



# Standard Test Method for Freezing Point of Aviation Fuels<sup>1</sup>

This standard is issued under the fixed designation D 2386; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

# 1. Scope\*

1.1 This test method covers the determination of the temperature below which solid hydrocarbon crystals may form in aviation turbine fuels and aviation gasoline.

NOTE 1—The interlaboratory program that generated the precisions for this test method did not include aviation gasoline.

1.2 The values stated in acceptable metric units are to be regarded as the standard.

1.3 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 warning statements, see 5.4, Section 6, and 7.2.

# 2. Referenced Documents

2.1 ASTM Standards:

- D 910 Specification for Aviation Gasolines<sup>2</sup>
- D 1655 Specification for Aviation Turbine Fuels<sup>2</sup>
- D 3117 Test Method for Wax Appearance Point of Distillate Fuels<sup>2</sup>
- E 1 Specification for ASTM Thermometers<sup>3</sup>
- E 77 Test Method for Inspection and Verification of Thermometers<sup>3</sup>
- 2.2 IP Standard:
- IP Standards for Petroleum and Its Products, Part 1<sup>4</sup>

#### 3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *freezing point, n—in aviation fuels,* the fuel temperature at which solid hydrocarbon crystals, formed on cooling, disappear when the temperature of the fuel is allowed to rise under specified conditions of test.

# 4. Significance and Use

4.1 The freezing point of an aviation fuel is the lowest temperature at which the fuel remains free of solid hydrocarbon crystals that can restrict the flow of fuel through filters if present in the fuel system of the aircraft. The temperature of the fuel in the aircraft tank normally falls during flight depending on aircraft speed, altitude, and flight duration. The freezing point of the fuel must always be lower than the minimum operational tank temperature.

4.2 Freezing point is a requirement in Specifications D 910 and D 1655.

# 5. Apparatus

5.1 Jacketed Sample Tube—A double-walled, unsilvered vessel, similar to a Dewar flask, the space between the inner and outer tube walls being filled at atmospheric pressure with dry nitrogen or air. The mouth of the sample tube shall be closed with a stopper supporting the thermometer and moisture-proof collar through which the stirrer passes (Fig. 1).

5.2 *Collars*—Moisture-proof collars as shown in Fig. 2 shall be used to prevent condensation of moisture.

5.3 *Stirrer*—Shall be made of 1.6-mm brass rod bent into a smooth three-loop spiral at the bottom.

NOTE 2—The stirrer may be mechanically actuated as described in the apparatus section of Test Method D 3117.

5.4 Vacuum Flask—An unsilvered vacuum flask (Warning—Implosion hazard) having the minimum dimensions shown in Fig. 1 shall be used to hold an adequate volume of cooling liquid and permit the necessary depth of immersion of the jacketed sample tube.

5.5 *Thermometer*—A total immersion type, having a range from -80 to +20°C, designated as ASTM No. 114C/IP No. 14C. (See Specification E 1, or Appendix A, IP Standard

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

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This test method has been approved by the sponsoring committees and accepted by the Cooperating Societies in accordance with established procedures.

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 05.01. <sup>3</sup> Annual Book of ASTM Standards, Vol 14.03.

<sup>&</sup>lt;sup>4</sup> A illi for Luit to C D to 1 (D)

 $<sup>^4</sup>$  Available from Institute of Petroleum (IP), 61 New Cavendish St., London, WIG 7AR, U.K.



FIG. 1 Freezing Point Apparatus

Thermometers, Volume 2, IP Standard Methods for Analysis and Testing of Petroleum and Related Products.)

Note 3—The accuracy of this thermometer is to be checked in accordance with Test Method E 77, at temperatures of 0, -40, -60, and  $-75^{\circ}C.^{5}$ 

### 6. Reagents and Materials

6.1 *Acetone*—Technical Grade acetone is suitable for the cooling bath, provided it does not leave a residue on drying. (**Warning**—Extremely flammable.)

6.2 *Ethanol or Ethyl Alcohol*—A commercial or technical grade of dry ethanol is suitable for the cooling bath. (Warning—Extremely flammable.)

6.3 *Isopropyl Alcohol*—A commercial or technical grade of dry isopropyl alcohol is suitable. (**Warning**—Extremely flammable.)

6.4 *Methanol or Methyl Alcohol*—A commercial or technical grade of dry methanol is suitable for the cooling bath. (Warning—Extremely flammable and toxic.)

6.5 *Carbon Dioxide (Solid) or Dry Ice*—A commercial grade of dry ice is suitable for use in the cooling bath. (See Note 4.) (**Warning**—Extremely cold, -78°C.)

