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Designation: D2959 – 95 (Reapproved 2003)

Standard Test Method for Ethylene Oxide Content of Polyethoxylated Nonionic Surfactants[1]

This standard is issued under the fixed designation D2959; 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 ethylene oxide in polyethoxylated nonionic surfactants. It can also be used for compounds containing propylene oxide or any compounds (glycols and glycol and polyglycol ethers and esters) which form unstable 1,2-diiodides on reaction with hydriodic acid.

NOTE 1—Compounds in which the oxyalkylene group is connected to a nitrogen cannot be entirely decomposed. This can be used to determine the percent of an ethoxylated surfactant in a mixture, if the ethylene oxide content of the ethoxylated surfactant is known.

NOTE 2—This method reports results as percent ethylene oxide. If this method is applied to unknown compounds or compositions, the analyst should be aware of the possible presence of material other than ethylene oxide.

NOTE 3—For use on built syndet compositions the organic active ingredient must be isolated in accordance with Test Method D2358.

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.* 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]

D2358 Test Method for Separation of Active Ingredient from Surfactant and Syndet Compositions

3. Summary of Test Method

3.1 By cleaving nonionic surfactants containing polyethylene oxide chains with hydriodic acid, the unstable 1,2-diiodoethane is formed. This vicinal diiodide decomposes to form ethylene and iodine.

$$-(CH_2CH_2O)_n- + 2n\ HI \rightarrow n\ ICH_2CH_2I + n\ H_2O$$

$$ICH_2CH_2I \rightarrow CH_2{=}CH_2 + I_2$$

3.2 The percent ethylene oxide is determined by measuring the amount of free iodine formed, since one mole of iodine is formed for each mole of ethylene oxide in the polyoxyalkylene chain. The free iodine is titrated with a standard sodium thiosulfate solution.

4. Apparatus (Fig. 1)

4.1 *Heat Sources*, two. The source of heat should be an electric heater provided with a sliding rheostat or other means of heat control.

4.2 *Flasks*, two, 100-mL, round bottom. Each flask should be equipped with a standard ground-glass joint to accommodate a vertical condenser, and with a side arm through which carbon dioxide can be passed to blanket the reaction mixture.

4.3 *Condensers*, two, with standard joints to fit the flasks.

4.4 *Gas Bubbler*, filled with dibutyl phthalate.

4.5 *Buret*, 50-mL, with 0.1-mL graduations.

4.6 *Pipet*, 5-mL.

5. Reagents

5.1 *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.[4] 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.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type III reagent water conforming to Sections 1, 2, and 3 of Specification D1193.

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[1] This 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 of Soaps and Synthetic Detergents.

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

[3] *Annual Book of ASTM Standards*, Vol 15.04.

[4] *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.

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