



## Standard Test Method for Filter Flow of Aviation Fuels at Low Temperatures<sup>1</sup>

This standard is issued under the fixed designation D 4305; 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.

### INTRODUCTION

This test method describes a procedure for determining the simulated freezing point for aviation turbine fuels using an automated apparatus. The results from this test method have been found to be equivalent to Test Method D 2386, except for liquids which have a viscosity of more than 5 mm<sup>2</sup>/s (cSt) at – 20°C (see Test Method D 445) which can give a higher (warmer) result than Test Method D 2386.

The procedure can also be used to investigate the formation of wax crystals or the cold flow properties of other products.

### 1. Scope

1.1 This test method covers the determination of low-temperature flow behavior, through a screen-type test filter, of aviation turbine fuels which can contain separated solids as wax.

1.2 Procedure A employs a 26- $\mu$ m test filter and is the recommended procedure. Some existing instruments are fitted with a 42- $\mu$ m filter, and Procedure B is retained to enable their continued use.

1.3 The use of Procedure A (26- $\mu$ m test filter) with fuels that have a viscosity of greater than 5.0 mm<sup>2</sup>/s (cSt) at – 20°C, as determined by Test Method D 445, can affect the precision and the simulated freezing point obtained on such fuels and can give a higher (warmer) result than the conventional freezing point obtained by Test Method D 2386.

NOTE 1—The principle of this test method relies on flow through a fine-mesh test filter, and, hence, the result can be affected by the viscosity of the sample. When using Procedure A, a no-flow condition is reached when crystals block the test filter or the viscosity exceeds about 14 mm<sup>2</sup>/s (cSt); sample with a viscosity of greater than 5 mm<sup>2</sup>/s (cSt) at – 20°C may exceed the 14-mm<sup>2</sup>/s (cSt) threshold at a temperature before crystals are formed. If viscosity affects the result before crystals are formed, then the reported value of the no-flow temperature of the sample will always be warmer than the actual freezing point, and therefore fail-safe and an indicator of possible flow anomalies at low temperature.

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 appro-*

*priate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)<sup>2</sup>

D 1655 Specification for Aviation Turbine Fuels<sup>2</sup>

D 2386 Test Method for Freezing Point of Aviation Fuels<sup>2</sup>

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>3</sup>

### 3. Terminology

#### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *flow point*—represents the temperature corresponding to the unblockage of a test filter, that previously was blocked by separated solids.

3.1.2 *no-flow point*—represents the temperature corresponding to a specified degree of blockage of a test filter by separated solids.

### 4. Summary of Test Method

4.1 A 5-mL specimen of fuel is subjected to a programmed temperature cycle while a pump maintains an oscillating flow at constant rate across a metal mesh test filter. As the temperature falls, separated wax tends to restrict the test filter causing an increase in pressure. When this pressure exceeds 1.33 kPa for more than 0.95 s, the no-flow point is indicated and the cooling cycle is stopped. The pump continues to

<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 on Flow Properties.

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

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.02.

operate and exert pressure as the fuel specimen is warmed. At the point when the test filter is unplugged and pressure falls below 1.33 kPa for more than 0.95 s, the flow point is indicated and the temperature of the specimen displayed and held.

## 5. Significance and Use

5.1 The lowest temperature at which aviation fuels remain free of solid hydrocarbon crystals, which may restrict the flow of fuel through filters in an aircraft fuel system, is a key safety parameter in the specification and use of fuels. In Test Method D 2386, the freezing point is defined as the temperature at which all crystals disappear following a cooling and warming cycle. In this test method, a cooling and warming cycle is also employed, however, the test result is defined as the flow point, which is the temperature of the specimen at which a test filter becomes unblocked on warming.

## 6. Apparatus<sup>4</sup>

6.1 A detailed description is given in Annex A1.

6.2 The apparatus defined in this test method comprises a cooling/warming cell, a specimen pump, pressure sensor, test filter, syringe, sample filter, and associated electronic controls and displays.

## 7. Reagents and Materials

7.1 *Heptane*, technical grade.

NOTE 2—**Caution:** Extremely flammable, vapor harmful if inhaled (see Annex A2.1).

7.2 *Jet A or A1*.

NOTE 3—**Caution:** Combustible, vapor harmful (see Annex A2.2).

