Designation: D 1176 – 98 (Reapproved 2002)

Standard Practice for
Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes[1]

This standard is issued under the fixed designation D 1176; 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 practice covers information on sampling and preparing solutions of engine coolants and antirusts (Notes 1 and 2).

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are approximate equivalents given 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.*

NOTE 1—Antirust as referred to in this practice covers products intended for use in automotive/light-duty and heavy-duty applications in which water is used as the cooling medium.

NOTE 2—Antirust products are available in liquid and solid form. Solids should be handled as outlined in Annex A1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1122 Test Method for Relative Density of Engine Coolant Concentrates and Engine Coolants By the Hydrometer[2]

D 1193 Specification for Reagent Water[3]

D 5931 Test Method for Density and Relative Density of Engine Coolant Concentrates and Aqueous Engine Coolants by Digital Density Meter[2]

3. Significance and Use

3.1 This practice is used commonly by vehicle service or laboratory personnel for sampling and preparing aqueous solutions of engine coolants or antirust for further evaluation and testing.

3.2 This practice shall be followed when sampling and preparing aqueous solutions of products for evaluation in ASTM test methods.

3.3 Care must be taken to ensure that a representative sample of product is taken.

3.4 This practice describes several different test methods. The methods to be followed should be agreed to by the supplier and the customer.

4. Sampling Concentrated Liquid Products

4.1 When received, the material will preferably be in a sealed, marked container. Allow the container to stand at room temperature (not below 20°C) before attempting to obtain a sample. The container and its contents shall be shaken well before sampling (see Note 3). Any signs of solution separation should be noted and appropriate samples obtained by using a decanting or pipeting technique. Some samples that contain multiple phases may require the use of a separatory funnel to isolate a particular liquid layer or solids.

NOTE 3—In the case of larger containers such as those with a nominal volume of 20 or 200 L (5 or 55 gal), the contents should be mixed thoroughly with a stirrer for at least 5 min to ensure a homogeneous mixture. The stirrer should be capable of dispersing any separated solids and supernatant phases without drawing air into the mixture.

4.2 Remove a representative sample by pipeting or siphoning.

5. Separation of Multiphase Products

5.1 If the product contains separated solids or liquids, place a representative sample obtained as described in Section 4 in a separatory funnel. Stopper the funnel and allow to stand for at least 16 h. Separate the various phases as follows:

5.1.1 Remove settled liquids or solids, if present, by allowing them to drain out together with a minimum amount of the major liquid phase.

5.1.2 Then drain the major liquid phase into a suitable container, taking care not to include any supernatant liquid phase.

5.1.3 Finally, drain the supernatant phase into a separate container.

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[1] This practice is under the jurisdiction of ASTM Committee D15 on Engine Coolants and is the direct responsibility of Subcommittee D15.06 on Glassware Performance Tests.

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

[3] *Annual Book of ASTM Standards*, Vol 11.01.

5.1.4 If the solids will not drain out from the separatory funnel, siphon off the various liquid phases in sequence from the top of the funnel.

5.1.5 If the solid does not separate from the liquid phase but remains in suspension, separate by passing a representative sample of the product through an appropriate filter paper, using suction, and transfer the liquid to a storage container.

6. Preparation of Aqueous Solutions

6.1 Concentration is generally expressed as the percentage by volume of the product at a specified temperature, usually 20°C (68°F).

6.2 *Gravimetric Procedure (Weight/Volume %)*:

6.2.1 Determine the relative density of the product at 20/20°C (68/68°F) to the nearest 0.0005 in accordance with Test Method D 1122.

6.2.2 Calculate the weight of the product required to give the volume at 20°C (68°F).

NOTE 4—*Example*—For a 30 % solution and a final volume of 100 mL, a product that has a relative density of 1.0149 at 20/20°C (68/68°F) would require that the following amount be weighed:

$$0.30 \times 100 \times (1.0149 \times 0.99715) = 30.360 \text{ g of product} \tag{1}$$

where:
0.99715 = the density of water at 20°C (68°F).

6.2.3 Weigh the sample in a clean, dry container on a balance at room temperature. The sample shall be weighed to an accuracy of ±0.02 %. Record the weight and transfer the sample to a clean, dry volumetric flask. Care should be taken to ensure that all of the sample is transferred from the weight container by rinsing with Specification D 1193, Type IV water (or ASTM corrosive water, if required by the test). Fill to just below the mark.

6.2.4 Mix the contents of the volumetric flask and bring to 20°C (68°F) by placing the flask in a constant temperature bath set at 20 ± 0.1°C. Adjust the volume to the mark using the specified water when the contents have had a chance to come to temperature. Transfer the solution to the test vessel or storage container.

6.3 *Volumetric Procedure (Volume %)*:

6.3.1 For many applications, sufficient accuracy can be obtained by measuring the sample in a graduated cylinder.

6.3.1.1 Measure the required volume of engine coolant or antirust into a graduated cylinder.

6.3.1.2 Add water of the type to be used for the test to just below the final volume. Mix the solution well. Add enough water of the type to be used for the test to bring the contents of the graduated cylinder to the final volume and mix well again. The test concentration shall be expressed as the ratio of the initial volume of the product to the final volume of the mixture of the product and water, both measured at 20°C.

6.3.2 More accurate concentrations can be prepared by the following procedure:

6.3.2.1 Suspend the sample in a constant temperature water bath set at 20 ± 0.1°C. Allow 15 to 30 min for the sample to equilibrate to this temperature.

6.3.2.2 When the sample has reached bath temperature, transfer the required volume by pipet (see Note 5) to a clean, dry volumetric flask calibrated at 20 ± 0.1°C.

NOTE 5—The pipet should be calibrated using the test solution at 20 ± 0.1°C.

6.3.2.3 Bring the level in the flask almost to the graduated mark with the type water to be used for the test. Mix the contents of the flask by inverting several times, taking care to avoid loss of solution through the stopper. Proceed as directed in 6.2.4.

7. Preparation of Test Solutions that Contain Solids or Liquid Mixtures, or Both, in the Sample to be Tested

7.1 It is difficult to handle and obtain accurate specific gravity measurements on samples containing solids and separated layers. They are generally handled best by following the recommendation outlined in 4.1.

8. Treatment of Mixtures Resulting from Separation of Secondary Phases on Dilution of Product with Water

8.1 If a representative portion of the mixture is desired, proceed as directed in 4.1 before a portion is removed by pipeting or siphoning. Stirring should be continued during removal of the sample.

8.2 If secondary phases are to be separated, proceed as directed in 5.1.

9. Keywords

9.1 antirust; aqueous solutions; engine coolant; gravimetric procedure; sampling; solutions; volumetric procedure

ANNEX

(Mandatory Information)

A1. HANDLING SOLID ANTIRUSTS

A1.1 Antirusts supplied as powder or granules shall be sampled by taking portions from various points of the container. In the case of solids or caked material, the entire package shall be broken up, mixed thoroughly, and quartered, if necessary. The sample shall be dried at 100°C to a constant weight and stored in a desiccator until used. Samples shall not be taken from those portions of the inhibitor in which caking has been caused by absorption of moisture and carbon dioxide through the container.

A1.2 Dried samples shall be weighed without delay to an accuracy of ±0.02 %, placed in a volumetric flask, and diluted to the specified concentration with whatever water is required for the given test according to the procedure for a liquid product.

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