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Standard Test Methods for Sampling and Chemical Analysis of Soaps and Soap Products¹

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These methods are identical in substance with the standard methods of the American Oil Chemists' Society which were developed by the Committee on Soap Analysis A-1 of that Society, and with those of the American Chemical Society.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the sampling and chemical analysis of cake, powdered, flake, liquid, and paste soaps, and soap products.

1.2 The test methods appear in the following order:

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1.3 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be approximate.

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.* For specific hazard statements, see Section 10. 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
- D216 Method for Distillation of Natural Gasoline
 - D459 Terminology Relating to Soaps and Other Detergents
 - D1193 Specification for Reagent Water
 - E1 Specification for ASTM Liquid-in-Glass Thermometers

¹ 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 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. Significance and Use

3.1 Soap and soap products are widely used. These test methods are suitable for setting specifications and performing quality control on soap and soap products.

SAMPLING

4. General Requirements

4.1 The seller shall have the option of being represented at the time of sampling, and when he so requests shall be furnished with a duplicate sample.

5. Cake Soaps, Flake and Powdered Soap Products When Packed in Cans or Cartons

5.1 One cake (can or carton) shall be taken at random from not less than 1 % of the seller's shipping containers, provided each package contains not less than 50 lb (22.7 kg). In the case of smaller containers, a cake (can or carton) shall be taken at random from each lot of containers totaling not more than 5000 lb (2268 kg), or fraction thereof. The gross sample shall in all cases consist of not less than three cakes (cans or cartons) taken at random from separate containers. To illustrate, if a total shipment consists of 70 000 lb, all in 1400 containers weighing 50 lb each, then 14 containers are chosen at random and one cake taken from each for a total sample of 14 cakes. If a total shipment of 70 000 lb includes containers weighing less than 50 lb, then the shipment must be divided into 14 lots of containers weighing approximately 5000 lb each, and one cake taken from each lot, again for a total sample size of 14 cakes. The gross sample shall in all cases consist of not less than three cakes (cans or cartons) taken at random from separate containers. In the case of very large lots where the sample drawn as above will amount to more than 20 lb (9.1 kg), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 lb. The individual cakes (cans or cartons) shall be sealed at once in moisture-proof containers such as polyethylene bags, or tightly wrapped in paraffined paper and sealed by rubbing the edges with a heated iron. The inspector shall accurately weigh each wrapped cake (can or carton), and record its weight and the date of weighing on the wrapper. The wrapped cakes (cans or cartons) shall be placed in an airtight container, which should be nearly filled, and which shall then be sealed, marked, and sent to the laboratory for test. Samples shall be kept cool until tested.

6. Flake and Powdered Soap Products When in Bulk

6.1 A grab sample of not less than 0.5 lb (227 g) shall be taken at random from not less than 1 % of the seller's shipping containers, provided each package contains not less than 100 lb (45.4 kg). In the case of smaller containers, a grab sample of not less than 0.5 lb shall be taken at random from each lot of containers totaling not more than 10 000 lb (4536 kg) or fraction thereof. The gross sample shall in all cases consist of not less than three grab samples of 0.5 lb each taken at random from separate containers. In the case of very large lots where the sample drawn as above will amount to more than 20 lb (9.1 kg), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 lb. The inspector

shall rapidly mix the gross sample and place it in an airtight container, which shall be filled, sealed, marked, accurately weighed, its weight and the date of weighing recorded on the package, and sent to the laboratory for test. Samples shall be kept cool until tested.

7. Liquid Soap

7.1 A sample of not less than 0.5 pt (236.6 mL) shall be taken at random from not less than 1 % of the seller's shipping containers, provided each package contains not less than 10 gal (37.9 L). In the case of smaller containers, a sample of not less than 0.5 pt shall be taken at random from each lot of containers totaling not more than 1000 gal (3785.4 L) or fraction thereof. The gross sample shall in all cases consist of not less than three samples of 0.5 pt each taken at random from separate containers. Before drawing the sample from the container selected, the contents of the container shall be thoroughly agitated. The inspector shall thoroughly mix the gross sample, place it in clean, dry cans or bottles, which shall be completely filled and securely stoppered with clean corks or caps, then sealed, marked, and sent to the laboratory for test.

8. Paste Soap Products

8.1 *When Packed in Cans or Cartons of 5 lb (2.27 kg) or Less*—One can or carton shall be taken at random from not less than 1 % of the seller's shipping containers, provided each package contains not less than 50 lb (22.7 kg). In the case of smaller containers, a can or carton shall be taken at random from each lot of containers totaling not more than 5000 lb (2268 kg) or fraction thereof. The gross sample shall in all cases consist of not less than three cans or cartons taken at random from separate containers. In the case of very large lots where the sample drawn as above will amount to more than 20 lb (9.1 kg), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 lb. The samples shall be wrapped, sealed, marked, and sent to the laboratory for test.

8.2 *When Packed in Bulk*—A *trier* sample³ of not less than 0.5 lb (227 g) shall be taken at random from not less than 1 % of the seller's shipping containers, provided each package contains not less than 50 lb. In the case of smaller containers, a *trier* sample³ of not less than 0.5 lb shall be taken at random from each lot of containers totaling not more than 5000 lb or fraction thereof. The gross sample shall in all cases consist of not less than three 0.5-lb samples, each taken at random from separate containers. With very large lots where the sample drawn as above will amount to more than 10 lb (4.5 kg), the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 10 lb. The inspector shall promptly place the gross sample in a clean, dry, airtight and watertight container, which shall be filled, sealed, marked, and sent to the laboratory for test.

³ A trier sample is obtained by inserting a *trier* into the material. A trier is a half-round steel cylinder $\frac{1}{2}$ to $\frac{3}{4}$ in. (12.7 to 19.1 mm) in diameter, 6 to 36 in. (152 to 914 mm) in length, pointed on one end and having a grip handle on the other end. After insertion, the trier is turned two or three times, and upon removal a core of the material being sampled is obtained.

