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Standard Test Method for Synthetic Anionic Active Ingredient in Detergents by Cationic Titration Procedure[1]

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 detffected 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

1.4 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, cunene, or toluene interfere positively. Since the cationic titration is conducted under acidic conditions, soap does not interfere. Soap

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[1] This test method is under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergentsand is the direct responsibility of Subcommittee D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components.

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[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.

must not be present in the anionic surfactant characterized in accordance with Sections 17 – 23.

6. Reagents

6.1 *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.[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.

6.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193.[3]

7. Hazards

7.1 All reagents and chemicals should be handled with care. Before using any chemical, read and follow all safety precautions and instructions on the manufacturer's label or MSDS (Material Safety Data Sheet).

SEPARATION OF ALCOHOL-SOLUBLE MATTER

8. Solvent

8.1 *Ethyl Alcohol*—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 must be used if alkali absorption is more than 0.2 mL of 100 mL of 0.1 *N* NaOH solution.

9. Procedure

9.1 Weigh out a sample, to the nearest 0.01 g, to correspond to surfactants with the following ranges of active ingredient contents and transfer to a 600-mL beaker:

Active Ingredient, wt %	Weight of Sample, g
10 to 25	30
25 to 40	15
40 to 60	10
60 to 80	7
Over 80	5.5

9.2 Add 300 to 350 mL of hot alcohol. Cover with a watch glass and heat on the steam bath for at least 2 h, stirring frequently to disperse solids and break up lumps. Have a prepared Gooch crucible or sintered glass filter inserted in the 1000-mL vacuum flask.

9.3 At the end of 2 h, remove the beaker from the bath and decant the alcohol solution rapidly through the filter, retaining as much as possible of the residue in the beaker. Add 50 mL of hot alcohol to the residue in the beaker. Heat to boiling on a hot plate, breaking up any lumps of the residue. Decant the alcohol through the filter as before. Repeat with another 50-mL portion of hot alcohol.

9.4 Evaporate the residual alcohol from the residue in the beaker on the steam bath, stirring at intervals, especially near the end. Dissolve the residue in the beaker with 10 mL of hot water, heating on the steam bath until solution is effected.

9.5 Dilute the water solution with 200 mL of hot alcohol, bring to a boil on the steam bath, and filter. Transfer the precipitate onto the filter with the aid of hot alcohol and policeman. Wash the beaker and the residue with hot alcohol three or four times. For samples being prepared for Part I of the characterization of anionic surfactant (Sections 18 – 20), continue as directed in 9.6. For samples being prepared for eventual use in Part II of the characterization of anionic surfactant (Sections 21 – 24), continue in accordance with the separation of oil-free sulfonate (Sections 10 and 11).

9.6 Transfer the filtrate to a 1000-mL beaker. Wash the filter flask with alcohol and 10 mL of water followed by an alcohol wash. Evaporate the filtrate to about 400 mL and transfer to a 1000-mL volumetric flask. Dilute with water to mark. Designate as Solution I and reserve for use in Part I of the characterization of the anionic surfactant (Sections 18 – 20).

SEPARATION OF OIL-FREE SULFONATE

10. Reagents

10.1 *Ethyl Alcohol*—See Section 8.

10.2 *Petroleum Ether,* having a boiling point of 30 to 60°C.

11. Procedure

11.1 Transfer the alcohol solution quantitatively to a 1000-mL beaker (with the 100-mL volume marked on the side) and concentrate to 100 mL on a steam bath or hot water bath. Transfer the concentrate quantitatively to a 500-mL, pear-shaped separatory funnel by rinsing the beaker with 100 mL of water in several portions. Add the remainder of water to the funnel to bring the total volume to 200 mL.

