



Standard Test Method for Synthetic Anionic Active Ingredient in Detergents by Cationic Titration Procedure¹

This standard is issued under the fixed designation D1681; 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 Direct titration of an anionic surfactant with a standardized cationic reagent is a simple and convenient method for the quantitative determination of the content of active ingredient. The end point is detected by the transfer of a colored complex from an organic solvent phase to an aqueous phase. The relationship between anionic and cationic agents is not always stoichiometric, and for maximum accuracy the anionic type of interest should first be characterized and then used to standardize the cationic reagent. In most cases, however, the different anionic surfactants likely to be encountered react in the same proportions. That is, a cationic titrating solution standardized against a characterized anionic agent can be used to analyze other anionics of known molecular weights.

1.2 This test method is applicable to alkylaryl sulfonates and fatty alkyl sulfates. Low results are obtained with alkylbenzene sulfonates having the alkyl chain length less than eight carbon atoms. Low results are also obtained for alkyl sulfates with the alkyl chain length of less than twelve carbon atoms. The anionic surfactants characterized in accordance with Sections 17 – 23 should be the sodium salt and not amine, ammonium, or potassium salts. In case only amine or ammonium salts are available, they should be first converted to the sodium salt before proceeding with this analysis.

1.3 The analytical procedures appear in the following order:

	Sections
Separation of Alcohol-Soluble Matter	8 and 9
Separation of Oil-Free Sulfonate	10 and 11
Determination of Sodium Chloride (NaCl) Content	12 – 17
Characterization of Anionic Surfactant Standard:	
Part I. Determination of Surfactant, SO ₃ Content, and Solution Molarity	18 – 20
Part II. Determination of Surfactant, SO ₃ and Active Ingredient Contents Combining Weight, and Solution Molarity	21 – 24
Standardization of Cationic Reagent	25 – 29
Quantitative Determination of Anionic Surfactant by Cationic Titration	30 – 33

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 Jan. 1, 2014. Published February 2014. Originally approved in 1959. Last previous edition approved in 2005 as D1681 – 05. DOI: 10.1520/D1681-05R14.

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

1.5 *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.* For a specific hazards statement, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

3. Summary of Test Method

3.1 This test method involves isolating alkylaryl sulfonate or fatty alkyl sulfate by extraction with ethyl alcohol to remove inorganic salts, extracting the alcohol-soluble material with petroleum ether to remove unsulfonatable matter, correcting for sodium chloride present, and determining the active ingredient combining weight. The characterized surfactant is used to standardize a cationic reagent which in turn is used to titrate similar anionic surfactants.

4. Significance and Use

4.1 This test method determines anionic detergents commonly found in laundry, dishwashing, and other cleaning materials. Accurate determination of the anionic active substance is highly important in assessing the cost and effectiveness of such cleaning substances.

5. Interferences

5.1 With the exception of picrate, perchlorate, thiocyanate, nitrate, dichromate, and chromate, common inorganic anions and low molecular weight organic anions do not interfere with this analysis. However, sulfonates of xylene, cumene, or toluene interfere positively. Since the cationic titration is conducted under acidic conditions, soap does not interfere. Soap

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.