Designation: ~~D4171 – 11~~ D4171 – 16

An American National Standard

Standard Specification for Fuel System Icing Inhibitors[1]

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 ~~(see~~ (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 material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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
D1353 Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products
D1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)
D1476 Test Method for Heptane Miscibility of Lacquer Solvents
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
D7547 Specification for Hydrocarbon Unleaded Aviation Gasoline
E1 Specification for ASTM Liquid-in-Glass Thermometers
E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

[1] This specification is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.J0.04 on Additives and Electrical Properties.

Current edition approved ~~May 1, 2011~~June 15, 2016. Published ~~June 2011~~ July 2016. Originally approved in 1982. Last previous edition approved in ~~2010~~2011 as ~~D4171–03(2010).~~D4171 – 11. DOI: ~~10.1520/D4171-11.~~10.1520/D4171-16.

[2] 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.

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

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)[3]
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

3. Classification

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

3.1.1 *Type I*—Ethylene glycol monomethyl ether is used as an anti-icing additive in both aviation gasoline and aviation turbine fuels.

NOTE 1—Ethylene glycol monomethyl ether (EGME) was previously included in this specification, 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.

3.2 *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.)

3.3 *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.)

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

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 ~~The~~ Determine the properties enumerated in this specification ~~shall be determined~~ in accordance with the following ASTM methods:

[3] The last approved version of this historical standard is referenced on www.astm.org.

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:		
~~20/20°C~~	~~0.785 to 0.787~~	~~D268~~
~~25/25°C~~	~~0.782 to 0.784~~	~~D268~~
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
Distillation range, max, °C	1.5 (including 82.3°C)	D1078
~~Nonvolatile matter, max, mg/100 mL~~	~~5~~	~~D1353~~
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 99 % heptane~~	~~D1476~~
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~~
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 DiEGME ~~(Type~~ (Type III)	ASTM Test Method
Acid number, max, mg KOH/g	0.09	D1613
Color, platinum-cobalt, max	10	D1209 or E450
Purity, min, mass %	99.0	Annex A1
~~pH of 25 % solution in water (25 ± 2°C)~~	~~5.5–7.5~~	~~E70[A]~~
pH of 25 % solution in water (25 °C ± 2 °C)	5.5–7.5	E70[A]
~~Relative density, 20°/20°C~~	~~1.020–1.025~~	~~D891 (Method A or B) or D4052~~
Relative density, 20 °/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	[B]

[A] ~~Twenty-five millilitres~~Pipette 25 mL of the inhibitor ~~shall be pipetted~~ into a ~~100-ml~~100 mL volumetric flask and filled with freshly boiled and cooled distilled water having a pH of 6.5 to 7.5. ~~The~~ Measure the pH value ~~shall be measured~~ with a pH meter calibrated in accordance with Test Method E70.

[B] 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.

6.1.1 *Relative Density*—Determine the relative density (that is, specific gravity) at ~~20~~20 °C or ~~25°C~~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 or E450.

6.1.3 *Distillation Range*—Test Method D1078 using ASTM Solvents Distillation Thermometers (40C with a range from ~~72~~72 °C to ~~126°C~~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, resistance temperature detectors (RTDs) or one conforming to Specification E2251 may be used that provides equivalent or better accuracy and precision than ASTM 40C.

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 INHIBITORS (TYPES I AND 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*—~~Means must be provided~~ 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.~~0.1 mg.

A1.5 Reagents

A1.5.1 *Purity of Reagents*—~~Reagent~~ Use reagent grade chemicals ~~will be used~~ 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