11.2 Extract the aqueous-alcoholic solution with three 50-mL portions of petroleum ether, using additional separatory funnels. Combine the ether extracts and wash with 3 to 50- mL portions of 50 % aqueous ethanol. Add the aqueous ethanol washings to the extracted aqueous alcohol solution. Discard the petroleum ether extracts. Transfer the oil-free aqueous alcoholic solution quantitatively to a 1000-mL beaker by rinsing the separatory funnel with small portions of water. Heat the solution in a 400-mL beaker in a water bath at 40 to 50°C in a hood to vent off petroleum ether fumes. When free of ether, transfer the oil-free salt-free solution quantitatively to a 1000-mL volumetric flask. Add 300 mL of alcohol and dilute to volume with water. Designate as Solution II and reserve for use in Part II of the characterization of the anionic surfactant (Sections 21 – 24).

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[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.

DETERMINATION OF SODIUM CHLORIDE (NaCl) CONTENT

12. Scope

12.1 Sections 13 – 17 cover the determination of the sodium chloride content of the surfactant in order that appropriate corrections can be made in the values obtained in the characterization of the anionic active ingredients and the quantitative determination of an unknown anionic active ingredient.

13. Apparatus

13.1 *Stirrer Motor and Small Glass Rod Stirrer.*

13.2 *Potentiometer.*[4]

13.3 *Calomel Reference Electrode,* saturated.

13.4 *Silver Wire Electrode,* 1mm in diameter by 120 mm in length.

14. Reagents and Materials

14.1 *Acetone.*

14.2 *Ethyl Alcohol*—Freshly boiled ethyl alcohol conforming to Formula No. 3A or No. 30 of the U.S. Bureau of Internal Revenue.

14.3 *Methyl Orange Indicator.*

14.4 *Nitric Acid (1+1)*—Mix 1 volume of HNO_3 (sp gr 1.42) containing 0.3 % sodium nitrite ($NaNO_2$) with 1 volume of water.

14.5 *Nitric Acid (1+4)*—Mix 1 volume of nitric acid (HNO_3, sp gr 1.42) with 4 volumes of water.

14.6 *Silver Nitrate, Standard Solution (0.2 N)*—Prepare and standardize a 0.2 *N* silver nitrate ($AgNO_3$) solution as follows: Weigh 17 g of $AgNO_3$ to the nearest 1 mg. Dissolve in water and transfer to a 500-mL volumetric flask. Dilute to the mark. Standardize as follows:

14.6.1 Dry about 10 g of NaCl at 110°C to constant weight. Weigh about 2.00 g of the dried NaCl to the nearest 1 mg. Dissolve in a solvent consisting of 60 % water and 40 % alcohol. Transfer to a 100-mL volumetric flask and dilute to the mark with solvent. Pipet 100 mL of the NaCl solution to a beaker and titrate with the $AgNO_3$ solution as described in Section 15.

14.6.2 Calculate the normality of the $AgNO_3$ solution as follows:

$$\text{Normality} = (A \times 100)/(B \times 58.45) \tag{1}$$

where:

A = grams of NaCl used, and
B = millilitres of $AgNO_3$ solution required for titration of the NaCl.

15. Procedure for Qualitative Determination

15.1 Place 5 mL of Solution I or II in a test tube and acidify with HNO_3 (1+4). Add several drops of $AgNO_3$ solution. If the solution becomes turbid, it contains NaCl. Presence of soap will also cause turbidity. Soap must not be present with the surfactant to be characterized.

16. Procedure for Quantitative Determination

16.1 Pipet 100-mL portions of Solution I or II into each of two 400-mL beakers. Add 2 drops of methyl orange indicator solution and acidify to the acid color by using HNO_3 (1+4). Warm slightly and stir to effect maximum solution. Add 50 mL of acetone.

16.2 Clean the silver electrode in the HNO_3 (1+1) containing $NaNO_2$. Set up the titration cell with the silver electrode connected to the top terminal, the saturated calomel cell connected to the bottom terminal. Set the pH meter on + mV. Start the stirring and titrate the solution potentiometrically as follows:

16.2.1 Add 0.5 mL of $AgNO_3$ solution and measure the emf. If appreciable chloride is present, the emf should be in the range of 100 mV.

