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An American National Standard

Standard Test Method for Measurement of Fuel System Icing Inhibitors (Ether Type) in Aviation Fuels[1]

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 U.S. 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. A measured volume of fuel, extracted with a fixed ratio of water, is tested with a suitable refractometer to determine the concentration of fuel system icing inhibitor (FSII) 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 % by volume 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 standard. No other units of measurement are included in this standard.

1.4 **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.

1.5 *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

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *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 *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

4. Summary of Test Method

4.1 In order to determine the concentration of DiEGME in aviation fuel, a measured volume of fuel is extracted with a

[1] This test method 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.

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 actual 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 sheets.)

NOTE 1—*Iso*propanol is not detected because of the similarity of *iso*propanol/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*—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 °C 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 °C to 45 °C.

6.1.4 *Gammon HB2D Refractometer*—A digital refractometer that provides a direct reading of DiEGME concentration and is automatically temperature compensated within the range of 10 °C to 40 °C.

[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,[1] which you may attend.

6.2 *Extraction Vessel*—Any suitable vessel of at least 200 mL with provisions for isolating a small column of water extract at the bottom. 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 mL ± 0.2 mL may be used. For the Brix refractometer, the pipette must measure 1.0 mL ± 0.1 mL.

6.5 *Thermometer*—The thermometer must have suitable range to measure air and fuel temperature in the field. Accurate to ±1 °C and meeting 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 1C.

7. 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. 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. Sample Preparation and Extraction

9.1 *Extraction Ratios for Both Analog and Digital Refractometers with Direct Reading DiEGME Scales:*

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 Ratios for Analog Brix Refractometers With or Without Automatic Temperature Compensation:*

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

9.2.2 Measure 1.0 mL of water into the extraction vessel.

10. Sample Extraction

10.1 Shake the extraction vessel vigorously for a minimum of 5 min for all fuels, preferably with the cap facing down.

10.2 Mechanical shakers may be used, provided that 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.)

10.3 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.

11. Sample Testing

11.1 *Measurement of Samples Using Digital Refractometers with DiEGME Scales:*

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

11.1.2 Isolate several drops of the water extract from the extraction vessel, and transfer to the sample well of the digital refractometer.

11.1.3 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.

11.1.4 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 into the refractometer well.

11.1.5 If using the MISCO Jet Fuel Refractometer, close the evaporation cover to help prevent evaporation of the sample during testing.

11.1.6 Allow some time for the temperature of the refractometer, fluid, and ambient environment to equalize.

11.1.7 Initiate the reading by pressing the “GO” Button on the MISCO Jet Fuel Refractometer or the “READ” Button on the Gammon HB2D Refractometer.

11.1.8 Record the ambient temperature reading displayed on the thermometer to the nearest degree Celsius.

11.1.9 Make certain that the temperature displayed on the thermometer is within the temperature compensation range of the refractometer.

11.1.10 Record the reading on the refractometer digital display to two significant figures in volume percent DiEGME.

11.1.11 Take four more readings of the same sample, and average the results.

11.1.12 Properly dispose of test fluids, wash apparatus with soap and water, and dry all items. (**Warning—**Treat the refractometer as an optical instrument and avoid damage to the lens and window elements. Store the refractometer in a protective cover or case.)

11.2 *Measurement of Samples Using Analog HB or Analog Brix Refractometers:*

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

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

11.2.3 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.

11.2.4 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.

11.2.5 *Slowly* lower the prism cover into place, point the refractometer at a light source, and look into the eyepiece. (**Warning—**Fuel entrained in the water may cause an indistinct refractometer reading. In most cases fuel residue on an analog refractometer can be eliminated by *slowly* lowering the refractometer cover. The surface tension of water should sweep fuel off the prism surface.)

11.2.6 Take the reading at the point the shadowline intersects the scale.

11.2.7 If using a HB refractometer, record the reading to two significant figures in volume percent DiEGME.

11.2.8 If using a Brix refractometer, record the Brix value and perform the conversion calculation in 12.2.

11.2.9 Record the ambient temperature to the nearest degree Centigrade using a thermometer.

11.2.10 Make certain that the temperature displayed on the thermometer is within the operational range, or the temperature compensation range, of the refractometer.

11.2.11 Properly dispose of test fluids, wash apparatus with soap and water, and dry all items. (**Warning—**Treat the refractometer as an optical instrument and avoid damage to the lens and window elements. Store the refractometer in a protective cover or case.)

12. Calculation

12.1 For both analog and digital refractometers with direct reading DiEGME scales, report the reading obtained to two significant figures as the final result in volume percent DiEGME. If multiple determinations are made, average the results that fall within the specified repeatability and reproducibility tolerances. For rounding off of significant figures, Practice E29 shall apply.

NOTE 2—For analog HB refractometers produced prior to July 2003 that have both an EGME and DiEGME scale on the reticle, report the reading in volume percent from the left hand scale marked DiEGME or M. The scale is printed on the reticule in the eyepiece of the refractometer.

