



Standard Test Method for Measurement of Fuel System Icing Inhibitors (Ether Type) in Aviation Fuels¹

This standard is issued under the fixed designation D5006; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers a technique for measuring the concentration of Diethylene Glycol Monomethyl Ether (DiEGME) in aviation fuels. ~~The HB and Brix scale refractometers are specified. A measured volume of fuel, extracted with a fixed ratio of water, is tested with a suitable refractometer to determine the concentration of this fuel system icing inhibitor (FSII) by measuring the refractive index of a water extract in fuel.~~ Precision estimates have been determined for the DiEGME additive using specific extraction ratios with a wide variety of fuel types. The extraction ratios are high enough that portable handheld refractometers can be used, but not so high as to sacrifice accuracy or linearity, or both, in the 0.01 to 0.25 vol % range of interest.

1.2 DiEGME is fully described in Specification D4171 and in other specifications.

~~1.3 The values stated in SI units are to be regarded as the standard.~~

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

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

D4171 Specification for Fuel System Icing Inhibitors

E1 Specification for ASTM Liquid-in-Glass Thermometers

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 ~~Brix scale—a refractometer with a refractive index scale calibrated to weight percent cane sugar (sucrose). analog refractometer, n —a traditional-style refractometer which visually projects a shadowline onto a scale etched into a glass reticle.~~

3.1.1.1 ~~Discussion—The scale, which is magnified by an eyepiece, displays either a direct reading of DiEGME concentration, as is the case with the analog HB refractometer, or may display Brix units which must be converted into DiEGME concentration.~~

3.1.2 ~~HB—a refractometer that can be used in a temperature range from 18 to 35°C without incorporating a temperature correction factor. Brix refractometer, n —a refractometer which displays readings on the Brix scale.~~

3.1.3 ~~Brix scale, n —an expression of the mathematical relationship between refractive index and the concentration by weight of pure sucrose in water.~~

3.1.4 digital refractometer, n —A refractometer which relies on a solid-state image sensor to measure the refractive index of a solution, convert the refractive index reading into a particular unit of measure (percent DiEGME), and outputs the results on a digital display.

3.2 *Acronyms:*

3.2.1 *DiEGME*—Diethylene Glycol Monomethyl Ether

3.2.2 *FSII*—fuel system icing inhibitor

¹ This test method 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.

Current edition approved Dec. Aug. 1, 2003-2010. Published January 2004-August 2010. Originally approved in 1989. Last previous edition approved in 2002-2003 as D5006-02-D5006-03. DOI: 10.1520/D5006-103.

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.

4. Summary of Test Method

4. In order to determine the concentration of DiEGME in aviation fuel, a measured volume of fuel is extracted with a fixed ratio of water. The extraction procedure includes sufficient agitation and contacting time to ensure that equilibrium distributions are attained. With the HB refractometer, several drops of the water extract are placed on the prism face and the volume percent DiEGME is read directly from a custom graduated scale printed on the reticule. If the Brix refractometer is used, a temperature correction factor is first applied to the reading, multiplied by 2 and divided by 100 to calculate volume percent DiEGME. (**Warning**—Diethylene glycol monomethyl ether (DiEGME), Slightly toxic material. This material caused slight embryo-fetal toxicity (delayed development) but no increase in birth defects in laboratory animals. Consult the suppliers' material safety data sheet.)

4.1 In order to determine the concentration of DiEGME in aviation fuel, a measured volume of fuel is extracted with a fixed ratio of water. The extraction procedure includes sufficient agitation and contacting time to ensure that equilibrium distributions are attained. If using an Analog Refractometer, place several drops of water extract on the measuring surface, point it towards a light source, and take a reading on the internal scale. The analog HB refractometer will display the actually percent volume of DiEGME on its scale. Users of a Brix refractometer will follow a similar procedure, but will have to convert the Brix reading into DiEGME percent volume. If the Brix refractometer is not automatically temperature compensated, then a temperature correction must first be applied to the Brix reading before converting it to percent DiEGME. If using a Digital Refractometer, place several drops of water extract in the sample well, press a button to initiate the reading, and the percent volume of DiEGME will be displayed on the LCD display. (**Warning**—Diethylene glycol monomethyl ether (DiEGME), Slightly toxic material. This material caused slight embryo-fetal toxicity (delayed development) but no increase in birth defects in laboratory animals. Consult the suppliers' material safety data sheet.)

