



Standard Test Method for Freezing Point of Aqueous Engine Coolants¹

This standard is issued under the fixed designation D 1177; 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 the determination of the freezing point of an aqueous engine coolant solution in the laboratory.

NOTE 1—Where solutions of specific concentrations are to be tested, they shall be prepared from representative samples as directed in Practice D 1176. Secondary phases separating on dilution need not be separated.

NOTE 2—These products may also be marketed in a ready-to-use form (prediluted).

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

D 1176 Practice for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes

E 1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 *Definitions:*

3.1.1 *freezing point*—the temperature at which crystallization begins in the absence of supercooling, or the maximum temperature reached immediately after initial crystal formation in the case of supercooling, or the temperature at which solid crystals, formed on cooling, disappear when the temperature of the specimen is allowed to rise.

¹ This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants 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.

4. Summary of Test Method

4.1 This test method involves the determination of the time-temperature curve prior to freezing and the determination of the horizontal or flattened portion of the freezing curve. The freezing point is taken as the intersection of projections of the cooling curve and the freezing curve. If the solution supercools, the freezing point is the maximum temperature reached after supercooling.

5. Significance and Use

5.1 The freezing point of an engine coolant indicates the coolant freeze protection.

5.2 The freezing point of an engine coolant may be used to determine the approximate glycol content, provided the glycol type is known.

6. Apparatus

6.1 *Freezing Point Apparatus*, shown assembled in Fig. 1, consisting of the following:

6.1.1 *Cooling Bath*, in which the refrigerant is contained, consisting of a standard 1.9-L (2-qt) Dewar flask. The flask may be silvered or unsilvered, and is supported in a close-fitting container. A pad of glass wool is placed in the bottom of the flask to protect it from damage by tip of freezing tube.

6.1.2 *Freezing Tube*³ consisting of a 200-mL (6.8-oz.) unevacuated, unsilvered Dewar flask. The tube is closed by a cork having a central hole for the thermocouple or thermometer, a second hole placed to one side for passage of the stirring rod, and a third hole for introducing wire for seeding at appropriate time.

6.1.3 *Stirring Mechanism*, consisting of a five-coil stirrer formed of stainless steel wire 1.6 mm ($1/16$ in.) in diameter. The coils are so spaced that, in the extreme upward position during operation, no coils are exposed above the surface of the sample. The stirrer is agitated by means of an ordinary windshield wiper motor or other motor devices, operating through suitable linkages so as to provide linear motion of the

³ For routine work, a tube with a seeding tip as described in the paper by R. E. Mallonee and F. L. Howard, "The Determination of Freezing Point of Engine Antifreeze," in the February 1951 issue of the *ASTM Bulletin* may be used. (See Fig. 2.)

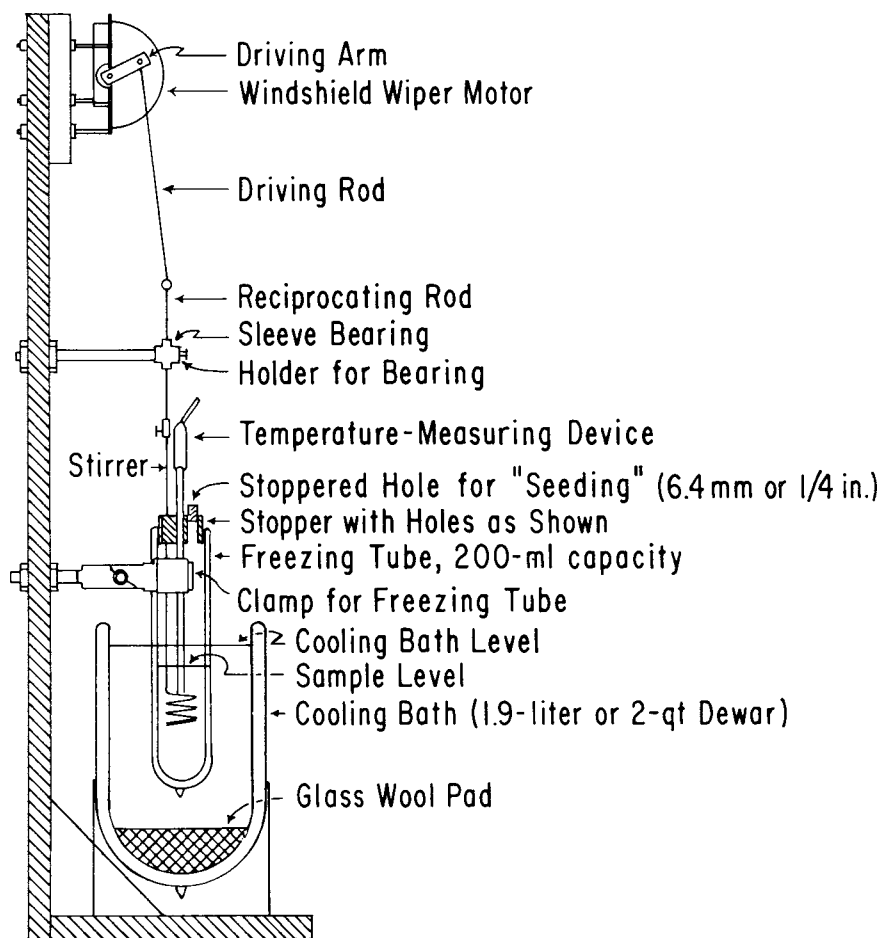


FIG. 1 Assembly of Freezing Point Apparatus

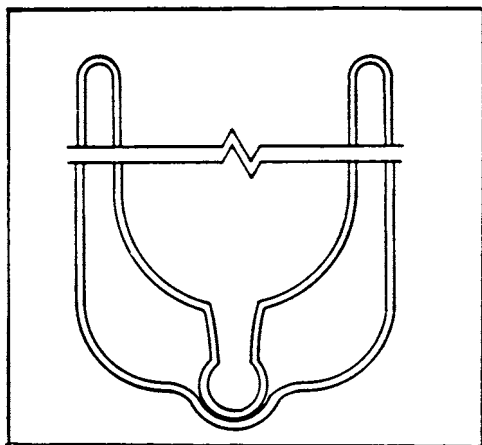


FIG. 2 Bottom of Freezing Tube Showing Seeding Tip

stirrer. The length of the stroke is adjusted so that the coil just clears the bottom of the freezing-point tube at low point of the stroke.

