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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.

¹ NOTE—Footnote 3 was updated editorially in July 2005.

—The IP logo and affiliation were removed editorially in August 2008.

1. Scope 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~~

1.2 This test method is designed to cover the temperature range of -80 to 20°C; however, 2003 Joint ASTM / IP Interlaboratory Cooperative Test Program 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 has only demonstrated the test method with fuels having freezing points in the range of -42 to -60°C.~~

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

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 For specific warning statements, see 7.1, 7.3, and 7.5.

2. Referenced Documents

2.1 ASTM Standards:²

D 2386 Test Method for Freezing Point of Aviation Fuels

3. Terminology

3.1 Definitions:

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.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *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.

3.2.2 *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.

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 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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³ 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 Vol 05.01, volume information, refer to the standard's Document Summary page on the ASTM website.

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



4. Summary of Test Method

4.1 A specimen is cooled at a rate of $15 \pm 5^\circ\text{C}/\text{min}$ 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 $10 + 0.5^\circ\text{C}/\text{min}$ 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 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 $+20^\circ\text{C}$ 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.)

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.

³ The sole source of supply of the Phase Technology Freezing Point Analyzer Model Series 70, 70V, and 70X known to the committee at this time is Phase Technology, No. 135-11960 Hammersmith Way, Richmond, B.C. Canada, V7A 5C9. 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. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

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 To verify the performance of the apparatus, an aviation turbine fuel sample for which extensive data has been obtained by freeze point, Test Method D 2386, may be used. Samples such as those used in the ASTM interlaboratory cross-check program would meet this criterion. Such verification materials can also be prepared from intracompany cross-checks. Alternatively, high-purity n-octane or n-nonane with known freezing points can be used to verify the calibration of the temperature-measuring device in the apparatus.

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 $15 \pm 5^\circ\text{C}/\text{min}$. 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 $10 \pm 0.5^\circ\text{C}/\text{min}$ 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.

10.8 Unlock and open the test chamber lid and clean the specimen out of the specimen cup with a cotton swab.

11. Report

11.1 Report the temperature recorded in 10.7 as the freezing point, Test Method D 5972.

12. Precision and Bias

12.1 *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 this test method, exceed 0.69°C to 0.54°C only in one case in 20 twenty.

12.1.1 *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 this test method, exceed 1.30°C to 0.80°C only in one case in 20 twenty.

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

12.3 *Relative Bias*—The results for all the samples from the interlaboratory program⁴ were examined for biases relative to Test Method D 2386. Some differences were observed for some fuels, but there was no consistent bias among all the fuels. All the differences were within the reproducibility of Test Method D2386 (2.5°C) except for two. These two were the only samples of fuels of Grades JP4 and Jet B, and for these the average results were 2.5°C and 2.8°C warmer than those of Test Method D2386. Therefore, the user should exercise appropriate caution when this test method is used in testing Jet B and JP4 samples. There was no lab bias demonstrated in the 2003 Joint ASTM / IP Interlaboratory Cooperative Test Program.⁵

12.4 The precision statements were derived from a 1994 interlaboratory cooperative test program. Participants analyzed 14 sample sets comprised of various aviation fuels over the temperature range of -45 to -65°C . Nine laboratories participated with the automatic phase transition apparatus and 15 participated with the manual Test Method D2386 test method. The precision statistics were compiled and calculated based on the 0.1°C resolution offered by the automatic phase transition method. Information on the types of samples and their respective average freezing point is contained in the research report.⁴ The precision statements were derived from a 2003 Joint ASTM / IP interlaboratory cooperative test program. Participants analyzed 13 samples

⁴ Supporting data (the results of the 1994 Interlaboratory Cooperative Test Program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1385.

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1398.

⁶ Supporting data (the results of the 2003 Joint ASTM/IP Interlaboratory Cooperative Test Program) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1572.