NOTE 1—Isopropanol is not detected because of the similarity of isopropanol/water refractive indices, and the presence of *iso* propanol in fuel containing other additives results in lower than true values.

5. Significance and Use

5.1 DiEGME is miscible with water and can be readily extracted from the fuel by contact with water during shipping and in storage. Methods are therefore needed to check the additive content in the fuel to ensure proper additive concentration in the aircraft.

5.2 This test method is applicable to analyses performed in the field or in a laboratory.

6. Apparatus

6.1 **Refractometer**—The HB temperature compensated, direct reading refractometer and the 0 to 30 or 0 to 16 Brix have been found satisfactory for use.—An optical instrument used to measure the physical properties of a solution. Refractometers suitable for use in this test method include:

6.1.1 **HB Refractometer**³—An analog refractometer with a direct reading scale for percent DiEGME. This instrument is automatically temperature compensated from 18 to 35°C.

6.1.2 **Brix Refractometer**—An analog refractometer with a Brix scale which may or may not be automatically temperature compensated.

6.1.3 **MISCO Jet Fuel Refractometer (p/n JPX-DiEGME)**—A digital refractometer that provides a direct reading of DiEGME concentration and is automatically temperature compensated within the range of 10 to 45°C.

6.1.4 **Gammon HBD2 Refractometer**—A digital refractometer that provides a direct reading of DiEGME concentration and is automatically temperature compensated within the range of 10 to 40°C.

6.2 **Extraction Vessel**—Any suitable vessel of at least 200 mL with provisions for isolating a small column of water extract. Examples are separatory funnels, (glass or plastic), or plastic dropping bottles.

6.3 **Measuring Vessel**—Any vessel capable of measuring up to 160 mL of fuel to an accuracy of ± 2 mL, such as a 250-mL graduated cylinder, or other calibrated container.

6.4 **Water Dispenser**—2.0-mL pipettes are preferred, but syringes or burettes not exceeding 5.0-mL capacity that can dispense 2.0 ± 0.2 mL may be used. For the Brix refractometer, the pipette must measure 1.0 ± 0.1 mL.

6.5 **Thermometer**—The thermometer must have suitable range to measure air and fuel temperature in the field. Accurate to $\pm 1^\circ\text{C}$ and meeting Specification E1.

³ The sole source of supply of the HB refractometer known to the committee at this time is Gammon Technical Products, Inc., 2300 Hwy 34, P.O. Box 400, Manasquan, NJ 08736. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

³ The analog HB refractometer and the digital HB2D refractometer are available from Gammon Technical Products, Inc., 2300 Hwy 34, P.O. Box 400, Manasquan, NJ 08736. The MISCO Jet Fuel Refractometer (p/n JPX-DiEGME) and Brix refractometers are available from MISCO Refractometer, 3401 Virginia Rd., Cleveland, Ohio 44122 USA. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

7. ~~Reagents and Materials.~~ Reagents and Materials

7.1 *Water*—Distilled or deionized water is preferred for the extraction procedure, and for refractometer calibration, but potable water may be used.

8. Calibration

~~8.1 Calibration of the HB or Brix scale refractometer consists of setting the reading obtained with water at ambient temperature to 0.0 with the zero adjustment.~~

~~8.2 The calibration step is incorporated into the procedure to minimize the effect of temperature changes between the time of calibration and measurement. (**Warning**—The extraction, calibration, and measurement steps should be done at ambient conditions. Avoid placing the refractometer on hot or cold surfaces, in pockets on your person, or other locations that would change the temperature of the instrument from ambient. When setting zero or making a measurement, take care not to heat or cool the refractometer from ambient.)~~ Refractometer Calibration

8.1 All refractometers should be zero-set to water before use. The exact procedure for zero-setting a refractometer varies based on the type and make of instrument. Consult the user manual for specific instructions on zero-setting each make of refractometer.

