Designation: D1177 – 22

Standard Test Method for Freezing Point of Aqueous Engine Coolants[1]

This standard is issued under the fixed designation D1177; 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 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 D1176. 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 standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1176 Practice for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes

E1 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.

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 or glycerin 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 the 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 resistance 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.

[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.

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

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

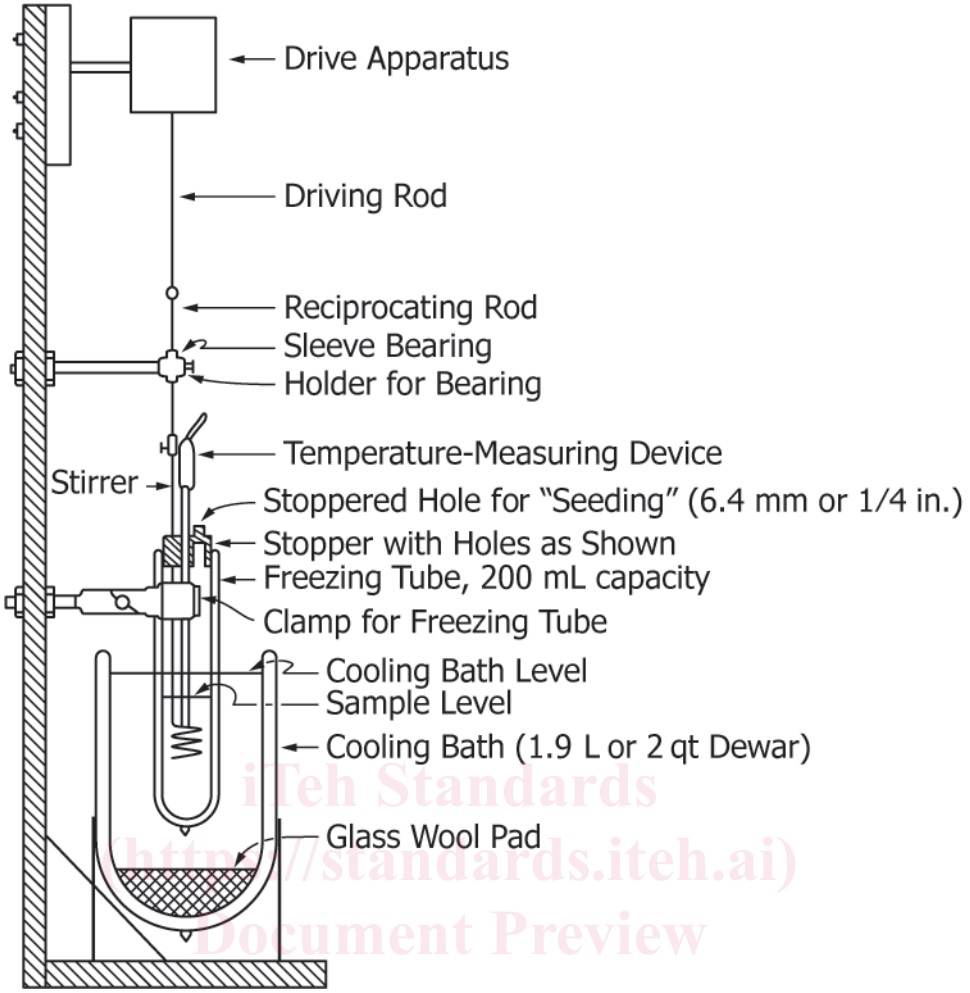

FIG. 1 Assembly of Freezing Point Apparatus

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 to provide linear motion of the 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 overall accuracy of ±0.2 °C (0.4 °F) or an ASTM Coolant Freezing Point Temperature Measuring Instrument conforming to the requirements in Specification E1 for Thermometer 75F, having a range from −37 °C to +2 °C (−35 °F to +35 °F) with a max scale error of ±0.3 °C (0.5 °F) or Thermometer 76F, having a range from −54 °C to −15 °C (−65 °F to +5 °F) with a max scale error of ±0.5 °C (1.0 °F). Platinum resistance thermometers have been adopted as a standard by the National Institute of Standards and Technology (NIST) and are recommended for this standard. If a result is in question, the resistance thermometer or a multi-junction copper-constantan thermocouple is the preferred test method.

7. Refrigerant

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

7.2 Liquid nitrogen may be used as the refrigerant especially 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.)

NOTE 3—A layer of dry ice, at least 13 mm (½ 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. When using liquid nitrogen, add as required to ensure the stirrer remains below the liquid level throughout the course of the test.

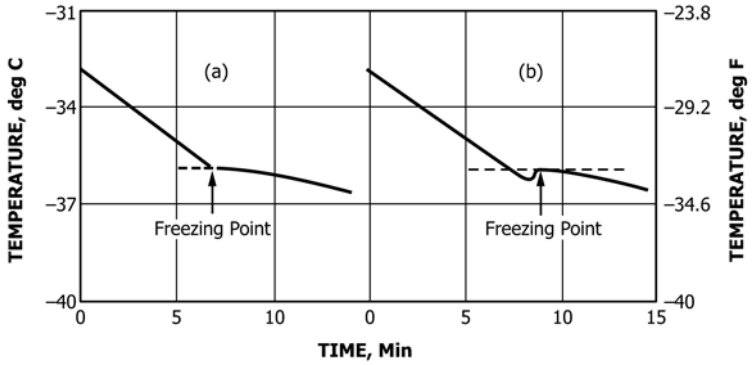

FIG. 2 Time-Temperature Cooling Curves for Determining the Freezing Point of an Engine Coolant

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, liquid nitrogen or by adding pieces of solid carbon dioxide from time to time to maintain conditions mentioned in Note 3. Temporarily remove the stopper from the freezing tube and introduce 75 mL to 150 mL (2.65 oz to 5.1 oz) of the sample.

NOTE 4—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 strokes per min to 80 strokes per min (Note 5). As soon as stirring is begun, observe and record the temperature at regular intervals of time. Electronic data acquisition systems may also be used in place of manual observation and recording. As the expected freezing point is approached, the time intervals shall be 10 s to 15 s. At the expected freezing point, seeding shall be started to prevent supercooling. This shall be accomplished by introducing a wire which has a small portion of the solution being tested frozen on its tip. It is convenient to freeze this solution in a small test tube inserted directly into the cooling bath. The cooling rate shall be less than 1 °C (2 °F)/min at the time the solution is seeded.

NOTE 5—A stroke is considered as a complete cycle of one upward and one downward motion of the stirrer.

8.4 Continue temperature readings at regular intervals for at least 5 min after the apparent freezing point.

9. Report

9.1 Report the following information:

9.1.1 If the temperature-measuring instrument does not automatically plot temperature versus time, manually plot the observed temperature against time. 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. 2(*a*)). If the solution supercools, the freezing point is the maximum temperature reached immediately after supercooling (see Fig. 2(*b*)).

NOTE 6—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[4]

10.1 Precision and Bias results are as follows:

	°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; freezing point

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D15-1010. Contact ASTM Customer Service at service@astm.org.