Designation: D 800 – 05

Standard Test Methods of Chemical Analysis of Industrial Metal Cleaning Compositions[1]

This standard is issued under the fixed designation D 800; 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.

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

1. Scope

1.1 These test methods describe the procedures for the chemical analysis of industrial metal cleaning compositions in solid, paste, or liquid form. These cleaning compositions may contain caustic alkalies, silicates, phosphates, chromates, carbonates, bicarbonates, borates, sulfates, sulfites, nitrates, chlorides, soaps, rosin, sulfonated wetting agents, anti-foaming agents, organic bases, organic solvents, organic coupling agents, and sometimes inorganic acid salts or organic acids. Occasionally, such cleaning compositions may contain sulfonated oils; in such cases, reference should be made to Method D 500. The examination under a low power microscope or magnifying glass, supplemented by a few qualitative tests, will often indicate the number of components and their identity.

1.2 The analytical methods appear in the following order:

	Section
Preparation of Sample	4
Total Alkalinity as Na_2O	5-8
Total Fatty Acids (and Rosin)	9 and 10
Na_2O Combined with Fatty Acids (and Rosin)	11 and 12
Anhydrous Soap	13
—Rosin (McNicoll Test Method):	
Qualitative	14 and 15
Quantitative	16-19
Total Silica Calculated as SiO_2	20-23
Phosphates:	
Qualitative	24 and 25
Quantitative	26-18
Combined Sodium and Potassium Oxides	34-37
Chlorides	38-40
Sulfates	41-43
Water, Distillation Test Method	44-47
Carbon Dioxide by Evolution-Absorption Test Method	48-52
Loss at 105°C	53 and 54
Total Matter Insoluble in Alcohol	55-57
Free Alkali	58-60
Synthetic Detergent or Wetting Agent	61 and 62
Volatile Hydrocarbons	63-67
Loss on Ignition	68 and 69
Report	70 and 71

1.3 *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. See 6.1 for a specific hazards statement.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 500 Test Methods of Chemical Analysis of Sulfonated and Sulfated Oils
D 841 Specification for Nitration Grade Toluene
D 843 Specification for Nitration Grade Xylene
D 1193 Specification for Reagent Water

3. Purity of Reagents and Materials

3.1 *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.

3.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

4. Preparation of Sample

4.1 Samples of metal cleaning compositions shall be well mixed. Excessive exposure to the air shall be avoided to

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[1] These test methods are under the jurisdiction of ASTM Committee D-12 on Soaps and Other Detergents and are the direct responsibility of Subcommittee D12.12 on Analysis of Soaps and Synthetic Detergents.

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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] *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.

prevent the sample from absorbing moisture and carbon dioxide and, if a volatile solvent is present, to prevent evaporation.

4.2 *Solid Cleaners*—If the cleaner is a solid and appears to contain no volatile solvent, dissolve 10.000 g in freshly boiled water and dilute to 500 mL as the sample for analysis.

4.3 *Liquid or Paste Cleaners*—If the cleaner is a paste or liquid dissolve a sufficient portion in freshly boiled water to give the equivalent of 10.000 g of the dry material in solution, and dilute to 500 mL as the sample for analysis.

NOTE 1—In order to prepare this sample it is first necessary to determine the water content (Sections 44-47), loss at 105°C (53 and 54), and volatile hydrocarbons (Sections 63-67).

TOTAL ALKALINITY AS Na_2O

5. Reagents

5.1 *Ethyl Ether.*

5.2 *Hydrochloric Acid, Standard (0.5 N)*—Prepare and standardize 0.5 *N* hydrochloric acid (HCl).

5.3 *Methyl Orange Indicator Solution*

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

6. Hazards

6.1 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).

7. Procedure

7.1 Pipet 50 mL of the sample (1.000 g) prepared in accordance with Section 4, into a 250-mL Erlenmeyer flask. Add methyl orange indicator solution and a measured excess of 0.5 *N* HCl, and boil to expel carbon dioxide (CO_2). Cool, add 25 mL of ethyl ether, and titrate the excess HCl with 0.5 *N* NaOH solution. When the water layer is neutral to methyl orange, it should be clear unless sodium metasilicate (or other silicate) is present in large quantities. If silicates are present, they will form a gelatinous mass which will produce a troublesome emulsion. All the soap will have been decomposed during the reaction and the corresponding fatty acid will be found in the ether layer.

