



Designation: **D6371—17** **D6371 – 17a**

## Standard Test Method for Cold Filter Plugging Point of Diesel and Heating Fuels<sup>1</sup>

This standard is issued under the fixed designation D6371; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

### 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney, and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—<http://www.epa.gov/mercury/faq.htm>—for additional information. Users should be aware that selling mercury and/or mercury containing products in your state or country may be prohibited by law.

1.6 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific warning statements, see Section 7.

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

- [D2500 Test Method for Cloud Point of Petroleum Products and Liquid Fuels](#)
- [D4057 Practice for Manual Sampling of Petroleum and Petroleum Products](#)
- [D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products](#)
- [D5771 Test Method for Cloud Point of Petroleum Products and Liquid Fuels \(Optical Detection Stepped Cooling Method\)](#)
- [D5772 Test Method for Cloud Point of Petroleum Products and Liquid Fuels \(Linear Cooling Rate Method\)](#)
- [D5773 Test Method for Cloud Point of Petroleum Products and Liquid Fuels \(Constant Cooling Rate Method\)](#)
- [D7962 Practice for Determination of Minimum Immersion Depth and Assessment of Temperature Sensor Measurement Drift](#)
- [E1 Specification for ASTM Liquid-in-Glass Thermometers](#)
- [E1137E644 Specification—Test Methods for Testing Industrial Platinum-Resistance Thermometers](#)
- [E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids](#)
- [E2877 Guide for Digital Contact Thermometers](#)

#### 2.2 IP Standards:<sup>3</sup>

- [IP 309 Diesel and domestic heating fuels—Determination of cold filter plugging point Specifications for IP Standard Thermometers](#)

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved May 1, 2017. Published June 1, 2017. Originally approved in 1999. Last previous edition approved in 2016 as D6371—16. DOI: 10.1520/D6371-17.10.1520/D6371-17A.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., <http://www.energyinst.org.uk>.

\*A Summary of Changes section appears at the end of this standard

2.3 *ISO Standards*:<sup>4</sup>

**IP 3310** Test sieves—Technical requirements and testing—Part 1: Metal cloth

2.4 *European Standards*:<sup>5</sup>

**EN 116** Diesel and domestic heating fuels—Determination of cold filter plugging point

### 3. Terminology

3.1 *Definitions*:

3.1.1 *digital contact thermometer (DCT), n*—an electronic device consisting of a digital display and associated temperature sensing probe.

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<sup>4</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

<sup>5</sup> Available from European Committee for Standardization (CEN), 36 rue de Stassart, B-1050, Brussels, Belgium, <http://www.cenorm.be>.

3.1.1.1 *Discussion*—

This device consists of a temperature sensor connected to a measuring instrument; this instrument measures the temperature-dependent quantity of the sensor, computes the temperature from the measured quantity, and provides a digital output. This digital output goes to a digital display and/or recording device that may be internal or external to the device. These devices are sometimes referred to as a “digital thermometer.”

3.1.1.2 *Discussion*—

PET is an acronym for portable electronic thermometers, a subset of digital contact thermometers (DCT).

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *certified reference material, n*—a stable petroleum product with a method-specific nominal CFPP value established by a method-specific interlaboratory study following RR:D02-1007<sup>6</sup> guidelines or ISO Guides 34 and 35.<sup>4</sup>

3.2.2 *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.

### 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.

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 **D2500**, **D5771**, **D5772**, or **D5773**. 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 mm ± 0.5 mm, a wall thickness of 1.25 mm ± 0.25 mm and a height of 120 mm ± 5 mm. The jar shall have a permanent mark at the 45 mL ± 1 mL level.

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<sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1007. Contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org).

NOTE 2—Test jars of the required dimensions may be obtained by selection from jars conforming to Test Method **D2500**, 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 mm ± 0.25 mm, outside diameter of 48 mm ± 0.25 mm, and a height of 115 mm ± 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 mm + 0.3 mm - 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 (see **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 **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 nonmetallic, nonabsorbent, 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**), the upper part of the stopper shall have an indentation to permit the thermometer (see **6.1.9**) 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 *Pipet*, of clear glass with a calibration mark corresponding to a contained volume of 20 mL ± 0.2 mL at a point 149 mm ± 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 *Filter Unit* (see **Fig. 7**), containing the following elements:

(1) *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 mm ± 0.1 mm.