## 8. Sampling

8.1 Obtain a sample in accordance with Practice D 4057.

8.2 Store the sample in a cool place (0 to 20°C) away from direct heat or sunlight.

## 9. Preparation of Apparatus

9.1 If the apparatus previously contained an unknown specimen, follow the manufacturer's instructions to clean the cell, using a paraffinic solvent such as heptane; oxygenated solvents such as acetone shall not be used as they may damage the cell. If the prior specimen is similar to the test material, the latter can be used to flush the cell.

9.2 The cleaning cycle shall be repeated at least twice, the last cycle involving flushing with the test material.

9.3 The cell temperature shall be warmer than 0°C before the test specimen is injected.

9.4 The syringe shall be clean and dry before use.

## 10. Calibration and Quality Control Checks

10.1 Follow the manufacturer's instructions.

10.1.1 *Pressure Transducer*—This should be checked once monthly by direct comparison with a water manometer, or a traceable pressure gage.

<sup>4</sup> The Setpoint Detector has been found to be satisfactory for use in this test method, and it is available from Stanhope-Seta, Chertsey, Surrey KT16 8AP, England.

10.1.2 *Specimen Pump*—Calibration and operation of the pump should be checked once monthly.

10.1.3 *Electronic Thermometer (PRT)*—Calibration and operation of the PRT should be checked against a traceable standard every six months.

10.1.4 *Control Sample*—Confirm the calibration of the instrument each day it is used by running a control sample of known flow point.

## 11. Procedures A and B

NOTE 4—Procedures A and B are identical except that Procedure A uses the 26- $\mu$ m test filter, while Procedure B uses the 42- $\mu$ m test filter.

11.1 Set the RAMP control to a temperature approximately 5°C above the expected flow point of the specimen. If the flow point is unknown, set the control to a temperature warmer than any possible flow point.

11.2 Set the cooling rate control to 1.75°C/min.

11.3 Set the instrument to determine *flow*. Wait until the cell temperature is above 0°C.

11.4 Fill the syringe with 5 mL of sample, taking care not to introduce any air into the syringe.

11.5 Introduce the 5-mL specimen into the instrument through the integral sample filter, using the syringe. Leave the syringe in place attached to the sample filter.

11.6 The specimen will be rapidly cooled until its temperature reaches the *ramp* setting.

11.7 When the *ramp* temperature of the sample is reached, the cooling rate will change to  $1.75 \pm 0.2^\circ\text{C}/\text{min}$ .

11.8 When the *no-flow* point is reached (pressure greater than 1.33 kPa for longer than 0.95 s) the cooling will be switched off. The specimen will then be warmed at a rate of  $1.75 \pm 0.2^\circ\text{C}/\text{min}$  until the *flow* point is reached (pressure less than 1.33 kPa for longer than 0.95 s).

11.9 The instrument will display the *flow* temperature of the sample.

11.10 Follow the manufacturer's instructions to drain the sample and clean the syringe.

## 12. Report

12.1 Record the *flow* temperature to the nearest 0.1°C. When determining compliance with aviation fuel specifications, the flow temperature can be reported as the freezing point of the fuel.

12.2 Record the *no-flow* temperature to the nearest 0.1°C (optional).

12.3 Record whether Procedure A or Procedure B was used.

## 13. Precision and Bias<sup>5,6,7</sup>

13.1 The precision of this test method, as determined by statistical analyses of interlaboratory results, is as follows:

<sup>5</sup> Data showing the equivalence of the Mark I, II, and III Setpoint Detector instruments (Procedure B) have been filed at ASTM Headquarters. Request RR:D02-1216.

<sup>6</sup> The results of the cooperative test program, for Procedure A, from which these results have been derived are filed at ASTM Headquarters. Request RR:D02-1385.

<sup>7</sup> The results of the cooperative test program, for Procedure B, from which these results have been derived are filed at ASTM Headquarters. Request RR:D02-1168.

13.1.1 *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 this test method, exceed the following only in one case in 20.

13.1.1.1 *Procedure A, 26- $\mu$ m Test Filter*— $r = 0.53^{\circ}\text{C}$ .

13.1.1.2 *Procedure B, 42- $\mu$ m Test Filter*— $r = 1.2^{\circ}\text{C}$ .

13.1.2 *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 correct operation of this test method, exceed the following only in one case in 20.

13.1.2.1 *Procedure A, 26- $\mu$ m Test Filter*— $R = 2.21^{\circ}\text{C}$ .

13.1.2.2 *Procedure B, 42- $\mu$ m Test Filter*— $R = 2.6^{\circ}\text{C}$ .

