the conditions prescribed in this test method.

3.1.2 certified reference material, n—a stable petroleum product with a method-specific nominal CFPP value established by a method-specific interlaboratory study following RR:D02-10079 guidelines or ISO Guides 34 and 35.

4. Summary of Test Method

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

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4.2 The indicated temperature at which the last filtration was commenced is recorded as the CFPP.

5. Significance and Use

5.1 The CFPP of a fuel is suitable for estimating the lowest temperature at which a fuel will give trouble-free flow in certain fuel systems.

5.2 In the case of diesel fuel used in European light duty trucks, 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 value in accordance with Test Method D 2500, D 5771, D 5772, or D 5773. Domestic heating installations are usually less critical and often operate satisfactorily at temperatures somewhat lower than those indicated by the test results.

5.3 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.

6. Apparatus

6.1 Manual Apparatus:

6.1.1 The apparatus, as detailed in 6.1.2-6.1.13, shall be arranged as shown in Fig. 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 ± 1 mL level.

NOTE 2—Test jars of the required dimensions may be obtained by selection from jars conforming to Test Method D 2500, which specifies a wider diameter tolerance.

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, outside diameter of 48 ± 0.25 mm, and a height of 115 ± 3 mm (see Fig. 2).

6.1.4 Insulating Ring, made from oil-resistant plastics or other suitable material, to be placed in the bottom of the jacket (see 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 - 0.0 mm.

6.1.5 Spacers (two), approximately 5-mm thick, made of oil-resistant plastics or other suitable material, to be placed as shown in Fig. 1 around 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 Fig. 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 (see 6.1.3) in a stable and upright position in the cooling bath and to provide a concentric location for the stopper (see 6.1.7). A design is shown in Fig. 4 for guidance, but this design 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 as shown in Fig. 5. It shall have three holes to accommodate the pipet (see 6.1.8) and the thermometer (see 6.1.9) and to allow venting of the system. If necessary, when using the high-range thermometer (see 6.1.9),

NOTE 1—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

NOTE 2—All dimensions are in millimetres, and the comma (,) is used as the decimal point.
the upper part of the stopper shall have an indentation to permit the thermometer (see 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 Pipet with Filter Unit:

6.1.8.1 A Pipet, 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 pipet (see Fig. 6). It shall be connected to the filter unit (see 6.1.8.2).

6.1.8.2 A Filter Unit (see Fig. 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 (see 6.1.8.2) to the lower part of the pipet (see 6.1.8.1) to ensure a leak-free joint. An example of satisfactory connection is shown in Fig. 7.

(c) A Disc, 15 ± 0.1-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 ± 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 1—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

FIG. 3 Spacers

FIG. 4 Supporting Ring

FIG. 5 Stopper with Holes for Thermometer, Pipet, and Vent

Note 1—All dimensions are in millimetres, and the comma (,) is used as the decimal point.

Note 3—The requirements for the wire mesh are taken from ISO 3310, to which reference may be made for methods for testing the gauze.
6.1.9 Thermometers, having ranges shown below and conforming to the requirements prescribed in Specification E 1 or Specifications for IP Standard Thermometers.

<table>
<thead>
<tr>
<th>Thermometer</th>
<th>Temperature Range</th>
<th>ASTM</th>
<th>IP</th>
</tr>
</thead>
<tbody>
<tr>
<td>High-range for CFPP down to</td>
<td>−38°C to +50°C</td>
<td>5C</td>
<td>1C</td>
</tr>
<tr>
<td>−30°C</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Low-range from CFPP below −30°C</td>
<td>−80°C to +20°C</td>
<td>6C</td>
<td>2C</td>
</tr>
<tr>
<td>Cooling bath</td>
<td>−80°C to +20°C</td>
<td>6C</td>
<td>2C</td>
</tr>
</tbody>
</table>

6.1.10 Cooling Bath:

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

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

6.1.11.3 The bath temperature shall be maintained at the required value and tolerance 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. Table 1 lists the bath temperature set-points required in the CFPP procedure. If only one bath is utilized, it must have the ability to change down to the next lower set-point temperature in a time period not exceeding 2 min 30 s.
TABLE 1 Cooling Bath Temperatures