9. Preparation of Samples

9.1 *Cake Soap*—Grind all bars through a suitable food chopper. In the case of large samples, it is permissible to quarter the bars and grind one quarter from each bar. However, each ground sample should consist of at least 3 lb (1.36 kg). Mix all ground samples thoroughly on a clean, dry, nonabsorbent, impervious surface with a spatula. Divide into four quarters and discard the two opposite quarters. Combine, remix, and requarter the remaining two quarters. Continue in this manner until the sample is reduced to approximately 2 lb (0.91 kg). Place this portion in a clean, dry sample container. Close tightly and label completely for identification. This is the sample for analysis and must be preserved in a cool dry place.

9.2 *Powdered and Chip Soaps*—Rapidly disintegrate and mix the sample of powdered, flake, or chip soap. If desired,

quarter down to about 1 lb (454 g). Weigh at once all portions for analysis, preserving the remainder in an airtight container in a cool place.

9.3 *Liquid Soap*—No preparation of the sample of liquid soap, other than a thorough mixing, is necessary unless it is received during very cold weather, when it should be allowed to stand at least 1 h after it has warmed to room temperature (20 to 30°C) before it is tested, particularly for its lathering qualities.

9.4 *Paste Soap Products*—Mix the sample of paste soap products thoroughly by kneading and quarter down to about 1 lb (454 g). Weigh at once all portions for analysis, preserving the remainder in an airtight container in a cool place.

METHODS FOR CHEMICAL ANALYSIS

10. Hazards

10.1 **Precaution**—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. Clean up any spill immediately. Consult the Material Safety Data Sheet for specific handling and disposal information.

10.2 Use of glass wool in place of asbestos cloth is recommended where applicable.

11. Purity of Reagents

11.1 *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.⁴ 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.

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

12. Duplicate Tests

12.1 When a determination shows nonconformity with the specifications a duplicate test shall be made.

MOISTURE

13. Choice of Test Method

13.1 The oven method described in Section 14 is generally applicable to all soaps, but certain exceptions to this method

must be made if accurate results are desired. These exceptions include the following:

13.1.1 For soaps containing appreciable amounts of sodium silicate, the distillation method (Sections 15-18) is preferred.

13.1.2 Soaps of linseed and other oxidizing oils absorb oxygen, and if the oven method is used may gain weight near the end of the test. Therefore, either an inert atmosphere or vacuum oven should be used. The distillation method is also applicable to these types of soap.

13.1.3 Soaps containing appreciable amounts of glycerin, such as cold-made and semiboiled (including paste soaps), usually give high results by the oven method. The distillation method is preferred for most accurate results on these types of soaps.

Method A—Matter Volatile at 105°C (Oven Method)

14. Procedure

14.1 Weigh 5 ± 0.01 g of the sample in a porcelain or glass dish about 6 to 8 cm in diameter and about 2 to 4 cm in depth, and dry to constant weight in an air oven at a temperature of $105 \pm 2^\circ\text{C}$. Constant weight is attained when successive heating for 1-h periods shows a loss (or gain) of not more than 0.1 %.

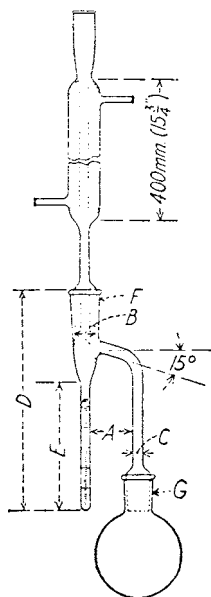
Method B—Distillation Method

15. Apparatus

15.1 The apparatus required consists of a glass flask heated by suitable means and provided with a reflux condenser discharging into a trap and connected to the flask. The connections between the trap and the condenser and flask shall be interchangeable ground joints. The trap serves to collect and measure the condensed water and to return the solvent to the flask. A suitable assembly of the apparatus is illustrated in **Fig. 1**.

15.1.1 *Flask*, 500-mL, of either the shortneck, round-bottom type or the Erlenmeyer type.

⁴ *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.



- A = 45 to 55 mm
- B = 22 to 24 mm inside diameter
- C = 9 to 11 mm inside diameter
- D = 235 to 240 mm
- E = 146 to 156 mm
- F and G are interchangeable joints, standard taper 24/40.

FIG. 1 Assembly at Distillation Apparatus

15.1.2 *Heat Source*—The source of heat may be either an oil bath (stearic acid, paraffin wax, etc.), or an electric heater provided with a sliding rheostat or other means of heat control.

15.1.3 *Condenser*—A water-cooled glass reflux condenser (Fig. 1), having a jacket approximately 400 mm in length with an inner tube 9.5 to 12.7 mm in outside diameter. The end of the condenser to be inserted in the trap may be ground off at an angle of 30° from the vertical axis of the condenser. When inserted into the trap, the tip of the condenser shall be about 7 mm above the surface of the liquid in the trap after the distillation conditions have been established. Fig. 1 shows a conventional sealed-in type of condenser, but any other condenser fulfilling the detailed requirements above may be used.

15.1.4 *Trap*—For greatest accuracy several trap sizes are allowable, depending upon the percentage of moisture expected:

Moisture Expected, %	Size of Trap, mL
0 to 5, incl	5
Over 5 to 17, incl	10
Over 17 to 30, incl	10
Over 30 to 50, incl	25
Over 50 to 70, incl	25
Over 70 to 85, incl	25

Traps made of well-annealed glass, constructed essentially as shown in Fig. 1 and graduated to contain one of the following specified volumes at 20°C shall be used:

15.1.4.1 *5-mL Trap*, subdivided into 0.1 mL divisions with each 1-mL line numbered (5 mL at top). The error in any indicated capacity may not be greater than 0.05 mL.

15.1.4.2 *10-mL Trap*, subdivided from 0 to 1 mL in 0.1-mL divisions and from 1 to 10 mL in 0.2-mL divisions.

15.1.4.3 *25-mL Trap*, subdivided from 0 to 1 mL in 0.1-mL divisions and from 1 to 25 mL in 0.2-mL divisions.

