Designation: D4171 – 16a

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 (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
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

[1] 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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[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.

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

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

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 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.

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
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
Purity, min, mass %	99.0	Annex A1
pH of 25 % solution in water (25 °C ± 2 °C)	5.5–7.5	E70[A]
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	[B]

[A] 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.
[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. 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 or E450.

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 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*—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.

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

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.[4] 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.)

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.)

A1.5.4 *Helium*—Use helium (hydrocarbon free) as the carrier gas for the chromatograph. (**Warning—**Helium is usually supplied as a compressed gas under high pressure.)

A1.5.5 *Ethylene Glycol*—Use ethylene glycol (anhydrous, 99 + %) as a calibration standard for analysis of diethylene glycol monomethyl ether. (**Warning—**Toxic, irritant.)

A1.5.6 *Ethylene Glycol Monomethyl Ether*—Use EGME (anhydrous, 99 + %) as a calibration standard for analysis of diethylene glycol monomethyl ether. (**Warning—**See Note 1.) (**Warning—**Ethylene glycol monomethyl ether (EGME).

[4] *Reagent Chemicals, American Chemical Society Specifications*, 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.

Combustible, toxic material.[5]) (**Warning—**In addition to other precautions, EGME has been shown to be a teratogen in animals. Avoid inhalation. Do not get in eyes, on skin, or on clothing. Wash thoroughly after handling.)

A1.5.7 *Triethylene Glycol Monomethyl Ether*—This material is used as a calibration standard for analysis of diethylene glycol monomethyl ether. The purity of this material should be determined and the standard adjusted for this purity.

A1.6 Preparation of Apparatus

A1.6.1 Install the gas chromatographic instrumentation in accordance with the manufacturer's instructions. System operating conditions will depend upon the column used and optimization of performance. See Table A1.1 for recommended conditions.

NOTE A1.1—The position of the capillary column in the injection port and in the detector is very important. Consult the instrument manufacturer's instruction manual for specific instructions. In general the column should be installed in such a manner that one end extends into the injection port and into the bottom of the glass liner and the other end extends into the detector up to within a few mm of the exit end of the flame jet.

A1.6.2 *System Performance*—System operating conditions must be used that effect baseline separation of the components of interest. A minimum resolution of 1.5 is required to accurately determine icing inhibitor purity. The resolution is calculated according to the following equation:

$$R = \frac{2(t_2 - t_1)}{W_1 + W_2} \qquad \text{(A1.1)}$$

where:

t_1 = time (seconds) for peak 1 at apex,
t_2 = time (seconds) for peak 2 at apex,
W_1 = peak width at base (seconds) for peak 1, and
W_1 = peak width at base (seconds) for peak 2.

A1.7 Procedure

A1.7.1 *Calibration*—Determine the response factor for each component of interest by preparing and analyzing samples of known composition. As any one component used in the calibration standard may contain one of the other components, it is best to prepare one calibration standard for each component in a pure solvent at the expected concentration range (in this case, approximately 0.05 % by mass). A "pure" solvent in this case means one of high purity (>99 %) which does not contain the components of interest.

A1.7.1.1 Calibration standards for ethylene glycol, EGME, and triethylene glycol monomethyl ether should be prepared for analysis of DiEGME. The purity of triethylene glycol monomethyl ether used to prepare the standard should be determined and used to correct the actual component mass in the standard. For example, the purity of a sample of triethylene glycol monomethyl ether is determined to be 95.0 %. A calibration standard for this component is prepared by weighing 0.05 g (to the nearest 0.1 mg) of triethylene glycol monomethyl ether into a suitable container to which is added 99.95 g of 99 + % pure isopropanol, given a total mass of 100 g. The actual mass percentage triethylene glycol monomethyl ether in the standard may now be computed as:

$$\frac{0.05 \cdot 95.0/100}{(0.05 + 99.95)}\,100\,\% = 0.0475\ \%\ \text{by mass} \qquad \text{(A1.2)}$$

This calibration standard should now be analyzed by capillary gas chromatography using conditions such as those specified in Table A1.1. The external standard response factor for the component may then be computed as:

$$Ai/Mi = \text{response factor for individual component } i,\ Fi \qquad \text{(A1.3)}$$

$$Ai = \text{area of individual component } i$$

$$Mi = \text{mass percent of individual component } i$$

A1.7.2 *Analysis*—Analyze the sample according to parameters such as those provided in Table A1.1.

A1.8 Calculations

A1.8.1 Calculate the mass percent of each individual component using an external standard procedure:

$$AiFi = \text{component } i,\ \%\ \text{by mass} \qquad \text{(A1.4)}$$

$$Ai = \text{peak area of component } i$$

$$Fi = \text{response factor for component } i$$

A1.8.2 For the analysis of diethylene glycol monomethyl ether (DiEGME—Type III), calculate the purity of the component using the following equation:

$$\text{DiEGME},\ \%\ \text{by mass} = 100 - C \qquad \text{(A1.5)}$$

where:

C = the sum of all impurities, including water, as determined by an alternate method (such as Test Method D1364) when using an HFID detector.

A1.8.3 If the analysis is to be performed on a field sample, sum all of the impurities, excluding water, and subtract from 100 to calculate purity. The purity of the DiEGME must be ≥99 % to meet use limits.

A1.9 Precision and Bias[6]

A1.9.1 The precision of this test method was determined by the statistical examination of interlaboratory test results obtained from ten coded samples analyzed in seven laboratories.

A1.9.2 *Repeatability*—The difference between successive 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 the test method, exceed the following values only in one case in twenty.

$$\text{Repeatability} = 0.09033\,(100.021 - X)\ \%\ \text{by mass} \qquad \text{(A1.6)}$$

where:

X = average of two percent by mass purities.

[5] For more detailed information on ethylene glycol monomethyl ether, refer to the *Federal Register*, Vol 51, No. 97, dated Tuesday, May 20, 1986. Consult the supplier's material safety data sheet.

[6] Supporting data can be obtained from ASTM Headquarters. Request RR:D02-1408.

For example, a sample that averages 99.50 % by mass purity in two tests has a repeatability of 0.05 % by mass.

A1.9.3 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical material would, in the long run, exceed the following values only in one case in twenty.

$$\text{Reproducibility} = 0.2184\,(101.364 - X)\ \%\ \text{by mass} \quad (A1.7)$$

where:

X = average of two percent by mass purities.

For example, a sample that averages 99.50 % by mass purity in two tests has a reproducibility of 0.41 % by mass.

A1.9.4 *Bias*—There was no significant bias between results obtained from this analysis and the known purity of samples used in the interlaboratory program.

SUMMARY OF CHANGES

Subcommittee D02.J0 has identified the location of selected changes to this standard since the last issue (D4171 – 16) that may impact the use of this standard. (Approved Dec. 1, 2016.)

(1) Revised subsection 6.1.3; added Guide E2877 to Referenced Documents.

Subcommittee D02.J0 has identified the location of selected changes to this standard since the last issue (D4171 – 11) that may impact the use of this standard. (Approved June 15, 2016.)

(1) Added Specification D7547 to the Referenced Documents and to subsection 1.1.