



Standard Test Method for Hydrogen Ion Concentration of Dry Adhesive Films¹

This standard is issued under the fixed designation D1583; 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 test method covers determination of the hydrogen ion concentration (pH), acidity, or alkalinity, of organic adhesives in the cured dry film form. This test method is not designed to apply to pressure-sensitive adhesives.

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

2. Referenced Documents

2.1 *ASTM Standards:*²

D907 Terminology of Adhesives

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

3. Terminology

3.1 *Definitions*—Many of the terms in this test method are defined in Terminology D907.

4. Summary of Test Method

4.1 Collect samples of the adhesive to be tested with any hardener necessary.

4.2 Dry adhesive films are prepared.

4.3 The adhesive films are ground and mixed with distilled water.

¹ This test method is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.30 on Wood Adhesives

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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.

4.4 The pH is determined using a pH meter in accordance with Test Method E70.

5. Significance and Use

5.1 The pH of an adhesive may be a factor whereby the adherends or the adhesive itself may be adversely affected in time by the acidity or alkalinity.

6. Apparatus

6.1 *pH Meter*, capable of making measurements to ± 0.05 pH.

6.2 *Glass Vial*, capable of properly immersing the electrode of the pH meter when filled with the specified test solution.

6.3 *Distilled Water*, with a pH between 5.6 and 7.0 at $23 \pm 1^\circ\text{C}$ ($73.4 \pm 1.8^\circ\text{F}$).

6.4 *Glass Sheet*, free from scratches, approximately 200 by 250 mm (8 by 10 in.) in size for use in oven.

6.5 *Circulating-Air Oven*, capable of maintaining temperatures of $23 \pm 1^\circ\text{C}$ ($73.4 \pm 1.8^\circ\text{F}$) to $150 \pm 1^\circ\text{C}$ ($302 \pm 1.8^\circ\text{F}$).

7. Sampling

7.1 Except in special cases, take a composite sample when possible from three or more separate containers, chosen at random. Also take samples from containers which appear to be nonrepresentative, and test such samples separately. Place the samples immediately in airtight glass containers filled to prevent excessive air space above the adhesive and transport them to the testing laboratory in these containers. Take precautions to reduce evaporation or drying to a minimum. Thoroughly mix the adhesive in the container if there is a tendency for the materials to separate before a sample is taken. Use the required catalysts and hardeners from the same shipment as the other adhesive components if the components are furnished as a unit by the adhesive manufacturer.

8. Test Specimens

8.1 *Room-Temperature Setting Adhesives*—Use adhesives that form dry films at room temperature and mix in accordance with the recommendations of the adhesive manufacturer. Spread the adhesive on glass or other inert material to which

the adhesive does not adhere to a film thickness of less than 0.51 mm (0.020 in.) and allow it to dry at $23 \pm 1^\circ\text{C}$ ($73.4 \pm 1.8^\circ\text{F}$).

8.2 *Elevated-Temperature Setting Adhesives*—Mix adhesives that require heat to form a dry film in accordance with the recommendations of the manufacturer. Prepare a film of the adhesive on glass or other inert material to which the adhesive does not adhere, and cure it in accordance with the recommendations of the manufacturer. The adhesive film should be less than 0.51 mm (0.020 in.) in thickness. In the absence of specific recommendations for curing the adhesive, place the films on the glass plates in a circulating-air oven at $66 \pm 1^\circ\text{C}$ ($150 \pm 1.8^\circ\text{F}$) until most of the solvent is evaporated; this usually requires 4 h. Then raise the temperature to $150 \pm 1^\circ\text{C}$ ($302 \pm 1.8^\circ\text{F}$) and continue to heat the films until cured. This process usually requires less than 1 h.

8.3 Grind the adhesive specimen so that all the particles pass through a No. 40 (425- μm) sieve.

8.4 Test at least three specimens from each sample.

9. Procedure

9.1 Immediately after grinding, weigh 2 g or a multiple thereof of the ground particles, and place them in a small standard-size glass vial that will permit adequate immersion of the electrode when the 10 mL, or a multiple thereof, of freshly boiled, cooled distilled water is added. Maintain the ratio of powder to water at 1 to 5. Stir the mixture thoroughly. Tightly seal the container with a rubber stopper at all times except when pH determinations are being made. Allow the suspension of ground particles to stand for 72 h at room temperature. Then stir the solution and determine the pH by means of a suitable pH meter. Standardize the pH meter in accordance with the

instructions of the manufacturer of the instrument or, in the absence of such instructions, in accordance with Test Method E70. Repeat the determination at intervals of 24 h thereafter until the difference between consecutive readings is not greater than 0.05 pH units. Record the last readings as the equilibrium film pH value of the adhesive.

9.2 Measure the pH at $23 \pm 1^\circ\text{C}$ ($73.4 \pm 1.8^\circ\text{F}$).

10. Report

10.1 Report the following information:

10.1.1 Complete identification of the adhesive tested including type, catalyst, hardener, source, lot number, condition, and so forth,

10.1.2 The pH of the distilled water used, and

10.1.3 The pH and the time required to reach equilibrium of each test specimen.

11. Precision and Bias

11.1 The standard deviation of the average (based on three specimens) obtained by the same analyst on different days was estimated to be 0.02 pH unit relative at 53 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.06 pH unit.

11.2 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicate determinations) obtained by analysts in different laboratories has been estimated to be 0.04 pH unit at 12 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.12 pH unit.

12. Keywords

12.1 adhesive films; pH

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