



Designation: ~~D4171–21~~ D4171 – 22

## Standard Specification for Fuel System Icing Inhibitors<sup>1</sup>

This standard is issued under the fixed designation D4171; 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. Scope\*

1.1 This specification covers additives for aviation fuels (for example, Specifications **D910**, **D7547**, and **D1655**) used to inhibit ice formation in aircraft fuel systems.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use Caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

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

**D56** Test Method for Flash Point by Tag Closed Cup Tester

**D93** Test Methods for Flash Point by Pensky-Martens Closed Cup Tester

**D268** Guide for Sampling and Testing Volatile Solvents and Chemical Intermediates for Use in Paint and Related Coatings and Material

**D891** Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals

**D910** Specification for Leaded Aviation Gasolines

**D1078** Test Method for Distillation Range of Volatile Organic Liquids

**D1209** Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)

**D1296** Test Method for Odor of Volatile Solvents and Diluents (Withdrawn 2021)<sup>3</sup>

**D1353** Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee **D02** on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee **D02.J0.04** on Additives and Electrical Properties.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

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

- D1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)
- D1476 Test Method for Heptane Miscibility of Lacquer Solvents (Withdrawn 2021)<sup>3</sup>
- D1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products
- D1655 Specification for Aviation Turbine Fuels
- D1722 Test Method for Water Miscibility of Water-Soluble Solvents
- D3828 Test Methods for Flash Point by Small Scale Closed Cup Tester
- D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter
- D5006 Test Method for Measurement of Fuel System Icing Inhibitors (Ether Type) in Aviation Fuels
- D5386 Test Method for Color of Liquids Using Tristimulus Colorimetry
- D7547 Specification for Hydrocarbon Unleaded Aviation Gasoline
- D8005 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)
- E1 Specification for ASTM Liquid-in-Glass Thermometers
- E70 Test Method for pH of Aqueous Solutions With the Glass Electrode
- E203 Test Method for Water Using Volumetric Karl Fischer Titration
- E300 Practice for Sampling Industrial Chemicals
- ~~E450 Test Method for Measurement of Color of Low-Colored Clear Liquids Using the Hunterlab Color Difference Meter (Withdrawn 1993)<sup>3</sup>~~
- E1064 Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration
- E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids
- E2877 Guide for Digital Contact Thermometers

### 3. Classification

3.1 Two types of fuel system icing inhibitors are provided as follows:

3.1.1 *Type II*—Anhydrous isopropanol, also described as 99 % grade 2-Propanol or isopropyl alcohol, is used as an anti-icing additive in aviation gasoline. (**Warning**—Isopropanol (2-Propanol) is both flammable and an irritant; use with caution.)

3.1.2 *Type III*—Diethylene glycol monomethyl ether (DiEGME) is used as an anti-icing additive in both aviation gasoline and aviation turbine fuel. (**Warning**—Diethylene glycol monomethyl ether, (DiEGME). Combustible, toxic material.)

3.1.2.1 Test Method **D5006** can be used to determine the concentration of DiEGME in aviation fuels.

NOTE 1—Ethylene glycol monomethyl ether (EGME) was previously included in this specification as Type I, last appearing in D4171 – 94. EGME is considered technically satisfactory for this application, but has been generally replaced by DiEGME due to availability, reduced toxicological concerns, and lack of widely available methodology to determine FSII concentration in aviation fuels when a mixture is known to be present, or when the identity of the FSII present in the fuel is not clearly known.

### 4. Properties

4.1 *Type II*—Isopropanol anti-icing additive shall conform to the requirements of **Table 1**, as manufactured.

4.2 *Type III*—Diethylene glycol monomethyl ether shall conform to the requirements of **Table 2**, as manufactured.

