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Standard Test Method for pH of Engine Coolants and Antirusts¹

This standard is issued under the fixed designation D1287; 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 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 E70.

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

1.3 *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

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

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 combination electrode or associated glass-saturated calomel electrode pair.

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 D15.04 on Chemical 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.

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 The pH range includes values from 0 to 14. Values from 0 to 7 represent the acidic half of the scale. Values from 7 to 14 represent the alkaline or basic half of the scale. The pH value 7 is considered neutral, as it is neither acidic nor alkaline.

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

4.4 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.

NOTE 4—In pH operation, the meter may be set up to perform one-, two-, or up to five-point standardizations or may be set up to automatically recognize U.S. standard (pH 4, 7 and 10) buffers, NIST/International (pH 1.68, 4.01, 6.86, 9.18 and 12.46) buffers, or a custom user-centered set of buffers, or both.

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

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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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

NOTE 5—Commercially available buffer tablets or prepared buffer solutions may be used.

6.4 Standard Buffer Solution pH = 1.68, (Buffer pH 1.68 from Fisher Scientific FC Part Number 13-620-836) or equivalent could be used.

6.5 *Potassium Hydrogen Phthalate Buffer Solution* (0.05 M, pH = 4.01 at 25 °C)—Dissolve 10.21 g of potassium hydrogen phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$) in water, and dilute to 1 L. Standard Buffer Solution pH = 4.0, Potassium Hydrogen Phthalate (Red, pH 4.01) from Fisher Scientific (FC Part Number: SB 101-500; NIST-SRM 185 g) or equivalent could be used.

6.6 *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 (KH_2PO_4) and 3.55 g of anhydrous disodium hydrogen phosphate (Na_2HPO_4) in water and dilute to 1 L. Standard Buffer Solution pH = 6.86, Potassium Phosphate (pH 6.86 from Fisher Scientific, or NIST-SRM 186 le/lle) or equivalent could be used.

6.7 Standard Buffer Solution pH = 7.0, Potassium Phosphate Monobasic (Yellow, pH 7.00) from Fisher Scientific (FC Part Number: SB 107-500) or equivalent could be used.

6.8 *Borax Buffer Solution* (0.01 M, pH = 9.18 at 25 °C)—Dissolve 3.81 g of disodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) 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. Standard Buffer Solution pH=9.18, Sodium Tetraborate $\times 10 \text{ H}_2\text{O}$ (borax) (pH 9.18) from Fisher Scientific or NIST-SRM 187c) or equivalent could be used.

6.9 Standard Buffer Solution pH=10.00, Potassium Carbonate, Potassium (Blue, Buffer pH 10.00 from Fisher Scientific FC Part Number SB 115-500) or equivalent could be used.

6.10 *Alkaline Phosphate Buffer Solution* (0.01 M trisodium phosphate, pH = 11.72 at 25 °C)—Dissolve 1.42 g of anhydrous disodium hydrogen phosphate (Na_2HPO_4) in 100 mL of a 0.1 M carbonate-free solution of sodium hydroxide and dilute to 1 L with water.

6.11 Standard Buffer Solution pH=12.46, (Buffer pH 12.46 from Fisher Scientific FC Part Number 13-620-837) or equivalent could be used.

6.12 *Potassium Chloride Electrolyte*—Prepare a saturated solution of potassium chloride (KCl) in water.

7. Sampling

7.1 Sample material in accordance with Practice D1176.

8. Preparation of Electrode System

8.1 *Maintenance of Electrodes*—Clean the glass electrode or combination 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.

8.1.1 A combination electrode system can also be used for this test method.

8.2 *Preparation of Electrodes*—Before and after using, wipe the electrode or combination 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 more than one of the standard buffer solutions. A single standardization point serves to establish the electrodes actual zero potential, but the electrode's slope is assumed to be a default or already known value. A pH measurement with one-point standardization should be restricted to a narrow pH range, very near the standardization point. 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.

9.2 Multi-point standardization allows measurements to be performed over a very broad pH range, but required the additional effort of carrying out further standardizations. With multiple buffer points, the meter uses the electrode slope applicable to the pH range of the current sample. This linear segments method is always in pH operation. When standardization by auto recognition of U.S. standard buffers or custom buffers is selected, three buffer points are usable. With auto-recognition of NIST buffers or a combination of U.S. standard, custom, and NIST buffers, or manual entry of buffers, up to five standardization buffers are permitted.

10. Procedure

10.1 Make the pH determination on (1) 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 **D1176**.

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 6—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 determination is made.

NOTE 7—In the event that the glass electrodes become coated with an oil film, as the result of testing two-phase products, it is necessary to remove such film with a suitable solvent, such as for inorganic deposits—wash with EDTA, ammonia, or acids; for grease and similar films—wash with acetone or methanol.

NOTE 8—With silicate-containing engine coolant, it is important to specify that the pH reading be taken either immediately after preparing the dilution or after a specified period of time, such as 30 min. In contrast to an immediate pH, the 30 min value has been shown to be approximately 0.2 units lower and a measurement made after 24 h can be from 0.3 to 0.5 pH units lower.

