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Designation: 16/15

Standard Test Method for Freezing Point of Aviation Fuels[1]

This standard is issued under the fixed designation D2386; 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.

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

ε^1 NOTE—A mercury caveat was removed from the scope editorially in July 2015.

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 SI units are to be regarded as standard. No other units of measurement are included in this 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 8.3.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D910 Specification for Leaded Aviation Gasolines
D1655 Specification for Aviation Turbine Fuels
D3117 Test Method for Wax Appearance Point of Distillate Fuels (Withdrawn 2010)[3]
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
E1 Specification for ASTM Liquid-in-Glass Thermometers
E77 Test Method for Inspection and Verification of Thermometers

2.2 *Energy Institute Standard:*

IP Standards for Petroleum and Its Products IP 16/15[4]

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.

3.1.2 *crystallization point, n*—the temperature at which crystals of hydrocarbons first appear when the test sample is cooled.

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 D910 and D1655.

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). A cork stopper is recommended.

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[1] 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.

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

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K., http://www.energyinst.org.

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

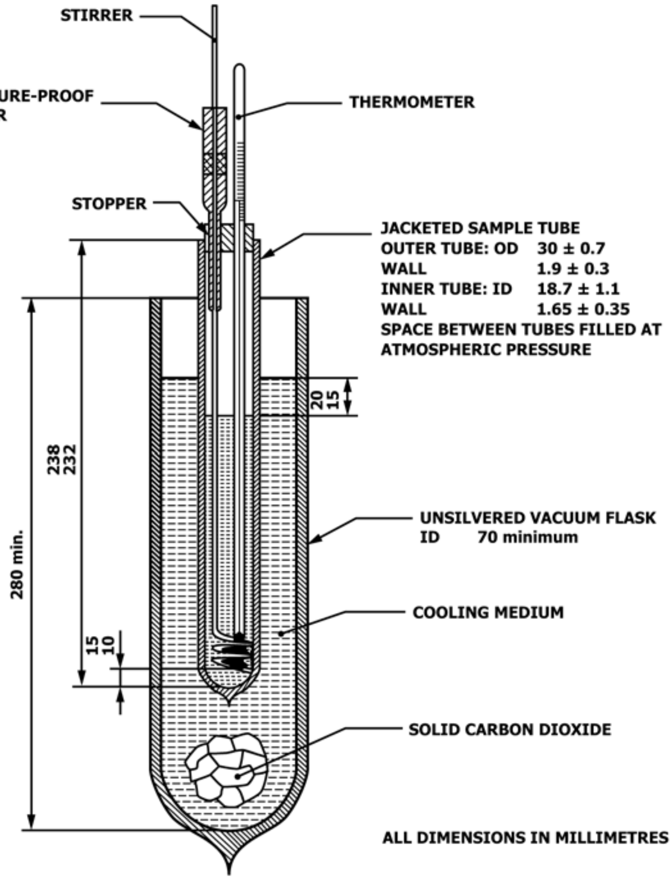

FIG. 1 Freezing Point Apparatus

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 or stainless steel 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 D3117.

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 °C to +20 °C, designated as ASTM No. 114C/IP No. 14C. (See Specification E1, or Appendix A, IP Standard 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 E77, at temperatures of 0 °C, −40 °C, −60 °C, and −75 °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. (**Warning**—Extremely cold, −78 °C. Carbon dioxide (solid) liberates gases that can cause suffocation. Contact with skin causes burns, freezing, or both.)

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. Liquid nitrogen liberates gases that can cause suffocation. Contact with skin causes burns, freezing, or both.)