### 5. Sampling

5.1 The material shall be sampled in accordance with Practice **E300**.

### 6. Test Methods

6.1 Determine the properties enumerated in this specification in accordance with the following ASTM methods:

6.1.1 *Relative Density*—Determine the relative density (that is, specific gravity) at 20 °C or 25 °C with respect to water by a method accurate to the third decimal place. See Section 5 of Test Method **D268**, Test Method **D4052**, or Method A or B of Test Methods **D891**.

6.1.2 *Color*—Test Method **D1209**, **D8005**, or ~~E450~~**D5386**.

**TABLE 1 Detailed Requirements for Isopropanol (99 % Grade)  
(Type II) FSII**

Property	Requirement	ASTM Test Method
Acidity, max, mg KOH/g	0.019	D1613
Relative density:		
20 °C/20 °C	0.785 to 0.787	D268
25 °C/25 °C	0.782 to 0.784	D268
Color, platinum-cobalt, max	10	D1209 or E450
Color, platinum-cobalt, max	10	D1209, D8005, or D5386
Distillation range, max, °C	1.5 (including 82.3 °C)	D1078
Nonvolatile matter, max, mg/100 mL	5	D1353
Odor	characteristic, nonresidual	D1296
Water, max, mass %	0.2	D1364
Heptane miscibility at 20 °C	miscible without turbidity with 19 vol heptane (99 % Grade)	D1476
Water miscibility at 25 °C	miscible without turbidity when diluted with 10 vol distilled water	D1722

**TABLE 2 Detailed Requirements for Fuel System Icing Inhibitors  
(Type III)**

Property	Requirement		ASTM Test Method
	DiEGME (Type III)		
Acid number, max, mg KOH/g	0.09		D1613
Color, platinum-cobalt, max	10		D1209 or E450
Color, platinum-cobalt, max	10		D1209, D8005, or D5386
Purity, min, mass %	99.0		Annex A1
pH of 25 % solution in water (25 °C ± 2 °C)	5.5–7.5		E70 <sup>A</sup>
Relative density, 20 °C/20 °C	1.020–1.025		D891 (Method A or B) or D4052
Water, max, mass %			D1364, E1064, or E203
Point of manufacture	0.10		
Point of use	0.8		
Flash point, min, °C	85 °C		D93, D56, or D3828
Antioxidant, mg/kg	50–150		<sup>B</sup>

<sup>A</sup> Pipette 25 mL of the inhibitor into a 100 mL volumetric flask and filled with freshly boiled and cooled distilled water having a pH of 6.5 to 7.5. Measure the pH value with a pH meter calibrated in accordance with Test Method E70.

<sup>B</sup> Acceptable antioxidants are: 2,6-ditertiary-butyl-4-methylphenol, 2,4-dimethyl-6-tertiary-butyl phenol, 2,6-ditertiary-butyl phenol, and 75 % min 2,6-ditertiary-butyl phenol plus 25 % max tertiary and tritertiary butyl phenols.

6.1.3 *Distillation Range*—Test Method D1078 using ASTM Solvents Distillation Thermometers (40C with a range from 72 °C to 126 °C for isopropanol) conforming to the requirements of Specification E1 or any other temperature measuring device that cover the temperature range of interest, such as thermocouples, thermistors, or resistance temperature detectors (RTDs). An instrument meeting Guide E2877 or Specification E2251 may be used in preference to 40C if the instrument provides equivalent or better accuracy and precision.

6.1.4 *Nonvolatile Matter*—Test Method D1353.

6.1.5 *Odor*—Test Method D1296.

6.1.6 *Water*—Test Method D1364, E1064, or E203.

6.1.7 *Heptane Miscibility*—Test Method D1476.

6.1.8 *Acidity*—Test Method **D1613**.

6.1.9 *Water Miscibility*—Test Method **D1722**.

6.1.10 *Flash Point*—Test Methods **D56**, **D93**, or **D3828**.