TABLE 1 Temperature Correction Factors for Brix Refractometer

			Reading													
			0	5	10	15	20	25	30	35	40	45	50	55	60	65	70
	10		0.50	0.54	0.58	0.61	0.64	0.66	0.68	0.70	0.72	0.73	0.74	0.75	0.76	0.78	0.79
	11		0.46	0.46	0.53	0.55	0.58	0.60	0.62	0.64	0.63	0.66	0.67	0.68	0.64	0.70	0.71
	12		0.42	0.45	0.48	0.50	0.52	0.54	0.56	0.57	0.58	0.59	0.60	0.61	0.61	0.63	0.63
	13		0.37	0.40	0.42	0.44	0.46	0.48	0.49	0.50	0.51	0.52	0.53	0.54	0.54	0.53	0.55
	14	Deduct	0.33	0.35	0.37	0.39	0.40	0.41	0.42	0.43	0.44	0.45	0.45	0.46	0.46	0.47	0.48
	15	from	0.27	0.29	0.31	0.33	0.34	0.34	0.35	0.36	0.37	0.37	0.38	0.39	0.39	0.40	0.40
	16	reading	0.22	0.24	0.25	0.26	0.27	0.28	0.28	0.29	0.30	0.30	0.30	0.31	0.31	0.32	0.32
	17		0.17	0.18	0.19	0.20	0.21	0.22	0.21	0.22	0.22	0.23	0.23	0.23	0.23	0.24	0.24
	18		0.12	0.13	0.13	0.14	0.14	0.14	0.11	0.15	0.15	0.15	0.15	0.16	0.16	0.16	0.16
	19		0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08
Temperature °C	20		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	21		0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08
	22		0.13	0.13	0.14	0.14	0.15	0.15	0.15	0.15	0.15	0.16	0.16	0.16	0.16	0.16	0.16
	23		0.19	0.20	0.21	0.22	0.22	0.23	0.23	0.23	0.28	0.24	0.24	0.24	0.24	0.24	0.24
	24		0.26	0.27	0.28	0.29	0.30	0.30	0.31	0.31	0.31	0.31	0.31	0.32	0.32	0.32	0.32
	25	Add to	0.33	0.35	0.36	0.37	0.38	0.38	0.39	0.39	0.40	0.40	0.40	0.40	0.40	0.40	0.40
	26	reading	0.40	0.42	0.43	0.44	0.45	0.46	0.47	0.47	0.48	0.48	0.48	0.48	0.48	0.48	0.48
	27		0.48	0.50	0.52	0.53	0.54	0.55	0.55	0.55	0.55	0.56	0.56	0.56	0.56	0.56	0.56
	28		0.56	0.57	0.60	0.61	0.62	0.63	0.63	0.63	0.64	0.64	0.64	0.64	0.64	0.64	0.64
	29		0.64	0.66	0.68	0.69	0.72	0.72	0.72	0.72	0.74	0.73	0.73	0.73	0.73	0.73	0.73
	30		0.71	0.74	0.77	0.78	0.79	0.80	0.80	0.81	0.81	0.81	0.81	0.81	0.81	0.81	0.81

12.2 For a non-temperature compensated analog refractometer with a Brix scale, first apply the temperature correction factor from Table 1. No temperature correction is necessary for Brix refractometers with automatic temperature compensation, as long as the measurements are taken within the temperature compensation range of the refractometer.

12.2.1 Calculate the volume percent DiEGME as follows:

$$\text{Vol \% FSII} = \frac{2 \times \text{Temperature Corrected Scale Reading}}{100} \quad (1)$$

13. Report

13.1 Report the following information:

13.1.1 The type of fuel analyzed,

13.1.2 The volume percent DiEGME found, and

13.1.3 The temperature (°C) of the analysis.

14. Precision and Bias[4]

14.1 The precision of this test method as determined by statistical examination of interlaboratory results according to RR:D02-1007[5] is as follows:

14.1.1 *Repeatability*—The difference between two test 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 in only one case in twenty:

HB temperature compensated refractometer: (2)

$$\text{repeatability} = 0.009 \text{ volume \%}$$

Brix scale refractometer: $\text{repeatability} = 0.005 \text{ volume \%}$ (3)

14.1.2 *Reproducibility*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in normal and correct operation of the test method, exceed the following values only in one case in twenty:

HB temperature compensated refractometer: (4)

$$\text{reproducibility} = 0.018 \text{ volume \%}$$

Brix scale refractometer: $\text{reproducibility} = 0.021 \text{ volume \%}$ (5)

NOTE 3—Accuracy and precision in the field can be lower than a similar test done under controlled laboratory conditions using a temperature controlled precision refractometer. The MISCO Jet Fuel Refractometer (p/n JPX-DIEGME) (6.1.3) and the Gammon HB2D Refractometer (6.1.4) digital instruments have been found to provide accuracy and precision equal to the original analog instrument.

14.2 *Bias*—The HB temperature compensated refractometer gave results, on average, greater than the true value by 0.0018 % by volume. The Brix scale refractometer gave results, on average, less than the true value by 0.0051 % by volume.

15. Keywords

15.1 aviation fuel; diethylene glycol monomethyl ether; fuel system icing inhibitor; refractometry

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1251.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1007.