11. Report

11.1 Report the pH.

12. Precision and Bias

12.1 *Repeatability*—Duplicate results by the same operator should not be considered suspect unless they differ by more than ± 0.1 pH units.

12.2 *Reproducibility*—The results submitted by two or more laboratories should not be considered suspect unless the results differ by more than ± 0.2 pH units.

13. Keywords

13.1 antirusts; engine coolants; pH of engine coolants

ANNEX

(Mandatory Information)

A1. APPARATUS

A1.1 Meter

A1.1.1 A voltmeter or potentiometer that will operate with an accuracy of ± 0.005 V and a sensitivity of ± 0.002 V, over a range of at least ± 0.5 V, when the meter is used with the electrodes specified in **A1.2** and **A1.3**, and when the resistance between the electrodes falls within the range from 0.2 M Ω to 20 M Ω . The meter shall be protected from stray electrostatic

fields so that no permanent change in the meter readings over the entire operating range is produced by touching with a grounded lead, any part of the exposed surface of the glass electrode, the glass electrode lead, the titration stand, or the meter. A desirable apparatus may consist of a continuous-reading electronic voltmeter with specified range, accuracy, and sensitivity, that is designed to operate on an input of less

than 5×10^{-12} A when an electrode system having 1000 M Ω resistance is connected across the meter terminals; that is provided with a metal shield connected to the ground; and is provided with a satisfactory terminal to connect the shielded connection wire from the glass electrode to the meter without interference from the presence of external electrostatic fields.

A1.2 Glass Electrode

A1.2.1 A pencil-type glass electrode 125 mm to 180 mm in length and 8 mm to 14 mm in diameter. The body of the electrode shall be made of a chemically resistant glass tube with a wall thickness of 1 mm to 3 mm. The end dipping into the solution shall be closed with a hemisphere of glass⁴ sealed on the electrode tube and the radius of this hemisphere shall be above 7 mm. The thickness of the glass in the hemisphere shall be great enough so that resistance of the hemisphere is 100 m Ω to 1000 m Ω at 25 °C. The electrode shall contain a reproducible, permanently sealed liquid cell for making electrical connections with the inner surface of the hemisphere. The entire electrical connection, from the sealed contact cell to the meter terminal, shall be surrounded by an electrical shield that will prevent electrostatic interference when the shield is grounded. The shield shall be insulated from the electrical connection by insulating material of the highest quality, such as rubber and glass, so that the resistance between the shield and the entire length of electrical connection is greater than 50 000 M Ω .

A1.3 Calomel Electrode

A1.3.1 A pencil-type calomel electrode 125 mm to 180 mm in length and 8 mm to 14 mm in diameter. This electrode shall be made of glass and shall be provided with an external, removable glass sleeve on the sealed end that is dipped into the titration solution. The glass sleeve shall be 8 mm to 25 mm in length, shall be slightly tapered, and shall be ground to fit the electrode, so that the sealed end of the electrode protrudes 2 mm to 20 mm beyond the sleeve. The ground surface shall be continuous and free of smooth spots. At a point midway

between the extremities of the ground surface, the electrode shall be pierced by a hole or holes 1 mm in diameter. The electrode shall contain the necessary mercury, calomel, and electrical connection to the mercury, all arranged in a permanent manner. The electrode shall be filled almost to capacity with saturated KCl electrolyte and shall be equipped with a stoppered port through which the electrolyte may be replenished. When suspended in the air, and with the sleeve in place, the electrode shall not leak electrolyte at a rate greater than 1 drop in 10 min.

A1.4 Combination Electrode

A1.4.1 Combination electrodes offer the convenience of having the reference and measuring electrodes combined in a single housing. They are offered in a wide variety of configurations to meet most application needs. Basically, the design consists of a glass pH indicating electrode coaxially joined to a silver/silver chloride reference electrode. The glass pH indicating electrode is located at the center of the probe. The outer annular space contains annular space of the probe. The outer annular space contains the silver/silver chloride reference element, electrolyte (4 M KCL saturated with AgCl or gelled 4 M KCl saturated with AgCl) and the junction (ceramic or porous plug). When immersed in a solution, the reference electrode makes contact with the sample through the junction thus completing electrical contact between the reference electrode and pH indicating electrode. Combination electrodes are responsible over the full 0 to 14 pH range.

A1.5 Stirrer

A1.5.1 A variable-speed mechanical stirrer of any suitable type (**Note A1.1**), equipped with a glass, propeller-type stirring paddle. A propeller with blades 6 mm in radius and set at a pitch of 30° to 45° is satisfactory. If electrical stirring apparatus is used, it must be electrically correct and grounded so that connecting or disconnecting the power to the motor will not produce a permanent change in meter reading during the course of the titration.

NOTE A1.1—Magnetic stirrers may be used in place of the mechanically driven types.

⁴ Corning 015 glass has been found satisfactory for this purpose.

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