



Designation: **D6660 – 01 (Reapproved 2007) D6660 – 01 (Reapproved 2014)^{ε1}**

Standard Test Method for Freezing Point of Aqueous Ethylene Glycol Base Engine Coolants by Automatic Phase Transition Method¹

This standard is issued under the fixed designation D6660; 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.

^{ε1} NOTE—Units information was corrected and Note 4 was changed to a warning statement and included in Section 7.3 editorially in July 2014.

1. Scope

1.1 This test method covers the determination of the freezing point of an aqueous engine coolant solution.

1.2 This test method is designed to cover ethylene glycol base coolants up to a maximum concentration of 60 % (v/v) in water; however, the ASTM interlaboratory study mentioned in 12.2 has only demonstrated the test method with samples having a concentration range of 40 to 60 % (v/v) water.

NOTE 1—Where solutions of specific concentrations are to be tested, they shall be prepared from representative samples as directed in Test Method D1176. Secondary phases separating on dilution need not be separated.

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

1.3 The values stated in SI units are to be regarded as the standard. The values given in parenthesis are for information only. No other units of measurement are included in this 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. Some specific hazards statements are given in 7.3.*

2. Referenced Documents

2.1 *ASTM Standards:*²

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

D1177 Test Method for Freezing Point of Aqueous Engine Coolants

D3306 Specification for Glycol Base Engine Coolant for Automobile and Light-Duty Service

D6210 Specification for Fully-Formulated Glycol Base Engine Coolant for Heavy-Duty Engines

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *automatic phase transition method, n*—in this standard, the procedures of automatically cooling an engine coolant sample until solid crystals appear, followed by controlled warming and recording the temperature at which the crystals redissolve into the liquid phase.

3.1.2 *freezing point, n*—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.

3.1.3 *peltier device, n*—a solid state thermoelectric device constructed with dissimilar semiconductor materials, configured in such a way that it will transfer heat to and away from a test specimen dependent on the direction of electric current applied to the device.

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.

4. Summary of Test Method

4.1 A specimen is cooled by a Peltier device while continuously being illuminated by a light source. The specimen is continuously monitored by an array of optical detectors for the first formation of crystals. Once the crystals are formed, the specimen is then warmed at controlled rates until all the crystals return to the liquid phase. The detectors are sufficient in number to ensure that any crystals are detected. The specimen temperature at which the crystals return to the liquid phase is recorded by the temperature sensor as the freezing point.

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.

5.3 Freezing point as measured by Test Method D1177 or approved alternative method is a requirement in Specifications D3306 and D6210.

5.4 This test method provides results that are equivalent to Test Method D1177 and expresses results to the nearest 0.1°C with improved reproducibility over Test Method D1177.

5.5 This test method determines the freezing point in a shorter period of time than Test Method D1177.

5.6 This test method removes most of the operator time and judgement required by Test Method D1177.

6. Apparatus

6.1 *Automatic Apparatus*³—The apparatus described in this method consists of a test chamber controlled by a microprocessor that is capable of cooling and heating the test specimen, optically observing the appearance and disappearance of solid crystals and recording the temperature of the specimen.

6.2 The apparatus shall be equipped with a specimen cup, optical detector array, light source, digital display, Peltier device and a specimen temperature measuring device.

6.3 The temperature measuring device in the specimen cup shall be capable of measuring the temperature of the test specimen from -80°C to +50°C at a resolution of 0.1°C.

6.4 The apparatus shall be equipped with fittings to permit the circulation of liquid cooling media to remove heat generated by the Peltier device and other electronic components of the apparatus.

7. Reagents and Materials

7.1 *Cooling Media*—Liquid heat exchange media to remove the heat generated by the Peltier device and other electronic components from the apparatus.

NOTE 3—Some apparatus are designed to use tap water as cooling media to bring specimen temperature to -60°C. To achieve cooling of specimen to -80°C, provide circulation of liquid cooling media at -30°C or lower to the apparatus. Refer to manufacturer's operating instructions on the relationship between cooling media temperature and the minimum specimen temperature.

7.2 *Adjustable Volume Pipette*,⁴ capable of dispensing 0.15 ± 0.01 ml of sample.