NOTE 1—The condenser and trap should be thoroughly cleaned before use.

16. Solvent

16.1 *Toluene*—Saturate the toluene with water by shaking with a small quantity of water and distill. Use the distillate for the determination.

17. Procedure

17.1 For soaps containing from 5 to 25 % moisture and volatile matter, use 20 ± 0.04 g of the sample. For soaps containing more than 25 % moisture and volatile matter, use 10 ± 0.02 g of the sample. Carefully transfer the weighed sample to the 500-mL flask. Add approximately 10 g of anhydrous, fused sodium acetate to prevent violent frothing, and then follow with 100 mL of xylene (or toluene) which has been saturated with water. Attach the flask to the trap which is connected to the condenser. Prior to starting the determination, fill the receiver with saturated xylene (or toluene) by pouring in through the reflux condenser. So that the refluxing will be under better control, wrap the flask and tube leading to the receiver with asbestos cloth. Heat the oil bath with a gas burner or other source of heat, or apply heat directly to the flask with an electric heater and distill slowly. The rate at the start should be approximately 100 drops per min. When the greater part of the water has distilled over, increase the distillation rate to 200 drops per min until no more water is collected. Purge the reflux condenser during the distillation with 5-mL portions of xylene (or toluene) to wash down any moisture adhering to the walls of the condenser. The water in the receiver may be made to separate from the xylene (or toluene) by using a spiral copper wire. Move the wire up and down in the condenser occasionally, thus causing the water to settle at the bottom of the receiver. Reflux for at least 2 h, and shut off the heat at the end of this period. Adjust the temperature of the distillate to 20°C and read the volume of water.

18. Calculation

18.1 Calculate the percentage of moisture in the soap, as follows:

$$\text{Moisture, \%} = [(V \times 0.998)/W] \times 100 \quad (1)$$

where:

V = millilitres of water at 20°C, and
W = grams of sample used.

TOTAL MATTER INSOLUBLE IN ALCOHOL

19. Reagent

19.1 *Ethyl Alcohol (95 %)*—Freshly boiled, reagent grade, ethyl alcohol, 95 % or higher, neutral to phenolphthalein, and containing only volatile denaturants⁵ plus 5 mL of water.

⁵ Fisher Scientific A962, (Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA 15219), or its equivalent, is suitable for this purpose.

20. Procedure

20.1 Digest 2 to 10 ± 0.01 g of the sample with 200 mL of freshly boiled ethyl alcohol in a covered vessel on a steam bath until the soap is dissolved. Filter through a tared filter paper neutral to phenolphthalein. Tare the filter paper by heating at 100 to 105°C for 30 min., cooling and weighing. Or filter through a weighed Gooch crucible with suction, protecting the solution from carbon dioxide and other acid fumes during the operation by covering with a watch glass. Wash the residue on the paper or in the crucible with hot neutral ethyl alcohol until free from soap, and reserve the filtrate and washings. Dry the filter paper or crucible with the residue at 100 to 105°C for 3 h, cool, and weigh the total matter insoluble in alcohol.

NOTE 2—The matter insoluble in alcohol will contain most of the alkaline salts, such as carbonates, borates, silicates, phosphates, and sulfates, as well as starch, and may be used for the approximate determination of these constituents. These salts are not entirely insoluble in alcohol, so for accurate determinations separate portions of the soap should be used. For determination of carbonates, see Sections 61-68; phosphates, Sections 69-76; sulfates, Sections 80 and 81; silicates, Sections 59 and 60; borax, Sections 56-58; and starch, Sections 87 and 88.

NOTE 3—In the case of soap products containing a high level of matter insoluble in alcohol, see 25.4 for an approximate determination of soap content.

FREE ALKALI OR FREE ACID

21. Procedure

21.1 Heat the reserved filtrate from the determination of total matter insoluble in alcohol (Section 20) to incipient boiling, add 0.5 mL of a 1 % solution of phenolphthalein, titrate with standard acid or alkali solution, and calculate to sodium hydroxide (NaOH) (or potassium hydroxide (KOH)) if alkaline, or to oleic acid, if acid.

NOTE 4—In the analysis of soaps known to contain little or no alkaline salts, it is unnecessary to filter the hot alcoholic soap solution as described in Section 20. However, the filtration should be carried out in all cases where alkaline salts such as silicates, phosphates, borates, and similar salts are present, since these are known to affect the free alkali determination. Free alkali values in soap or surfactant mixtures containing borax are unreliable due to solubility of borax in hot alcohol. In this case, see Sections 56-58 to determine the percentage of borax present, titrate an equivalent amount of borax with the standard acid, and subtract this titer from the one obtained in 21.1 before calculating alkalinity as NaOH or KOH.

MATTER INSOLUBLE IN WATER

22. Procedure

22.1 Proceed as in the determination of matter insoluble in alcohol (starting with a fresh sample of soap and omitting the drying and weighing of matter insoluble in alcohol) (Section 20). After filtering and thoroughly washing the residue, change the receivers, extract the residue with water at 60°C and wash the filter thoroughly. (When the matter insoluble is all inorganic, boiling water may be used for the extraction and washing.) Reserve the water solution. Dry the filter and residue at 100 to 105°C for 3 h, cool, and weigh the matter insoluble in water. The nature of this matter may be determined by further examination.

TOTAL ALKALINITY OF MATTER INSOLUBLE IN ALCOHOL, ALKALINE SALTS

23. Procedure

23.1 Titrate the water solution obtained in the determination of matter insoluble in water (Section 22) with standard acid, using methyl orange as indicator. Calculate the alkalinity to sodium oxide (Na₂O) and, if desired, to any other basis agreed upon by the purchaser and the seller.

COMBINED ALKALI, TOTAL ANHYDROUS SOAP

24. Reagents

24.1 *Ethyl Alcohol, Neutral*, carbon dioxide (CO₂)-free.

24.2 *Ethyl Ether*.