6.6 *Liquid Nitrogen*—A commercial or technical grade of liquid nitrogen is suitable for the cooling bath when the freezing point is lower than -65°C. (**Warning**—Extremely cold, -196°C.)

NOTE 4— Carbon dioxide (solid) and liquid nitrogen liberate gasses that can cause suffocation. Contact with skin causes burns, freezing, or both.

#### 7. Procedure

7.1 Measure out  $25 \pm 1$  mL of the fuel and transfer it to the clean, dry, jacketed sample tube. Close the tube tightly with the cork holding the stirrer, thermometer, and moisture proof collar and adjust the thermometer position so that its bulb does not touch the walls of the tube flask and is approximately in the center. The bulb of the thermometer should be 10 to 15 mm from the bottom of the sample tube.

7.2 Clamp the jacketed sample tube so that it extends as far as possible into the vacuum flask (**Warning**—Implosion hazard) containing the cooling medium (Note 5). The surface of the sample should be approximately 15 to 20 mm below the level of the coolant. Unless the medium is cooled by mechanical refrigeration, add solid carbon dioxide as necessary throughout the test to maintain the coolant level in the vacuum flask.

NOTE 5—Acetone and either methyl, ethyl, or isopropyl alcohols are suitable. All of these require cautious handling. Liquid nitrogen may also be used as a coolant instead of liquids cooled with solid carbon dioxide for

<sup>&</sup>lt;sup>5</sup> The U.S. National Bureau of Standards, Gaithersburg, MD, and the British National Physical Laboratory, Teddington, England are able to certify thermometers at these temperatures.

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FIG. 2 Moistureproof Collars for Freezing Point Apparatus

fuel samples which have a freezing point below  $-65^{\circ}$ C. Mechanical refrigeration is permitted. Where used the refrigerant temperature should be  $-70^{\circ}$ C to  $80^{\circ}$ C.

7.3 Stir the fuel continuously, moving the stirrer up and down at the rate of 1 to 1.5 cycles/s, except when making observations, taking care that the stirrer loops approach the bottom of the flask on the downstroke and remain below the fuel surface on the upstroke (Note 6). Disregard any cloud that appears at approximately –  $10^{\circ}$ C and does not increase in intensity as the temperature is lowered, because this is due to water. Record the temperature at which hydrocarbon crystals appear. Remove the jacketed sample tube from the coolant and allow the sample to warm, stirring it continuously at 1 to 1.5 cycles/s. Record the temperature at which the hydrocarbon crystals completely disappear.

NOTE 6—Because the gases released by the coolant can obscure vision, the sample tube can be removed to observe the appearance of the wax crystals. The tube can be removed for periods no longer than 10 s. If crystals have already formed, the temperature should be noted and the sample allowed to be reheated to  $5^{\circ}$ C above the point where the crystals disappear. The sample should then be reimmersed and allowed to cool.

Remove the sample slightly above the noted temperature and observe the wax appearance point.

# 8. Report

8.1 The observed freezing point determined in Section 7 shall be corrected by applying the relevant thermometer correction resulting from the checks described in Note 3. Where the observed freezing point falls between two calibration temperatures, the correction at the observed temperature shall be obtained by linear interpolation. Report the corrected temperature of crystal disappearance to the nearest 0.5°C as the freezing point, Test Method D 2386.

NOTE 7—When results are desired in degrees Fahrenheit, test results obtained in degrees Celsius should be converted to the nearest whole degree Fahrenheit. Interim Celsius freezing points should carry the best precision available for subsequent conversion to degrees Fahrenheit.

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# 9. Precision and Bias<sup>6</sup>

9.1 *Precision*—The precision of this test method was obtained by the statistical examination of the results of 14 samples of fuel consisting of Jet A, Jet A1, Jet B, JP-4, and JP-5 tested by 16 laboratories.

9.1.1 *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 0.8°C only in one case in twenty.

9.1.2 *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 2.3°C only in one case in twenty.

9.2 *Bias*—Because there are no liquid hydrocarbon mixtures of "known" freezing point, which simulate aviation fuels, bias cannot be established.

# 10. Keywords

10.1 aviation gasoline; aviation turbine fuels; crystallization point; determination; freezing point; low temperature tests; manual method; petroleum products; physical tests

# SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D 2386–01) that may impact the use of this standard.

(1) Revised definition of freezing point in 3.1.1.

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<sup>&</sup>lt;sup>6</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1175.