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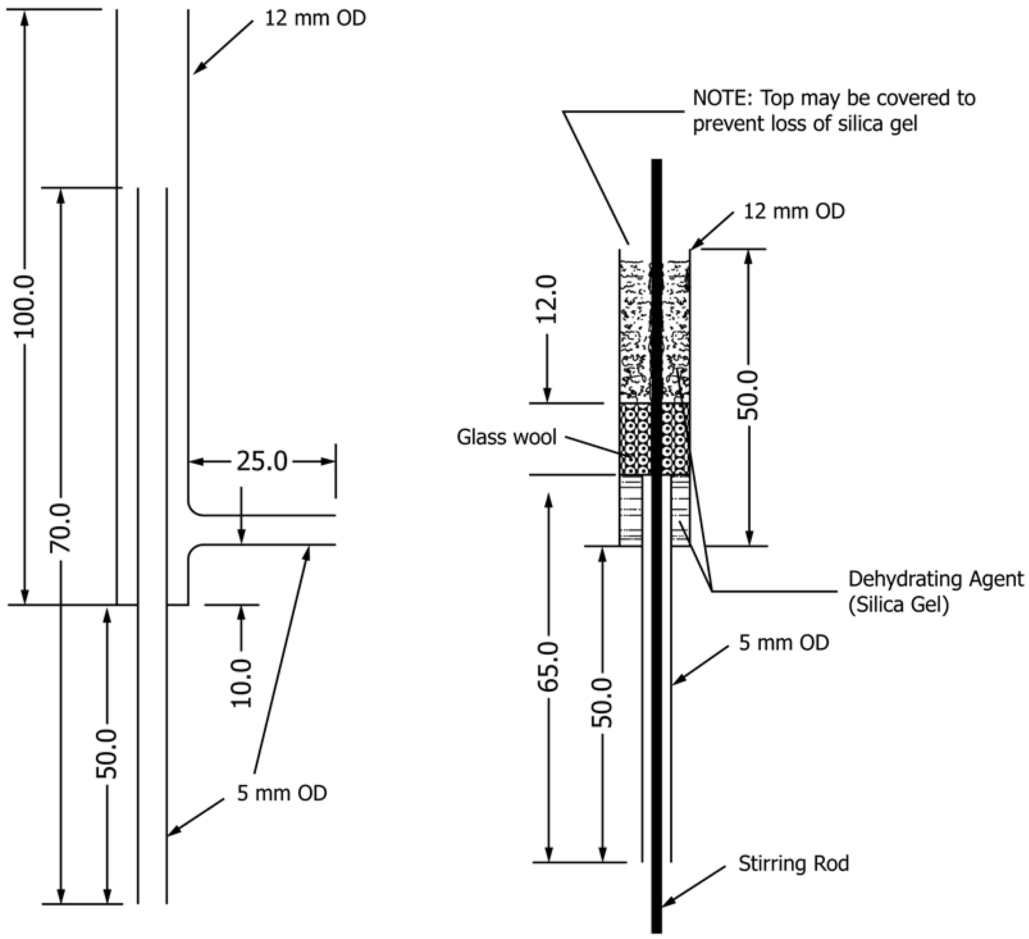
[5] The U.S. National Bureau of Standards, Gaithersburg, MD, and the British National Physical Laboratory, Teddington, England are able to certify thermometers at these temperatures.



Type A
Nitrogen Collar

Type B Modified
Moistureproof
Collar

NOTE 1—All dimensions are in mm and ±0.1 mm glass wall thickness is 1 mm.

FIG. 2 Moistureproof Collars for Freezing Point Apparatus

6.7 *Fiberglass*—commercial grade, for use in moisture-proof collar B.

6.8 *Dehydrating Agent*—Use one of the following:

6.8.1 Calcium sulfate ($CaS0_4$), granulated anhydrous calcium sulfate, for use as a desiccant in moisture-proof collar B, or to assist in drying the nitrogen gas or air (5.2) used with collar A.

6.8.2 Silica gel, 1.7 mm, for use as a desiccant in moisture-proof collar B, or to assist in drying the nitrogen gas or air (5.2) used with collar A. (**Warning—**Silica gel dyed with cadmium salts can cause cancer by inhalation.)

7. Sampling

7.1 Obtain a sample in accordance with Practice D4057 or Practice D4177.

7.2 At least 25 mL of specimen is required for each test.

7.3 Keep the sample in a tightly sealed container at ambient room temperature to minimize introduction of any moisture. Minimize exposure of the sample to sources of heat.

8. Procedure

8.1 *Collar*—Assemble the collar (5.2), thermometer (5.5) and stirrer (5.3) into the cork. To prevent the ingress of water into the test portion, it is important that an effective moisture-proof collar as prepared in accordance with 8.1.1 or 8.1.2 is used.

8.1.1 Collar type A, flush with nitrogen or dry air before fitting to the jacketed sample tube, and throughout the entire determination.

NOTE 4—The air can be effectively dried by passing through absorbent tubes filled with dehydrating agents (6.8.1 and 6.8.2).