24.3 *Methyl Orange Indicator*.

24.4 *Phenolphthalein Indicator Solution*.

24.5 *Sodium Hydroxide, Standard Solution (0.5 N)*—Prepare and standardize a 0.5 N sodium hydroxide (NaOH) solution.

24.6 *Sulfuric Acid (1 + 1)*—Gradually pour 10 g of concentrated sulfuric acid (H₂SO₄, sp gr 1.84) onto 10 g of cracked ice made from distilled water, gently swirling the mixing vessel, or gradually pour the acid down the sides of the mixing vessel into an equal weight of water, swirling gently, while submersing the vessel in an ice bath.

25. Procedure

25.1 Dissolve 5 to 10 ± 0.01 g of the sample, depending upon the anhydrous soap content, in 100 mL of water in a 250-mL Erlenmeyer flask. The flask may be heated to not more than 60°C to effect solution. When the solution is complete, add H₂SO₄ (1 + 1) in slight excess, insert a small funnel in the neck of the flask, and heat the flask to a temperature not exceeding 60°C until the fatty acids separate as a clear layer. Transfer to a separatory funnel, draw off the acid layer into a second separatory funnel, and shake the acid aqueous liquid with three 30-mL portions of ethyl ether. Dissolve the fatty acids in the ether used for washing the aqueous liquid, and shake with 10-mL portions of water until they are no longer acid to methyl orange. Unite the water portions used for washing and shake with 30 mL of ether. Wash this ether until the wash water is neutral to methyl orange. Reserve the acid water for the determination of chlorides (Sections 53 and 54).

25.2 Unite the ether solutions (if necessary, filter, washing the paper with ether) in a suitable weighed vessel, add 100 mL of neutral ethyl alcohol, add phenolphthalein, and titrate to exact neutrality with 0.5 N NaOH solution. Evaporate the alcohol and ether solution on a steam bath, and dry to constant weight as in the determination of matter volatile at 105°C, and calculate the percentage of soda soap. This naturally includes any mineral oil and neutral fat which, if determined separately, must be deducted from the result to obtain A, the percentage of the true anhydrous soap. See also 43.2. Calculate the combined sodium oxide (Na₂O) and deduct from the weight of soda soap to give the acid anhydrides. Calculate the weight percentage of combined sodium oxide as follows:

$$\text{Na}_2\text{O, weight \%} = 3.099\text{MN/W} \quad (2)$$

If the original soap was potash soap, titrate directly with 0.5 N KOH solution, or make the proper calculation to reduce to K_2O as follows (Note 4):

$$\text{K}_2\text{O, weight \%} = 4.710\text{MN/W} \quad (3)$$

If the weight percent soap, *A*, need be converted to percent total fatty acids, calculate as follows:

$$F = A - \text{MNZ/W} \quad (4)$$

where:

- F* = weight percent total fatty acids,
- M* = mL of standardized NaOH solution,
- N* = normality of the standardized NaOH per 24.5,
- A* = weight percent sodium or potassium soap,
- W* = sample weight in grams per 25.1, and
- Z* = 2.20 for soda soap, and 3.81 for potash soap.

In case the soap shows an excess of free acid, proper corrections must be made in calculating the combined alkali in the original soap. (A blank test should be made on the NaOH or KOH solution for neutral salts and the proper corrections made if necessary.)

25.3 In the case of soaps containing a large amount of soluble silicates and soap products containing a high percentage of finely divided material insoluble in water, the foregoing procedure cannot be applied as given. In such cases the filtrate obtained in the determination of total matter insoluble in alcohol (Section 20) may be used after neutralizing any free acid or alkali. Evaporate the alcohol on a steam bath, take up with water, and proceed according to the procedure described in 25.1 and 25.2.

25.4 In the case of soap products containing a high percentage of matter insoluble in alcohol where approximate results will suffice, such as cleansers, soap powders, scouring compounds, pastes, etc., and where agreed upon by the purchaser and the seller, the alcoholic solution obtained after filtering off and washing the matter insoluble in alcohol (Section 20) may be evaporated directly in a weighed vessel on a steam bath, then dried at 105°C to constant weight, and the result reported as soap.

COMBINED SODIUM AND POTASSIUM OXIDES

NOTE 5—The total combined alkali present in the soap is determined by the method described in Section 25, and calculated as sodium oxide (Na_2O). Determine the combined potassium oxide (K_2O) by the following method, calculate it to the equivalent Na_2O , and subtract this from the total combined alkali calculated as Na_2O ; the remainder is the combined Na_2O .

26. Reagents

26.1 *Ammonium Chloride Solution*—Dissolve 100 g of ammonium chloride (NH_4Cl) in 500 mL of water, add 5 to 10 g of pulverized potassium chloroplatinate (K_2PtCl_6), and shake at intervals for 6 to 8 h. Allow the mixture to settle overnight and filter. (The residue may be used for the preparation of a fresh supply of NH_4Cl solution.)

26.2 *Ethyl Alcohol* (80 %).

26.3 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

26.4 *Hydrochloric Acid* (1 + 1)—Mix equal volumes of HCl (sp gr 1.19) and water.

26.5 *Platinum Solution*—Prepare a solution containing the equivalent of 1 g of metallic platinum (2.1 g of chloroplatinic acid (H_2PtCl_6)) in each 10 mL of solution. For materials containing less than 15 % of K_2O , a solution containing 0.2 g of metallic platinum (0.42 g of H_2PtCl_6) in each 10 mL of solution is recommended.

27. Preparation of Sample

27.1 Weigh 10 ± 0.01 g of the sample and sinter it in an evaporating dish below a dull red heat. Leach the ash with hot water, filter into a 100-mL volumetric flask, and wash the paper with three 5 to 10-mL portions of hot water. Complete the ashing after returning the filter paper and residue to the original dish and sintering as before. Excessive heating shall be avoided. Removal of most of the alkali present by thoroughly washing the ash with hot water before completion of the ashing will aid in preventing overheating of the greater portion of the sample. Add a few drops of HCl (1 + 1) to the ash and wash the contents of the dish into the volumetric flask. Acidify the solution in the volumetric flask with HCl, dilute to 100 mL, mix thoroughly, and pass through a dry filter.