(2) *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**.

(3) *Disc*, 15 mm ± 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:

No aperture size shall exceed the nominal size by more than 22 µm.

The average aperture size shall be within ± 3.1 µm of the nominal size.

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

(4) *Filter Holder of Brass*, in which the disc of wire mesh gauze (see **6.1.8.2** (3)) is firmly clamped by a retaining ring pressed into the filter holder. The diameter of the exposed part of the gauze shall be 12 mm + 0.1 mm - 0.0 mm (see **Fig. 8**).

(5) *Brass Cylinder*, threaded on the outside, that can be screwed into the cavity of the body (see **6.1.8.2** (1)) to clamp the filter holder (see **6.1.8.2** (4)) against the O-ring (**6.1.8.2** (1)), The lower end shall have four slots to allow the specimen to flow into the filter unit.

NOTE 3—The requirements for the wire mesh are taken from **IP 3310**, to which reference may be made for methods for testing the gauze.

6.1.9 *Temperature Measuring Device*—Either a liquid-in-glass thermometer as described in **6.1.9.1** or a digital contact thermometer (DCT) meeting the requirements described in **6.1.9.2**.

6.1.9.1 *Liquid-in-glass Thermometers*, having ranges shown below and conforming to the requirements prescribed in Specifications **E1** or **E2251**, or Specifications for IP Standard Thermometers.

Thermometer	Temperature Range	Thermometer Number	
		ASTM	IP
High-range for CFPP down to -30 °C	-38 °C to +50 °C	5C, S5C	1C
Low-range from CFPP below -30 °C	-80 °C to +20 °C	6C	2C
Cooling bath	-80 °C to +20 °C	6C	2C

### 6.1.9.2 Digital contact thermometer requirements:

Parameter	Requirement
BCT	Guide <a href="#">E2877</a> Class G or better
DCT	Guide <a href="#">E2877</a> Class F or better
Temperature Range	–80 °C to +50 °C
Nominal Temperature Range <sup>A</sup>	–38 °C to +50 °C for CFPP down to –30 °C –80 °C to +20 °C for CFPP below –30 °C –80 °C to +20 °C for cooling bath
Display Resolution	0.1 °C, minimum
Accuracy <sup>B</sup>	±500 mK (±0.5 °C) for combined probe and sensor
Sensor Type	Platinum Resistance Thermometer (PRT)
Sensor Sheath <sup>C</sup>	4.2 mm OD maximum
Sensor Sensor Length <sup>D</sup>	3 mm o.d. sheath with a sensing element less than 10 mm in length Less than 10 mm
Minimum Immersion Immersion Depth <sup>E</sup>	Less than 40 mm per Practice <a href="#">D7962</a> . Less than 40 mm per Practice <a href="#">D7962</a> .
Accuracy Measurement Drift <sup>E</sup>	±500 mK (±0.5 °C) for combined probe and sensor Less than 500 mK (0.5 °C) per year.
Response Time Response Time <sup>E</sup>	Less than or equal to 25 s as defined in Specification <a href="#">E1137</a> . Less than or equal to 4 s per Footnote <sup>F</sup>
Drift	Less than 500 mK (0.5 °C) per year.
Calibration Error	Less than 500 mK (0.5 °C) over the range of intended use.
Calibration Range Calibration Range	–80 °C or lower to +50 °C Consistent with temperature range of use
Calibration Data Calibration Data	4 data points evenly distributed over calibration range with data included in calibration report. Four data points evenly distributed over the calibration range that is consistent with the range of use. The calibration data is to be included in calibration report.
Calibration Report	From a calibration laboratory with demonstrated competency in temperature calibration which is traceable to a national calibration laboratory or metrology standards body.

<sup>A</sup>The nominal temperature range may be different than the values shown provided the calibration and accuracy criteria are met.

<sup>B</sup>Accuracy is the combined accuracy of the DCT unit which is the display and sensor.

<sup>C</sup>Sensor sheath is the tube that holds the sensing element. The value is the outside diameter of the sheath segment containing the sensor element.

