Designation: D3321 – 13

Standard Test Method for Use of the Refractometer for Field Test Determination of the Freezing Point of Aqueous Engine Coolants[1]

This standard is issued under the fixed designation D3321; 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 test method covers the use of a portable refractometer for determining the approximate freezing protection provided by ethylene and propylene glycol-based coolant solutions as used in engine cooling systems and special applications.

NOTE 1—Some instruments have a supplementary freezing protection scale for methoxypropanol coolants. Others carry a supplemental scale calibrated in density or specific gravity readings of sulfuric acid solutions so that the refractometer can be used to determine the charged condition of lead acid storage batteries.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1177 Test Method for Freezing Point of Aqueous Engine Coolants

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 These coolant testers are critical-angle refractometers designed for rapid, approximate measurement of ethylene and propylene glycol coolant freezing point protection. Only a few drops of test solution are required. Some testers automatically correct for ambient air temperature and the temperature of the solution being tested. The instrument is rugged, simple to read, and easy to clean and maintain.

3.2 The coolant freezing point readings are taken at points where the dividing line between light and dark crosses the scales. Some refractometers have a coolant scale for indicating the freezing point of aqueous ethylene glycol coolants only, while other refractometers also have a scale for indicating the freezing point of aqueous propylene glycol coolants. The range of the scales varies from one device to another.

3.3 Freezing point measurements are concentration-related values and are in turn directly related to refractive index. It has been empirically determined that freezing point measurements are accurate within 1°C (2°F).

4. Significance and Use

4.1 This practice is commonly used by vehicle service personnel to determine the freezing point, in degrees Celsius or Fahrenheit, of aqueous solutions of commercial ethylene and propylene glycol-based coolant. A durable hand-held refractometer is available that reads the freezing point, directly, in degrees Celsius or Fahrenheit, when a few drops of engine coolant are properly placed on the temperature-compensated prism surface of the refractometer. This refractometer is for glycol and water solutions, and is not suitable for other coolant solutions.

4.2 The hand-held refractometer should be calibrated before use (see Section 7).

4.3 Care must be taken to use the correct glycol freezing point scale for the glycol type being measured. Use of the wrong glycol scale can result in freezing point errors of 18 and more degrees Fahrenheit.

[1] This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants and Related Fluids and is the direct responsibility of Subcommittee D15.03 on Physical 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.

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

4.4 Ethylene glycol/propylene glycol mixtures will result in inaccurate freezing point measurements using either freezing point scale.

5. Interferences

5.1 Interference can occur if the mixture is contaminated or if the prism surface is not clean. The presence of other glycols such as diethylene glycol in small amounts will not cause interference.

6. Apparatus

6.1 The hand-held critical angle refractometer is a rugged die-cast portable instrument that is covered with a high-impact plastic to minimize damage to the eyepiece lens if dropped. A polished glass prism is opposite the viewing end. A hinged plastic cover is moved over the prism (sampling end) to allow for even sample distribution and prevent liquid sample spillage during the test. No eyepiece or prism adjustments are required for sample testing.

6.2 The telescopic recessed eyepiece is located at one end and the graduated, translucent prism on the opposite end (see Fig. 1).

7. Calibration

7.1 Calibration of these coolant testers should periodically be verified by testing a water sample in accordance with the procedure outlined in Section 8.

7.2 If the sample tested deviates from 0°C (+32°F) the coolant tester is out of calibration and should be recalibrated.

7.3 This calibration test is best performed with the coolant tester and water sample at room temperature. If the instrument used is designed to be automatically temperature compensated, work within the stated temperature-compensated range.

8. Procedure

8.1 *Cleaning*—Before using, swing back the plastic cover at the slanted end of the tester exposing both the measuring window and the bottom of the plastic cover. *Wipe both clean and dry* with tissue or clean soft cloth. Close the plastic cover (see Fig. 2).

8.2 *Testing Coolant Solution*—Commercial instruments are usually equipped with a small suction pipet for sampling solutions. This tube should be used to remove a sample (from below the coolant surface) and eject a few drops on the measuring window (see Fig. 3). Flush suction pipet with test solution before withdrawing sample for testing.

8.3 *Readings:*

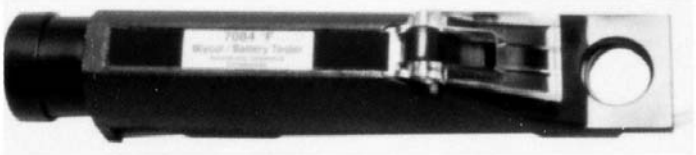


FIG. 1 Hand-Held Critical Angle Refractometer

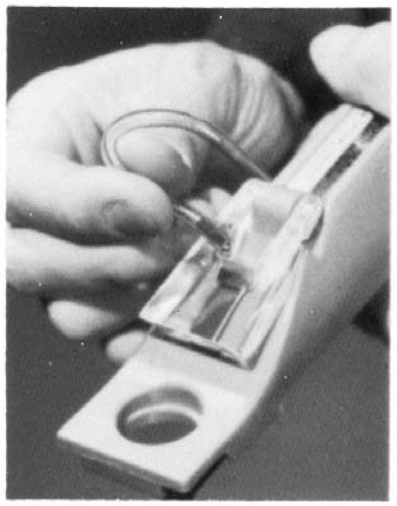


FIG. 2 Cleaning

8.3.1 Point the instrument toward any light source (for example, a headlight) and look into the eyepiece (Fig. 4).

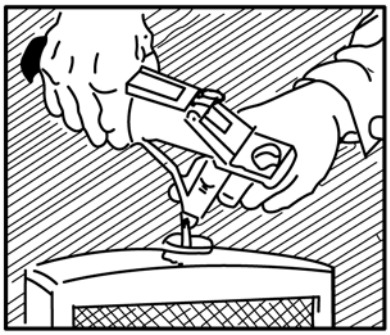
8.3.2 The freeze point protection is the point where the dividing line between light and dark (edge of the shadow) crosses the scale; read the scale marked for the type of coolant being tested (see Fig. 5).