28. Procedure

28.1 Acidify an accurately measured 10-mL aliquot of the solution obtained as described in Section 27 with a few drops of HCl and add 10 mL of the platinum solution. Evaporate the solution on a water bath to a thick paste which will become solid on cooling to room temperature. Avoid exposure to ammonia fumes while heating the solution. Treat the residue with approximately 6 mL of ethyl alcohol and add 0.6 mL of HCl (sp gr 1.19). Filter on a Gooch crucible and wash the precipitate thoroughly with ethyl alcohol both by decantation and on the filter, continuing the washing until after the filtrate is colorless. Then wash the residue five or six times with 25-mL portions of the NH_4Cl solution to remove the impurities from the precipitate. Wash again thoroughly with ethyl alcohol, dry the precipitate at 100°C for 30 min, and weigh.

29. Calculation

29.1 Calculate the percentage of K_2O as follows:

$$\text{K}_2\text{O, \%} = A \times 19.376 \quad (5)$$

where *A* = grams of K_2PtCl_6 weighed.

FREE ALKALI AND POTASSIUM CARBONATE IN POTASH PASTE SOAPS

30. Reagents

30.1 *Ethyl Alcohol, Neutral (Absolute)*—Freshly boiled absolute ethyl alcohol, neutral to phenolphthalein, conforming to either Formula No. 3A or 30 of the U.S. Bureau of Internal Revenue.

30.2 *Methyl Orange Indicator Solution (1 g/L)*—Prepare solution of 0.1 g methyl orange in 100 mL of water.

30.3 *Phenolphthalein Indicator Solution (10 g/L)*—Prepare a solution of 1 g phenolphthalein in 100 mL of neutral ethyl alcohol (95 %).

30.4 *Sodium Hydroxide, Standard Solution (0.5 N)*—Prepare and standardize a 0.5 N sodium hydroxide (NaOH) solution.

30.5 *Sulfuric Acid, Standard Solution (0.5 N)*—Prepare and standardize a 0.5 N sulfuric acid (H₂SO₄) solution.

31. Procedure

31.1 Weigh 10 ± 0.01 g of the sample into a 250-mL beaker and dissolve in 100 mL of freshly boiled water. Add a measured amount of 0.5 N H₂SO₄ sufficient to make the solution acid to methyl orange indicator, and bring to incipient boiling until fatty acids separate out in a clear layer. Excessive boiling should be avoided to preclude the possibility of volatilization of any low molecular weight fatty acids that may be present.

31.2 Add 0.5 mL of phenolphthalein indicator solution, and while stirring the contents of the beaker, titrate with NaOH solution until the solution is almost neutral but still slightly on the acid side to phenolphthalein.

31.3 Evaporate to dryness on a steam bath and dissolve in 200 mL of neutral alcohol (absolute). Titrate with NaOH solution to neutrality to phenolphthalein.

31.4 Determine carbon dioxide (CO₂) on a separate sample by the train-absorption method (Section 64) or the evolution-volumetric method (Section 65).

32. Calculation

32.1 Calculate the percentages of potassium carbonate (K₂CO₃) and free potassium hydroxide (KOH) as follows:

$$K = C \times 3.140 \quad (6)$$

$$K_2CO_3, \% = (K/W_1) \times 100$$

$$V_k = K/0.03455$$

$$V_t = V_1 - (V_2 + V_3)$$

$$\text{Free KOH, \%} = [(V_t - V_k) \times 0.02805 \times 100]/W_2$$

where:

- K = grams of K₂CO₃ equivalent to C ,
- C = grams of CO₂ (31.4),
- W_1 = grams of sample used for determination of CO₂ (31.4),
- V_k = millilitres of 0.5 N H₂SO₄ equivalent to K₂CO₃,
- V_t = millilitres of 0.5 N H₂SO₄ equivalent to free KOH + K₂CO₃,
- V_1 = millilitres of 0.5 N H₂SO₄ used to acidify sample (31.1),
- V_2 = millilitres of 0.5 N NaOH solution required for titration of aqueous solution (31.2),
- V_3 = millilitres of 0.5 N NaOH solution required for titration of alcoholic solution (31.3), and
- W_2 = grams of sample used for titration procedure (31.1).

UNSAPONIFIED PLUS UNSAPONIFIABLE MATTER

NOTE 6—In the case of superfatted soaps, free fatty acids, which are the superfating agents in highest percentage, plus this unsaponified and unsaponifiable matter, constitute the major portion of the superfating agents used.

33. Apparatus

33.1 *Extraction Cylinder*—The extraction cylinder shall be a 250-mL graduated glass-stoppered cylinder about 39 mm in diameter and about 35.5 cm (14 in.) in length.

34. Reagents

34.1 *Ethyl Alcohol (95 %)*

34.2 *Ethyl Alcohol (50 %)*

34.3 *Ethyl Alcohol (10 %)*

34.4 *Petroleum Ether*—The solvent used shall be of the pentane type, containing a minimum amount of isopentane, isohexane, and hexane, and boiling in the range 35 to 60°C.⁶

Distillation test:^A

Initial boiling point	35 to 38°C
Dry flask end point	52 to 60°C
Distilling under 54°C, min	95 %
Distilling under 40°C, max	60 %
Specific gravity at 15.5/15.5 °C (60/60°F)	0.630 to 0.660
Color	water white
Doctor test	sweet
Evaporation residue, 100 mL, max	0.0011 g
Copper-strip corrosion test ^B	noncorrosive
Unsaturated compounds ^C	trace only permitted
Residue in distilling flask	neutral to methyl orange
Blotter-strip odor test ^D	odorless within 12 min
Aromatic compounds ^E	no nitrobenzene odor
Saponification value	less than 1.0 mg KOH/100 mL

^A Distillation test shall be made in accordance with ASTM Method D216. As a check on the evaporation residue, 250 mL of the petroleum ether and 0.25 g of stearin or other hard fat (previously brought to constant weight by heating) when dried as in the actual determination shall not show an increase in weight exceeding 0.003 g.