### 13.2 Test Programs:

13.2.1 *Procedure A (26- $\mu$ m Test Filter)*—An international interlaboratory test program<sup>7</sup> involving 14 aviation fuels and seven laboratories was carried out in 1993-1994, comparing flow point with freezing point by Test Method D 2386. Four fuels were excluded from the analysis because their viscosities exceeded 5.0 mm<sup>2</sup>/s (cSt) at  $-20^{\circ}\text{C}$ .

13.2.2 *Procedure B (42- $\mu$ m Test Filter)*—A 1980 interlaboratory test program involving 19 aviation fuels among eight laboratories was carried out comparing flow point results with freezing point by Test Method D 2386. Two fuels were excluded from analysis because their flow points exceeded the low temperature limit of the instrument (about  $-70^{\circ}\text{C}$ ).

13.3 *Bias*—Since there is no accepted reference material suitable for determining the bias of the procedures in this test method, bias cannot be determined.

13.4 *Relationship to Test Method D 2386*—The following relative bias was determined when compared to the results of Test Method D 2386.

13.4.1 *Procedure A (26- $\mu$ m Filter)*—The interlaboratory program indicated no relative bias between this test method and Test Method D 2386.

13.4.2 *Procedure B (42- $\mu$ m Test Filter)*—The correlation between FR (freezing point in Test Method D 2386<sup>2</sup>), and FP (flow point in this test method), is given as follows:

$$FR (^{\circ}\text{C}) = 1.04 (FP^{\circ}\text{C}) + 2.67^{\circ}\text{C} \quad (1)$$

NOTE 5—*Procedure A*: Aviation fuels with a viscosity of greater than 5 mm<sup>2</sup>/s (cSt) at  $-20^{\circ}\text{C}$  (determined by Test Method D 445) can give a result which is warmer than the freezing point determined by Test Method D 2386.

NOTE 6—*Procedure B*: Fuels of similar composition and wax precipitation characteristics can exhibit constant bias from the correlation line between freezing point and flow point. Where this occurs, such as production from a single refinery or crude source, a different correlation may exist.

NOTE 7—*Procedure B*: Results of up to  $10^{\circ}\text{C}$  colder than those obtained by Test Method D 2386 have been reported when testing hydrocracked fuels.

## 14. Keywords

14.1 aviation fuels; filter flow; flow point; freezing point

## ANNEXES

### (Mandatory Information)

#### A1. APPARATUS

A1.1 *Cell Assembly*—Comprises a cylindrical sample chamber bored in the center of an aluminum block with an illuminated viewing window provided across its diameter. The chamber is fitted with an airtight cover to the underside of which is attached a transparent cylinder, thus forming an inner and outer cell to the chamber. The cells are separated by a stainless steel test filter which is attached to the lower end of the inner cell cylinder. A combined fill and drain point is fitted to the outer chamber (see Fig. A1.1).<sup>5</sup>

A1.2 *Cooling System*—Temperature range from 5 to  $-65^{\circ}\text{C}$ .

A1.3 *Specimen Pump*—Comprises a cam-operated diaphragm pump driven by a synchronous motor. The pump alternately applies pressure and vacuum to the inner cell, thus expelling and drawing the specimen through the filter at a rate of 20 cycles/min.

A1.4 *Pressure Sensor*—A pressure sensor with a minimum accuracy of 0.05 kPa is required to measure the pressure differential across the test filter. This increases when wax crystals form or when there is a change in state sufficient to impede the flow through the test filter. The unit senses when the

pressure differential across the filter exceeds 1.33 kPa for more than  $1 \pm 0.05$  s. When this occurs, the cooling cycle is stopped. Pressure and vacuum are still applied as the specimen warms and when the pressure differential has decreased to a point below 1.33 kPa for  $1 \pm 0.05$  s, the electronic thermometer display is held at the (flow) test result reading.

A1.5 *Mode Selection*—To change the operation of the apparatus so that completion of the test occurs at the *plugging* (no-flow) point of the test filter.

A1.6 *Drain Valve*—Connected to the cell assembly allowing the sample to be drained at the conclusion of the test.

A1.7 *Syringe*—Preset at  $5 \pm 0.1$  mL with a fitting that matches the socket provided on the instrument. The syringe is left in the socket for the duration of the test.

A1.8 *Electronic Thermometer*—Reading to  $0.1^{\circ}\text{C}$ , with a minimum accuracy of  $0.1^{\circ}\text{C}$ , permanently fitted to the instrument and electronically linked to the programmed cycle. A platinum resistance temperature sensing probe shall be connected by means of screened leads to the thermometer.