NOTE 2—Tester temperature scales are reversed from standard thermometer scales. Below zero readings are located on upper half of scale.

8.3.3 A little experience is required to obtain the best contrast between the light and dark portions of the scale. Tilt the instrument towards the light source until best results are obtained.

NOTE 3—Refractometer freezing point scales are available in °C and °F for both propylene glycol and ethylene glycol-base coolants.

8.3.4 If the *edge of the shadow* is not sharp, the measuring surfaces were not sufficiently well cleaned or dried or an insufficient amount of coolant sample was used. Clean and dry the window. Conduct a new test.



NOTE 1—Do not remove clear plastic pump from tester. Release tip of pump from tester housing and insert into radiator filler neck or coolant reservoir tank. Be sure to insert well below level of fluid. Press and release bulb to draw up a sample of coolant. Eject the liquid to flush the tube. Repeat filling to obtain sample. Bend plastic tube around tester so that tip can be inserted in cover plate opening. Eject a few drops of coolant onto measuring surface by pressing bulb. Ensure that the sample is not contaminated with oil.

FIG. 3 Sampling

FIG. 4 Reading

NOTE 4—Oil contamination will reduce the sharpness of the dividing line.

8.3.5 A completely dark scale indicates insufficient coolant sample was used. Completely light scale indicates that the coolant freezing point is below the scale range.

8.3.6 There is a danger of the loss of water vapor from the mixture, due to the small amount of sample required, if the solution is sampled at elevated temperatures. Under these circumstances the reading should be taken immediately. More accurate readings are obtained when testing at ambient temperature.

9. Precision and Bias

9.1 The precision of this test method is based on an interlaboratory study conducted in 2011. Twelve laboratories tested four different materials for two different properties. Each "test result" represents an individual determination. Each laboratory reported triplicate test results for each analysis. Practice E691 was followed for the design and analysis of the data; the details are given in RR:D15-1032.[3]

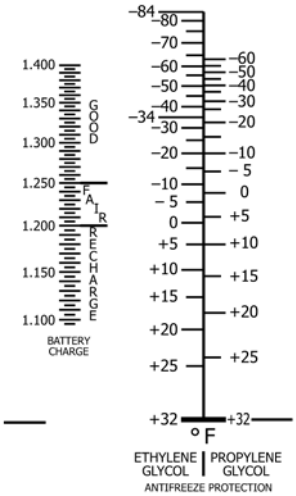


FIG. 5 Tester Temperature Scale

9.1.1 *Repeatability Limit, r*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the *r* value for that material; *r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

9.1.1.1 Repeatability limits are listed in Tables 1 and 2.

9.1.2 *Reproducibility Limit, R*—Two test results shall be judged not equivalent if they differ by more than the *R* value for that material; *R* is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

9.1.2.1 Reproducibility limits are listed in Tables 1 and 2.

9.1.3 The above terms (*repeatability limit* and *reproducibility limit*) are used as specified in Practice E177.

9.1.4 Any judgment in accordance with statements 9.1.1 and 9.1.2 would have an approximate 95% probability of being correct.

9.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

9.3 The precision statement was determined through statistical examination of 288 results, from twelve laboratories, on four different materials.

10. Keywords

10.1 engine coolants; freezing point; refractometer

[3] Supporting data are available from ASTM Headquarters. Request RR:D15-1032.

TABLE 1 Ethylene Glycol (EG), %

Material	Average, $\bar{\chi}^A$	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
Sample No. 1: Ethylene Glycol in Water	50.50	0.00	1.00	0.00	2.80
Sample No. 2: Conventional Fully Formulated EG	49.53	0.17	0.63	0.47	1.75
Sample No. 3: ELC #1	50.44	0.17	0.87	0.47	2.43
Sample No. 4: ELC #2	51.58	0.29	0.83	0.81	2.32

[A]The average of the laboratories' calculated averages.

TABLE 2 Freeze Point, °C

Material	Average, $\bar{\chi}^A$	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, r	Reproducibility Limit, R
Sample No. 1: Ethylene Glycol in Water	-35.67	0.33	2.09	0.93	5.84
Sample No. 2: Conventional Fully Formulated EG	-32.92	0.47	1.86	1.32	5.21
Sample No. 3: ELC #1	-35.03	0.37	1.85	1.04	5.17
Sample No. 4: ELC #2	-38.44	0.29	2.05	0.81	5.74

[A]The average of the laboratories' calculated averages.

SUMMARY OF CHANGES

Committee D15.03 has identified the location of selected changes to this standard since the last issue (D3321–12) that may impact the use of this standard.

(1) Remove note 3 sole source reference.

(2) Change note 4 to note 3.