8. Calculation

8.1 Calculate the total alkalinity as percentage of sodium oxide (Na_2O) as follows:

$$\text{Total alkalinity as } Na_2O, \% = [(A - B) \times 0.0155 \times 100]/W \quad (1)$$

where:

A = millilitres of 0.5 *N* HCl (1 mL of 0.5 *N* HCl = 0.0155 g of Na_2O),
B = millilitres of 0.5 *N* NaOH solution, and
W = grams of sample used.

TOTAL FATTY ACIDS (AND ROSIN ACIDS)

9. Procedure

9.1 Pour into a separatory funnel the neutral ethyl ether-water mixture obtained from the titration in Section 7, using additional ether to wash all the fat or oil into the separatory funnel. Allow the two layers to separate, and draw off the water layer into a second separatory funnel. Wash the water layer with two 10-mL portions of ethyl ether. Combine the ether extracts, and wash with successive portions of water until they are no longer acid to methyl orange. Transfer the ether layer to a weighed flask, evaporate the ether over a steam bath, and finally dry to constant weight at 105°C.

10. Calculation

10.1 Calculate the percentage of fatty acids (and rosin acids) as follows:

$$\text{Total fatty acids (and rosin acids), } \% = (E/W) \times 100 \quad (2)$$

where:

E = grams of ether residue, and
W = grams of sample used.

Na_2O COMBINED WITH FATTY ACIDS (AND ROSIN ACIDS)

11. Procedure

11.1 Dissolve the fatty acid (and rosin acid) residue obtained under Section 9 in warm neutral alcohol, add six drops of phenolphthalein indicator solution, and titrate with 0.5 *N* NaOH solution.

12. Calculation

12.1 Calculate the percentage of Na_2O combined with the fatty acids (and rosin acids) as follows:

$$Na_2O \text{ combined with fatty acids (and rosin acids), } \%$$

$$= (B \times 0.0155)/W \times 100 \quad (3)$$

where:

B = millilitres of 0.5 *N* NaOH solution (1 mL of 0.5 *N* NaOH = 0.0155 g of Na_2O), and
W = grams of sample used.

ANHYDROUS SOAP

13. Calculation

13.1 Calculate the percentage of anhydrous soap as follows:

$$H = C \times 0.29 \quad (4)$$

$$F = R - H$$

$$P = F + C$$

where:

C = percentage of Na_2O combined with fatty acids (and rosin acids) (Section 11),
F = percentage of fatty acid anhydride,
H = percentage of water equivalent,
R = percentage of fatty acids (and rosin acids) (Section 10), and
P = percentage of anhydrous soap.

ROSIN (McNICOLL TEST METHOD) Qualitative

14. Reagents

14.1 *Acetic Anhydride* (99 to 100 %).

14.2 *Sulfuric Acid (34.7 + 35.7)*—Prepare by carefully adding 34.7 mL of H_2SO_4 (sp gr 1.84) to 35.7 mL of water.

15. Procedure

15.1 The presence of rosin may be determined qualitatively by testing a portion of the residue obtained under Section 9 by the Liebermann-Storch reaction as follows:

15.1.1 Dissolve a portion of the ether extract by warming in several times its weight of acetic anhydride, and then cool the solution. Allow several drops of H_2SO_4 to flow slowly into the acetic anhydride-fat mixture. Rosin is indicated by the development of a fugitive violet coloration changing to a brownish tinge at the margin of contact of the reagents. The test should be checked by testing a sample of fatty acids to which rosin has been added. If the test for rosin is positive the quantitative procedure should be carried out as described in Sections 16-19.

Quantitative

16. Apparatus

16.1 The apparatus shall consist of a glass flask connected, preferably by a ground-glass joint, to a reflux condenser.

16.1.1 *Esterification Flask*—A 150-mL flask of either the round-bottom or Erlenmeyer type shall be used.

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

17. Reagents

17.1 *Naphthalene-β-Sulfonic Acid Solution*—Dissolve 40 g of Eastman grade or equivalent reagent in 1 L of absolute methyl alcohol.