16.2.2 Add $AgNO_3$ solution slowly in 2 to 3-mL portions until the emf reaches 200 mV. Stir well.

16.2.3 Add $AgNO_3$ solution in 0.1-mL portions, allowing sufficient time after each addition for the solution to reach equilibrium (60 to 80 s). Measure the emf (stirrer off) at each 0.1-mL point.

16.2.4 Calculate the end point by the rate of change method (Note 1). The end point is usually in the range from 260 to 270 mV.

NOTE 1—*Example*—The method for determining the maximum rate of change is as follows:

mL	emf	ΔE	ΔE'
21.2	210}	10}	10
21.3	220}	20}	17[A]
21.4	240}	37}	12
21.5	277}	25	
21.6	302		

[A] Maximum rate of change.

$$\text{End point} = 21.3 + \frac{17}{17+12} \times 0.1 \tag{2}$$

$$= 21.46 \text{ mL}$$

16.3 Run a blank and subtract the value obtained from the value calculated in 16.2.4.

17. Calculations

17.1 Calculate the grams of NaCl present in 250 mL of Solution I or II as follows:

$$A = (B \times N \times 0.05845 \times 250)/100 \tag{3}$$

where:

A = grams of NaCl present in 250 mL of Solution I or II,
B = millilitres of $AgNO_3$ solution required for titration of the NaCl, and
N = normality of the $AgNO_3$ solution.

17.2 Calculate the grams of NaCl equivalent to sodium sulfate (Na_2SO_4) present in 250 mL of Solution I or II as follows:

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[4] The Beckman Model G pH meter, or its equivalent, has been found satisfactory for this purpose.

$$A = (B \times 71.03)/58.45 \quad (4)$$

where:

A = grams of Na_2SO_4 equivalent to the NaCl in 250 mL of Solution I or II, and

B = grams of NaCl present in 250 mL of Solution I or II.

CHARACTERIZATION OF ANIONIC SURFACTANT STANDARD

Part I. Determination of Surfactant, SO_3 Content, and Solution Molarity

18. Reagents

18.1 *Ethyl Alcohol*—See Section 6.

18.2 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H_2SO_4).

19. Procedure

19.1 Pipet 250 mL of Solution I (9.6) into each of two 250-mL beakers. Evaporate to near dryness on a steam bath or under an infrared lamp. Transfer quantitatively the concentrated solution from the beaker into a tared, ignited, platinum crucible. Wash the beaker with small amounts of alcohol and water to ensure complete transfer to the crucible.

19.2 Evaporate the contents in the crucible to dryness on a steam bath or under an infrared lamp, adding small portions of alcohol to aid in removal of water.

19.3 Carefully heat the crucible, supported on a quartz triangle, over a small flame until the sample is charred and no longer smoking. *Avoid spattering*. Cool and add 3 to 4 drops of H_2SO_4. Heat carefully again with a small flame until fuming ceases. *Avoid spattering*. The residue should be gray or white. In case it is dark or dark spotted, cool and add about 1 mL of H_2SO_4.

19.4 Slowly heat over a small flame and fume off H_2SO_4. Then place the crucible in a muffle furnace at 800°C for 20 min (or over a Meker burner at full heat for 20 min), desiccate, and weigh.

19.5 Add 3 to 4 drops of H_2SO_4, fume off acid over a small flame, repeat heating in muffle furnace at 800°C for 20 min, desiccate, and weigh the crucible. First and second weights should agree within 1 mg. In case of poor agreement, repeat the acid treatment, fuming, and heating steps. Record weights to the nearest 0.1 mg.

20. Calculations

20.1 Calculate the molarity, M_I, of Solution I as follows:

$$M_I = (A \times 80.07 \times 4)/(71.03 \times 80.07) \quad (5)$$

where:

A = grams of ash (Note 2).

