Designation: D 848 – 03

Standard Test Method for Acid Wash Color of Industrial Aromatic Hydrocarbons[1]

This standard is issued under the fixed designation D 848; 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 the determination of the acid wash color of benzene, toluene, xylenes, refined solvent naphthas, and similar industrial aromatic hydrocarbons.

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.* For specific hazard statements, see Sections 8 and 12.1.

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]

D 4790 Terminology of Aromatic Hydrocarbons and Related Chemicals[3]

2.2 *Other Document:*

OSHA Regulations. 29 CFR, paragraphs 1910.1000 and 1910.1200[4]

3. Terminology

3.1 See Terminology D 4790 for definitions of terms used in this test method.

4. Summary of Test Method

4.1 A mixture of the aromatic hydrocarbon and sulfuric acid is vigorously shaken and the color of the acid layer is compared with that of color standards prepared from $CoCl_2$ and $FeCl_3$.

5. Significance and Use

5.1 This test method is suitable for setting specifications on the materials referenced in 1.1. It may also be used as an internal quality control tool and in development or research work.

5.2 The color developed in the acid layer gives an indication of impurities which if sulfonated would cause the material to be discolored.

6. Apparatus

6.1 *Containers for Color Standards*—Clear and unblemished, clean, French square, flint-glass, flat-bottom, glass-stoppered, 1-oz capacity bottles holding 31 to 33 mL when filled to the neck.[5] The bottles shall be labeled with the reference number of the color standard they contain (see 11.2).

6.2 *Test Containers*—Containers exactly like those described in 6.1 except that each French square bottle shall be marked by etching to show when the bottle contains the volume of 7 and 28 mL, respectively. Colored crayons and similar markers shall not be used for marking the bottles.

7. Reagents

7.1 *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.[6] 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.

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

[5] The sole source of supply of the apparatus known to the committee at this time is Ramin USA Corporation, 39019 FM 149 Rd., Magnolia, TX 77354. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

[6] *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.

***A Summary of Changes section appears at the end of this standard.**

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water, Type I or II as described in Specification D 1193.

7.3 *Cobalt Chloride* ($CoCl_2 \cdot 6H_2O$).

7.4 *Ferric Chloride* ($FeCl_3 \cdot 6H_2O$).

7.5 *Hydrochloric Acid* (1 + 39)—Mix 25 mL of hydrochloric acid (31 weight % HCl) with 975 mL of water.

7.6 *Potassium Chromate* (K_2CrO_4).

7.7 *Potassium Dichromate* ($K_2Cr_2O_7$).

7.8 *Sulfuric Acid* (96 ±0.5 weight % H_2SO_4).

7.9 *Sulfuric Acid* (78 ±0.5 weight % H_2SO_4).

8. Hazards

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

8.2 When handling strong acids or acid cleaning solutions, wear proper personnel protective equipment.

9. Sampling

9.1 Sample the material in accordance with Practice D 3437.

10. Cleaning of Containers

10.1 Clean new containers (Section 6) with a cleaning solution that will not impact the results, such as a chromic acid substitute, rinse with tap water followed by distilled water, and dry in an oven set at a minimum of 105°C for at least 1 h. Likewise, clean all other glassware used in this test method.

11. Preparation of Reference Color Standards

NOTE 1—Purchase of solutions or reference color standards, or both, is allowed. The user of this standard assumes the responsibility of ensuring any purchased solutions or standards are prepared with materials that meet the requirements expressed in the Reagents section of this standard. Likewise, the user of this standard assumes the responsibility of ensuring any purchased solutions or standards are prepared as expressed in this section.

11.1 *Stock Solutions*—Prepare the following basic reagent solutions for use in preparing the reference color standards:

11.1.1 *Solution A*—Dissolve 59.50 g of $CoCl_2 \cdot 6H_2O$ in HCl (1 + 39) and make up to 1 L in a volumetric flask with HCl (1 + 39).

11.1.2 *Solution B*—Dissolve 45.054 g of $FeCl_3 \cdot 6H_2O$ in HCl (1 + 39) and make up to 1 L in a volumetric flask with HCl (1 + 39).

11.1.3 *Solution C*—Mix 3½ volumes of Solution A with 36½ volumes of Solution B and dilute with 90 volumes of water.

11.1.4 *Solution D*—Mix 3½ volumes of Solution A with 36½ volumes of Solution B.

11.1.5 *Solution E*—Prepare an aqueous solution of K_2CrO_4 saturated at 21°C.

11.1.6 *Solution F*—Prepare an aqueous solution of $K_2Cr_2O_7$ saturated at 21°C and dilute with an equal volume of water.

11.2 Prepare reference color standard solutions having the following compositions and numbered as specified below. It is not required to make each color standard. Only those reference color standards that bracket the samples being evaluated must be utilized.

No. 0—Distilled water.
No. 1—1 volume of Solution C plus 1 volume of water.
No. 2—5½ volumes of Solution C plus 2 volumes of water.
No. 3—Solution C.
No. 4—1 volume of Solution D plus 1 volume of water.
No. 5—5½ volumes of Solution D plus 2 volumes of water.
No. 6—Solution D.
No. 7—5 volumes of Solution E plus 2 volumes of water.
No. 8—Solution E.
No. 9—7 volumes of Solution E plus ½ volume of Solution F.
No. 10—6½ volumes of Solution E plus 1 volume of Solution F.
No. 11—5½ volumes of Solution E plus 2 volumes of Solution F.
No. 12—1 volume of Solution E plus 1 volume of Solution F.
No. 13—2 volumes of Solution E plus 5 volumes of Solution F.
No. 14—Solution F.

