Designation: D1768 – 89 (Reapproved 2016)

Standard Test Method for Sodium Alkylbenzene Sulfonate in Synthetic Detergents by Ultraviolet Absorption[1]

This standard is issued under the fixed designation D1768; 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.

1. Scope

1.1 This test method covers commercial synthetic detergents and built detergent formulations that do not contain organic additives such as amides. Optical dyes and materials normally found in formulated detergents do not interfere; however, materials other than sulfonates that possess strong ultraviolet absorptions must be absent. The indicated sample size and aliquoting scheme is based on samples containing 30 to 40% of active ingredient.

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.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D460 Test Methods for Sampling and Chemical Analysis of Soaps and Soap Products

D1568 Test Methods for Sampling and Chemical Analysis of Alkylbenzene Sulfonates

D3049 Test Method for Synthetic Anionic Ingredient by Cationic Titration

D4251 Test Method for Active Matter in Anionic Surfactants by Potentiometric Titration

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Summary of Test Method

3.1 This test method involves measurement of the ultraviolet absorbance of the sample at the maximum absorbance point for sodium alkylbenzene sulfonate (224 nm) and at a selected background point of 270 nm in order to determine the amount of active ingredient present. The percentage of active ingredient is calculated from the above absorbances and data on the pure active ingredient.

4. Apparatus

4.1 *Spectrophotometer,*[3] equipped for liquid samples and for measurements in the ultraviolet region. The instrument shall be capable of measuring absorbance with a repeatability of ±0.5 % or better from an average at the 0.4 absorbance level in the spectral region between 220 and 270 nm. The spectral positions shall be accurate to ±0.5 nm and repeatable to ±0.2 nm.

4.2 *Absorption Cells,*[4] silica, matched pairs of optical path length 1.000 ± 0.005 cm.

5. Reagent

5.1 *Ethyl Alcohol* (95 %) —Conforming to either Formula No. 3A or No. 30 of the U.S. Bureau of Internal Revenue.

6. Reference Standard

6.1 A previously analyzed sample or solution may be used to check performance of the cell and instrument.[3] Guard such reference samples from contamination and renew periodically (quarterly).

7. Calibration and Standardization of Cells[3]

7.1 The cells in a pair, when filled with distilled water, should match within 1 % transmittance at 224 nm. Otherwise, calibrate the cells as directed by the manufacturer and use a correction factor for each cell.

[1] This test method is under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components.

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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.

[3] For information on performance testing of spectrophotometers, refer to Practice E275.

[4] One-centimetre silica cells manufactured by either the Pyrocell Manufacturing Co., 270 E. 84th St., New York, NY, Catalog No. S22-240, or by Beckman Instruments, Inc., Fullerton, CA, Catalog No. 40736 have been found satisfactory for this purpose.

7.2 Before each day of operation, load the quartz cells with distilled water and make certain that the cells match within 1 % transmittance. This practice is necessary to check cleanliness of the cells. Clean cells, if necessary, using dichromate cleaning solution, until the desired transmittance is obtained.

NOTE 1—All glassware must be rinsed with freshly prepared distilled water before use. *Do not dry* the rinsed glassware. This is necessary, especially after the first dilution, to avoid errors due to contamination.

8. Determination of Purity of Sodium Alkylbenzene Sulfonate Standard from its Organic Alcohol-Soluble Matter

8.1 Determine triplicate values for the percentage of organic alcohol-soluble matter in the alkylbenzene sulfonate standard (or particular alkylbenzene sulfonate being determined) in accordance with 8.2 – 8.5.

NOTE 2—The purity of the standard, or percentage of active ingredient, can also be determined by the cationic titration of Test Method D3049 or by the specific ion electrode titration of Test Method D4251.

8.2 Determine moisture in accordance with Moisture by the Distillation Method sections of Test Methods D1568.

8.3 Determine alcohol-insoluble matter in accordance with Sections 19 to 20 (Total Matter Insoluble in Alcohol) of Test Methods D460 or Total Matter Insoluble in Alcohol 20 to 22 of Test Methods D1568.

8.4 Determine chlorides calculated as sodium chloride in accordance with Chlorides Calculated as Sodium Chloride (NaCl) sections of Test Methods D1568.

8.5 Determine unsulfonated matter (neutral oil) in accordance with Neutral Oil sections of Test Methods D1568.

9. Determination of Active Ingredient

9.1 *Powders and Solid Samples*—Weigh, to the nearest 0.1 mg, three 0.9 to 1.1-g portions of a representative sample. Dissolve each portion of the sample in water (Note 3) and dilute to 500 mL with water at room temperature in a volumetric flask. Mix well. Treat each portion of the sample solution in accordance with 9.3 and 9.4.

NOTE 3—It is desirable for the sample to be completely dissolved. It may be dissolved as follows:

(1) Transfer the weighed sample to the volumetric flask and dilute to volume with water at room temperature. Carefully insert a TFE-fluorocarbon-covered stirring magnet and agitate vigorously on a magnetic stirrer for 15 to 20 min. Carefully invert the flask several times to ensure thorough mixing. If insoluble matter remains, allow the flask to stand for several hours, or preferably overnight. Then continue in accordance with 9.3 by pipetting the 5-mL aliquot from the top of the supernatant solution.