17.2 *Phenolphthalein Indicator Solution (5 g/L)*—Dissolve 0.5 g of phenolphthalein in 50 mL of neutral redistilled alcohol and then mix with 50 mL of water.

17.3 *Potassium Hydroxide 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).

17.4 *Sulfuric Acid (1 + 2)*—Carefully mix 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) into 2 volumes of water.

18. Procedure

18.1 *Preparation of Fatty and Rosin Acids*—Dissolve a sufficient portion of the sample to give the equivalent of 50 g of dry material in 500 mL of hot water. (If the cleaner to be tested contains alcohol, the alcohol should be completely removed by evaporation from the solution.) Add 100 mL of H_2SO_4 (1 + 2), and 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. Wash the acids in this manner three times. 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 rosin determination.

18.2 *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.

19. Calculation

19.1 Calculate the results as follows:

$$R = [(S - B) \times N \times 0.346 \times 100]/W \tag{5}$$

$$R_1 = R - 1.0$$

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

$$R_S = R_2 \times 1.064$$

where:

R = percentage of rosin acids in fatty acids,
R_1 = corrected percentage of rosin acids in fatty acids (Note 2),
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 the KOH solution,
W = grams of sample used,
F = percentage of total fatty acids (and rosin acids) in the cleaner, and
A = percentage of total anhydrous soap (Section 13).

19.2 If true fatty acid soap is desired, subtract the rosin soap from the total anhydrous soap.

NOTE 2—Cooperative studies have shown that the McNicoll test 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.

TOTAL SILICA CALCULATED AS SiO_2

20. Reagents

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

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

20.3 *Hydrofluoric Acid (sp gr 1.15)*—Prepare a solution of hydrofluoric acid (HF) having a specific gravity of 1.15.

20.4 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (CH_2SO_4).

21. Preparation of Sample

21.1 *When Soap is Present*—If soap is present in the cleaner, draw off the water solution from the total alkalinity titration (Section 7) into a porcelain evaporating dish. Add an excess of HCl (sp gr 1.19), evaporate the mixture to dryness on a steam bath, and carry out the procedure in Section 22<usb>. The weight of the sample will be 1.000 g.

21.2 *When Soap is Not Present*—If soap is *not* present in the cleaner, transfer a 100-mL aliquot of the sample solution obtained as described in Section 3 to a 400-mL evaporating dish, neutralize with HCl (sp gr 1.19) using methyl orange as the indicator, evaporate to apparent dryness on a steam bath, and carry out the procedure as described in Section 22. The weight of the sample will be 2.000 g.

22. Procedure

22.1 Triturate the dehydrated residue, obtained as described in 21.1 or 21.2, with the smooth end of a stirring rod, moisten the residue with 10 mL of HCl (1 + 1), and again evaporate to apparent dryness on the steam bath. Dehydrate at 110°C for 1 h, take up the residue with 10 mL of HCl (1 + 1) and 20 mL of water, and digest a short time on the steam bath to effect solution of the soluble salts. Filter the silica on a fine-texture paper by washing the dish with hot water. Scrub the dish with a rubber policeman and again wash thoroughly with hot water. Wash the residue and paper free of acid with hot water and reserve.

22.2 Evaporate the filtrate and washings on the steam bath in the porcelain dish used before, moisten the residue with 10 mL of HCl (1 + 1), and again evaporate to dryness. Dehydrate at 110°C for 1 h, take up the residue with 10 mL of HCl (1 + 1) and 20 mL of water, digest as before to dissolve soluble salts, and filter off any additional silica on a separate filter paper. Scrub the dish and wash the residue and filter paper free of acid as before. Reserve the filtrate for the determination of phosphates (31.2) and sulfates (Section 42).

22.3 Transfer both papers and residues to a platinum crucible, previously ignited and weighed without cover, and ignite until free of carbon, heating slowly at first. Cover the crucible with a platinum cover, heat to the highest temperature of a blast lamp for 15 min, cool in a desiccator, and weigh without the crucible cover.

22.4 Moisten the weighed contents of the crucible with water, add 10 mL of HF and 4 drops of concentrated H_2SO_4, evaporate to dryness over a low flame, ignite at the highest temperature of the blast lamp for 2 min, cool in a desiccator, and weigh.

