

Designation: D 1287 – 91 (Reapproved 1997)^{€1}

Standard Test Method for pH of Engine Coolants and Antirusts¹

This standard is issued under the fixed designation D 1287; 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.

 ϵ^1 Note—Editorial corrections were made throughout in June 1998.

1. Scope

1.1 This test method covers the determination of the pH of unused engine coolants and antirusts, and used or unused aqueous dilutions of the concentrated products.

Note 1—Antirusts in solid form are not included in this test method. Note 2—It is believed that this procedure gives all the information required for determining the pH of samples of engine coolant or antirust likely to be encountered. Should additional information on pH determinations be needed, reference should be made to Test Method E 70.

1.2 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 Practice for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes²

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode²

3. Summary of Test Method

3.1 A sample, as received or after dilution with a specified volume of distilled water, is placed in a beaker or sample cup and the pH measured with a pH meter and associated glass-saturated calomel electrode pair.

Note 3—The readings of the meter in the case of the concentrated products are not pH values according to the accepted definition, but are apparent pH values which are useful in the interpretation of the condition of the product.

4. Significance and Use

4.1 pH is a measure of the hydrogen ion concentration and

indicates whether an engine coolant, antirust, or a solution of these compounds is acidic, alkaline, or neutral.

4.2 pH is sometimes used for production quality control. It is generally desirable that engine coolants have an alkaline pH.

4.3 pH is not significant from the standpoint of predicting service life. The pH of used engine coolants or antirust solutions is not a dependable indication of either existing effectiveness or remaining life of the solution.

5. Apparatus

5.1 The pH meter, glass electrode, and calomel electrode, shall be as specified in the Annex.

6. Reagents

6.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 Purity of Water— Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity. Distilled water shall be boiled thoroughly, or purged with carbon dioxide-free air, to remove carbon dioxide and shall be protected with a soda-lime tube or its equivalent while cooling and in storage. The pH of the water shall be between 6.2 and 7.2 at 25°C.

6.3 Standard Buffer Solutions—The buffer solutions for calibrating the pH meter and electrode pair shall be prepared from salts sold specifically for use, either singly or in combination, as pH standards. The salts shall be dried for 1 h at 110°C before use, except in the case of borax which shall be used as the decahydrate. The solutions with pH less than 9.5

¹ This test method is under the jurisdiction of ASTM Committee D-15 on Engine Coolants and is the direct responsibility of Subcommittee D15.04 on Chemical Properties.

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² Annual Book of ASTM Standards, Vol 15.05.

³ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.



shall be stored in chemically resistant bottles. The alkaline phosphate solution shall be stored in glass bottles coated on the side with paraffin.

Note 4—Commercially available buffer tablets or prepared buffer solutions may be used.

- 6.4 Potassium Hydrogen Phthalate Buffer Solution (0.05 M, pH = 4.01 at 25°C)—Dissolve 10.21 g of potassium hydrogen phthalate (KHC $_8$ H $_4$ O $_4$) in water, and dilute to 1 L.
- 6.5 Neutral Phosphate Buffer Solution (0.025 M with respect to each phosphate salt, pH = 6.86 at 25°C)—Dissolve 3.40 g of potassium dihydrogen phosphate (KH₂PO₄) and 3.55 g of anhydrous disodium hydrogen phosphate (Na₂HPO₄) in water and dilute to 1 L.
- 6.6 Borax Buffer Solution (0.01 M, pH = 9.18 at 25°C)—Dissolve 3.81 g of disodium tetraborate decahydrate (Na₂B₄O $7 \cdot 10H_2$ O) in water, and dilute to 1 L. Protect this solution from atmospheric carbon dioxide; stopper the bottle at all times except when actually in use.
- 6.7 Alkaline Phosphate Buffer Solution (0.01 M trisodium phosphate, pH = 11.72 at 25°C)—Dissolve 1.42 g of anhydrous disodium hydrogen phosphate (Na₂HPO₄) in 100 mL of a 0.1 M carbonate-free solution of sodium hydroxide and dilute to 1 L with water.
- 6.8 Potassium Chloride Electrolyte— Prepare a saturated solution of potassium chloride (KCl) in water.

