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An American National Standard

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Standard Test Method for Freezing Point of Aviation Fuels (Automatic Phase Transition Method)[1]

This standard is issued under the fixed designation D 5972; 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.

ε[1] NOTE—Note 1 was updated and Note 2 was included editorially in November 2001.

1. Scope

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

1.2 This test method is designed to cover the temperature range of −80 to 20°C; however, the ASTM interlaboratory study mentioned in 12.4 has only demonstrated the test method with fuels having freezing points in the range of −45 to −65°C.

1.3 The user shall exercise appropriate caution when this test method is used in testing Jet B and JP 4 samples (see 12.3).

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *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 7.1, 7.3, and 7.5.

2. Referenced Documents

2.1 *ASTM Standards:*

D 2386 Test Method for Freezing Point of Aviation Fuels[2]

3. Terminology

3.1 *Definitions:*

3.2 *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.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *automatic phase transition method*, *n—in this test method*, the procedures of automatically cooling a liquid aviation fuel specimen until solid hydrocarbon crystals appear, followed by controlled warming and recording of the temperature at which the solid hydrocarbon crystals completely redissolve into the liquid phase.

3.3.2 *Peltier device*, *n*—a solid-state thermoelectric device constructed with dissimilar semiconductor materials, configured in such a way that it will transfer heat to and away from a test specimen dependent on the direction of electric current applied to the device.

4. Summary of Test Method

4.1 A specimen is cooled at a rate of 15 ± 5°C/min by a Peltier device while continuously being illuminated by a light source. The specimen is continuously monitored by an array of optical detectors for the first formation of solid hydrocarbon crystals. Once the hydrocarbon crystals are formed, the specimen is then warmed at a rate of 10 + 0.5°C/min until the last hydrocarbon crystals return to the liquid phase. The detectors are sufficient in number to ensure that any solid hydrocarbon crystals are detected. The specimen temperature at which the last hydrocarbon crystals return to the liquid phase is recorded as the freezing point.

5. Significance and Use

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

5.2 Petroleum blending operations require precise measurement of the freezing point.

5.3 This test method produces results which have been found to be equivalent to Test Method D 2386 and expresses results to the nearest 0.1°C, with improved precision over Test

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07.0D on Wax-Related Viscometric Properties of Fuels and Oils.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

Method D 2386. This test method also eliminates most of the operator time and judgment required by Test Method D 2386.

5.4 When specification requires Test Method D 2386, do not substitute this test method or any other test method.

6. Apparatus

6.1 *Automatic Apparatus*[3]—This apparatus consists of a microprocessor-controlled test chamber that is capable of cooling and heating the test specimen, optically observing the appearance and disappearance of solid hydrocarbon crystals, and recording the temperature of the specimen. A detailed description of the apparatus is provided in Annex A1.

6.2 The apparatus shall be equipped with a specimen cup, optical detector array, light source, digital display, Peltier device, and a specimen temperature measuring device.

6.3 The temperature measuring device in the specimen cup shall be capable of measuring the temperature of the test specimen from −80 to +20°C at a resolution of 0.1°C and accuracy of 0.1°C.

6.4 The apparatus shall be equipped with fittings to permit the circulation of a liquid medium to remove heat generated by the Peltier device and other electronic components of the apparatus.

6.5 The apparatus shall be equipped with fittings to permit the circulation of purge gas to purge the test chamber containing the specimen cup of any atmospheric moisture.

7. Reagents and Materials

7.1 *n-Octane*—Reagent grade is suitable. (**Warning**—Flammable. Harmful if inhaled. Keep away from heat, sparks, and open flame.)

7.2 *Cooling Medium*—Liquid heat exchange medium to remove the heat generated by the Peltier device and other electronic components from the apparatus.

NOTE 1—Some apparatus are designed to use tap water as a cooling medium to bring the specimen temperature to −60°C. To achieve cooling of the specimen to −80°C, provide circulation of the cooling medium at −30°C or lower to the apparatus. Since water freezes at 0°C, a commercial or technical grade isopropanol is suitable as the cooling medium. Refer to the manufacturer's operating instructions on the relationship between the cooling medium temperature and the minimum specimen temperature.

7.3 *Purge Gas*—A gas such as air, nitrogen, helium, or argon with a dew point below the lowest temperature attained by the specimen under the conditions of the test. (**Warning**—Compressed gas under high pressure.) (**Warning**—Inert gas can be an asphyxiant when inhaled.

7.4 *Pipette*, capable of dispensing 0.15 ± 0.01 mL of sample.

7.5 *Cotton Swabs*—Plastic- or paper-shaft cotton swabs to clean the specimen cup. (**Warning**—The use of swabs with wooden shafts may damage the mirrored surface of the specimen cup.)

[3] Phase Technology Freezing Point Analyzer Model Series 30, 50, and 70, available from Phase Technology, No. 135-11960 Hammersmith Way, Richmond, B.C. Canada, V7A 5C9, has been found suitable for use in this test method. All the model series previously mentioned have identical test chambers and electronics. The distinction between different model series is the low temperature limit. Refer to manufacturer's product information on the low-temperature limit of various models.

8. Preparation of Apparatus

8.1 Install the analyzer for operation in accordance with the manufacturer's instructions.

8.2 Turn on the liquid cooling medium and ensure its temperature is appropriate for the specimen being tested in accordance with the manufacturer's instructions (see Note 1).

8.3 Turn on the purge gas and ensure that it is regulated to the appropriate pressure in accordance with the manufacturer's instructions.

8.4 Turn on the main power switch of the analyzer.

NOTE 2—Some apparatus are capable of generating a source of dry purge gas, thus eliminating the need for an external supply of a compressed gas.

9. Calibration and Standardization

9.1 Ensure that all of the manufacturer's instructions for calibrating, checking, and operating the apparatus are followed.

9.2 An aviation turbine fuel sample which has been extensively tested in a Test Method D 2386 interlaboratory study can be used to verify performance of the apparatus within the precision of the test method.

9.3 Reagent grade *n*-octane with a known freezing point value can be used to verify the calibration of the specimen temperature measuring device within the precision of this test method. The literature value[4] for the freezing point of pure *n*-octane is −56.8°C.

10. Procedure

10.1 Open the test chamber lid and clean the specimen cup inside the test chamber with a cotton swab.

10.2 Rinse the specimen cup by pipetting 0.15 ± 0.01 mL of specimen into the cup. Clean the specimen out of the cup by using a cotton swab. The cup should be cleaned to the point where no visible droplets of specimen remain in the cup.

10.3 Rinse the cup a second time by repeating 10.2.

10.4 Carefully measure 0.15 ± 0.01 mL of specimen into the specimen cup.

10.5 Close and lock the test chamber lid.

10.6 Start the operation of the apparatus according to the manufacturer's instructions. From this point up to and including the termination of the measurement, the apparatus automatically controls all operations. Purge gas and liquid cooling medium will begin to flow through the apparatus. The Peltier device cools the specimen at a rate of 15 ± 5°C/min. The optical detectors continuously monitor the specimen for the formation of hydrocarbon crystals. The temperature of the specimen is continuously monitored by the apparatus and displayed on its front panel. Once hydrocarbon crystals are detected, the specimen is then warmed at 10 ± 0.5°C/min until all the crystals redissolve into the liquid phase. When the disappearance of the last crystals is detected, the specimen temperature is recorded and the measurement is terminated.

10.7 The freezing point value will be displayed by the apparatus.

[4] CRC Handbook of Chemistry and Physics, 64th Edition, CRC Press, Inc., Boca Raton, FL, 1984. p C-404.