23. Calculation

23.1 Calculate the weight of the total silica as SiO_2 as follows:

$$\text{Total SiO}_2, \% = [(A - B)/C] \times 100 \qquad (6)$$

where:

A = grams of ignited residue before treatment with HF (22.3),

B = grams of ignited residue after treatment with HF (22.4), and

C = grams of sample used.

PHOSPHATES
Qualitative

24. Reagents

24.1 *Nitric Acid (1 + 5)*—Mix 1 volume of concentrated nitric acid (HNO_3, sp gr 1.42) with 5 volumes of water.

24.2 *Silver Nitrate Solution (85 g/L)*—Dissolve 85 g of silver nitrate ($AgNO_3$) in water and dilute to 1 L.

24.3 *Sodium Hydroxide Solution (1 + 1)*—Dissolve sodium hydroxide (NaOH) in an equal weight of water. When using, decant the solution from the settled carbonate.

25. Procedure

25.1 The presence of an orthophosphate (Na_3PO_4) or pyrophosphate ($Na_4P_2O_7$) may be confirmed as follows: Acidify a sample of the cleaner with HNO_3(1 + 5). Extract fatty acids, if formed, with ether, remove carbon dioxide by aeration of the solution, and neutralize with clarified NaOH solution until faintly pink to phenolphthalein. Add $AgNO_3$ solution to the sample solution. The appearance of either the yellow silver orthophosphate precipitate or the white silver pyrophosphate precipitate indicates the presence of phosphates. Precipitates may also be obtained with chlorides and chromates. If phosphates are present, determine them in accordance with the quantitative procedure described in Section 32.

Quantitative

26. Application

26.1 This test method is applicable to any species of alkali metal phosphates free of interfering ions. The test method can be used for the analysis of soap and synthetic detergent builders if the sample is properly prepared (see Section 31).

27. Summary of Test Method

27.1 All of the phosphate present is converted, by acid hydrolysis, to the ortho form and titrated between pH 4.3 and 8.8 with sodium hydroxide solution.

28. Interferences

28.1 Heavy metals such as iron, aluminum, calcium, magnesium, etc., that will precipitate, either as insoluble phosphates or hydroxides, before the upper end point is reached, will interfere. Interference also occurs if borates, sulfites, carbonates, or other buffering materials are present. The last two and much of the borate will be expelled during the acid hydrolysis boil. Ammonia or other weak bases also will interfere. The most common interference is from silicic acid. Experiment and experience in analysis of spray-dried synthetics, have shown that unless the ratio of the percentage of SiO_2 to the percentage of P_2O_5 approaches or exceeds 0.2, the interference by silicates will be so slight that it may be neglected. Larger amounts must be dehydrated but need not be removed by filtration during preparation of the sample.

29. Apparatus

29.1 *Electrometric Titration Apparatus*, equipped with glass and calomel electrodes. Any standard pH meter, capable of performing titrations accurate to ±0.1 pH and accurately standardized at pH 4.0 and 8.0 is suitable.

29.2 *Gas Burners*, preferably of the chimney or Argand type.

29.3 *Muffle Furnace*, with suitable pyrometer and controls for maintaining temperatures up to 550°C.

29.4 *Motor Stirrer*, air or electric.

30. Reagents

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

30.2 *Mixed Indicator (optional)*—Prepare the following solutions:

30.2.1 *Methyl Orange Solution (0.5 g/L)*—Dissolve 0.05 g of methyl orange in water and dilute to 100 mL.

30.2.2 *Phenolphthalein, Alcohol Solution (5.0 g/L)*—Dissolve 0.50 g of phenolphthalein in alcohol (50 %) and dilute to 100 mL with alcohol.

30.2.3 *Thymol Blue Solution (0.4 g/L)*—Dissolve 0.04 g of thymol blue in water and dilute to 100 mL.

30.2.4 *Methylene Blue Solution (1.0 g/L)*—Dissolve 0.10 g of methylene blue in water and dilute to 100 mL.

30.2.5 *Alcohol (90 %)*—Prepared from alcohol conforming to Formula No. 3A or No. 30 of the U. S. Bureau of Internal Revenue.