<table>
<thead>
<tr>
<th>Expected CFPP</th>
<th>Required Cooling Bath Temperature(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Down to −20°C</td>
<td>−34 ± 0.5°C</td>
</tr>
<tr>
<td>Between −20°C and −35°C</td>
<td>−34 ± 0.5°C then −51 ± 1°C</td>
</tr>
<tr>
<td>Below −35°C</td>
<td>−34 ± 0.5°C then −51 ± 1°C then −67 ± 2°C</td>
</tr>
</tbody>
</table>

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

6.1.12 **Vacuum Source**, vacuum pump or water pump powerful enough to ensure an air flow rate in the vacuum regulator 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 of water (2 ± 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 a vacuum reservoir to ensure a constant depression. The arrangement is shown in Fig. 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 Apparatus**:

6.2.1 The automated apparatus shall include elements conforming to 6.1.1-6.1.8, platinum resistance thermometers, cooling bath(s), vacuum pump, and suitable electronic control and measurement devices.

6.2.2 **Cooling Bath**, a refrigeration unit capable of maintaining the cooling bath at the required temperature and also of automatically changing the bath temperature within 2 min 30 s at the appropriate stage (see 12.2.5).

6.2.3 **Vacuum Pump**, powerful enough to ensure an air flow rate in the vacuum regulator of a minimum of 15 ± 1 L/h, and to maintain a constant vacuum of 200 ± 1 mm (2 ± 0.05 kPa) for the duration of the test. For multi-position testers using the same vacuum pump, the flow rate shall be checked when several positions are operating simultaneously.

7. **Reagents and Materials**

7.1 **Heptane**, clean commercial or reagent grade.

**Note**: 4—**Warning**: Flammable. Harmful if inhaled.

7.2 **Acetone**, clean commercial or reagent grade.

**Note**: 5—**Warning**: Extremely flammable.

7.3 **Lintless Filter Paper**, (5 ± 1 µm retention).

7.4 **Certified Reference Materials**.

8. **Sampling**

8.1 Unless otherwise specified in the commodity specification, samples shall be taken as described in Practice D 4057 or D 4177 in accordance with the requirements of national standards or regulation for the sampling of the product under test, or both.

9. **Preparation of Test Specimen**

9.1 Filter approximately 50 mL of the sample (see 8.1) at laboratory ambient temperature, but in any case not at a temperature less than 15°C, through dry filter paper (see 7.3).

10. **Preparation of Apparatus**

10.1 Prepare the manual apparatus or the automated apparatus for operation in accordance with the manufacturer’s instructions for calibrating, checking, and operating the equipment. See Fig. 1 for manual apparatus.

10.2 Before each test, dismantle the filter unit (see 6.1.8.2) and wash the pieces and the test jar (see 6.1.2), the pipet (see 6.1.9), then rinse with acetone (see 7.2) and dry in a stream of filtered air. Check the cleanliness and dryness of all elements, including the jacket (see 6.1.3). Examine the wire mesh (see 6.1.8.2(c)) and the joints (see 6.1.8.2(a) and 6.1.8.2(b) for damage; if necessary renew them.

10.3 Check that the screw cap (see 6.1.8.2(b)) is tight enough to prevent leakage.

11. **Calibration and Standardization**

11.1 Adjust the automated CFPP apparatus (when used) in accordance with the manufacturer’s instructions.

11.2 Calibrate the temperature measuring device in accordance with the manufacturer’s instructions.

11.3 Periodically verify the correct functioning of manual and automated apparatus using a certified reference material or in-house secondary reference material, such as fuel of known CFPP value.

**Note** 6—It is preferable that verification be carried out at least two times a year, where possible, using certified reference materials. The apparatus should be checked more frequently (for example, weekly) using a secondary verification material.

11.4 When the CFPP values obtained using a verification material deviate by more than the test repeatability (see 15.2), or an unacceptable statistical quality control bias is observed, check the condition and operation of the apparatus to ensure conformity with the specification as stated in this test method. The manufacturer’s instruction manual should provide guidance on ensuring that the apparatus is correctly set up and calibrated.

12. **Procedure**

12.1 **Manual Apparatus**:

12.1.1 Establish the cooling bath temperature at −34 ± 0.5°C.

12.1.2 Place the insulating ring (see 6.1.4) on the bottom of the jacket (see 6.1.3). If spacers (see 6.1.5) are not mounted on the insulating ring (see 6.1.4), position them approximately 15 and 75 mm above the bottom of the test jar (see 6.1.2).