7.3 *Cotton Swabs*⁵—Plastic or paper shaft cotton swabs to clean the specimen cup. (**Warning**—The use of swabs with wooden shafts may damage the mirrored surface of the specimen cup.)

NOTE 4—**Caution:** The use of swabs with wooden shafts may damage the mirrored surface of the specimen cup.

8. Preparation of Apparatus

8.1 Install the analyzer for operation in accordance with manufacturer's instructions.

8.2 Make liquid cooling media connections and ensure that they do not leak.

8.3 Turn on the liquid cooling media.

8.4 Turn on the main power switch of the analyzer. After the automatic self diagnostics start-up sequence is completed, the instrument will display a "READY" message.

³ The following instrument has been found suitable for use in this test method: Phase Technology Freezing Point Analyzer model series 70 and 70V. The sole source of supply of the apparatus known to the committee at this time is Phase Technology, 1168 Hammersmith Gate, Richmond, B.C. Canada, V7A 5H8. 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 A suitable pipette is an Eppendorf pipette.

⁵ Suitable cotton swabs are Q-tips or equivalent with paper or plastic shafts.

9. Calibration and Standardization

- 9.1 Ensure that all of the manufacturer's instructions for calibrating, checking and operating the apparatus are followed.
- 9.2 A sample with a mutually agreed upon freezing point can be used to verify performance of the apparatus.

10. Procedure

- 10.1 Open the test chamber lid and clean the specimen cup inside the test chamber with a cotton swab.
- 10.2 Use the pipette to deliver $0.15 \text{ ml} \pm 0.01 \text{ ml}$ of specimen into the specimen cup. Clean the specimen out of the cup by using a cotton swab. The cup should be cleaned to the point where no visible droplets of specimen remain in the cup.
- 10.3 Repeat step 10.2.
- 10.4 Carefully measure $0.15 \text{ ml} \pm 0.01 \text{ ml}$ of specimen into the specimen cup.
- 10.5 Close and lock the test chamber lid.
- 10.6 Push the "RUN" button located on the front panel of the apparatus. The specimen is cooled by the Peltier device while the appearance of solid crystals is continuously monitored by the optical detectors. The temperature of the specimen is continuously monitored and displayed on the front panel of the apparatus. Once the crystals are detected, the specimen is then warmed until all the crystals re-dissolve into the liquid phase. The measurement is automatically terminated once the freezing point is detected.
- 10.7 When the measurement is complete the freezing point value per Test Method D6660 will be displayed on the front panel of the apparatus.
- 10.8 Unlock and open the test chamber lid and clean the specimen out of the specimen cup with a cotton swab.

11. Report

- 11.1 Report the temperature recorded in 10.7 as the freezing point, Test Method D6660.

12. Precision and Bias

12.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory⁶ test results 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 0.6°C (1.1°F) only in one case in twenty.

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 the normal and correct operation of the test method, exceed 0.8°C (1.4°F) only in one case in twenty.

12.2 *Relative Bias*—The automatic method displayed a mean bias of 0.67°C relative to the manual procedure, Test Method D1177. It is not statistically significant at the 95 % confidence level.

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The precision statements were derived from a 1999 interlaboratory cooperative test program. Participants analyzed ~~5~~ five sample sets comprised of ethylene glycol base coolant the concentration range of 40 to 60 volume in 5 % volume increments. Eight laboratories participated with the automatic phase transition apparatus and seven participated with the manual D1177 test method. The interlaboratory program was conducted double blind. Two analysts measured different sets of samples by each method at seven of the laboratories. One laboratory performed only the automatic method. Each laboratory received two sets of randomized samples labelled A through E and 1 to 5. A total of ~~twenty five~~ 25 repeat measurements were distributed over the laboratories. The precision statistics were compiled and calculated based on the 0.1°C resolution offered by the automatic phase transition method. Information on the types of samples and their respective average freezing point is contained in the research report.

13. Keywords

- 13.1 aqueous engine coolants; crystals; engine coolants; freezing point; Peltier; thermoelectric

⁶ The results of the 1999 Interlaboratory Cooperative Test Program are available from ASTM Headquarters. Request ASTM RR:D15-1020.