<sup>D</sup>The physical length of the temperature sensing element.

<sup>E</sup>As determined by Practice [D7962](#) or an equivalent procedure.

<sup>F</sup>Response Time—The time for a DCT to respond to a step change in temperature. The response time is 63.2 % of the step change time as determined per Section 9 of Test Method [E644](#). The step change evaluation begins at 20 °C ± 5 °C air to 77 °C ± 5 °C with water circulating at 0.9 m/s ± 0.09 m/s past the sensor.

NOTE 4—A DCT display mounted on the end to the probe's sheath is likely not suitable due to temperature exposure of the electronics. Consult manufacturer for temperature limitations.

NOTE 5—When making measurements below –40 °C with a PRT, it may be necessary to use a 1000 ohm sensor in order to obtain accurate measurements.

6.1.9.3 The DCT calibration drift shall be checked at least annually by either measuring the ice point or against a reference thermometer in a constant temperature bath at the prescribed immersion depth to ensure compliance with [6.1.9.2](#). See Practice [D7962](#).

NOTE 6—When a DCT's calibration drifts in one direction over several calibration checks, it may be an indication of deterioration of the DCT.

#### 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.

6.1.10.3 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.

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 L/h  $\pm$  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 mm  $\pm$  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 mm  $\pm$  1 mm of water (2 kPa  $\pm$  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 L/h  $\pm$  1 L/h, and to maintain a constant vacuum of 200 mm  $\pm$  1 mm (2 kPa  $\pm$  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. (**Warning**—Flammable. Harmful if inhaled.)

7.2 *Acetone*, clean commercial or reagent grade. (**Warning**—Extremely flammable.)

7.3 *Filter Paper*, (approximately 4  $\mu$ m to 6  $\mu$ m 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 D4057 or D4177 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 6.1.8.1) and the thermometer (see 6.1.9 for manual apparatus and 6.2 for platinum resistance used in automated equipment) with heptane (see 7.1), 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(3)) and the joints (see 6.1.8.2(1) and 6.1.8.2(2) for damage; if necessary renew them.

10.3 Check that the screw cap (see 6.1.8.2(2)) 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 7—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 14.2), 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  $-34\text{ °C} \pm 0.5\text{ °C}$ .

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 mm 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  $-30\text{ °C}$ . 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(5)) rests on the bottom of the test jar, and position the thermometer so that its lower end is  $1.5\text{ mm} \pm 0.2\text{ mm}$  above the bottom of the test jar. Take care to ensure that no part of the thermometer is not in contact with the side of the test jar or the filter body.

NOTE 8—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  $1.5\text{ mm} \pm 0.2\text{ mm}$  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  $85\text{ mm} \pm 2\text{ mm}$  in the cooling bath (see 6.1.10), which is maintained at the temperature of  $-34\text{ °C} \pm 0.5\text{ °C}$ .

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  $200\text{ mm} \pm 1\text{ mm}$  of water depression ( $2\text{ kPa} \pm 0.05\text{ kPa}$ ).

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 of not less than  $5\text{ °C}$  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  $1\text{ °C}$  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 filtration was commenced as CFPP (see Section 13).

NOTE 9—A small minority of samples may exhibit anomalous aspiration behavior, which can be detected by examining the observed aspiration times. This behavior is marked by 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.

12.1.13 If the filter has not plugged when the temperature of the specimen reaches  $-20\text{ °C}$ , continue the test by using a second cooling bath maintained at  $-51\text{ °C} \pm 1\text{ °C}$ , 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  $-51\text{ °C} \pm 1\text{ °C}$ . 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  $1\text{ °C}$  decrease of the specimen temperature.

12.1.14 If the filter has not plugged when the temperature of the specimen reaches  $-35\text{ °C}$ , continue the test by using a third cooling bath maintained at  $-67\text{ °C} \pm 2\text{ °C}$  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  $-67\text{ °C} \pm 2\text{ °C}$ . 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  $1\text{ °C}$  decrease of the specimen temperature.

12.1.15 If the filter has not plugged when the temperature of the specimen reaches  $-51\text{ °C}$ , 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(5)) rests on the bottom of the test