20.2 Calculate the percentage of sulfur trioxide (SO_3) in the sample as follows:

$$SO_3, \text{wt\%} = [(A \times 160.14 \times 1000)/(B \times 142.06 \times 250)] \times 100 \quad (6)$$

where:

A = grams of ash (Note 2), and

B = grams of sample used.

NOTE 2—The weight of the ash must be corrected for NaCl content which is converted to Na_2SO_4 in accordance with 17.2.

NOTE 3—A sulfur analysis on the dried residue from Solution I may be run in addition to the ash content.

Part II. Determination of Surfactant, SO_3 and Active Ingredient Contents, Combining Weight, and Solution Molarity

21. Apparatus

21.1 *Vacuum Oven.*

22. Reagents

22.1 *Ethyl Alcohol*—See Section 6.

22.2 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H_2SO_4).

23. Procedure

23.1 Pipet 25 mL of Solution II (11.2) into each of two 250-mL beakers. Evaporate to near dryness on a steam bath or under an infrared lamp. Transfer the concentrated solution quantitatively from the beaker into a tared, ignited, platinum crucible. Wash the beaker with small amounts of alcohol and water to ensure complete transfer to the crucible.

23.2 Evaporate the contents in the crucible to dryness on a steam bath or under an infrared lamp, adding small portions of alcohol to aid in removal of water.

23.3 Dry the residue to constant weight (within 1 mg) in a vacuum oven at 60°C. (This may require as long as 2 days.) Record the weight of the purified active ingredient to within 0.1 mg.

23.4 Carefully heat the crucible, supported on a quartz triangle, over a small flame until the sample is charred and no longer smoking. *Avoid spattering*. Cool and add 3 to 4 drops of $H_2 SO_4$. Heat carefully again with a small flame until fuming ceases. *Avoid spattering*. The residue should be gray or white. In case it is dark or dark spotted, cool and add about 1 mL of H_2SO_4.

23.5 Slowly heat over a small flame to fume off H_2SO_4. Then place the crucible in a muffle furnace at 800°C for 20 min (or over a Meker burner at full heat for 20 min), desiccate, and weigh.

23.6 Add 3 to 4 drops of H_2SO_4, fume off acid over a small flame, and repeat heating in a muffle furnace at 800°C for 20 min, desiccate, and weigh the crucible. First and second weighings should agree within 1 mg. In case of poor agreement repeat the acid treatment, fuming, and heating steps. Record weights to the nearest 0.1 mg.

24. Calculations

24.1 Calculate the percentage of purified active ingredient in the sample as follows:

$$\text{Active ingredient, \%} = [(A \times 4)/B] \times 100 \quad (7)$$

where:

A = grams of dried, purified active ingredient in the sample (Note 4), and
B = grams of sample used.

NOTE 4—The weight of the dried ingredient must be corrected for NaCl content in accordance with 17.1.

24.2 Calculate the combining weight of the active ingredient as follows:

$$\text{Combining weight} = (A/B) \times 71.03 \quad (8)$$

where:

A = grams of dried, purified active ingredient (Note 4), and
B = grams of ash (Note 2).

24.3 Calculate the percentage of SO_3 in the sample as follows:

$$SO_3,\ \text{wt\%} = [(A \times 160.14 \times 1000)/(B \times 142.06 \times 23)] \times 100 \quad (9)$$

where:

A = grams of ash (Note 2), and
B = grams of sample used.

24.4 Calculate the molarity, M_{II}, of the oil-free, salt-free aqueous alcohol Solution II as follows:

$$M_{II} = (A \times 80.07 \times 4)/(71.03 \times 80.07) \quad (10)$$

where:

A = grams of ash (Note 2).

NOTE 5—A sulfur analysis of the dried residue from Solution II may be run in addition to the ash content.

STANDARDIZATION OF CATIONIC REAGENT

25. Apparatus

25.1 *Extraction Cylinders,* 100-mL, glass-stoppered.

25.2 *Microburet,* 23-mL.

