Designation: D2358 – 89 (Reapproved 2009)

Standard Test Method for Separation of Active Ingredient from Surfactant and Syndet Compositions[1]

This standard is issued under the fixed designation D2358; 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 procedure for the separation and purification of active ingredient from surfactants and syndet compositions. The separated active ingredient may be used for qualitative examinations. This test method also permits the estimation of total active ingredient level present in the sample under test.

1.2 This test method yields the active ingredient together with other alcohol-soluble materials and therefore is useful only in estimating the actual active ingredient level. Correction for the amount of the most common contaminant, sodium chloride, is shown by a separate determination.

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.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1681 Test Method for Synthetic Anionic Active Ingredient in Detergents by Cationic Titration Procedure

3. Summary of Test Method

3.1 The test method involves the extraction of the active ingredient with alcohol. Reprecipitation of the insolubles is specified to remove the last traces of active ingredient. Dilution of the alcoholic extract to a known volume and the evaporation of a suitable aliquot permits measurement of total alcohol-soluble matter. An estimation of sodium chloride content is made so that a corrected total active ingredient level may be obtained. Provision is made for purification of the active ingredients in Section 14.

4. Reagents

4.1 *Purity of Reagents*:

4.1.1 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.[3] 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 distilled water or water of equal purity.

SEPARATION OF TOTAL ALCOHOL-SOLUBLE MATTER

5. Reagents

5.1 *Ethyl Alcohol (95 percent)*—Freshly boiled ethyl alcohol conforming to Formula No. 3A or No. 30 of the U. S. Bureau of Internal Revenue. The alcohol should not be neutralized. Redistilled alcohol shall be used if alkali absorption is more than 0.2 mL of 0.1 *N* NaOH solution/100 mL of alcohol.

5.2 *Ethyl Alcohol (Absolute)*—Freshly boiled 200-proof ethyl alcohol conforming to either Formulas No. 3A or No. 30 of the U. S. Bureau of Internal Revenue.

5.3 *Phenolphthalein Indicator Solution (10 g/L)*—Dissolve 1 g of phenolphthalein in 50 mL of ethyl alcohol (95 %) and then mix with 50 mL of water.

5.4 *Sulfuric Acid (1 + 100)*—Add 1 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) to 100 mL of water.

---

[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 Oct. 1, 2009. Published December 2009. Originally approved in 1965 as D2358 – 65 T. Last previous edition approved in 2003 as D2358 – 89 (2003). DOI: 10.1520/D2358-89R09.

[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.

[3] *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.

6. Safety Precautions

6.1 Formulas No. 3A and No. 30 ethyl alcohols are denatured alcohols. They are health hazards and flammable liquids. See manufacturer's label warning as to use, safe handling, and disposal.

6.2 Sulfuric acid is corrosive. Use proper protective equipment including adequate eye protection. If acid contacts the body or is splashed in the eyes, flush the affected parts with water for at least 15 min. Obtain medical attention.

7. Procedure

7.1 Weigh out a sample, to the nearest 0.01 g, to correspond with the levels of active ingredient prescribed in Table 1 and transfer to a 600-mL beaker. Liquid samples containing high levels of water should be evaporated to a pasty consistency after weighing. Samples containing high levels of hydrated alkaline salts should be dried in an oven at 105°C for 1 h after weighing.

7.2 Add 300 to 350 mL of hot 95 % alcohol. Cover with a watch glass and heat on a steam bath for approximately 1½ to 2 h, stirring frequently to disperse the solids and break up any lumps.

7.3 Remove the beaker from the steam bath and permit the insolubles to settle for a few minutes. Add 2 drops of phenolphthalein indicator solution. If the solution is pink, neutralize with 1 *N* H_2SO_4 using no more than 1 mL. Samples containing high levels of free alkali, requiring more than 1 mL of 1 *N* H_2SO_4, should be neutralized with H_2SO_4 of higher concentration to limit the amount added to less than 1 mL. Before proceeding, make the solution just alkaline with 0.1 *N* NaOH solution. If the solution is acid originally, neutralize the free acid with alcoholic NaOH solution to the phenolphthalein end point. Decant the alcoholic solution through a suitable filter, retaining as much as possible of the insoluble matter in the beaker. Collect the clear filtrate in a suitable filter flask or beaker.

