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An American National Standard

Standard Specification for Fuel System Icing Inhibitors[1]

This standard is issued under the fixed designation D 4171; 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 Specifications D 910 and D 1655) used to inhibit ice formation in aircraft fuel systems.

1.2 The values stated in SI units are to be regarded as standard.

1.3 *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.*

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

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2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 56 Test Method for Flash Point by the Tag Closed Tester
D 93 Test Methods for Flash Point by Pensky-Martens Closed Tester
D 268 Test Methods of Sampling and Testing Volatile Solvents and Chemical Intermediates for Use in Paint and Related Coatings and Materials
D 891 Test Methods for Specific Gravity, Apparent, of Liquid Industrial Chemicals
D 910 Specification for Aviation Gasolines
D 1078 Test Method for Distillation Range of Volatile Organic Liquids
D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)
D 1296 Test Method for Odor of Volatile Solvents and Diluents
D 1353 Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer and Related Products
D 1364 Test Method for Water in Volatile Solvents (Karl Fischer Reagent Titration Method)
D 1476 Test Method for Heptane Miscibility of Lacquer Solvents
D 1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products
D 1655 Specification for Aviation Turbine Fuels
D 1722 Test Method for Water Miscibility of Water-Soluble Solvents
D 3828 Test Methods for Flash Point by Small Scale Closed Tester
D 4052 Test Method for Density and Relative Density of Liquids by Digital Density Meter
D 5006 Test Method for Measurement of Fuel System Icing Inhibitors (Ether Type) in Aviation Fuels
E 1 Specification for ASTM Thermometers
E 70 Test Method for ~~PH~~ pH of Aqueous Solutions with the Glass Electrode
E 203 Test Method for Water Using Karl Fischer Reagent
E 300 Practice for Sampling Industrial Chemicals
E 450 Method for Measurement of Color of Low-Colored Clear Liquids Using the Hunterlab Color Difference Meter[3]
E 1064 Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration

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 D 4171–94. 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.~~

~~NOTE 2—Isopropanol, (2-Propanol). Flammable material. Irritant.~~ 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.~~

~~NOTE 3—**Warning:** Diethylene~~ fuel. (**Warning**— Diethylene glycol monomethyl ether, (DiEGME). Combustible, toxic material.)

3.3.1 Test Method D 5006 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 E 300.

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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*, ~~Vol 05.01.~~ volume information, refer to the standard's Document Summary page on the ASTM website.
~~*Annual Book of ASTM Standards*, Vol 06.04.~~

[3] Withdrawn.

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

Property	Requirement	ASTM Test Method
Acidity, max, mg KOH/g	0.019	D 1613
Relative density:		
20/20°C	0.785 to 0.787	D 268
25/25°C	0.782 to 0.784	D 268
Color, platinum-cobalt, max	10	D 1209 or E450
Distillation range, max, °C	1.5 (including 82.3°C)	D 1078
Nonvolatile matter, max, mg/100 mL	5	D 1353
Odor	characteristic, nonresidual	D 1296
Water, max, mass %	0.2	D 1364
Heptane miscibility at 20°C	miscible without turbidity with 19 vol 99 % heptane	D 1476
Water miscibility at 25°C	miscible without turbidity when diluted with 10 vol distilled water	D 1722

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	D 1613
Color, platinum-cobalt, max	10	D 1209 or E450
Purity, min, mass %	99.0	Annex A1
pH of 25 % solution in water (25 ± 2°C)	5.5–7.5	E 70[A]
Relative density, 20°/20°C	1.020–1.025	D 891 (Method A or B) or D4052
Water, max, mass %		D 1364, E1064, or E 203
Point of manufacture	0.10	
Point of use	0.8	
Flash point, min, °C	85°C	D 93, D 56, or D 3828
Antioxidant, mg/kg	50–150	[B]

[A] Twenty-five milliliteres of the inhibitor shall be pipetted into a 100-ml volumetric flask and filled with freshly boiled and cooled distilled water having a pH of 6.5 to 7.5. The pH value shall be measured with a pH meter calibrated in accordance with Test Method E 70.