## **7. Keywords**

7.1 additives; aircraft fuel systems; aviation fuels; fuel system icing inhibitors; ice formation

## **ANNEX**

### **(Mandatory Information)**

#### **A1. TEST METHOD FOR DETERMINING PURITY OF FUEL SYSTEM ICING INHIBITOR (TYPE III)**

##### **A1.1 Scope**

A1.1.1 This test method measures the purity of fuel system icing inhibitors (Type III). The test results are used to determine if the inhibitor meets the purity requirements listed in **Table 2**.

##### **A1.2 Summary of Test Method**

A1.2.1 A representative sample of fuel system icing inhibitor (Type III) is injected into a capillary gas chromatograph and the components of the inhibitor are separated and measured with a flame ionization detector. Quantitation is made by peak area measurement using external standardization and a computing integrator. As the linear dynamic range of many gas chromatographic detectors is often exceeded for the major component, the sum of all impurities (all components other than the inhibitor) are subtracted from 100 to calculate the purity of the icing inhibitor.

##### **A1.3 Significance and Use**

A1.3.1 Fuel system icing inhibitor performance (Type III) is based upon test results using the pure inhibitor in a specific concentration range. Impurities affect inhibitor solubility in the fuel and reduce the effective concentration. Methods are therefore needed to check additive purity to ensure adequate performance in the aircraft.

##### **A1.4 Apparatus**

A1.4.1 *Gas Chromatograph*—Any gas chromatographic instrumentation can be used that meets the requirements described below.

A1.4.2 *Temperature Control*—The chromatograph must be capable of programmed temperature operation.

A1.4.3 *Sample Inlet System*—An automatic sampler with split injection is recommended, however, manual split injection is

acceptable if care is taken to assure injected sample volume and rate of injection is constant. On-column injection is acceptable, however, modifications to the procedure are required which are not specified here.

A1.4.4 *Detector*—A hydrogen flame ionization detector (HFID) is recommended, however, any detector can be used that has the sensitivity to measure the purity of the icing inhibitors at the levels listed in [Table 2](#).

A1.4.5 *Column*—Any gas chromatographic column can be used that provides separation of the impurities from the fuel system icing inhibitor (Type III). Columns and conditions that have been used successfully are shown in [Table A1.1](#).

A1.4.6 *Integrator*—Provide means for the determination of peak areas for the impurities and the icing inhibitors. This can be accomplished with a computer or electronic integrator.

A1.4.7 *Analytical Balance*—Capable of measuring 0.1 mg.

## A1.5 Reagents

A1.5.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A1.5.2 *Air*—Use air (hydrocarbon free) as the HFID oxidant. (**Warning**—Air is usually supplied as a compressed gas under high pressure and supports combustion.)

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<https://standards.iteh.ai/catalog/standards/sist/7bd16153-4167-49b9-97ee-b01d63916710/astm-d4171-22>

A1.5.3 *Hydrogen*—Use hydrogen (hydrocarbon free) as the fuel for the flame ionization detector. (**Warning**—Extremely flammable. Hydrogen is usually supplied as a compressed gas under high pressure.)

**TABLE A1.1 Recommended Operating Conditions**

Column	30 M by 0.32 mm bonded phase 86 % methyl, 14 cyanopropyl '1701' (1.0 µm film thickness) fused-silica capillary column
Column temperature	100 °C initial temperature, programmed to 250 °C at 12 °C/min
Injection system	Split injection system which contains a glass insert liner that is firmly packed with silylated glass wool. The split ratio is 50:1 and the injection temperature is 250 °C
Detector	Hydrogen flame ionization at 250 °C
Sample volume	0.5 µL with a 5 µL syringe
Carrier gas	Helium at an average flow velocity of 20 cm/second (propane elutes in 2.5 min with a column temperature of 60 °C) to give a flow rate of ~1 mL/min
Make-up gas	Helium at 20 mL/min
Air flow	350 mL/min
Hydrogen flow	30 mL/min

<sup>4</sup> ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.