12.1.3 Pour the filtered specimen (see Section 9) into the clean and dry test jar to the mark (45 mL).

12.1.4 Close the test jar with the stopper (see 6.1.7) carrying the pipet with filter unit (see 6.1.8) and the appropriate thermometer (see 6.1.9). Use a low-range thermometer if the expected CFPP 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 (see 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 no part of the thermistor is not in contact with the side of the test jar or the filter body.

Note 7—The precise positioning of the thermometer in the test jar is a critical parameter of this test method. The position of the lower end of the thermometer above the bottom of the test jar can be indirectly measured by marking the stem of the thermometer flush with the stopper (see 6.1.7) when the lower end of the thermometer is just touching the bottom of the test jar, and then pulling the thermometer up such that the reference line is 1.5 ± 0.2 mm above the top of the stopper.

12.1.5 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 (see 6.1.10), which is maintained at the temperature of –34 ± 0.5°C.

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

12.1.7 With the stopcock (see 6.1.11) open to atmosphere, connect the pipet to the vacuum system (see 6.1.12 and 6.1.13) by means of flexible tubing attached to the stopcock (see Fig. 1). Switch on the vacuum source and regulate to ensure an air flow rate of 15 L/h in the vacuum regulator (see 6.1.13). Before starting a test, check that the U-tube manometer indicates a 200 ± 1 mm of water depression (2 ± 0.05 kPa).

12.1.8 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 specimen has cooled to a temperature not less than 5°C above its cloud point.

12.1.9 When the specimen temperature reaches a suitable integer value, turn the stopcock (see 6.1.11) so that the filter assembly is connected to the vacuum source, causing the specimen to be drawn through the wire mesh into the pipet; simultaneously start the stopwatch.

12.1.10 When the specimen reaches the mark on the pipet, stop the stopwatch and turn the stopcock to its initial position to vent the pipet and so allow the specimen to return to the test jar.

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

12.1.12 Repeat the operations (see 12.1.9 to 12.1.10) for each 1°C decrease of the specimen temperature until the temperature is reached at which the pipet is not filled to the 20 mL mark within 60 s. Record the temperature at which this last portion, starting at a higher temperature.

12.1.13 If the filter has not plugged when the temperature of the specimen reaches –20°C, continue the test by using a second cooling bath maintained at –51 ± 1°C, quickly transferring the test jar and filtration assembly to a new jacket placed on the second cooling bath. Alternatively, for single bath apparatus, adjust the refrigeration unit to –51 ± 1°C. The new temperature must be reached within 2 min 30 s of the adjustment. Repeat the operations 12.1.9 to 12.1.10 to each 1°C decrease of the specimen temperature.

12.1.14 If the filter has not plugged when the temperature of the specimen reaches –35°C, continue the test by using a third cooling bath maintained at –67 ± 2°C by quickly transferring the test jar and filtration assembly to a new jacket placed on the second cooling bath. Alternatively, for single bath apparatus, adjust the refrigeration unit to –67 ± 2°C. The new temperature must be reached within 2 min 30 s of the adjustment. Repeat the operations 12.1.9 to 12.1.10 at each 1°C decrease of the specimen temperature.

12.1.15 If the filter has not plugged when the temperature of the specimen reaches –51°C, discontinue the test (see Section 13).

12.1.16 If, after cooling in accordance with 12.1.12, 12.1.13, and 12.1.14, the specimen fills the pipet to the mark in less than 60 s, but does not flow back completely into the test jar when the pipet is vented to atmosphere through the stopcock (see 6.1.11) before the start of the next aspiration, record the temperature at the commencement of the filtration as the CFPP (see Section 13).

12.2 Automated Apparatus:

12.2.1 Check that the cooling bath is operating and has reached the temperature required as specified in the manufacturer’s instructions.

12.2.2 Pour the filtered specimen (see Section 9) into the clean and dry test jar to the 45 mL mark.

12.2.3 Close the test jar with the stopper (see 6.1.7) carrying the pipet with filter unit (see 6.1.8) and the platinum resistance thermometer. Adjust the apparatus in such a way that the bottom of the filter unit (see 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 no part of the thermometer is in contact with the side of the test jar or the filter body.