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

6.1.1 *Relative Density*— Determine the relative density (that is, specific gravity) at 20 or 25°C with respect to water by a method accurate to the third decimal place. See Section 5 of Test Method D 268, Test Method D 4052, or Method A or B of Test Methods D 891.

6.1.2 *Color*—Test Method D 1209 or E 450.

6.1.3 *Distillation Range*—Test Method D 1078 using ASTM Solvents Distillation Thermometers (40C with a range from 72 to 126°C for isopropanol) conforming to the requirements of Specification E 1.

6.1.4 *Nonvolatile Matter*—Test Method D 1353.

6.1.5 *Odor*—Test Method 1296.

6.1.6 *Water*—Test Method D 1364, E 1064, or E 203.

6.1.7 *Heptane Miscibility*—Test Method D 1476.

6.1.8 *Acidity*—Test Method D 1613.

6.1.9 *Water Miscibility*—Test Method D 1722.

6.1.10 *Flash Point*— Test Methods 56, D 93, or D 3828.

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 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*—Reagent grade chemicals will be used in all tests. Unless otherwise indicated, it is intended that all

TABLE A1.1 Recommended Operating Conditions

Column	30M by 0.32 mm bonded phase 86 % methyl, 14 cyanopropyl '1701' (1.0 µ 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 microliter with a 5 microliter syringe~~
Sample volume	0.5 microlitre with a 5 microlitre 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~~
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

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*—Air (hydrocarbon free) is used as the HFID ~~oxidant.~~

~~NOTE A1.1—*Air:* **Warning:** Air~~ oxidant. (**Warning—**Air is usually supplied as a compressed gas under high pressure and supports combustion.)

A1.5.3 *Hydrogen*—Hydrogen (hydrocarbon free) is used as the fuel for the flame ionization ~~detector.~~

~~NOTE A1.2—*Hydrogen:* **Warning:**~~ **detector. (Warning—Extremely flammable. Hydrogen is usually supplied as a compressed gas under high pressure.)**

A1.5.4 *Helium*—Helium (hydrocarbon free) is used as the carrier gas for the ~~chromatograph.~~

~~NOTE A1.3—*Helium:* **Warning:**~~ **chromatograph. (Warning—Helium is usually supplied as a compressed gas under high pressure.)**

A1.5.5 *Ethylene Glycol*— Ethylene glycol (anhydrous, 99 + %) is used as a calibration standard for analysis of diethylene glycol monomethyl ~~ether.~~

~~NOTE A1.4—*Ethylene glycol:* **Warning:**~~ **ether. (Warning—Toxic, irritant.)**

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

~~NOTE A1.5—**Warning:**~~ 1.) (**Warning—**Ethylene glycol monomethyl ether (EGME). Combustible, toxic material. ~~NOTE A1.6—**Precaution:** In~~[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.~~7~~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:

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

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

[4] ~~*Annual Book*~~*Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For suggestions on the testing of ~~ASTM~~ reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, ~~Vol 15.05.~~ BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.
~~*Annual Book of ASTM Standards*~~

[5] For more detailed information on ethylene glycol monomethyl ether, refer to the *Federal Register*, Vol ~~05.02.~~ 51, No. 97, dated Tuesday, May 20, 1986. Consult the supplier's material safety data sheet.

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:

$$\frac{0.05 * 95.0/100}{(0.05 + 99.95)} 100\ \% = 0.0475 \text{ mass } \% \quad \text{(A1.2)}$$

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:

$$\text{A}i/\text{M}i = \text{response factor for individual component } i, Fi \quad \text{(A1.3)}$$

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

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

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:

$$\text{A}i\text{F}i = \text{component } i, \text{ \% by mass} \quad \text{(A1.4)}$$

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

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

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

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

where:

C = the sum of all impurities, including water, as determined by an alternate method (such as Test Method D 1364) 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.

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

where:

X = average of two mass % purities.

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

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

where:

X = average of two mass % purities.

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

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[6] Supporting data can be obtained from ASTM ~~Standards, Vol 14.03.~~ Headquarters. Request RR:D02-1408.