26. Reagents and Materials

26.1 *n-Butanol.*

26.2 *Cetyltrimethylammonium Bromide* (CTAB) (0.005 *M*, or 1.80 g/L)[5]—The reagent should be filtered after being made up to 0.005 *M*. Standardize the solution in accordance with Section 26. This solution must be standardized monthly.

NOTE 6—Hyamine 1622[6] (0.005 *M*) may be used as an alternative. In some cases sharper titration end points are obtained with the Hyamine 1622.

26.3 *Chloroform.*

26.4 *Indicator Solution*—Prepare the following solution:

26.4.1 *Methylene Blue Chloride (3.00 g/L)*—Dissolve 0.300 g of methylene blue chloride in 100 mL of water.

26.4.2 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H_2SO_4).

26.4.3 *Sodium Sulfate* (Na_2SO_4), anhydrous. Mix the reagents in the following proportions, diluting the final solution to 1 L with water:

Methylene blue chloride	10.0 mL
Sulfuric acid	6.6 mL
Sodium sulfate	50.0 g

27. Procedure

27.1 Prepare a 0.0045 to 0.0050 ± 0.00001 *M* solution (Solution III) of characterized anionic surfactant solution as follows:

27.1.1 Pipet the following amount of Solution I or Solution II into a 250-mL beaker:

$$A = (250 \times 0.0045)/(M_I \ or \ M_{II}) \quad (11)$$

where:

A = millilitres of solution to be used, to the nearest 3 mL,
M_I = molarity of Solution I, and
M_{II} = molarity of Solution II.

27.1.2 Heat the solution on a steam bath to about a 10- mL volume to remove ethanol.

27.1.3 Transfer the contents of the beaker to a 250-mL volumetric flask. Rinse beaker well with water and add the rinsings to the flask. Add 15 mL of *n*-butanol, mix well, and dilute to the mark with water. Call this Solution III.

27.1.4 Pipet 10-mL aliquots of Solution III into each of two 100-mL stoppered cylinders. Add by pipet 25 mL of indicator solution and 15 mL of chloroform. Add 5 mL of CTAB solution from a 10-mL microburet.

27.1.5 Shake the mixture well. Allow the two layers to separate and then continue the titration. Add small increments of CTAB, followed by vigorous shaking until the end point is reached. The end point of the titration is reached when the color is the same intensity in the two layers. Make comparisons by reflected light using a white sheet of paper as the background. Allow the cylinder to stand 1 min before viewing the layers.

27.1.6 About 15 to 20 mL of CTAB should be required to titrate the standard anionic solutions. In case more or less than this amount is required, vary the volume of Solution III to bring the CTAB in range.

28. Calculations

28.1 Calculate the molarity, M_{III}, of Solution III as follows:

$$M_{III} = M_I \ or \ M_{II} \times A/250 \quad (12)$$

where:

M_I = molarity of Solution I,
M_{II} = molarity of Solution II, and
A = millilitres of Solution I or II used.

28.2 Calculate the molarity, M_{CTAB}, of the CTAB solution as follows:

$$M_{CTAB} = (M_{III} \times A / B) \quad (13)$$

where:

M_{III} = molarity of Solution III,
A = millilitres of Solution III used, and
B = millilitres of CTAB solution used.

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[5] Reagent No. T-5650 supplied by the Eastman Kodak Co., or its equivalent, has been found satisfactory for this purpose.

[6] Hyamine 1622 supplied by Rohm and Haas, or its equivalent, has been found satisfactory for this purpose.

29. Precision

29.1 Duplicate titrations should agree within 0.05 mL of CTAB.

QUANTITATIVE DETERMINATION OF ANIONIC SURFACTANT BY CATIONIC TITRATION

30. Apparatus

30.1 *Extraction Cylinders,* 100-mL, glass-stoppered.

30.2 *Microburet,* 10-mL.

31. Procedure

31.1 Estimate the active ingredient content of an "as-received" sample as the difference between the sum of the alcohol-insoluble and volatile contents and 100 wt %, or by a trial titration.

