



Designation: ~~D4584 – 05 (Reapproved 2010)~~ **D4584 – 05 (Reapproved 2015)**

## Standard Test Method for Measuring Apparent pH of Electrocoat Baths<sup>1</sup>

This standard is issued under the fixed designation D4584; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

- 1.1 This test method covers the measurement of the free hydrogen ion concentration of electrocoat baths and their ultrafiltrates.
- 1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 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:*<sup>2</sup>
  - [D1193 Specification for Reagent Water](#)
  - [D1293 Test Methods for pH of Water](#)
  - [E70 Test Method for pH of Aqueous Solutions With the Glass Electrode](#)
  - [E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals \(Withdrawn 2009\)](#)<sup>3</sup>

### 3. Summary of Test Method

- 3.1 A specimen of a well-agitated electrocoat bath is placed in a stirrer-equipped container and the pH measured with a pH meter and associated glass and reference electrodes.

### 4. Significance and Use

- 4.1 The pH is the measure of the free hydrogen ion concentration of a sample, and it indicates whether an electrocoat bath is acidic, neutral, or basic. Since pH measurements of good precision are made in aqueous solutions, it is suggested that the pH measurements of electrocoat baths are only semi-quantitative, and therefore such measurements should be referred to as apparent pH measurements.
- 4.2 The pH of electrocoat paints is used for research, production, and quality control or electrocoat bath process control.
- 4.3 Other related methods for determining the pH of water or aqueous systems are described in Test Methods [D1293](#) and [E70](#).

### 5. Apparatus

- 5.1 *pH Meter,*
- 5.2 *Glass and Reference Electrodes.*

NOTE 1—Due to the polarity of electrocoat materials it is desirable to use a separate set of electrodes for each bath polarity, cathodic and anodic, because a bath of opposite polarity poisons the electrodes. A desired practice is to rinse the electrodes after each measurement with an appropriate solvent for the electrocoat material.

- 5.3 *Thermometer,* capable of 0.5°C accuracy with a –2 to 32°C range.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee [D01](#) on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee [D01.21](#) on Chemical Analysis of Paints and Paint Materials.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the [standard's Document Summary](#) page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

## 6. Reagents

6.1 *Reference Standard Solutions*, commercial standards of pH 4.0, 7.0, and 10.0.

6.2 *Purity of Water*—References to water shall be understood to mean water conforming to Type II of Specification **D1193**.

## 7. Sampling and Sample Preparation

7.1 The sample should be obtained while the electrocoat bath is under proper circulation so that a uniform material is obtained. In case of an ultrafiltrate, the material should be thoroughly mixed or stirred prior to sampling to assure uniformity.

7.2 After sampling and prior to removing a test specimen it is mandatory that the sample be shaken or stirred until it is homogeneous and free of any settled material. This is particularly important if there is any delay between the sampling of the bath and the preparation of the specimens for the test. The absence of settled material can be ascertained visually (in a transparent container) or by inserting a spatula, scraping the bottom of the container to make sure that there is no settled matter. Shake or stir the samples until specimens are taken for measurement; *this point is very important*.

## 8. Procedure

8.1 *Standardization*—Turn on the pH meter and allow it to warm up. Wash the electrodes with a stream of water. Use the pH meter manufacturer's instructions to calibrate the electrodes with buffer solutions of pH 4 and 7 for cationic systems and 7 and 10 for anionic systems. Rinsing the electrodes thoroughly after each calibration. The reference buffer solutions should thoroughly match the temperature of the samples to be measured within 2°C.

8.2 *Apparent pH Measurement*:

8.2.1 Place a suitable quantity of a well-mixed electrocoat specimen in a clean and dry container equipped with a stirrer (preferably magnetic). Operate the stirrer fast enough to avoid separation of the paint but slow enough to prevent splashing or vortexing. Allow the temperature of the specimen to come to equilibrium (that is, the temperature should show a drift of less than 0.5°C/min). Record the temperature, and dial in the temperature correction on the pH meter. Place the electrodes in the specimen and determine the pH value in accordance with the pH meter manufacturer's instructions. Record the pH value and the temperature of the specimen.

8.2.2 Run two successive specimens of the electrocoat bath sample; the pH value should be within 0.1 units/min and should show drifts of less than 0.1 units/min. If these drift limits are not met, check the pH meter and repeat the whole procedure.

## 9. Precision and Bias

9.1 In an interlaboratory study of the test method, with five laboratories measuring five electrocoat bath samples with pH ranging from 2.6 to 8.0, the intralaboratory standard deviation (in accordance with Practice **E180**) was found to be 0.03 units at 25 df, and the interlaboratory coefficient of variation was 0.08 units at 20 df. Based on these standard deviations, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

9.1.1 *Repeatability*—Two results, each the mean of duplicate determinations, obtained by the same operator on different days should be considered suspect if they differ by more than 0.1 units.

9.1.2 *Reproducibility*—Two results, each the mean of duplicate determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 0.3 units.

9.1.3 *Bias*—Bias cannot be determined for this method because there are no accepted standards for the free hydrogen ion content of electrocoat baths and their ultrafiltrates.

## 10. Keywords

10.1 electrocoat baths; pH; pH measurement; ultrafiltrates

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