Mix the solutions in the following proportions:

Methyl orange solution	32 mL
Phenolphthalein solution	32 mL
Thymol blue solution	8 mL
Methylene blue solution	4 mL
Alcohol	24 mL

The individual components are stable indefinitely. The mixed indicator should be prepared at least weekly. In practice, 3 mL of this mixed indicator are used in a final volume of approximately 250 mL of solution to be titrated. The lower end point is taken as the first change from gray to a definite green; the upper end point is the change from pink to a bright purple.

30.3 *Sodium Hydroxide, Standard Solution* (0.5 or 1.0 *N*)—Prepare an 0.5 or 1.0 *N* carbonate-free solution of sodium hydroxide (NaOH) and standardize accurately.

30.4 *Sodium Hydroxide Solution (1 + 1)*—See 24.3. A moredilute solution may be used. NaOH solutions must be protected from carbon dioxide (CO_2) contamination.

31. Preparation of Sample Solution

31.1 Commercial sodium or potassium phosphates need no special preparation except solution in water. Weigh a portion of the well-mixed sample to the nearest 0.001 g, transfer directly to a 400-mL beaker, and dissolve in about 100 mL of water. Neutralize to litmus paper with HCl and add 10 mL excess. The optimum size of sample is given by the formula:

$$\text{Grams of sample} = (N \times 280)/P \tag{7}$$

where:
N = normality of the NaOH solution to be used in the titration, and
P = percentage of P_2O_5 expected in the sample.

31.2 Soap products may be analyzed by using the filtrate from the silicon dioxide (SiO_2) determination (see 22.2). Use care not to exceed the sample weight prescribed in 31.1. Alternatively the sample may be prepared as described in 31.3.

31.3 Built synthetic products may be analyzed by using the alcohol-insoluble portion, but the following procedure is more rapid and quite as accurate. Weigh a sample, of size chosen by the formula in 31.1 (but not to exceed 10 g) to the nearest 0.001 g. Place the sample in a porcelain or silica evaporating dish, or large crucible, and ignite gently over a low gas burner until most of the volatile combustible matter is burned off. Transfer to a muffle, operated at not over 550°C, for 10 to 15 min. The ignited residue need not be free of carbon and usually is of a grayish color. Cool and add cautiously 10 mL of HCl. Evaporate to dryness, take up with 50 mL of water and 10 mL of HCl, and transfer to a 400-mL beaker.

32. Procedure

32.1 Each solution in a 400-mL beaker, prepared as described in 31.1, should have a volume of about 100 mL and contain an excess of at least 10 mL of HCl (sp gr 1.19). Cover with a watch glass and boil gently for a minimum of 30 min. Up to 60 min may be necessary for phosphates of the glass type. All phosphates must be in the ortho form. Cool to room temperature (20 to 30°C).

32.2 Dilute to 200 mL, place on an electrometric titration stand (Note 3), and neutralize to a pH of 4.3. Most of the neutralization may be made with NaOH solution, but final adjustment should be made with the standard NaOH solution (0.5 or 1.0 *N*) to be used in the titration. Cool again, if necessary, to maintain the temperature below 30°C. Titrate carefully to the upper end point (pH 8.8) recording the titration between end points (*T*).

NOTE 3—The mixed indicator may be used for this titration but with some small sacrifice of accuracy. If the samples have been prepared by the ignition test method, they must be filtered and the paper washed thoroughly, after the acid hydrolysis, as particles of carbon obscure the visual end point. The color changes can be checked by comparison with pH meter readings to acquire familiarity with the exact shade required. For greatest accuracy, titration with a pH meter is recommended.

33. Calculation

33.1 Calculate the percentage of total P_2O_5 as follows:

$$\text{Total } P_2O_5, \% = (TN \times 7.098)/W \tag{8}$$

where:
T = millilitres of NaOH solution required for titration of the sample,
N = normality of the NaOH solution, and
W = grams of sample in the sample solution.

COMBINED SODIUM AND POTASSIUM OXIDES

34. Reagents

34.1 *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.)

34.2 *Ethyl Alcohol (80 %)*—Ethyl alcohol conforming to either Formula No. 3A or No. 30 of the U. S. Bureau of Internal Revenue.

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

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

34.5 *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 potassium oxide (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.

35. Preparation of Sample

35.1 Weigh a sufficient portion of the sample to give the equivalent of 10 ± 0.01 g of the dry material 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. Avoid excessive heating. 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 (sp gr 1.19), dilute to 100 mL, mix thoroughly, and pass through a dry filter.