31.2 Prepare a 6 % *n*-butanol solution of the sample as follows: Dissolve the amount of sample (combining weight about 348) given in Fig. 1 (weighed to the nearest 1 mg) in 100 mL of water in a 250-mL beaker. Transfer to a 250-mL volumetric flask. Thoroughly rinse the beaker and add rinsings to flask. Add 15 mL of *n*-butanol and dilute the contents of the flask to the mark with water. Mix well.

31.3 Pipet a 10-mL aliquot into each of the two 100-mL cylinders, add the other ingredients, and titrate as described in 27.1.4 and 27.1.5.

31.4 About 20 mL of CTAB solution should be required for the titration. If the volume used is less, a higher aliquot of the test solution shall be taken and the titration repeated, or 1 or 2 mL more of the test solution added to the two-phase system and the titration simply continued to the new end point. If the volume is greater than 20 mL, another run must be made with a smaller aliquot.

32. Calculations

32.1 Calculate the percentage of sulfur trioxide (SO_3) in the sample as follows:

$$SO_3,\ wt\,\% = [(A \times B \times 0.0801 \times 250)/(C \times D)] \times 100 \quad (14)$$

where:

A = millilitres of CTAB solution required for titration of the sample,
B = molarity of the CTAB solution,
C = grams of sample used, and
D = millilitres in aliquot.

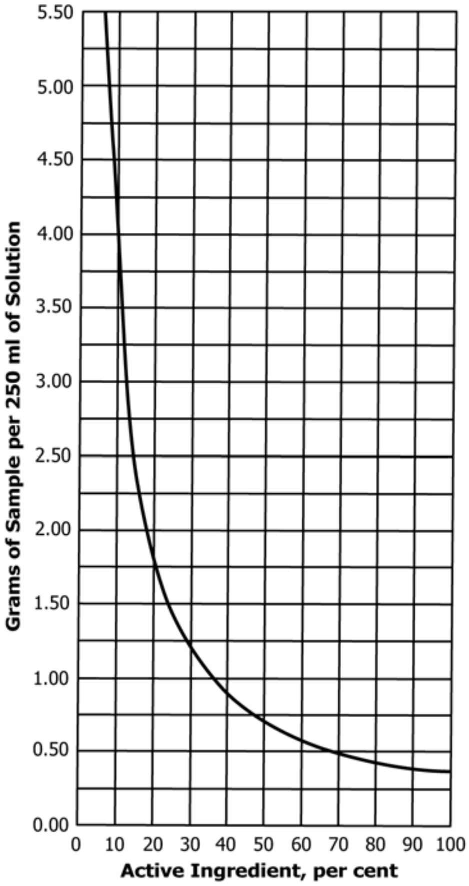

FIG. 1 Sample Size in Relation to Percentage Active Ingredient

32.2 Calculate the weight percentage of active ingredient in the sample as follows:

$$\text{Active ingredient, wt\,\%} = AB/80.01 \quad (15)$$

where:

A = weight percentage of SO_3 present in the sample (32.1), and
B = combining weight of sample.

33. Precision

33.1 The data given in Table 1 were obtained from cooperative analysis of samples by four to eight laboratories. The values are based on averages of duplicate determinations.

TABLE 1 Precision Data

	Standard Deviation		98 % Confidence Limit	
	Within One Laboratory	Between Two Laboratories	Within One Laboratory	Between Two Laboratories
Sulfur trioxide (SO_3) in sample, wt % (for a sample with 9.2 % SO_3)	0.09	0.13	0.23	0.33
Purified active ingredient in sample, wt % (for a sample with 40 % active ingredient)	0.12	0.17	0.31	0.44
Combining weight (for a sample with combining weight of 350)	3.0	4.2	7.8	10.8
CTAB molarity	0.000065	0.000092	0.00019	0.00027
Active ingredient by cationic titration (based on 100 wt % active sample)	0.59	0.83	1.93	2.30

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