Note 9—The precise positioning of the thermometer in the test jar is a critical parameter of this test method. The position of the lower end of the thermometer above the bottom of the test jar can be indirectly measured by marking the stem of the thermometer flush with the stopper (see 6.1.7) when the lower end of the thermometer is just touching the bottom of the test jar, and then pulling the thermometer up such that the reference line is 1.5 ± 0.2 mm above the top of the stopper.

12.2.4 If necessary, reconnect the pipet to the vacuum system. Switch on the vacuum source and regulate to ensure an air flow rate of 15 L/h in the vacuum regulator. Check that the U-tube manometer (if used) indicates a 200 ± 1 mm depression (2 ± 0.05 kPa) or that the electronic vacuum regulator indicates a pressure of 2 ± 0.05 kPa.

12.2.5 Press the start button immediately after insertion of the test jar assembly. If the cloud point is known, aspiration of the specimen through the filter may be set to start when it has cooled to a temperature not less than 5°C above the cloud point. The apparatus will carry out the test procedure filtering the specimen at each 1°C decrease if temperature and measuring the filtering time. If the time to reach the 20 mL mark exceeds 60 s on the first filtration, the test is to be abandoned and repeated on a fresh specimen starting at a higher temperature. The apparatus will record the first temperature at which
the specimen fails to reach the 20 mL mark in less than 60 s or fails to flow back into the test jar when the vacuum is cut off as CFPP (see Section 13). The test will be discontinued if the specimen reaches −51°C without plugging (see Section 13). During the procedure, the apparatus will automatically change the cooling bath temperature as indicated below.

<table>
<thead>
<tr>
<th>Bath Temperature</th>
<th>Start of test</th>
<th>When (if) specimen reaches −20°C</th>
<th>When (if) specimen reaches −35°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>−34 ± 0.5°C</td>
<td>−51 ± 1°C</td>
<td>−67 ± 2°C</td>
</tr>
</tbody>
</table>

Note 10—A small minority of samples may exhibit anomalous aspiration behavior, which can be detected by examining the aspiration times recorded in the test printout for signs of an unexpected reduction in the time taken to fill the pipet, after which aspiration time again continues to increase progressively until the failure limit of 60 s is reached.

12.2.6 If the automated CFPP apparatus used does not incorporate a lower light sensor, it shall only be used if the test sequence is observed as in the manual procedure (see 12.1.16), so that any fuels not flowing back into the test jar as described are detected and reported accordingly.

13. Report

13.1 Report the temperature read or indicated at the beginning of the last filtration to the nearest 1°C (see 12.1.12, 12.1.16, and 12.2.5) as the CFPP.

13.2 If the specimen has reached −51°C without plugging (see 12.1.15 and 12.2.5) report as “Not plugged at −51°C.”

13.3 The report shall contain at least the following information:

13.3.1 The type and identification of the product under test;
13.3.2 A reference to this test method;
13.3.3 The sampling procedure used (see Section 8);
13.3.4 The result of the test (13.1 or 13.2);
13.3.5 Any deviation from the procedure described (see Note 8 and Note 10); and
13.3.6 the date of the test.

14. Precision and Bias

14.1 The precision of this procedure as determined by the statistical examination of the interlaboratory test results is as follows:

14.2 Repeatability—The difference between results obtained on the same day by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, with normal and correct operation of the test method, exceed 1.76°C only in one case in twenty.

14.3 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 indicated by the formula:

\[ 0.102 (25−X)°C \]

where: \( X \) is the average of the two results being compared, only in one case in twenty.

Note 11—The interlaboratory test program used to determine the precision of this test method was carried out in 1988 by the IP. The program involved 46 laboratories and 5 samples, ranging in CFPP values from 0°C to −33°C. Extrapolations to measurements more than a few degrees outside this range are unsupported by the data. The raw data from the 1988 program was re-analyzed in 1997 using the ASTM D2PP program. The report of the re-evaluation is available from ASTM Headquarters RR:D02-1452.

14.4 Bias—The procedure in this test method has no bias because the value of CFPP can be defined only in terms of a test method.

14.5 Relative Bias—The current interlaboratory tests confirm that there is no relative bias between the manual and automated apparatuses. Both apparatuses are suitable for reference purposes.

15. Keywords

15.1 automated cold filter plugging point; cold filter plugging point (CFPP); diesel; domestic heating fuels; filterability; manual cold filter plugging point