8.2 The calibration step is incorporated into the procedure to minimize the effect of temperature changes between the time of calibration and measurement. (**Warning**—The extraction, calibration, and measurement steps should be done at ambient conditions. Avoid placing the refractometer on hot or cold surfaces, in pockets on your person, or other locations that would change the temperature of the instrument from ambient. When zero-setting or making a measurement, take care not to heat or cool the refractometer from ambient.)

8.3 For the most accurate possible readings, the refractometer, the calibration fluid, and the ambient temperature should all be in equilibrium within the temperature compensation range, or the operational temperature range, of the refractometer. If there is a temperature disparity, allow some time for the temperatures to equalize before taking a reading.

9. Procedure Sample Preparation and Extraction

9.1 *Extraction Procedure for the HB Refractometer (Temperature Compensated, Direct Reading)* Extraction Ratios for Both Analog and Digital Refractometers with Direct Reading DiEGME Scales:

9.1.1 Locate the thermometer and refractometer where they will remain at ambient temperature during the test.

9.1.2 Measure 160 mL of fuel to be tested into the extraction vessel.

9.1.3 Measure 2.0 mL of water into the extraction vessel.

9.1.1 Measure 160 mL of fuel to be tested into the extraction vessel.

9.1.2 Measure 2.0 mL of water into the extraction vessel.

9.2 *Extraction Procedure for the Brix Scale Refractometer (Non-Temperature Compensated)* Extraction Ratios for Analog Brix Refractometers With or Without Automatic Temperature Compensation :

9.2.1 Locate the thermometer and refractometer where they will remain at ambient temperature during the test.

9.2.2 Measure 80 mL of the fuel to be tested into the extraction vessel.

9.2.3 Measure 1.0 mL of water into the extraction vessel.

9.3 *Procedure for the Determination of Fuel System Icing Inhibitor:*

9.3.1 Shake the extraction vessel vigorously for a minimum of 5 min for all fuels.

9.3.1.1 Mechanical shakers may be used provided thorough intermixing of the aqueous and fuel phases occurs, similar to that obtained by hand shaking. (**Warning**—Following the extraction procedures is most critical. Failure to extract for the specified time or failure to provide vigorous agitation can result in false readings. If lower than expected readings are obtained, a second test should be done with a longer extraction time.)

9.3.2 Allow the extraction vessel to sit undisturbed at ambient temperature for a period of at least 2 min to allow the water to settle to the bottom. (**Warning**—Fuel entrained in the water causes an indistinct refractometer reading. In most cases fuel residue can be eliminated by *slowly* lowering the refractometer cover. The surface tension of water will sweep fuel off the prism surface.)

9.3.3 Open the cover of the refractometer prism and wipe it clean with a tissue. Place several drops of the water used for the extraction on the prism face.

9.3.4 Close the cover and view the scale through the eyepiece. Adjust the focus if necessary to bring the numbered scale into focus. Observe the position of the shadow line on the numbered scale.

9.3.5 Rotate the zero adjustment knob or set screw so that the shadow line intersects at 0.0 on the HB or Brix scale refractometer.

9.3.6 Open the prism cover and wipe the surface clean with a tissue.

9.3.7 Isolate several drops of the water extract from the extraction vessel and place on the prism face.

9.3.7.1 If a separatory funnel is used, it may be necessary to collect some extract into a smaller container, and then transfer several drops to the prism face with a clean eyedropper, syringe or pipette.

9.3.7.2 If a dropping bottle is used as an extraction vessel, place it right side up, remove the cap, squeeze slightly, and replace the cap with the bottle under a slight vacuum. Invert the bottle and allow the water extract to settle to the bottom. Uncap the bottle and squeeze it gently until several drops of extract are collected on a tissue held in the same hand as the refractometer, and then allow several drops of the water extract to fall onto the prism face.