8.1.2 Collar type B, fill with fiberglass (6.7) and a suitable dehydrating agent (6.8.1 and 6.8.2) as shown in Fig. 2. The fiberglass shall be replaced every fourth test. The dehydrating agent should be renewed at intervals of not more than 3 months or when a color change shows it to be ineffective

8.2 Measure out 25 mL ± 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 mm to 15 mm from the bottom of the sample tube. (**Warning—**Do not add solid carbon dioxide to liquid nitrogen.)

NOTE 5—Performance of this test method can be difficult, since the specimen tube is immersed in a coolant medium that evolves gas bubbles during the test. This can interfere with visual observations. In addition, the crystals that are formed in the specimen can be difficult to recognize, and they can appear in a variety of manifestations. It is strongly suggested that operators seek guidance from experienced operators of this test method to assist them in the correct recognition of these crystals.

NOTE 6—This test method should be performed under laboratory conditions where there is an ample supply of light. Some crystals can be very faint in appearance and difficult to observe under inadequate lighting conditions.

8.3 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 7). The surface of the sample should be approximately 15 mm 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 7—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 fuel samples which have a freezing point below −65 °C. Mechanical refrigeration is permitted. Where used the refrigerant temperature should be −70 °C to 80 °C.

8.4 Stir the fuel continuously, moving the stirrer up and down at the rate of 1 cycles/s to 1.5 cycles/s, taking care that the stirrer loops approach the bottom of the flask on the downstroke and remain below the specimen surface on the upstroke. It is permissible for momentary interruption of stirring while performing some operations of the procedure (see Note 8). Observe the specimen continuously for the appearance of hydrocarbon crystals. Disregard any cloud that appears at approximately −10 °C and does not increase in intensity as the temperature decreases, because this cloud is due to water. Record the temperature at which hydrocarbon crystals appear. Remove the jacketed sample tube from the coolant and allow the specimen to warm by ambient air, stirring it continuously at 1 cycles/s to 1.5 cycles/s. Continue to observe the specimen continuously for the disappearance of hydrocarbon crystals. Record the temperature at which the hydrocarbon crystals completely disappear.

NOTE 8—Because the gases released by the coolant can obscure observations, the sample tube can be removed from the coolant for observations. The tube can be removed for periods no longer than 10 s. If crystals are observed to have already formed, the specimen temperature should be noted and the specimen allowed to be warmed by ambient air, with continued stirring, to at least 5 °C above the temperature at which the crystals disappear. The specimen should then be re-immersed in the coolant and allowed to cool. Remove the specimen from the coolant slightly above the noted temperature, and observe for appearance of the crystals.

NOTE 9—It is recommended to compare the crystal appearance temperature with the crystal disappearance temperature. The appearance temperature should be colder than the disappearance temperature. If this is not the case, this is an indication that the crystals were not correctly recognized. Also, the difference between these temperatures should typically be no greater than 6 °C.

9. Report

9.1 The observed freezing point determined in Section 8 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 D2386.

NOTE 10—False results can be obtained if the temperature of the test portion is not uniform during the cooling and warming cycles. To obtain a uniform temperature, the test portion must be stirred strictly in accordance with the procedure described.

NOTE 11—Contamination by other petroleum products can cause crystals to appear at much higher temperatures than normally expected for aviation fuel freeze points. In order to identify such contamination, it is important that observations are made throughout the test, both in the cooling and warming cycles.

NOTE 12—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.

10. Precision and Bias[6]

10.1 *Precision*—The precision of this test method was obtained by the statistical examination of the results of 13 samples of fuel consisting of Jet A, Jet A1, JP-5 and JP-8 tested by 15 laboratories.

10.1.1 *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 1.5 °C only in one case in twenty.

10.1.2 *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 2.5 °C only in one case in twenty.

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

11. Keywords

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

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1572.

SUMMARY OF CHANGES

Subcommittee D02.07 has identified the location of selected changes to this standard since the last issue (D2386 – 06 (2012)) that may impact the use of this standard. (Approved June 1, 2015.)

(1) Added new subsections 6.7, 6.8, and 8.1.
(2) Added new Note 4, Note 10, and Note 11.
(3) Added new term 3.1.2.
(4) Revised subsections 2.2, 5.1, 5.3, and 8.2.

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