Designation: D2023 – 89 (Reapproved 2016)

Standard Test Method of Analysis for Sodium Toluene Sulfonate in Detergents[1]

This standard is issued under the fixed designation D2023; 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 the analysis for the apparent sodium toluene sulfonate (NaTS) content of detergents.

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]

D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 This test method is based on the separation of the low molecular weight sulfonate from interfering higher molecular weight sulfonates and determination by ultraviolet absorption. The higher molecular weight sulfonates are extracted as sulfonic acids in a hydrochloric acid solution using ethyl ether as the solvent.

4. Significance and Use

4.1 This test method is of use to anyone engaged in compositional analysis of detergent formulations. This would include formulators, and analysts employed by companies that manufacture the components usually included in such formulations.

5. Interferences

5.1 Any low molecular weight substituted benzene sulfonate or benzene sulfonate (NaBS) itself will interfere and give an apparent toluene sulfonate figure. Since the method is standardized using *p*-toluene sulfonate, *o*-sulfonate will not be assayed correctly. When contamination is suspected, it should be checked by running a complete absorption curve and comparing with known samples.

6. Apparatus

NOTE 1—Absolute cleanliness of apparatus is essential. Use glass apparatus only. Contact with rubber, cork, or hands will introduce absorbance errors.

6.1 *Separatory Funnels,* 500-mL capacity, glass-stoppered, pear-shaped.

6.2 *Balance,* analytical.

6.3 *Spectrophotometer* (Note 2), with necessary ultraviolet accessories for working in the range from 230 to 300 nm. These include 1.00 and 5.00-cm absorption cells with quartz windows.

NOTE 2—Details of the test method as written are based on the use of the Beckman DU or Cary recording spectrophotometers. Equivalent spectrophotometers also may be used, provided suitable modifications can be made in the details of the method.

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.[3] 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193.

[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.

Current edition approved July 1, 2016. Published August 2016. Originally approved in 1962 as D2023 – 62 T. Last previous edition approved in 2009 as D2023 – 89(2009). DOI: 10.1520/D2023-89R16.

[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] *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.

7.3 *Ethyl Ether.*

7.4 *Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid (HCl).

7.5 *Hydrochloric Acid (1+3)*—Mix 1 volume of concentrated HCl with 3 volumes of water.

7.6 *p-Toluenesulfonic Acid Sodium Salt* (NaTS)[4]—Dry for 24 h at 105°C and store in a desiccator. Use for preparation of standards.

7.7 *Sodium Sulfate* (Na_2SO_4), anhydrous.

8. Precaution

8.1 This test method includes ethyl ether extraction from an acidic solution. Appropriate safety practices, such as those included in the Material Safety Data Sheets for ethyl ether, should be employed. Especially important is adequate eye protection to guard against spattering.

9. Preparation of Standards

9.1 Weigh 0.25 ± 0.0001 g of the standard NaTS, dissolve in water, and dilute to the mark in a 100-mL volumetric flask. Mix thoroughly. From this solution make pipettings as follows:

9.1.1 For the Cary spectrophotometer and 5-cm cells (Note 2), take 50 mL (b = cell length).

9.1.2 For the Beckman DU spectrophotometer and 5-cm cells (Note 2), take 10 mL (b = cell length).

NOTE 3—If 1.0-cm cells are used, the concentration must be increased accordingly.

9.2 Place the indicated volume of solution in 250-mL volumetric flask. Add to the flask 10.0 g of Na_2SO_4 and 60 mL of HCl. Dilute to the mark with water and mix thoroughly. From the 250-mL volumetric flask, pipet 25 mL into a 100-mL volumetric flask, dilute to volume with water and mix thoroughly. The concentration, c, in this final dilution will be 0.125 g/L for Cary and 0.025 g/L for Beckman instruments.

9.3 Prepare a blank solution with water, Na_2SO_4, and HCl, but without NaTS, exactly as described for standards.

9.4 Measure the absorbance of the dilute standard solution against the blank over the range 230 to 300 nm. Determine the maximum absorption near 261 nm and the absorption at wavelengths 19 nm above and below the maximum. If a non-recording instrument is used, determine the peak absorbance and read the reference points 19 nm above and below. Correct the absorbance near 261 nm by subtracting one half the sum of the absorbances at 19 nm below and 19 nm above the peak wavelength.

9.5 Calculate the absorptivity, a, of the standard solution as follows:

$$a = \text{corrected absorbance near 261 nm}/bc \qquad (1)$$

[4] Eastman Organic Chemical No. 524 has been found satisfactory for this purpose.

10. Procedure

10.1 Weigh 10.00 ± 0.01 g of the sample of synthetic detergent to be analyzed and dissolve in about 75 mL of hot water. Transfer to a 500-mL glass-stoppered, pear-shaped, separatory funnel; add 10.0 g of Na_2SO_4 and 40 mL of HCl. Swirl to dissolve and cool to room temperature.

10.2 Extract twice with 100-mL portions of ethyl ether, shaking vigorously during each extraction. **Caution:** see 8.1. Combine the ether extracts and wash them with three 25-mL portions of 1 + 3 HCl to ensure complete removal of sodium ions. Add these extracts to the extracted acid aqueous solution and extract once more with 100 mL of ether. Allow to settle for 15 to 30 min, or until clear, and draw off the acid water layer into a beaker and evaporate the ether, on a steam bath. Filter this solution if not clear, preferably through a glass fiber filter.

10.3 *Discard all ether extracts.* Transfer the acid water solution, free of ether, to a 250-mL glass-stoppered, volumetric flask. Dilute to the mark with water and mix thoroughly. This solution contains all of the NaTS or NaBS free of synthetic detergent sulfonates.

10.4 For the Cary spectrophotometer, pipet 10 mL of this solution into a 100-mL glass-stoppered volumetric flask and dilute to volume with water. Mix thoroughly and make absorbance measurements against a blank diluted in a similar manner which contains water, HCl, and Na_2SO_4 only. Absorbance measurements should be made in a 5-cm cell (Note 3) exactly as described in Section 5 (c = 4.00 g/L).

10.5 For the Beckman DU spectrophotometer, pipet 25 mL from the 250-mL volumetric flask into another 250-mL volumetric flask. Dilute to the mark with water, mix, and pipet 25 mL of this solution into a 100-mL volumetric flask. Dilute, mix, and make absorbance readings against a blank similarly prepared with reagents as described in Section 5 (c = 1.00 g/L).

11. Calculation

11.1 Calculate the absorptivity of the sample in the same manner as that used for the standard (9.5).

11.2 Calculate the weight percentage of sodium toluene sulfonate as follows:

$$\text{Sodium toluene sulfonate, weight \%} = (a_1/a_2) \times 100 \qquad (2)$$

where:

a_1 = absorptivity of the sample, and
a_2 = absorptivity of the standard.

12. Precision

12.1 The precision of this method is as follows:

Within-laboratory, %	0.085
Between-laboratory, %	0.200

These values were determined at the 0 to 3 % level.

13. Keywords

13.1 hydrotropes; sodium toluene sulfonate; ultraviolet absorption