7. Sampling

7.1 Sample material in accordance with Practice D 1176.

8. Preparation of Electrode System

- 8.1 Maintenance of Electrodes—Clean the glass electrode at frequent intervals (not less than once every week during continual use) in accordance with the manufacturer's instructions. Drain the calomel electrode at least once each week and fill with KCl electrolyte. Keep the electrolyte level in the calomel electrode above that of the solution being tested at all times. When not in use, immerse the lower halves of the electrodes in distilled water, or the appropriate buffer solution recommended by the manufacturer. Do not immerse the electrodes in the solution being tested for any appreciable period of time between determinations. Although the electrodes are not extremely fragile, they should be handled carefully at all times.
- 8.2 Preparation of Electrodes—Before and after using, wipe the electrode thoroughly with a clean cloth, or a soft absorbent tissue, and rinse with distilled water. Follow the manufacturer's recommendations if further preparation is required. Prior to each pH determination, soak the prepared electrode in distilled water for at least 2 min. Immediately before use, touch the tips of the electrodes with a dry cloth or tissue to remove excess water.

9. Standardization and Testing of Electrode System in pH Units

9.1 The assembly shall always be standardized with one of the standard buffer solutions. A second standard buffer solution shall be used to check the linearity of the response of the electrodes at different pH values and to detect a faulty glass electrode or incorrect temperature compensation. The two buffer solutions selected should bracket the anticipated pH of the solutions being tested.

- 9.1.1 Turn on the instrument, allow it to warm up, and adjust according to the manufacturer's instructions. Immerse the tips of the electrodes in the standard buffer solution chosen and allow sufficient time for the temperature of the buffer solution and the electrodes to equalize. Set the temperature knob at the temperature of the buffer solution. Adjust the standardization or asymmetry potential control until the meter registers a scale reading, in pH units, equal to the known pH of the standardizing buffer solution.
- 9.1.2 Rinse the electrodes with distilled water and touch the tips with a dry cloth or tissue to remove excess water. Immerse the electrodes in a second standard buffer solution. The reading of the meter shall agree with the known pH of the second standard buffer solution within ± 0.05 unit without changing the setting of the standardization of asymmetry potential control. If it does not agree or if the meter gives a slow response time and drifts, the electrode should be cleaned in accordance with the manufacturer's instructions.
- 9.1.3 The presence of a faulty electrode is indicated by failure to obtain a correct value for the pH of the second standard buffer solution after the meter has been standardized with the first. A cracked electrode will often yield pH values that are essentially the same for both standard buffer solutions.

10. Procedure

- 10.1 Make the pH determination on (*I*) the unused concentrated engine coolant or antirust (as received), or (2) any desired dilution, with distilled water, of the concentrated product, or (*3*) used engine coolant or antirust solution. For dilute solutions, suggested engine coolant concentrations are 50 and 33 volume %; antirust solution concentration should be in the range of proposed usage. Prepare solutions as follows: Unused engine coolant or antirust solution samples to be tested should be obtained following the applicable portions of the procedure outlined in Practice D 1176.
- 10.1.1 When a small, insoluble liquid layer is present, remove it prior to testing the unused sample or preparing diluted samples for testing. Using a 100-mL mixing cylinder and a pipet or other suitable measuring device, make up a solution of the concentration desired for testing by measuring the necessary amount of antifreeze or antirust solution into the cylinder and adding sufficient distilled water to bring the solution volume to 100 mL.
- 10.1.2 Thoroughly mix the contents of the cylinder by shaking, and transfer into a 250-mL tall-form spoutless or other suitable beaker.
- NOTE 5—Some pH apparatus is equipped with a sample cup. It is satisfactory to use these cups and the smaller sample required with them, as an alternative to the 100-mL sample and beaker specified.
- 10.2 Prepare the calomel and glass electrodes as directed in Section 8. Immerse the tip of the electrode in the solution and agitate. Where the instrument has a mechanical stirrer, this may be used. Allow the system to come to equilibrium, and record the pH value. Remove and wash the electrodes with distilled water and immerse them in distilled water until the next