11.3 Rinse the No. 0 container (5.1) and its glass stopper three times with water, fill with water, and stopper. Rinse the No. 1 container and its stopper three times with reference color standard solution No. 1 (Section 11.2), fill with this solution, and stopper. In this way, prepare the set of containers of color standards from 0 through 14 having the compositions shown for the corresponding color solution standards in 11.2. When filling the French square bottles, leave ¼ in. (6 mm) of vapor space below the neck of the bottle. Seal each container with paraffin to prevent loss by evaporation or seepage.

NOTE 2—It is recommended color standards be prepared annually from fresh solutions.

12. Procedure

12.1 Fill a clean, dry test container to the 7-mL mark with the acid of the strength specified in Table 1 for the type of sample to be tested . Add sufficient sample to bring the total volume to the 28-mL mark (Note 3). Insert the stopper, hold a finger over the stopper, and give vigorous shakes with a stroke of 13 to 25 cm (5 to 10 in.), shaking for a total of 150 cycles over a period of 40 to 50 s, that is at a rate of 3 to 3.75 cycles/s. (Use of an automatic shaker is allowed given it can be shown to produce comparable results to the manual technique.) (**Warning**—Concentrated sulfuric acid will cause severe burns on contact with the skin. As a precaution the test container should be wrapped in a towel or enclosed in a plastic bag during the shaking period. The test should be performed using appropriate personal protective equipment.)

NOTE 3—If the room temperature is above 85°F, maintain the acid, sample, and reference color standards at a temperature between 77 and

TABLE 1 Acid Strengths and Standing Times

	Sample	Sulfuric Acid Strength, %	Standing Time, min
Group 1	Benzene, all ASTM grades Toluene, all ASTM grades Xylene, nitration grade Xylene, 5° Xylene, 10° Any other more highly refined products	96	15
Group 2	Xylene, industrial grade Refined solvent naphtha	96	5
Group 3	Hi-flash solvent Heavy solvent naphtha	78	5

85°F (25 and 29°C) through the test, and insulate the test container in some convenient way, such as wrapping with a cloth, during the shaking period.

12.2 Allow the container to stand, protected from direct sunlight, for the period of time shown in Table 1. Without further delay, invert the container gently once or twice to obtain a uniform color in the acid layer, and compare the color of the acid layer with that of the standards (11.3). Make the comparison against a white background or against daylight, using transmitted light (Note 4). When testing samples in Group 1 (Table 1), observe the color of the oil layer as well as that of the acid layer. Standards used shall include standards one number above and one number below the sample, except for the samples reading 0 or 14.

NOTE 4—Agreement of results may be improved by using a color comparator of a suitable type for observing the color of the acid layer in comparison with the reference standard color solution.

12.3 Designate the color of the acid layer by the number of the nearest matching standard, following the number with a plus or minus sign if the sample is darker or lighter, respectively, than the standard. Disregard any difference in hue and determine only whether the color of the acid layer is darker or lighter than the color of the reference standard to which the sample most nearly corresponds. If the hue of the acid color is different from the hue of the reference color standard, record the color number followed by (X). Thus "No. 4 − (X)" means that the acid wash test color is slightly lighter than No. 4 color standard and that the hue of the No. 4 color standard is not the same as the hue of the acid layer.

12.4 Dispose of the acid and hydrocarbon properly before cleaning the container. Clean the test container by flushing thoroughly with water (tap grade or better) until traces of acid have been removed. The test container must be completely dry before being placed back into use.

NOTE 5—Suitable solvents, that will not impact the results, may be used after the water rinse step to assist in drying the container.

13. Interpretation of Results

13.1 Report Group 1 samples (Table 1) as passing the test only when the oil layer shows no change in color and when the acid layer is not darker than the specified color standard. A cloudiness or haze in the oil layer should not be interpreted as a change in color.

13.2 When testing samples of Groups 2 or 3, disregard the color of the oil layer and report the sample as passing the test when the acid layer is not darker than the specified color standard.

14. Precision and Bias

14.1 Precision data have not been established for all types of samples on which this test method is used. Limited cooperative tests were conducted in 1961, principally to establish equality with the previously used shaking procedure. Precision estimates taken from these data are as follows:

Average Acid Wash Color		Repeatability		Reproducibility	
		Degrees of Freedom	95 % Repeatability	Degrees of Freedom	95 % Reproducibility
Benzene	1.4	11	0.75	9	2.34
	6.1	12	1.85	10	4.47
Xylene	4.7	12	0.40	10	1.39
	10.2	12	1.14	10	3.52

15. Keywords

15.1 acid wash color; aromatic hydrocarbons

SUMMARY OF CHANGES

Committee D16 has identified the location of selected changes to this standard since the last issue (D 848 – 02) that may impact the use of this issue.

(1) Section 6.1 – Source of bottles added to Footnote 5.

(2) Section 12 – Portion of Note 5 extracted and made 12.4.