^B Copper-strip corrosion test shall be made by inserting a small polished copper strip into the petroleum ether in the distilling flask. There should be no appreciable darkening of the copper.

^C Unsaturated compounds shall be determined by the method for determining olefins as described in *Industrial and Engineering Chemistry*, Analytical Edition, March 15, 1938, p. 154.

^D Odor test: Immerse 1 in. of a strip of white unglazed blotting paper, approximately 1 by 4 by 0.166 in. in size, in the petroleum ether for 30 s, remove the strip, and allow to dry at room temperature in still air for 12 min.

^E Aromatic compounds: Add 5 drops of petroleum ether to 40 drops of sulfuric acid (H₂SO₄, sp gr 1.84) and 10 drops of nitric acid (HNO₃, sp gr 1.42) in a test tube, warm for 10 min, allow to cool for 30 min, transfer to a shallow dish, and dilute with water.

34.5 *Sodium Hydroxide, Standard Solution (0.1N)*—Prepare and standardize a 0.1 N NaOH solution.

34.6 *Sodium Hydroxide, Standard Solution (0.04 N)*—Prepare and standardize a 0.04 N sodium hydroxide (NaOH) solution.

35. Procedure

35.1 Weigh 5 ± 0.2 g of the sample and place it in a 250-mL Erlenmeyer flask or beaker that contains 0.1 g of sodium bicarbonate (NaHCO₃) to promote phase separation during the

⁶ J. T. Baker Analyzed Reagent 9268, (J. T. Baker, Inc. Phillipsburg, NJ) or its equivalent, is suitable for this purpose.

extractions, and dissolve in 100 mL of redistilled ethyl alcohol (50 %). Warm and shake to effect solution, keeping the temperature under 60°C, and filter off any undissolved residue on a Gooch crucible with a glass wool pad, or on a funnel using a glass wool pad deposited on a perforated porcelain disk. Wash three times with hot alcohol (50 %). Wash with a small amount of petroleum ether to remove any traces of unsaponified and unsaponifiable matter. Transfer the entire alcohol-water-and-ether filtrate to the extraction cylinder and make up to the 160-mL mark with redistilled, ethyl alcohol (50 %). Add 50 mL of petroleum ether, shake vigorously for 1 min (Note 5), and allow to settle until both layers are clear. The volume of the upper layer should be about 40 mL. Draw off the petroleum ether layer as closely as possible by means of a slender glass siphon into a separatory funnel of 500-mL capacity.

35.2 Repeat the extraction at least six times using 50 mL of petroleum ether each time (Note 8). To avoid extraction of free fatty and rosin acids, wash the combined ether extracts first with a mixture of 15 mL of 0.1 *N* NaOH solution and 15 mL of ethyl alcohol (95 %), and then three times with 25-mL portions of ethyl alcohol (10 %), shaking vigorously each time. Transfer the petroleum ether extract to a beaker and evaporate the petroleum ether on a steam bath by the aid of a current of air.

NOTE 7—Thorough and vigorous shaking is necessary in order to secure accurate results. The two phases must be brought into the most intimate contact possible; otherwise low and disagreeing results may be obtained. If any emulsion occurs, break it with 10 g of sodium sulfate.

NOTE 8—This method will not remove all the unsaponifiable matter in soaps to which lanolin has been added. More extractions are required when substances of this nature are present.

35.3 Test the residue for solubility by treating with 50 mL of petroleum ether at room temperature. Filter, and wash free from the insoluble residue, if any. Evaporate and dry in the same manner on a steam bath, and finally in an air oven at 100 to 101°C for 30 min. Weigh, and return to the oven, reweighing at 15-min intervals until constant weight is reached. Take up the residue in 50 mL of warm ethyl alcohol, neutralized to phenolphthalein, titrate to the same color as the original neutral alcohol with 0.04 *N* NaOH solution, and calculate to oleic acid. Deduct this figure from the gross weight previously found and report as “unsaponified and unsaponifiable matter.”

35.4 Make a blank test on the petroleum ether by evaporating 250 mL of the ether with about 0.25 g of stearin or some other hard fat previously brought to constant weight by heating and drying as in the actual determination. The blank must not exceed a few milligrams.

NOTE 9—Any blank from the petroleum ether must be deducted from the weight before calculating the unsaponified and unsaponifiable matter.

UNSAPONIFIABLE MATTER

36. Apparatus

36.1 *Extraction Cylinder*—See Section 33.

37. Reagents

37.1 See 34.1 and 34.4.

37.2 *Potassium Hydroxide Solution (50 %)*—Dissolve 500 g of potassium hydroxide (KOH) in 500 mL of water.

38. Procedure

38.1 Transfer 5 ± 0.2 g of the sample to a 200-mL Erlenmeyer flask. Add 30 mL of redistilled ethyl alcohol (95 %) and 5 mL of KOH solution, and boil for 1 h under a reflux condenser. Transfer to the extraction cylinder and wash to the 40-mL mark with redistilled ethyl alcohol (95 %). Complete the transfer, first with warm and then with cold water, until the total volume is 80 mL, and finally with a small quantity of petroleum ether. Cool the cylinder to room temperature and add 50 mL of petroleum ether; then proceed with the extraction as outlined in the procedure for unsaponified plus unsaponifiable matter (Section 35; see also Note 5 and Note 6), except that the alkaline wash may be omitted. Weigh the residue and correct for fatty acids in the usual manner. Report the result as unsaponifiable matter.

UNSAPONIFIED MATTER

39. Calculation

39.1 From the total unsaponified plus unsaponifiable matter determined in Section 35, deduct the unsaponifiable figure obtained in Section 38 and report the difference as “unsaponified matter.”

