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An American National Standard

**Designation: 435/98**

## Standard Test Method for Freezing Point of Aviation Fuels (Automatic Phase Transition Method)<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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~~<sup>ε1</sup> Note—Note 1 was updated and Note 2 was included editorially in November 2001.~~

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<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 D02.07.0D on Wax-Related Viscometric Properties of Fuels and Oils.

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### 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  $20^{\circ}\text{C}$ ; 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  $-65^{\circ}\text{C}$ .

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<sup>2</sup>

### 3. Terminology

3.1 *Definitions:*

3.21.1 *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.

3.32 *Definitions of Terms Specific to This Standard:*

3.32.1 *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.

3.32.2 *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  $15 \pm 5^{\circ}\text{C}/\text{min}$  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.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.01.

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*<sup>3</sup>—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.)

## 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 ~~At~~ To verify the performance of the apparatus, an aviation turbine fuel sample for which extensive data has been extensively tested in a obtained by freeze point, Test Method D 2386 interlaboratory study can D 2386, may be used. Samples such as those used to verify performance of in the apparatus within the precision of the test method.

<sup>3</sup> The sole source of supply of the Phase Technology Freezing Point Analyzer Model Series 30, 50, and 70, available from 70 known to the committee at this time is Phase Technology, No. 135-11960 Hammersmith Way, Richmond, B.C. Canada, V7A-5C9, ~~has been found suitable for use in this test method.~~ 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 <sup>1</sup>, which you may attend.

9.3 ~~Reagent grade n-octane~~ 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 a known freezing point value points can be used to verify the calibration of the specimen temperature measuring ~~temperature-measuring~~ device within in the precision of this test method. The literature value<sup>4</sup> for the freezing point of pure n-octane is  $-56.8^{\circ}\text{C}$ . 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  $0.15 \pm 0.01$  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  $0.15 \pm 0.01$  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  $0.69^{\circ}\text{C}$  only in ~~one~~ 1 case in ~~twenty~~ 20.

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  $1.30^{\circ}\text{C}$  only in ~~one~~ 1 case in ~~twenty~~ 20.

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 program<sup>4</sup> 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

<sup>4</sup>CRC Handbook

<sup>4</sup>Supporting data (the results of Chemistry the 1994 Interlaboratory Cooperative Test Program) have been filed at ASTM International Headquarters and Physics, 64th Edition, CRC Press, Inc., Boca Raton, FL, 1984. p C-404. may be obtained by requesting Research Report RR: D02-1385.

differences were within the reproducibility of Test Method D 2386 (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 D 2386. Therefore, the user should exercise appropriate caution when this test method is used in testing Jet B and JP4 samples.

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 D 2386 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.<sup>4</sup>

**13. Keywords**

13.1 aviation turbine fuels; freezing point; Peltier; thermoelectric; wax crystals

**ANNEX**

**(Mandatory Information)**

**A1. DETAILED DESCRIPTION OF APPARATUS**

A1.1 *Test Chamber*, comprised of optical detectors, lens, light source, specimen cup, temperature sensor, Peltier device, and heat sink arranged in a configuration as shown in Fig. A1.1. The lid of the test chamber can be opened to allow cleaning of specimen cup and introduction of new specimen. Once closed and locked, the chamber becomes airtight. An O-ring is used to seal the mating surfaces between the lid and the rest of the chamber. The air trapped in the closed chamber is purged by dry gas. The dry gas inlet and outlet are shown in Fig. A1.1. The test chamber wall is made of black-colored metal and plastic components to minimize light reflection.

A1.1.1 *Specimen Cup*, comprised of a black plastic wall and a highly polished metal bottom. The polished surface of the bottom serves as a reflective surface for light. The transfer of heat to and away from the specimen, through the metal bottom, is controlled by the Peltier device.

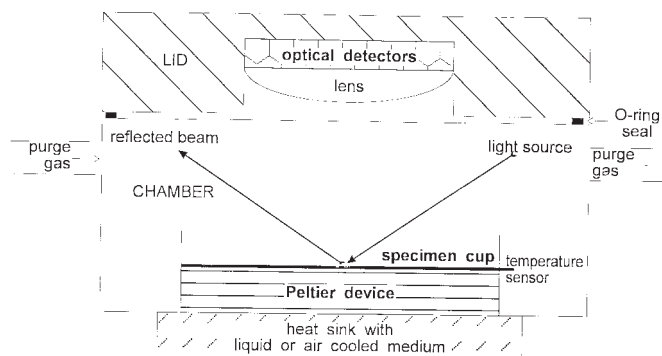
A1.1.2 *Temperature Sensor*, reading to 0.1°C and minimum accuracy to 0.1°C, permanently embedded into the bottom of the specimen cup and positioned less than 0.1 mm below the top surface of the cup bottom. This temperature sensor, which is made of a single strand of platinum, provides accurate measurement of the specimen temperature.

