

# StandardTest Method for Freezing Point of Aviation Fuels (Automatic Laser Method)<sup>1</sup>

This standard is issued under the fixed designation D7153; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

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

1.2 This test method is designed to cover the temperature range of -80 °C to 20 °C; however, the interlaboratory study mentioned in 12.4 has only demonstrated the test method with fuels having freezing points in the range of -60 °C to -42 °C.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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 to determine the applicability of regulatory limitations prior to use.

### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

D2386 Test Method for Freezing Point of Aviation Fuels D4057 Practice for Manual Sampling of Petroleum and 96-b5 Petroleum Products

- D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
- 2.2 Energy Institute Standard:
- **IP 16** Determination Freezing Point of Aviation Fuels<sup>3</sup>

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

3.3 Symbols:

Cd = the specimen temperature at which the appearance of the first crystals are detected in the specimen by an optical crystal detector under specified conditions of test.

*Co* = the specimen temperature at which the appearance of opacity in the specimen is detected by an optical opacity detector under specified conditions of test.

Do = the specimen temperature at which the disappearance of opacity in the specimen is detected by an optical bound opacity detector under specified conditions of test.

## 4. Summary of Test Method

4.1 A specimen is cooled at a rate of 10 °C/min  $\pm$  5 °C/min while continuously being illuminated by a laser light source. The specimen is continuously monitored by optical crystal and opacity detectors for the first formation of solid hydrocarbon crystals. Once the hydrocarbon crystals are detected by both sets of optical detectors, the specimen is then warmed at a rate of 3 °C/min  $\pm$  0.5 °C/min. When initial opacity in the specimen disappears, the specimen is then warmed at a rate of 12 °C/min  $\pm$  1 °C/min. The specimen temperature at which the last hydrocarbon crystals return to the liquid phase, as detected by the crystal detector, is recorded as the freezing point.

4.2 In certain circumstances, as measured by the apparatus, the specimen is reheated to approximately 10 °C, then cooled at the rate in 4.1 until hydrocarbon crystals are detected by the crystal detector. The specimen is then warmed at a rate of 12 °C/min  $\pm$  1 °C/min, until the last hydrocarbon crystals return to the liquid phase. The specimen temperature at which

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

<sup>&</sup>lt;sup>1</sup>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.

Current edition approved April 1, 2015. Published April 2015. Originally approved in 2005. Last previous edition approved in 2010 as D7153 – 05 (2010). DOI: 10.1520/D7153-15.

<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Annual Book of IP Standards Methods, Vol 1. Available from Energy Institute, 61 New Cavendish St., London, WIG 7AR, U.K.

the last hydrocarbon crystals return to the liquid phase, as detected by the crystal detector, 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 which, if present in the fuel system of the aircraft, can restrict the flow of fuel through filters. 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 shall 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 expresses results to the nearest 0.1  $^{\circ}$ C, and it eliminates most of the operator time and judgment required by Test Method D2386.

5.4 When a specification requires Test Method D2386, do not substitute this test method or any other test method.

#### 6. Apparatus

6.1 Automatic Apparatus<sup>4</sup>—This apparatus consists of a microprocessor-controlled test cell that is capable of cooling and heating the specimen, dual optical detectors to monitor the appearance and disappearance of crystals and opacity, 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 chamber, optical detectors, laser light source, digital display, cooling and heating systems, and a specimen temperature measuring device.

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

6.4 The apparatus shall be capable of cooling the specimen at a rate of 10 °C/min  $\pm$  5 °C/min, heating the specimen at rates of 3 °C/min  $\pm$  0.5 °C/min and 12 °C/min  $\pm$  1 °C/min over the temperature range of -80 °C to +20 °C.

Note 1—The apparatus described is covered by a patent. If you are aware of an alternative(s) to the patented item, please attach to your ballot return a description of the alternatives. All suggestions will be considered by the committee.

NOTE 2-The software version used in this apparatus is version V 5.3.

6.5 *Standard Syringe*, capable of injecting approximately 10 mL  $\pm$  2 mL of the specimen, with a tip or an adapter tip that will fit the inlet of the test cell. A disposable 10 mL syringe with a Luer type cone connection has been found suitable.

6.6 *Waste Receiving Container*, capable of collecting the overflow when the specimen is injected into the test cell. A 400 mL standard glass beaker has been found suitable.

## 7. Sampling

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

7.2 At least 30 mL of sample is required for each test.

### 8. Preparation of Apparatus

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

8.2 Turn on the main power switch of the analyzer.

## 9. Calibration and Standardization

9.1 Ensure that all of the manufacturer's instructions for calibration of the mechanical and electronic systems and operation of 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 Test Method D2386 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 intra-company cross-checks.

9.3 At intervals of not more than 12 months, check the temperature measuring system against a temperature measuring device that has a current calibration certificate.

9.4 Verify the correct functioning of the apparatus in accordance with the manufacturer's instructions using a verification liquid (9.2) when first installed and thereafter at intervals not exceeding one month.

## **10.** Procedure

10.1 Draw 10 mL  $\pm$  2 mL bubble-free portion of sample into a syringe. Connect the syringe to the inlet port (Fig. 1). Rinse the test cell by injecting 10 mL  $\pm$  2 mL of specimen into the test cell; the specimen excess will flow into the waste receiving container (Fig. 2)

10.2 Rinse the test cell a second time by repeating 10.1.

10.3 Draw a 10 mL  $\pm$  2 mL bubble-free portion of sample into a syringe.

10.4 Connect the syringe to the inlet port (Fig. 1). Dispense the specimen into the test cell; the specimen excess will flow into the waste receiving container (Fig. 2). Leave the syringe connected to the sample inlet for the entire duration of the test.

10.5 Start the operation of the apparatus according the manufacturer's instructions. From this point through Section 11, the apparatus automatically controls the procedure.

10.5.1 Cool the specimen at a rate of 10 °C/min  $\pm$  5 °C/min while continuously illuminating with a polarized laser light source. Monitor the specimen continuously with two optical detectors, an opacity detector and a crystal detector (Fig. 3), for the first formation of solid hydrocarbon crystals.

10.5.2 Once the appearance of the first crystals (Cd) is detected on the crystal detector and opacity (Co) is detected on

<sup>&</sup>lt;sup>4</sup> The sole source of supply of the apparatus known to the committee at this time is ISL model FZP 5G2s series Freezing Point Analyzer, available from PAC - ISL, BP 70285 - VERSON, 14653 CARPIQUET Cedex, France. 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.