ROSIN (McNICOLL METHOD)⁷

40. Apparatus

40.1 The apparatus required consists of a glass flask connected, preferably by a ground-glass joint, to a reflux condenser.

40.1.1 *Esterification Flask*, 150-mL of either the round-bottom or Erlenmeyer type.

40.1.2 *Reflux Condenser*—Any suitable water-cooled glass reflux condenser may be used.

41. Reagents

41.1 *Naphthalene- β -Sulfonic Acid Solution*—Dissolve 40 g of reagent⁸ in 1 L of absolute methyl alcohol.

41.2 *Phenolphthalein Indicator*—Prepare a 0.5 % solution of phenolphthalein in neutral redistilled alcohol.

41.3 *Potassium Hydroxide, Standard Alcoholic Solution (0.2 N)*—Accurately standardize a 0.2 *N* solution of potassium hydroxide (KOH) in neutral redistilled alcohol (due to volatility of alcohol, this solution should be restandardized frequently).

42. Procedure

42.1 *Preparation of Fatty and Rosin Acids*—For the preparation of the sample for this determination, follow the procedure described in Section 45.

⁷ Cox and Evers, “Report of British Standards Committee,” *Analyst*, Vol 62, No. 741, 1937, pp. 865 to 870; also D. McNicoll, “The Estimation of Rosin Acids in Fatty Mixtures,” *Journal, Soc. Chemical Industry*, Vol 40, 1921, p. 124 T.

⁸ Eastman grade or equivalent reagent has been found satisfactory for this purpose.

42.2 *Esterification and Titration*—Weigh about 2 ± 0.001 g of the fatty acids into the esterification flask. Add 25 mL of naphthalene- β -sulfonic acid solution. Add a few glass beads to ensure smooth boiling, attach the reflux condenser, and boil for 30 min; also, run a blank test using 25 mL of the reagent. At the end of the boiling period cool the contents of the flask, add 0.5 mL of phenolphthalein indicator, and titrate immediately with 0.2 *N* alcoholic KOH solution.

43. Calculation

43.1 Calculate the percentage of rosin as follows (see **Note 10**):

$$R = [(S - B) \times N \times 0.346 \times 100] / W \quad (7)$$

$$R_1 = R - 1.0$$

$$R_2 = (R_1 \times F) / 100$$

$$R_s = R_2 \times 1.064$$

where:

- R = percentage of rosin in fatty acids,
- R_1 = corrected percentage of rosin in fatty acids, (see **Note 11**),
- R_2 = percentage of rosin on basis of original sample,
- R_s = percentage of rosin soda soap on basis of original sample,
- S = millilitres of KOH solution required for titration of the sample,
- B = millilitres of KOH solution required for titration of the blank,
- N = normality of KOH solution,
- W = grams of sample
- F = percentage of total fatty acids in soap, (see **25.2**).

NOTE 10—In all cases where the rosin content is found to be less than 5 % the actual presence or absence of rosin should be checked qualitatively by the Liebermann-Storch test, as follows:

Transfer 1 to 2 mL of the sample of fatty acids to a test tube, treat with 5 to 10 mL of acetic anhydride, and warm on a steam bath. After cooling, pour 1 to 2 mL into a white porcelain dish and allow a drop or two of sulfuric acid (H_2SO_4 , sp gr 1.53) to run down the side of the vessel. (The H_2SO_4 (sp gr 1.53) is prepared by diluting 34.7 mL of H_2SO_4 (sp gr 1.84) with 35.7 mL of water.) If rosin is present, a fugitive violet coloration changing to a brownish tinge is immediately produced at the margin of contact of the reagents. The test should be checked with a sample of fatty acids to which a small amount of rosin has been added.

NOTE 11—Cooperative studies have shown that the McNicoll method gives results approximately 1 % higher than the amount of rosin present. Consequently, the committee recommends deducting 1 % from the percentage of rosin found in the fatty acids.

43.2 If true fatty acid soap is desired, subtract the rosin soap from the total anhydrous soap determined in **25.2**, as follows:

$$T = A - R_s \quad (8)$$

where:

- T = percentage of true fatty acid soap,
- A = percentage of total anhydrous soap, and

R_s = percentage of rosin soda soap.

PREPARATION OF TOTAL FATTY MATTER (FATTY AND ROSIN ACIDS, AND UNSAPONIFIED AND UNSAPONIFIABLE FATTY MATTER)

44. Reagent

44.1 *Sulfuric Acid (1 + 4)*—Slowly mix 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) with 4 volumes of water.

45. Preparation for Rosin and Titer Tests, Iodine and Acid Numbers

45.1 Dissolve about 50 g of the sample in 500 mL of hot water. (If soaps to be tested contain alcohol, the alcohol should be completely removed by evaporation from the soap solution.) Add 100 mL of H_2SO_4 (30 %), heat gently until the fatty matter collects in a clear layer. Siphon off the aqueous acid layer, add 300 mL of hot water, boil gently for a few minutes, and siphon off the aqueous acid layer. Repeat the washing of the acids in this manner until the final washing is neutral to methyl orange indicator (usually 3 to 6 washings). Complete this acidification and washing in a very short period of time, and keep the beaker covered to prevent oxidation of the acids. After the last washing, remove the last traces of water from the beaker with a pipet, filter the fatty acids through one or two thicknesses of filter paper, and dry at a temperature of 105°C for 45 to 60 min or heat rapidly to 130°C and allow to cool. Do not hold at 130°C, but if water is present, decant the clear fatty acids into another beaker, and again reheat them momentarily to 130°C. These acids may then be used for the titer and rosin determinations.