36. Procedure

36.1 Acidify an accurately measured 10-mL aliquot of the solution obtained in Section 34 with a few drops of HCl (sp gr 1.19) 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.

36.2 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, continuingthe 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.

37. Calculation

37.1 Calculate the percentage of potassium oxide (K_2O) as follows:

$$K_2O, \% = [(W \times 0.19376)/10] \times 100 \tag{9}$$

where:
W = grams of K_2PtCl_6 used.

37.2 Calculate the percentage of sodium oxide (Na_2O) as follows:

$$Na_2O, \% = C - (D \times 0.6582) \tag{10}$$

where:
C = percentage total alkalinity as Na_2O (Section 8), and
D = percentage combined K_2O (37.1) converted to equivalent Na_2O.

CHLORIDES

38. Reagents

38.1 *Magnesium Nitrate Solution (200 g/L)*—Dissolve 200 g of chloride-free magnesium nitrate ($Mg(NO_3)_2·6H_2O$) in water and dilute to 1 L.

38.2 *Potassium Chromate Indicator Solution*—Dissolve 5 g of potassium chromate (K_2CrO_4) in water and add a solution of $AgNO_3$ until a slight red precipitate is produced, filter the solution, and dilute to 100 mL.

38.3 *Silver Nitrate, Standard Solution (0.1 N)*—Prepare and standardize a 0.1 *N* silver nitrate ($AgNO_3$) solution.

39. Procedure

39.1 Dissolve a sufficient portion of the sample to give the equivalent of 5 ± 0.01 g of the dry material in 300 mL of water, boiling if necessary to effect solution. Add an excess of neutral, chloride-free, $MgNO_3$ solution (about 25 mL of $Mg(NO_3)_2·6H_2O$ solution (200 g/L)). Without cooling or filtering, titrate with 0.1 *N* $AgNO_3$ solution, using 2 mL of K_2CrO_4 indicator solution.

40. Calculation

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

$$\text{NaCl}, \% = [(S \times 0.00585)/W] \times 100 \tag{11}$$

where:
S = millilitres of 0.1 *N* $AgNO_3$ solution required for titration of the sample, and
W = grams of sample used.

SULFATES

41. Reagents

41.1 *Barium Chloride Solution (100 g/L)*—Dissolve 100 g of barium chloride ($BaCl_2·2H_2O$) in water and dilute to 1 L.

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

42. Procedure

42.1 The matter insoluble in alcohol obtained in accordance with Section 56 may suffice, or the filtrate from the determination of total silica as SiO_2(21.2) may be used as the sample.

42.2 If only a determination for sulfates is desired, ignite 10 ± 0.10 g of the sample and use the ash from the ignition. Digest with 100 mL of water, cover with a watch glass, and carefully neutralize to methyl orange with HCl. When neutralized, add 5 mL excess of HCl, filter, and wash the residue thoroughly. (Evaporation to dryness is unnecessary unless gelatinous silica should have separated. Evaporation should never be performed on a bath heated by gas.)

42.3 Dilute the filtrate to 250 mL in a beaker, and boil. To the boiling solution add 15 to 20 mL of $BaCl_2$ solution slowly, drop by drop, from a pipet. Continue boiling until the precipitate is well formed, or digest on a steam bath overnight. Set aside overnight or for a few hours, filter through a prepared Gooch crucible, ignite gently, and weigh as barium sulfate ($BaSO_4$).

43. Calculation

43.1 Calculate the sulfates as percentage of sodium sulfate (Na_2SO_4) as follows:

$$Na_2SO_4, \% = [(B \times 0.60858)/W] \times 100 \qquad (12)$$

where:

B = grams of $BaSO_4$, and
W = grams of sample used.

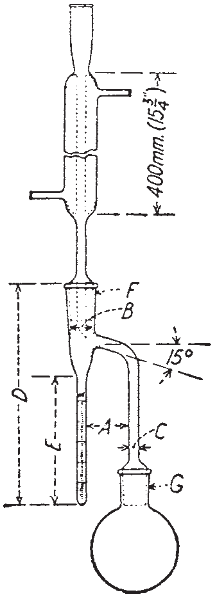
WATER BY DISTILLATION TEST METHOD

44. Apparatus

44.1 The apparatus shall consist 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, 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.

