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Standard Test Method for Anionic Detergent by *Para*-Toluidine Hydrochloride¹

This standard is issued under the fixed designation D 4224; 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 determination of the equivalent weight and the amount of total anionic ingredient in a surfactant. It is applicable to single organic sulfates and sulfonates of specified molecular weights, and in general wherever an aliphatic chain longer than six carbons is present.

1.2 *This test method includes the use of carbon tetrachloride, a hazardous solvent, that, while not flammable, is very harmful to the human body, particularly the liver. Breathing of vapors and repeated or prolonged contact with the skin is to be avoided. It is the responsibility of the user of this standard to establish appropriate safety practices and to 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.

1.3 The analytical procedures appear in the following order:

	Sections
Equivalent Weight	5 to 10
Percent Anionic Detergent	11 to 16

2. Referenced Documents

- 2.1 *ASTM Standards:*
D 1193 Specification for Reagent Water²
E 200 Practice for Preparation, Standardization, and Storage of Standard Solutions for Chemical Analysis²

3. Significance and Use

3.1 Other direct titration methods for making this determination require prior knowledge of the equivalent weight of the material. Different methods, while not contingent on knowledge of equivalent weight, depend on time-consuming and often imprecise determinations of many individual components.

3.2 The procedures described in this test method offer a relatively rapid and simple approach to determining both the equivalent weight and the amount of anionic surfactant present in a surfactant formulation.

4. Interferences

- 4.1 Soaps, fatty acids and fatty amides may interfere.
4.2 Low molecular weight sulfonates, such as those of toluene and xylene, do not interfere when present up to 15 % of active material, nor do normal inorganic components of

detergent formulations such as chloride, sulfate, borate, phosphates, perborate, and silicates.

EQUIVALENT WEIGHT

5. Summary of Test Method

5.1 Carbon tetrachloride extractable materials are removed from an alcohol-water solution of the sample which is then treated with *para*-toluidine hydrochloride. This reagent reacts with high molecular weight anionic surfactants to form amine salts that are soluble in and can be extracted quantitatively by carbon tetrachloride.

5.2 The carbon tetrachloride extract is dried and weighed to give the weight of the amine salt formed. The residue is dissolved in alcohol and titrated against standard base and standard silver nitrate. The equivalent weight is calculated from the weight of the amine salt and the two titrations.

6. Apparatus

- 6.1 *Analytical Balance.*
6.2 *Vacuum Oven.*
6.3 *Vacuum Pump or Aspirator.*
6.4 *Desiccator.*
6.5 *Steam Bath.*
6.6 *Automatic Titrator.*³ (Alternative: D 820:39–41 manual titration.)
6.7 *Combination Electrode*, silver with silver chloride coating.⁴
6.8 *Beaker*, tall form, 180 mL.⁵

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean water as defined by Type II of Specification D 1193.

³ The Metrohm E436 has been found satisfactory for this purpose.

⁴ The Metrohm E246 has been found satisfactory for this

⁵ The Pyrex beaker, No. 114, or its equivalent, has been found satisfactory for this purpose.

⁶ "Reagent Chemicals, American Chemical Society Specifications," Am. Chem. Soc., Washington, D.C. For suggestions on the testing of reagents not listed by The American Chemical Society, see "Reagents Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc. New York, N.Y. and the "United States Pharmacopeia."

¹ This test method is under the jurisdiction of ASTM Committee D-12 on Soaps and Other Detergents and is the direct responsibility of Subcommittee D12.12 on Analysis of Soaps and Synthetic Detergents.

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