Designation: D 847 – 96 (Reapproved 2000)

# Standard Test Method for Acidity of Benzene, Toluene, Xylenes, Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons[1]

This standard is issued under the fixed designation D 847; 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 is intended for the detection of acidity and for the quantitative determination of acidity of benzene, toluene, xylenes, solvent naphthas, and similar industrial aromatic hydrocarbons.

1.2 The following applies to all specified limits in this test method: for purposes of determining conformance with this test method, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29.

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.* For specific hazard statements see Section 8.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 3437 Practice for Sampling and Handling Liquid Cyclic Products[3]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[4]

2.2 *Other Documents:*

OSHA Regulations, 29 CFR, Paragraphs 1910.1000 and 1910.1200[5]

## 3. Terminology

3.1 *Definitions:*

3.1.1 *acidity*—the number of milligrams of sodium hydroxide consumed when 100 mL of the specimen are titrated under the conditions prescribed in this test method.

3.1.2 *acid reaction*—a characteristic of materials producing the acid-color of the indicator used under the conditions prescribed in this test method.

3.1.3 *alkaline or basic reaction*—a characteristic of the materials producing the alkali-color of the indicator used under the conditions prescribed in this test method.

## 4. Summary of Test Method

4.1 The acidity of aromatic hydrocarbons is detected and determined quantitatively using a sodium hydroxide titration and a color change in a phenolphthalein indicator.

## 5. Significance and Use

5.1 This test method is suitable for setting specifications, for use as an internal quality control tool, and for use in development or research work on industrial aromatic hydrocarbons and related materials. This test method gives an indication of residual acidity and is a measure of the quality of the finished product. It is an indication of the tendency of the product to corrode equipment.

## 6. Interferences

6.1 Tests for acidity are not applicable in the presence of contaminating acidic or alkaline gases, soaps, salts, or other compounds derived from the atmosphere or apparatus. The container holding the specimen, and the apparatus, water, indicator, and other materials used in the test shall be chosen so that they themselves shall not appreciably affect the results. Since new corks used in specimen bottles often are bleached with oxalic acid, it is advisable to rinse them thoroughly and check them for neutrality with the indicator used in the test. Glassware shall be of acid-resistant and alkali-resistant glass[6] and shall be rinsed with neutral distilled water before use. The room in which the test is performed shall be chosen so as to prevent undue contamination by carbon dioxide, ammonia, or other interfering substances that may be present in the atmosphere.

6.2 The distilled water used in the test shall not alter the

[1] This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.01 on Benzene, Toluene, Xylenes, Cyclohexane, and Their Derivatives.

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

[3] *Annual Book of ASTM Standards*, Vol 06.04.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

[6] Borosilicate glass or the equivalent has been found satisfactory for this purpose.

composition of the specimen nor otherwise interfere with the purpose of the work. Although inert impurities often may be neglected, care must be exercised to correct for impurities or to eliminate them entirely if they are likely to interfere. When the distilled water does not show an acid or alkaline reaction, it may be used without further adjustment to neutrality. However, if the water shows an acid or alkaline reaction, it shall be brought to a persistent pink end point before use by titration with standard 0.01 *N* NaOH solution (see 8.5) or standard 0.01 *N* $H_2SO_4$, respectively.

## 7. Apparatus

7.1 *Graduate*, 100-mL.[6]

7.2 *Bottle*, 500-mL glass-stoppered.[6]

7.3 *Buret*, 10-mL, graduated in 0.05-mL subdivisions.

## 8. Reagents

8.1 *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.[7] 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.

8.2 *Phenolphthalein Indicator Solution*— Dissolve 0.5 g of phenolphthalein in 100 mL of cp ethyl alcohol (95 %). Add 0.01 *N* NaOH solution cautiously until a faint pink color appears; then just remove the color with a drop or two of 0.01 *N* acid.

8.3 *Sodium Hydroxide, Standard Solution* (0.1 *N*).

8.4 *Sodium Hydroxide, Standard Solution* (0.01 *N*).

8.5 *Sulfuric Acid, Standard* (0.01 *N*).

8.6 *Purity of Water: Distilled Water, Neutral*——Boil vigorously for 30 min, 1 to 2 L of distilled water conforming to Type III of Specification D 1193. Insert a stopper carrying a guard tube of soda lime. Rinse a 200-mL flask with this distilled water, add a 100-mL portion, and titrate in a closed system with 0.01 *N* NaOH solution: or heat to boiling and titrate immediately, taking care that the temperature does not fall below 80°C during the titration. If more than 1 drop (0.05 mL) of 0.01 *N* NaOH solution is required to obtain an end point with phenolphthalein, adjust the pH of the water to be used by adding the calculated amount of NaOH solution. Repeat the blank titration, and readjust if necessary until the blank titration on 100 mL of the distilled water is 1 drop (0.05 mL) or less of the 0.01 *N* NaOH solution. The distilled water now will be neutral or very slightly acid to the phenolphthalein indicator.

## 9. Hazards

9.1 Consult current OSHA regulations, supplier's Material Safety Data Sheets, and local regulations for all materials used in this test method.

## 10. Sampling

10.1 Sample the material in accordance with Practice D 3437.

## 11. Procedure

11.1 Measure out 100 mL of the sample into the 500-mL bottle. Add 100 mL of neutral distilled water (see 8.2) and 2 drops of phenolphthalein indicator solution. Stopper and shake thoroughly. Without separating the layers, titrate the acidity, if any, with standard 0.1 *N* NaOH solution to the first persistent shade of pink, while shaking thoroughly.

11.2 If more than 2 drops (0.10 mL) of 0.1 *N* NaOH solution is required to produce a persistent pink end point, discard the results of the test. While taking precautions to avoid contamination from the apparatus and atmosphere, proceed as follows: Rinse the 100-mL graduated cylinder and the 500-mL bottle and glass stopper with neutral distilled water. To the bottle, add 100 mL of the sample and 100 mL of neutral distilled water. Add 2 drops of phenolphthalein indicator solution, and shake vigorously for 10 s. Bring the temperature to between 15 and 18.5°C (60 and 65°F). Add 1 drop of 0.1 *N* NaOH solution, stopper, and shake vigorously for 10 s. Repeat the addition of 1 drop of NaOH solution, stoppering, and shaking for 10 s, until a sharp pink end point is secured. Repeat this procedure on a blank run with 100 mL of the neutral distilled water.

## 12. Interpretation of Results and Calculation

12.1 Unless otherwise indicated in the applicable specifications, the test results shall be interpreted as follows:

12.1.1 A specimen shall be said to contain no free acid, that is, show no evidence of acidity, if 2 drops or less of 0.1 *N* NaOH solution produces a persistent pink end point in the test bottle.

12.1.2 When more than 2 drops (0.10 mL) of 0.1 *N* NaOH solution is required to produce a persistent pink end point in the test bottle, the acidity shall be reported in terms of milligrams of NaOH required for 100 mL of specimen, and shall be calculated as follows:

$$\text{Acidity, mg NaOH per 100 mL} = 4\,(A - B) \quad (1)$$

where:

$A$ = 0.1 *N* NaOH solution required for titration of the sample, mL, and

$B$ = 0.1 *N* NaOH solution required for titration of the blank, mL.

## 13. Precision and Bias

13.1 In the case of pass/fail data, no generally accepted method for determining precision is currently available.

## 14. Keywords

14.1 acidity

[7] *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.