



Designation: D4252 – 89 (Reapproved 2017)

# Standard Test Methods for Chemical Analysis of Alcohol Ethoxylates and Alkylphenol Ethoxylates<sup>1</sup>

This standard is issued under the fixed designation D4252; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover the various test methods used to evaluate those properties pertinent to the characterization of alcohol ethoxylates and alkylphenol ethoxylates with respect to suitability for desired uses.

1.2 The procedures for sampling and analysis appear in the following order:

	Sections
Sampling	
Liquids	6
Solids	7
Methods of Chemical Analysis	
Water or moisture	8
Refractive index	9 and 10
pH	11
Acidity or basicity	12 and 13
Hydroxyl number	14 – 20
Cloud point	21
Iodine number	22
Ash	23
Iron	24
Ethylene oxide content	25
Polyethylene glycols	26 – 32

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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. Specific hazard statements appear in Section 5 and [Note 1](#) and [Note 3](#).

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and are the direct responsibility of Subcommittee D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components.

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## 2. Referenced Documents

### 2.1 *ASTM Standards*:<sup>2</sup>

- D459 Terminology Relating to Soaps and Other Detergents
- D482 Test Method for Ash from Petroleum Products
- D1068 Test Methods for Iron in Water
- D1172 Guide for pH of Aqueous Solutions of Soaps and Detergents
- D1193 Specification for Reagent Water
- D1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)
- D1218 Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids
- D1613 Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products
- D1959 Test Method for Iodine Value of Drying Oils and Fatty Acids (Withdrawn 2006)<sup>3</sup>
- D2024 Test Method for Cloud Point of Nonionic Surfactants
- D2959 Test Method for Ethylene Oxide Content of Polyethoxylated Nonionic Surfactants
- E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)<sup>3</sup>
- E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis
- E203 Test Method for Water Using Volumetric Karl Fischer Titration
- E394 Test Method for Iron in Trace Quantities Using the 1,10-Phenanthroline Method

## 3. Significance and Use

3.1 Alcohol ethoxylates and alkylphenol ethoxylates are important surfactants in household and industrial cleaners.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

They may be used as either nonionic surfactants or sulfated and used as anionic surfactants. Careful control of the ethoxylate characteristics is desired as variations may result in either desirable or undesirable end-use properties.

#### 4. Purity of Reagents

4.1 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.<sup>4</sup> 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.

4.2 Unless otherwise indicated, references to water shall be understood to mean Type III reagent water conforming to Specification **D1193**.

#### 5. Safety Precautions

5.1 All reagents and chemicals should be handled with care. Before using any chemical, read and follow all safety precautions and instructions on the manufacturer label. Clean up any spill immediately. For information on cleaning up spills refer to the Laboratory Disposal Manual, Manufacturing Chemists Association, Washington, DC.

### SAMPLING

#### 6. Liquids

6.1 Thoroughly mix the sample before sampling. If a solid layer or crystals have formed on the bottom of the bottle, melt in an oven or water bath at 55°C and mix well before sampling.

#### 7. Solids

7.1 Melt in an oven or water bath at 55°C and mix well before sampling. If it is necessary to heat at temperatures above 60°C, replace the gas cap with an inert gas, such as oxygen-free nitrogen, helium, or argon before heating.

### WATER OR MOISTURE

#### 8. Procedure

8.1 Determine water or moisture in accordance with Test Method **E203**.

### REFRACTIVE INDEX

#### 9. Procedure

9.1 Measure the refractive index at 50°C in accordance with Test Method **D1218**.

<sup>4</sup> *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.

#### 10. Precision

10.1 The following criteria should be used for judging the acceptability of results:<sup>5,6</sup>

10.1.1 *Repeatability (Single Analyst)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.00021 units absolute at 36 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.00059 units absolute.

10.1.2 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.00059 units absolute at 7 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.0020 units absolute.

10.1.3 *Checking Limits for Duplicates*—Report the refractive index of the sample to the nearest 0.0001. Duplicate runs that agree within 0.00025 units are acceptable for averaging (95 % confidence level).

### pH

#### 11. Procedure

11.1 Determine the pH in accordance with Test Method **D1172**, except prepare the solution by transferring  $1 \pm 0.001$  g of the sample to a 100 mL volumetric flask and diluting in accordance with Test Method **D1172**. Measure the pH 10 min after diluting the sample solution to volume.

### ACIDITY OR BASICITY

#### 12. Procedure

12.1 Determine acidity or basicity in accordance with Test Method **D1613** using a 3+1 solution of isopropyl alcohol and water as the solvent and a 10-g sample.

#### 13. Precision

13.1 The following criteria should be used for judging the acceptability of results:<sup>6,7</sup>

13.1.1 *Repeatability (Single Analyst)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.017 meq/100 g at 27 df. Two such averages should be considered suspect (95 % confidence level) if they differ by more than 0.049 meq/100 g.

13.1.2 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been established to be 0.12 meq/100 g at 5 df. Two such averages should be

<sup>5</sup> The precision estimates are based on an interlaboratory study on ALFONIC® 1412-60, MAKON®-10, NEODOL® 25-9, PLURONIC® 25R1, and TERGITOL 15-S-3 by nine laboratories.

<sup>6</sup> Statistical analysis was performed in accordance with Practice **E180** for developing precision estimates. Data supporting the precision statements are on file at ASTM International Headquarters. Request RR: RR:D12-1004.

<sup>7</sup> The precision estimates are based on an interlaboratory study on ALFONIC® 1412-60, MAKON®-10, NEODOL® 25-9, PLURONIC® 25R1 and TERGITOL® 15-S-3 by six laboratories.