Designation: D 6660 – 01

Standard Test Method for Freezing Point of Aqueous Ethylene Glycol Base Engine Coolants by Automatic Phase Transition Method[1]

This standard is issued under the fixed designation D 6660; 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 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 D 1176. 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.

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:*

D 1176 Test Method for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes[2]

D 1177 Test Method for Freezing Point of Aqueous Engine Coolants[2]

D 3306 Specification for Glycol Base Engine Coolant for Automobile and Light Duty Service[2]

D 6210 Specification for Fully Formulated Ethylene Glycol Base Engine Coolant for Heavy Duty Engines[2]

3. Terminology

3.1 *Definitions:*

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.

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 D 1177 or approved alternative method is a requirement in Specifications D 3306 and D 6210.

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[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] *Annual Book of ASTM Standards,* Vol 15.05.

5.4 This test method provides results that are equivalent to Test Method D 1177 and expresses results to the nearest 0.1°C with improved reproducibility over Test Method D 1177.

5.5 This test method determines the freezing point in a shorter period of time than Test Method D 1177.

5.6 This test method removes most of the operator time and judgement required by Test Method D 1177.

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.

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.

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 ml ± 0.01 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 ml ± 0.01 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 D 6660 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 D 6660.

12. Precision and Bias

12.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory[6] 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 D 1177. It is not statistically significant at the 95 % confidence level.

[3] The following instrument has been found suitable for use in this test method: Phase Technology Freezing Point Analyzer model series 70 and 70V available from Phase Technology, i 1168 Hammersmith Gate, Richmond, B.C. Canada, V7A 5H8.

[4] A suitable pipette is an Eppendorf pipette.

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

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

The precision statements were derived from a 1999 interlaboratory cooperative test program. Participants analyzed 5 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 D 1177 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 though E and 1 to 5. A total of twenty five 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

ANNEX

(Mandatory Information)

A1. APPARATUS

A1.1 *Test Chamber*, comprised of optical detectors, lens, light source, specimen cup, temperature sensor, Peltier device and heat sink arranged in a configuration as shown in Fig. A1.1. The lid of the test chamber can be opened to allow cleaning of specimen cup and introduction of new specimen. Once closed and locked, the chamber becomes airtight. An O-ring is used to seal the mating surfaces between the lid and the rest of the chamber. The test chamber wall is made of black-colored metal and plastic components to minimize light reflection.

A1.1.1 *Specimen Cup*, comprised of black plastic wall and a highly polished metal bottom. The polished surface of the bottom serves as a reflective surface for light. The transfer of heat to and away from the specimen, through the metal bottom, is controlled by the Peltier device.

A1.1.2 *Temperature Sensor*, reading to 0.1°C, permanently embedded into the bottom of the specimen cup and positioned less than 0.1 mm below the top surface of the cup bottom. This temperature sensor, which is made of a single strand platinum, provides accurate measurement of the specimen temperature.

A1.1.3 *Peltier Device*, capable of controlling the specimen temperature over a wide range. The range varies depending on the model series. During specimen cooling, heat is transferred from the top of the device to the bottom. Since the top is in thermal contact with the bottom of the specimen cup the specimen will be chilled. The bottom of the Peltier device is in