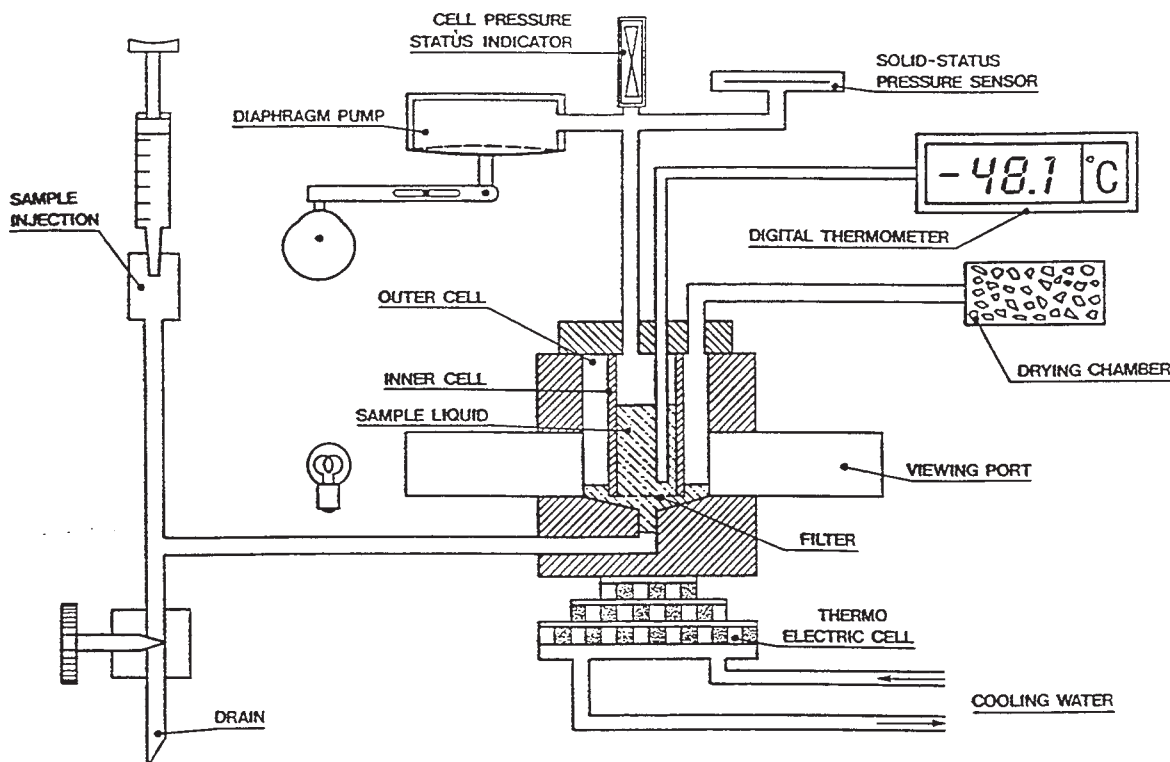


FIG. A1.1 Schematic of Cell Assembly and Cooling System

A1.9 *Drying Chamber*—A desiccant-filled drying chamber is connected permanently to the outlet from the cell assembly and ensures that the displaced air is kept moisture free.

A1.10 *Test Filters*:

A1.10.1 26- $\mu$ m, diameter  $4.25 \pm 0.25$  mm, stainless steel.

A1.10.2 42- $\mu$ m, diameter  $9 \pm 0.25$  mm, stainless steel.

A1.11 *Sample Filter*—26- $\mu$ m, stainless steel, fitted to the socket, provided for injection of the sample with the syringe.

## A2. PRECAUTIONARY STATEMENTS

A2.1 *n-Heptane*:

A2.1.1 Keep away from heat, sparks, and open flame.

A2.1.2 Keep container closed.

A2.1.3 Use with adequate ventilation. Avoid prolonged breathing of vapor or spray mist.

A2.1.4 Avoid prolonged or repeated skin contact.

A2.2 *Aviation Turbine Fuel (Jet A or A-1, see Specification D 1655)*:

A2.2.1 Keep away from heat, sparks and open flame.

A2.2.2 Keep container closed.

A2.2.3 Use with adequate ventilation.

A2.2.4 Avoid buildup of vapors and eliminate all sources of ignition.

A2.2.5 Avoid breathing vapor or spray mist.

A2.2.6 Avoid prolonged or repeated contact with skin.

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