(2) Transfer the weighed sample to a 600-mL beaker. Add 200 mL of water and place on a steam bath or hot plate for about 10 min with occasional stirring. Cool to room temperature and dilute to volume. A fine stream of water or a few drops of alcohol will aid in breaking any foam persisting in the neck of the volumetric flask. As in *(1)*, allow any insoluble matter to settle before continuing in accordance with 9.3 and 9.4.

9.2 *Slurry Sample*—Weigh, to the nearest 1 mg, three approximately 20-g portions of a representative sample. Add 50 mL of alcohol and mix to disperse the sample. Dissolve each portion of the sample in water (Note 3) and dilute to 1000 mL with water at room temperature, employing a 1000-mL volumetric flask. Mix well. Pipet a 50-mL aliquot of each solution into a 500-mL volumetric flask, dilute to volume with water at room temperature, and mix well. Treat each of the three sample solutions as directed in 9.3 and 9.4.

9.3 Pipet a 5-mL aliquot of the solution from 9.1 or 9.2 into a 250-mL volumetric flask and dilute to volume with water at room temperature. Mix well.

9.4 Using 1-cm cells in the spectrophotometer, measure the absorbance at 224 and 270 nm versus a water blank. With instruments having scanning capability, scan from 300 down to 210 nm. This facilitates obtaining the absorbance at the peak maximum near 224 nm. (**Caution**, Note 4. See also Note 5 and Note 6).

NOTE 4—**Caution:** There is danger of contamination of the sample with detergent dust in the production laboratory air; therefore, these measurements must be made immediately after the procedure described in 9.3. Contamination is evident when a high reading (above 0.1 absorbance) at 270 nm is obtained. Discard such contaminated samples and start with a new aliquot (9.3, Note 5 and Note 6).

NOTE 5—The observed absorbance readings should be between 0.2 and 0.9; otherwise weigh a new sample or take a new or different aliquot and dilute to a known volume. (A calibration curve in this absorbance range must be made with a sample of the sodium alkylbenzene sulfonate being quantitatively measured. See also Note 7.)

NOTE 6—When analyzing samples of doubtful origin, the absorption maximum at 224 nm should be checked. Measure the absorbance at 220, 224, 228, and 270 nm. The absorbance at 224 nm should be greater than the absorbance at either 220 nm or 228 nm. If the 224-nm absorption is not the maximum of all the absorbance readings, alkylbenzene sulfonate is not responsible for the observed absorption and the spectrophotometric method is invalid.

10. Calculation

10.1 *Alcohol-Soluble Matter*—All percentages appearing in 10 are weight percentages. Calculate the percentage of alcohol-soluble matter in the sodium alkylbenzene sulfonate standard (or particular alkylbenzene sulfonate being determined) as percent sodium alkylbenzene sulfonate as follows:

$$\text{Alcohol} - \text{soluble matter (sodium alkylbenzene sulfonate), \%} = 100 - (M + A + B + C) \quad (1)$$

where:

M = percentage of alcohol-insoluble matter,
A = percentage of moisture,
B = percentage of sodium chloride, and
C = percentage of neutral oil.

Average the results of the three determinations, which should agree within 0.5 %.

10.2 *Absorptivity Value*—Calculate the absorptivity value, a, for each of the three portions of the sodium alkylbenzesulfonate standard (or particular alkybenzene sulfonate being determined) as follows:

$$\text{Absorptivity value, } a = (A_{224} - A_{270})/M_s bc \times 100 \quad (2)$$

where:

A = observed absorbance,
M_s = percentage of organic alcohol-soluble matter in the sample (average of three determinations),
b = cell length in centimetres, and
c = concentration of final dilution in grams per 1000 mL.

Average the three results.

10.3 *Active Ingredient*—Calculate the percentage of active ingredient (sodium alkylbenzene sulfonate) as follows (Note 7):

$$\text{Active ingredient (sodium alkylbenzene sulfonate)}, \% = (A_{224} - A_{270}) \times 25/Wa \times 100 \quad (3)$$

where:

A = observed absorbance at 224 and 270 nm (average of three results),
W = grams of sample used (9.1) or grams of sample represented in the aliquot used (9.2), and
a = absorptivity value for the particular alkylbenzene sulfonate being determined (10.2).

NOTE 7—The calculation as written is based on the diluting and aliquoting scheme as described in 9.1 – 9.4 and the absorptivity value, a, of products made from commercially available dodecylbenzenes. The calculation may be adapted to general use as follows:

$$\text{Active ingredient (sodium sulfonate)}, \% = (A_{224} - A_{270})/cb \times 100/a \quad (4)$$

where:

A = observed absorbance,
b = cell length in centimetres,
c = concentration of final dilution in grams per 1000 mL and
a = absorptivity value.

11. Precision[5]

11.1 *Repeatability (Single Analyst)*—The standard deviation of results (each of the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.34 % absolute at 10 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 1.07 % absolute.

11.2 *Reproducibility (multilaboratory)*—The standard deviation of results (each of the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.95 % of absolute at 4 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 3.72 % absolute.

11.3 *Checking Limits for Duplicates*—Report the percent of sodium alkylbenzene sulfonate of the sample to the nearest 0.1. Duplicate runs that agree within 0.86 % absolute are acceptable for averaging (95 % confidence level).

12. Keywords

12.1 sodium alkylbenzene sulfonate; synthetic detergents; ultraviolet absorption

[5] Supporting data are available from ASTM Headquarters, 100 Barr Harbor Drive, West Conshohoken, PA 19428. Request RR:D12-1001.