6.1.4 *Temperature Measurement*—A resistance thermometer or a multi-junction copper-constantan thermocouple may be used with suitable measuring instruments, providing these give an over-all sensitivity of 0.1°C (0.2°F). The instrument shall be calibrated before each series of determinations by

using suitable reference standards. Platinum resistance thermometers have been adopted as a standard by the National Institute of Standards and Technology (NIST) and are recommended for this standard.

NOTE 3—ASTM Coolant Freezing Point Temperature Measuring Instruments having a range from -37 to $+2^{\circ}\text{C}$ (-35 to $+35^{\circ}\text{F}$) or -54 to -15°C (-65 to $+5^{\circ}\text{F}$) and conforming to the requirements for Thermometers 75F or 76F as prescribed in Specifications E 1, may be used where less accuracy is acceptable provided reference standards are used for calibration purposes.

7. Refrigerant

7.1 The refrigerant shall consist of solid carbon dioxide in alcohol or in other suitable bath liquids.

NOTE 4—A layer of dry ice, at least 13 mm ($\frac{1}{2}$ in.) thick, must be maintained in the bottom or on the top of the cooling bath during a determination, depending on the bath liquid used. Adequate precautions should be taken against fire hazards or toxic effects of bath liquids, or both.

7.2 Liquid nitrogen may be used as the refrigerant when the freezing point of the coolant is -46°C (-50°F) or lower. (**Warning**—The liquid nitrogen should be discarded after each day's use to avoid the possibility of explosion due to dissolved oxygen and inadvertent mixing with organic coolant materials.)

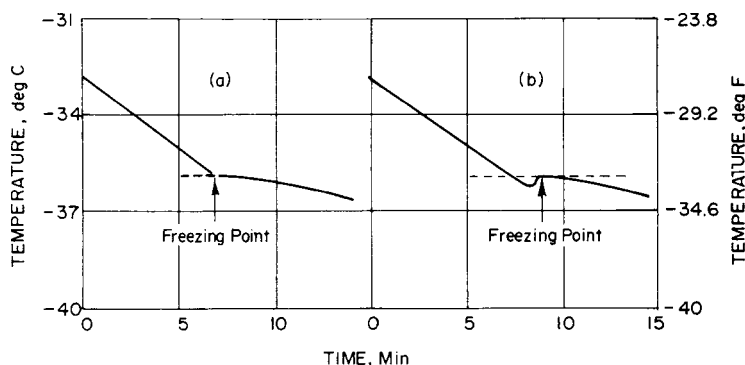


FIG. 3 Time-Temperature Cooling Curves for Determining the Freezing Point of an Engine Coolant

8. Procedure

8.1 Assemble the apparatus as shown in Fig. 1, with no refrigerant and no sample of coolant yet in place. Check the operation of the stirring mechanism after assembly to be sure that all parts operate freely.

8.2 Fill the Dewar flask surrounding the freezing tube with the refrigerant liquid, adding pieces of solid carbon dioxide from time to time to maintain conditions mentioned in Note 4. Temporarily remove the stopper from the freezing tube and introduce 75 to 100 mL (2.65 to 3.4 oz.) of the sample.

NOTE 5—The sample may be precooled to approximately 8°C (15°F) above the expected freezing point before introducing it into the freezing tube.

8.3 Start the stirrer and adjust it to operate at 60 to 80 strokes per min (Note 6). As soon as stirring is begun, observe and record the temperature at regular intervals of time. As the expected freezing point is approached, the time intervals should be quite short, 15 s if possible. At the expected freezing point, seeding should be started to prevent supercooling. This can be accomplished by introducing a wire which has a small portion of the solution being tested frozen on its tip. It is most convenient to freeze this solution in a small test tube inserted directly into the cooling bath.

NOTE 6—A stroke is considered as a complete cycle of one upward and one downward motion of the stirrer.

NOTE 7—The cooling rate must be less than 1°C (2°F)/min at the time the solution is seeded.

8.4 Continue temperature readings at regular intervals for at least 10 min after the apparent freezing point.

9. Report

9.1 Plot the observed temperature against time on rectangular coordinate paper. Where the curve shows a definite flat or plateau during freezing, the freezing point is taken as the intersection of projections of the cooling curve and the flat or horizontal plateau portion of the freezing curve (see Fig. 3(a)). If the solution supercools, the freezing point is the maximum temperature reached immediately after supercooling (see Fig. 3(b)).

NOTE 8—The amount of supercooling should be held to a minimum. If the supercooling exceeds 1°C (2°F) the test should be rejected.

10. Precision and Bias

10.1 Precision and Bias results are as follows:⁴

	°C	°F
Repeatability of Test for a Given Analyst	0.17	0.31
Intermediate Precision of Test for a Given Laboratory	0.88	1.58
Reproducibility of Test Across Laboratories	1.30	2.34

11. Keywords

11.1 aqueous engine coolants; engine coolants; engine coolants; freezing point

⁴ A research report is available from ASTM headquarters. Request RR:D15-1010.

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