Designation: D 5006 – 03

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 D 5006; 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 to determine the concentration of this fuel system icing inhibitor (FSII) by measuring the refractive index of a water extract. 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 D 4171 and in other specifications.

1.3 The values stated in SI units are to be regarded as the 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]

D 4171 Specification for Fuel System Icing Inhibitors

E 1 Specification for ASTM Thermometers

E 29 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).

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.

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 fixed ratio of water. The extraction procedure includes sufficient agitation and contacting time to ensure that equilibrium distributions are attained. With the HB refractometer,[3,4] 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.)

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

[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 on Aviation Fuels.

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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 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 [1], which you may attend.

[4] After July 1, 2003, all HB refractometers provided by Gammon Technical Products will contain only the DiEGME scale on the instrument reticle.

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.

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 ±1°C and meeting Specification E 1.

7. Reagents and Materials.

7.1 *Water*—Distilled or deionized water is preferred for the extraction procedure, 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.)

9. Procedure

9.1 *Extraction Procedure for the HB Refractometer (Temperature Compensated, Direct Reading)*:

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.

9.2 *Extraction Procedure for the Brix Scale Refractometer (Non-Temperature Compensated)*:

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.

9.3.8 *Slowly* lower the prism cover into place using the same technique described in 9.3.4. Observe and record the position of the shadow line.

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

9.3.10 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.)

10. Calculation

10.1 For the HB refractometer, report the reading obtained in 9.3.8 to two significant figures as the final result in volume

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

percent DiEGME. If multiple determinations are made, average results falling within the specified repeatability and reproducibility tolerances. For rounding off of significant figures, Practice E 29 shall apply.

NOTE 2—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.

10.2 For the non-temperature compensated refractometer with Brix scale, first apply the temperature correction factor from Table 1. Calculate the volume percent DiEGME as follows:

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

11. Report

11.1 Report the following information:

11.1.1 The type of fuel analyzed,

11.1.2 The volume percent DiEGME found, and

11.1.3 The temperature (°C) of the analysis.

12. Precision and Bias [5]

12.1 The precision of this test method as determined by statistical examination of interlaboratory results according to RR: D02-1007[6] is as follows:

12.1.1 *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:

$$\text{HB temperature compensated refractometer: repeatability} = 0.009 \text{ volume \%} \quad (2)$$

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

12.1.2 *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:

$$\text{HB temperature compensated refractometer: reproducibility} = 0.018 \text{ volume \%} \quad (4)$$

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

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.

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

13. Keywords

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

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

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