44.1.1 *Flask*—500-mL, of either the short-neck, round-bottom type or the Erlenmeyer type shall be used.

44.1.2 *Heat Source*—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.



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 of Distillation Apparatus

44.1.3 *Condenser*—A water-cooled glass reflux condenser (Fig. 1), having a jacket approximately 400 mm (15¾ in.) in length with an inner tube 9.5 to 12.7 mm (⅜ to ½ in.) in outside diameter shall be used. 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 (¼ in.) 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.

44.1.4 *Trap*—Made of well-annealed glass constructed in accordance with Fig. 1 and graduated as shown to contain 5 mL at 20°C shall be used. It shall be 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.

45. Reagents

45.1 *Sodium Acetate*—($NaC_2H_3O_2$), anhydrous, fused.

45.2 *Xylene (or Toluene)*—Xylene shall be used except for cleaners containing 1 % or more of glycerin, in which case toluene shall be used. Xylene shall conform to Specification D 843. Toluene shall conform to Specification D 841.

46. Procedure

46.1 For cleaners containing from 5 to 25 % moisture and volatile matter, use 20 ± 0.04 g of the sample. For cleaners 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) that has been saturated with water. Attach the flask to the trap which shall be 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.

46.2 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/min. When the greater part of the water has distilled over, increase the distillation rate to 200 drops/ 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.

47. Calculation

47.1 Calculate the percentage of water as follows:

$$\text{Water}, \% = [(V \times 0.998)/W] \times 100 \qquad (13)$$

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

CARBON DIOXIDE BY EVOLUTION-ABSORPTION TEST METHOD

48. Apparatus

48.1 *Extraction Flask*—A 150-mL wide-neck extraction flask.

48.2 *Condenser*—A 25-cm reflux condenser.

48.3 *Thistle Tube*—equipped with a two-way stopcock, the end to be drawn out to a fine point.

48.4 *U-Tubes*—Four U-tubes prepared as described in 50.2.

48.5 *Drying Tube*—Filled with a drying agent as described in 50.2.

48.6 *Aspirator*—A means of aspirating the apparatus to ensure a continuous stream of carbon dioxide (CO_2)-free air.

49. Reagents

49.1 *Methyl Orange Indicator Solution*.

49.2 *Sulfuric Acid (2 + 9)*—Carefully mix 2 volumes of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) with 9 volumes of water.

50. Preparation of Apparatus

50.1 *Apparatus Assembly*—Place the 150-mL extraction flask on a gauze over a burner. Fit the flask with a three-hole rubber stopper, one opening to carry the reflux condenser, the second to carry a thistle tube with a two-way stopcock for the introduction of acid into the flask, and the third to carry the tube for the introduction of a continuous stream of CO_2-free air into the flask. Draw out the ends of the thistle and air supply tubes to a small point, and place them in the stopper so that the points are very close to the bottom of the flask. Attach to the air supply tube, one of the U-tubes containing ascarite so that the air admitted to the flask will be free from CO_2.

50.2 *Preparation of Absorption Train*—Attach to the top of the reflux condenser a train consisting of the following:

50.2.1 A U-tube containing granulated zinc for the removal of acid gases,

50.2.2 A drying tube containing magnesium perchlorate, anhydrous calcium sulfate (Drierite), or anhydrous calcium chloride,

50.2.3 A weighed U-tube containing ascarite in the first half and the same drying agent in the second half as used in 50.2.2, and

50.2.4 A protective U-tube containing any of the above mentioned drying agents.

50.3 Attach the final tube to an aspirator.

51. Procedure

51.1 Aspirate with a stream of carbon dioxide (CO_2)-free air at a rate of approximately 20 to 30 mL/min until the train is free of CO_2 as determined by no further change in weight greater than 0.3 mg in the U-tube. Weigh a sufficient portion of the sample to give the equivalent of 10 ± 0.01 g of the dry material directly into the extraction flask, cover with 50 mL of freshly boiled water, add 2 drops of methyl orange indicator solution, and close the apparatus with the train in place.