45.2 In preparing the acids for the iodine and acid number determinations, the washed acids should be filtered through one or two thicknesses of filter paper at a temperature not

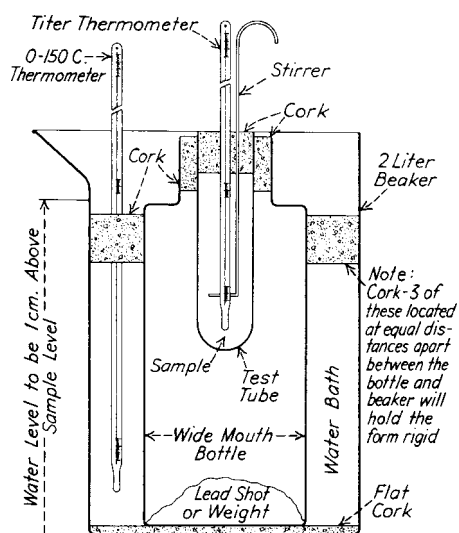


FIG. 2 Apparatus Assembly for Titer Test

exceeding 15°C above the titer point of the fatty acids. If the acids are not perfectly clear and dry, refilter.

TITER TEST

46. Apparatus

46.1 The apparatus required, shall be assembled as shown in Fig. 2 and shall consist of the following:

46.1.1 *Water Bath*—A 2-L Griffin low-form beaker.

46.1.2 *Wide-Mouth Bottle*—A wide-mouth 450-mL bottle, 190 mm in height, having a neck 38 mm in inside diameter, fitted with a cork to carry the test tube (46.1.3) and with sufficient weights or lead in the bottom of the bottle to hold it steady when placed in the water bath.

46.1.3 *Test Tube*—Test tube 100 mm in length, and 25 mm in diameter with or without rim. The tube may have an etched line extending around it at a distance of 57 mm from the bottom to indicate the height to which it is to be filled.

46.1.4 *Stirrer*—A stirrer 2 to 3 mm in outside diameter with one end bent in the form of a loop 19 mm in diameter. Glass, Nichrome, stainless steel, or Monel metal wire may be used. The upper end of the stirrer may be formed to accommodate hand stirring or for attachment to a mechanical stirrer.

46.1.5 *Laboratory Thermometer*—A laboratory thermometer having a range from 0 to 150°C.

46.1.6 *Thermometer*—An ASTM Titer Test Thermometer, having a range from -2 to +68°C and conforming to the requirements for Thermometer 36°C⁹ as prescribed in Specifications E1.

47. Procedure

47.1 Transfer the fatty acids (see Section 45), when cooled somewhat, to the test tube, filling it to the 57-mm line, and place the tube in the wide-mouth bottle. Set the bottle assembly in the water bath (the water should reach the neck of the bottle), as shown in Fig. 2, and adjust the temperature of the bath to 20°C for fatty acids with titers of 35°C and over, and to 15 to 20°C below the expected titer for all fatty acids with titers below 35°C.

47.2 Insert the titer test thermometer in the center of the sample and suspend it at such a height that the immersion mark coincides with the top of the sample.

47.3 When the titer test thermometer reads 10°C above the expected titer, stir with the stirring rod in a vertical manner at the rate of 100 completed up-and-down motions per minute. The stirrer should travel about 3.8 cm. Read the thermometer every 15 s, and continue stirring until the temperature remains constant (or begins to rise) for 30 s. Discontinue stirring immediately and note the rise in temperature. Report as the titer of the fatty acids, the highest temperature reached by the thermometer.

47.4 Duplicate determinations should not differ by more than 0.2°C.

⁹ Thermometers made to these specifications conform also with the requirements for the titer test thermometer of the American Oil Chemists Society and the Association of Official Agricultural Chemists, except for the special marking.

ACID NUMBER OF FATTY ACIDS

48. Procedure

48.1 For the preparation of the sample for this determination follow the procedure described in Section 45. In a 250-mL Erlenmeyer flask dissolve 2 g of the fatty acids, accurately weighed in 20 to 30 mL of neutral ethyl alcohol (95 %) near the boiling point. Titrate with standard alkali, using phenolphthalein as indicator.

49. Calculation

49.1 Calculate the acid number of the fatty acid, expressed as milligrams of KOH per gram of fatty acid, as follows:

$$\text{Acid number} = (AN \times 56.1)/B \quad (9)$$

where:

- A = millilitres of KOH solution required for titration of the sample,
 N = normality of the KOH solution, and
 B = grams of sample used.

IODINE NUMBER (WIJS METHOD)

50. Reagents

50.1 *Potassium Dichromate, Standard Solution (0.1 N)*—Dissolve 4.903 g of potassium dichromate (K₂Cr₂O₇) in water and dilute to 1 L at the temperature at which titrations are to be made.

NOTE 12—Occasionally K₂Cr₂O₇ is found containing sodium dichromate (Na₂Cr₂O₇), although this is rare. If the character of the K₂Cr₂O₇ is not certain, the purity can be ascertained by titration against freshly resublimed iodine. However, this is usually unnecessary.

50.2 *Potassium Iodide Solution (150 g/L)*—Dissolve 150 g of potassium iodide (KI) in water and dilute to 1 L.

50.3 *Sodium Thiosulfate, Standard Solution (0.1 N)*—Dissolve 24.8 g of sodium thiosulfate (Na₂S₂O₃·5H₂O) in freshly boiled water and dilute to 1 L at the temperature at which the titrations are to be made. To standardize, place 40 mL of K₂Cr₂O₇ to which has been added 10 mL of the solution of KI in a glass-stoppered flask, add 5 mL of concentrated hydrochloric acid (HCl, sp gr 1.19), dilute with 100 mL of water, and allow the Na₂S₂O₃·5H₂O solution to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste, and while shaking constantly, continue to add the Na₂S₂O₃·5H₂O solution until the blue color just disappears.

50.4 *Starch Paste*—Boil 1 g of starch in 200 mL of water for 10 min, and cool to room temperature.

NOTE 13—An improved starch solution may be prepared by autoclaving 2 g of starch and 6 g of boric acid dissolved in 200 mL of water at 15-psi pressure for 15 min. This solution has good keeping qualities.

50.5 *Wijs Iodine Solution*¹⁰—Dissolve 13.0 g of resublimed iodine in 1 L of glacial acetic acid and pass in washed and dried

¹⁰ Stock Wijs solution can be readily purchased from a number of chemical supply houses at nominal costs.