7.4 Add 50 mL of hot 95 % alcohol to the residue in the beaker and bring to boiling on a safety hot plate. Let the insolubles settle and decant the alcoholic solution through the filter as before. Repeat the extraction of the insolubles with an additional 50 mL of 95 % alcohol, retaining the bulk of the insolubles in the beaker.

7.5 Evaporate the residual alcohol from the residue in the beaker on a steam bath, stirring at intervals to permit complete evaporation of the solvent. Dissolve the residue in the beaker in a minimum volume of hot water, not to exceed 10 mL, heating on a steam bath to effect solution.

7.6 Dilute the water solution with 200 mL of hot absolute alcohol. Bring to a boil on a steam bath and filter through the previously used filter, combining the filtrate with the initial alcoholic filtrate. Wash the beaker and residue several times with hot 95 % alcohol, transferring the insolubles to the filter with several small portions of hot 95 % alcohol.

7.7 Evaporate the combined alcoholic filtrate and washings to approximately 450 mL and transfer to a 500-mL volumetric flask. Cool to room temperature and dilute to volume with 95 % alcohol. Mix well.

7.8 Pipet a 100-mL aliquot into a tared flask and evaporate to dryness on a steam bath. Place in an oven maintained at 105 ± 2°C and dry for half-hour periods to constant weight.

TABLE 1 Active Ingredient Level Expected

Active Ingredient Level Expected, %	Weight of Sample, g
10 to 25	20
25 to 40	15
40 to 60	10
60 to 80	7
Over 80	5

8. Calculation

8.1 Calculate the percentage of total alcohol-soluble matter as follows:

$$\text{Total alcohol}-\text{soluble matter, \%} = [(W - T)/S] \times 100 \quad (1)$$

where:
- W = weight of dish plus alcohol-soluble matter, g,
- T = weight of dish, g, and
- S = grams of sample in aliquot taken.

CORRECTION FOR SODIUM CHLORIDE CONTENT

9. Procedure

9.1 Pipet a suitable aliquot from the solution as prepared in 7.7 into a 400-mL beaker. Evaporate to a volume of about 30 mL and dilute to 100 mL with water. Add 2 drops of methyl orange indicator solution and acidify to the acid color by using HNO_3(1+4). Warm slightly, stir, and add 50 mL of acetone. Follow the procedure for quantitative determination, starting with 13.2 of Method D1681.

10. Calculation

10.1 Calculate the percentage of sodium chloride (NaCl) as follows:

$$\text{NaCl, \%} = [(B \times N \times 0.05845)/S] \times 100 \quad (2)$$

where:
- B = millilitres of $AgNO_3$ solution used,
- N = normality of the $AgNO_3$ solution, and
- S = grams of sample in aliquot taken.

TOTAL ACTIVE INGREDIENT

11. Calculation

11.1 Calculate the corrected alcohol-soluble matter, which is a measure of the total active ingredient, as follows:

$$\text{Alcohol}-\text{soluble matter, corrected, \%} = A - C \quad (3)$$

where:
- A = percentage of total alcohol-soluble matter (Section 8), and
- C = percentage of NaCl (Section 10.).

PURIFICATION OF TOTAL ALCOHOL-SOLUBLE MATTER

12. Reagent

12.1 *Acetone-Ether Mixture (1+1)*—Mix 1 part of acetone with 1 part of ether.

13. Safety Precautions

13.1 Acetone is a flammable solvent and a health hazard. This test should be performed in a well-ventilated explosion-proof area and protective gloves and garments should be used.

13.2 Ether is a skin, brain, and kidney irritant and an extreme fire and explosion hazard. All precautions should be taken to proceed with this test only in a special explosion-proof area and under a special explosion-proof hood. Protective gloves and garments should be used.

NOTE 1—See special instructions on manufacturer's label for acetone and ether warning and disposal.

14. Procedure

14.1 To the dried residue from 7.8 add 50 mL of acetoneether mixture and warm on a steam bath.

14.2 Agitate with a glass stirring rod and filter while warm through a general-purpose acid-washed filter paper. Collect the filtrate in a small beaker. Wash the paper with a few small volumes of acetone-ether mixture.

14.3 Evaporate the combined filtrate and washings on a steam bath. Dry the residue in an oven at 105 ± 2°C.

NOTE 2—The evaporated fraction may be used for infrared spectrophotometric or qualitative examination. If the sample size is insufficientfor these purposes, a larger aliquot may be taken for evaporation.

15. Keywords

15.1 active ingredient; alcohol extraction; surfactant compositions; syndet compositions