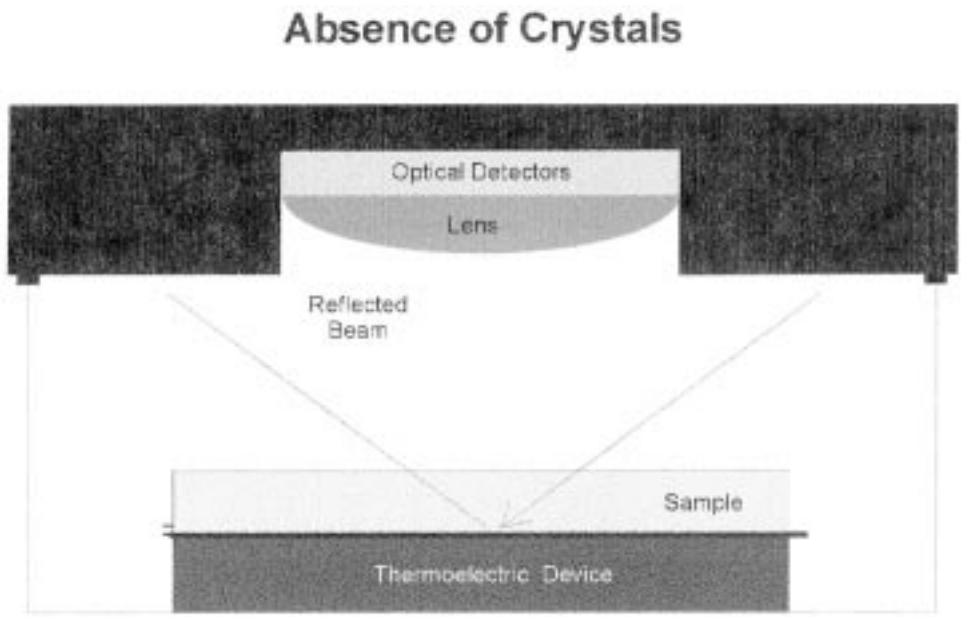

FIG. A1.1 Schematic of Test Chamber

thermal contact with the heat sink, where heat is dissipated to the cooling media. During specimen warming, the reverse process will take place.

A1.1.4 *Light Source*, to provide a beam of light with a wavelength of 660 ± 10 nm. The light source is positioned such that it provides an incident beam (Fig. A1.1) impinging onto the specimen at a specified angle. The light is reflected from the polished bottom of the specimen cup. When the specimen is a homogeneous liquid, the reflected beam impinges onto the chamber lid, which is black in color. The reflected light is then absorbed by the black surface. When solid crystals appear in the specimen, the reflected beam is scattered by the solid liquid phase boundaries. A significant amount of scattered light impinges onto the lens (Fig. A1.2)

A1.1.5 *Optical Detectors* positioned above the lens to monitor the clarity of the specimen. The distance between the optical detectors and the lens is adjusted such that the image of the specimen is projected onto the light sensitive surface of the optical detectors. Sufficient optical detectors are used to cover the image area.

A1.2 *Apparatus Exterior Interface* is composed of several displays and buttons as shown in Fig. A1.3 (the exact layout of the displays and buttons may vary slightly depending on the model series).

A1.2.1 *Message Display* provides information on the status of the apparatus. It displays a READY message when the apparatus is idle and no fault is found. At the end of a test, the result is displayed. It displays a diagnostic message if a fault is detected in any of the major components of the apparatus. Detailed explanation of the diagnostic messages are available in the manufacturer's users' manual.

A1.2.2 *Specimen Temperature Display* gives an update of the specimen temperature, recorded to 0.1 °C, every 2 s.

A1.2.3 *Light Signal Display* gives an update of the scattered light level received by the optical detectors every 2 s. This information is used by service personnel for troubleshooting purposes.

A1.2.4 *RUN Button* allows the operator to start the measurement sequence once the specimen is put inside the test chamber.

A1.2.5 *RESET Button* allows the operator to stop the measurement sequence. Upon pressing this button, the apparatus will immediately stop the measurement sequence and warm the specimen to about 20°C.

NOTE A1.1—A full description, installation, set up instructions and maintenance instruction, are contained within the manufacturer's manual supplied with each instrument and filed at ASTM Headquarters. Request RR:D15–1021.

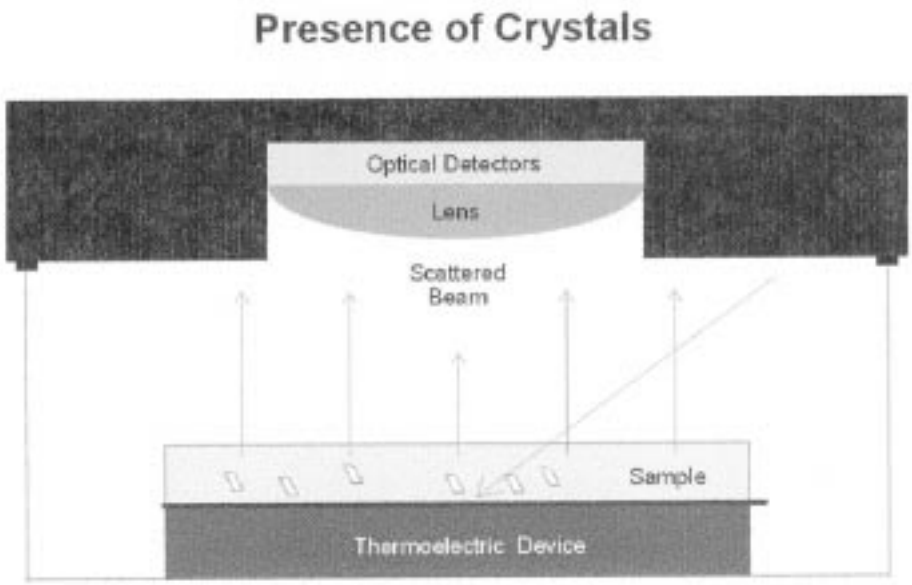

FIG. A1.2 Detection of Crystal Formation

FIG. A1.3 Apparatus Exterior