



# Standard Test Method for Inorganic Sulfate in Surfactants by Potentiometric Lead Titration<sup>1</sup>

This standard is issued under the fixed designation D 6174; 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 describes a potentiometric titration procedure for determining the inorganic sulfate content of surfactants. It is intended for the analysis of α-olefin sulfonates, alcohol sulfates, alcohol ether sulfates, alkylbenzene-sulfonates, and the like.

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:*  
D 1193 Specification for Reagent Water<sup>2</sup>

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *inorganic sulfate, n*—sulfate species present as sulfuric acid, ionic salts of this acid, or mixtures of these.

## 4. Summary of Test Method

4.1 A surfactant sample containing inorganic sulfate is titrated in ethanolic medium with a standard lead solution. Lead sulfate precipitate is formed during the titration. Ethanol and sodium perchlorate are present to decrease the solubility of lead sulfate, thus sharpening the endpoint. Acetic acid is added to remove possible interference from carbonate. The endpoint is signaled by an increase in lead ion activity, as measured by a lead-selective electrode.

## 5. Significance and Use

5.1 Anionic surfactants, such as those listed in 1.1, commonly are used in detergent formulations. Their acceptability for use depends on their purity. Sulfate content, as measured by this test method, can be used to estimate the purity of an anionic surfactant under test.

## 6. Apparatus

6.1 *Potentiometric Titration Assembly*, consisting of an automatic titrator fitted with a lead ion-selective electrode, a double-junction reference electrode, and a 10-mL buret. The reference electrode should be filled with the standard inner and outer filling solutions supplied with it. A TFE-fluorocarbon-coated magnetic stirring bar should be used for mixing during titration, with a separate magnetic stirring motor if the autotitrator is not so equipped.

NOTE 1—Proper care of the lead-selective electrode is essential for obtaining high-quality titration curves. Follow manufacturer's instructions.

## 7. Reagents

- 7.1 *Glacial Acetic Acid*.  
7.2 *Lead Nitrate*, reagent grade.  
7.3 *Sodium Sulfate*, anhydrous, reagent grade.  
7.4 *Sodium Perchlorate*, reagent grade.  
7.5 *Ethanol*, denatured, formula 3A.  
7.6 *Water*, Type III reagent water conforming to Specification D 1193.<sup>3</sup>

## 8. Preparation of Standard Solutions

- 8.1 *10 % Acetic Acid*—Dilute glacial acetic acid 1/10 with water.  
8.2 *Lead Titrant, 0.05 M*—Dissolve 16.6 g lead nitrate in 300 mL water. Pour into a 1-L bottle and fill with 3A ethanol. Mix well. Standardize according to 9.1.  
8.3 *Sulfate Standard, 0.05 M*—Dry 5 g anhydrous sodium sulfate at 110°C for 1 h. Accurately weigh about 3.5 g into a 500-mL volumetric flask, dilute to volume with water, and mix to dissolve. Calculate the exact concentration as follows:

$$\frac{G}{(142.02)(0.500)} = \text{Molarity} \quad (1)$$

where:

<sup>3</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 Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D12 on Soaps and Detergents and is the direct responsibility of Subcommittee D12.12 on Current edition approved Sept. 10, 1997. Published April 1998.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.