A1.1.3 *Peltier Device*, capable of controlling the specimen temperature over a wide range. The range varies depending on the model series. During specimen cooling, heat is transferred from the top of the device to the bottom. Since the top is in thermal contact with the bottom of the specimen cup, the specimen will be chilled. The bottom of the Peltier device is in thermal contact with the heat sink, where heat is dissipated to the cooling medium. During specimen warming, the reverse process will take place.

A1.1.4 *Light Source*, to provide a beam of light with a wavelength of  $660 \pm 10$  nm. The light source is positioned such that it provides an incident beam (Fig. A1.1) impinging onto the specimen at an acute angle. The light is reflected from the polished bottom of the specimen cup. When the specimen is a homogeneous liquid, the reflected beam impinges onto the chamber lid, which is black in color. The reflected light is then absorbed by the black surface. When wax crystals appear in the specimen, the reflected beam is scattered by the solid-liquid phase boundaries. A significant amount of scattered light impinges onto the lens (Fig. A1.2).

A1.1.5 *Optical Detectors*, positioned above the lens to monitor the clarity of the specimen. The distance between the optical detectors and the lens is adjusted such that the image of the specimen is projected onto the light-sensitive surface of the optical detectors. Sufficient optical detectors are used to cover the image area.

A1.2 *Apparatus Exterior Interface*—The exact layout may vary; however, the following displays and push buttons are



**FIG. A1.1 Schematic of Test Chamber**

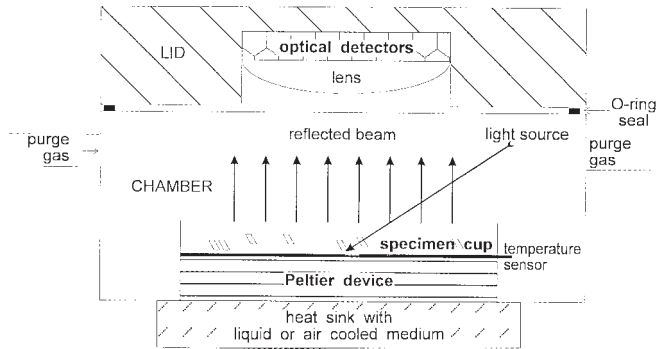


FIG. A1.2 Detection of Crystal Formation

recommended. A typical apparatus (see Fig. A1.3) is shown as an example.

A1.2.1 *Message Display*, provides information on the status of the apparatus. It shall display an appropriate message when the apparatus is idle and no fault is found. At the end of a test, the result is displayed. It displays a diagnostic message if a fault is detected in any of the major components of the apparatus. Detailed explanation of the diagnostic messages is available in the manufacturer's service manual.

A1.2.2 *Specimen Temperature Display*, gives an update of the specimen temperature during the measurement.

A1.2.3 *Light Signal Display*, gives an update of the scattered light level received by the optical detectors. This information may be used by service personnel for trouble-shooting purposes.

A1.2.4 *MENU Buttons*, allow the operator to change the temperature from Celsius to Fahrenheit and vice versa; noting that the Celsius scale is regarded as the standard.

A1.2.5 *RUN Button*, allows the operator to start the measurement sequence once the specimen is put inside the test chamber.



FIG. A1.3 Apparatus Exterior

A1.2.6 *RESET Button*, allows the operator to stop the measurement sequence. Upon pressing this button, the apparatus will immediately stop the measurement sequence and warm the specimen to ambient temperature.

NOTE A1.1—A full description, installation, setup instructions, and maintenance instructions are contained within the manufacturer’s manual supplied with each instrument. They are included in the research report.<sup>5</sup>

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<sup>6</sup> The results of the 1994 Interlaboratory Cooperative Test Program are available from

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters, ~~R~~ and may be obtained by request ~~ASTM~~ ASTM Research Report RR: D02–13985.

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