51.2 Start the aspiration at a rate of 20 to 30 mL/min, and slowly add through the thistle tube sufficient $H_2SO_4(2 + 9)$ to neutralize the NaOH and a sufficient excess to ensure the final acidity of the mixture as indicated by the methyl orange. Always leave some acid in the thistle tube as an air seal. Heat gently and continue until the contents of the flask have boiled for 5 min; remove the source of heat, and continue aspirating until the flask has cooled, or for about 30 min. Remove the U-tube containing ascarite and weigh using a tared U-tube as a counterpoise. The increase in weight represents CO_2.

52. Calculation

52.1 From the increase in weight of the tube calculate the percentage of sodium carbonate (Na_2CO_3) as follows:

$$Na_2CO_3, \% = [(A \times 2.409)/W] \times 100 \quad (14)$$

where:
A = grams of CO_2, and
W = grams of sample used.

52.2 Calculate the percentage of carbon dioxide (CO_2) as follows:

$$CO_2, \% = (B/G) \times 100 \quad (15)$$

where:
B = grams of CO_2, and
G = grams of sample used.

LOSS AT 105°C

53. Procedure

53.1 Dry 2 to 10 ± 0.01 g of the sample at 105°C to constant weight, care being taken to prevent the sample from absorbing carbon dioxide (CO_2). Reserve the residue for the determination of total matter insoluble in alcohol (Section 56).

54. Calculation

54.1 Calculate the percentage loss at 105°C as follows (Note 4):

$$\text{Loss at } 105°C, \% = [(A - B)/A] \times 100 \quad (16)$$

where:
A = grams of sample, and
B = grams of dried sample.

NOTE 4—Bicarbonates are converted to carbonates on drying at 105°C and lose 36.9 % of their weight. It is next to impossible to determine bicarbonates in the presence of caustic alkalies because of conversion to the carbonate.

TOTAL MATTER INSOLUBLE IN ALCOHOL

55. Reagents

55.1 *Neutral Ethyl Alcohol* (*absolute*)—Freshly boiled ethyl alcohol, neutral to phenolphthalein.[4]

55.2 *Neutral Ethyl Alcohol (95 %)*—Freshly boiled, reagent grade ethyl alcohol, 95 % or higher, neutral to phenolphthalein, and containing only volatile denaturants. A possible source is to add 5 mL of water to 95 mL of absolute alcohol (55.1).

56. Procedure

56.1 Weigh 2 ± 0.001 g of the sample into a 250-mL beaker. Add 100 mL of neutral ethyl alcohol (95 %), cover the beaker, and heat on the steam bath with frequent stirring and maceration of the sample until completely disintegrated. Let settle and filter the supernatant liquid through a tared sintered-glass crucible, with suction, into a 500-mL filtering flask, retaining as much of the residue as possible in the beaker. Repeat this extraction three times with 25-mL portions of hot neutral ethyl alcohol (95 %), each time retaining as much of the residue as possible in the beaker. Reserve the alcoholic filtrate for determination of free alkali (Section 58). Finally, evaporate any remaining alcohol and dissolve the residue in the smallest possible quantity of hot water (5 mL is sufficient). Reprecipitate the alcohol-insoluble matter by slowly adding, while stirring vigorously, 50 mL of neutral ethyl alcohol (absolute).

NOTE 5—Solution and reprecipitation of alcohol-insoluble matter is necessary for complete separation from alcohol-soluble matter.

56.2 Heat the solution to boiling on the steam bath, filter, and transfer the precipitate quantitatively to the sintered-glass crucible, washing several times with neutral ethyl alcohol (95 %). Combine all of the filtrate and washings together with those from 56.1, and reserve for determination of free alkali (Section 58). Evaporate on the steam bath any alcohol remaining in the precipitate in the sintered-glass crucible, and then dry to constant weight at 105 ± 2°C.

57. Calculation

57.1 Calculate the percentage of total matter insoluble in alcohol as follows:

$$T = (M/W) \times 100 \tag{17}$$

where:

T = percentage of total matter insoluble in alcohol,
M = grams of insoluble matter, and
W = grams of sample used.

FREE ALKALI

58. Reagents

58.1 *Hydrochloric Acid (0.5 N)*—Prepare and standardize a 0.5 *N* hydrochloric acid (HCl) solution.

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

59. Procedure

59.1 Heat the reserved filtrate from the determination of total matter insoluble in alcohol (Section 56) to incipient boiling, add 0.5 mL of phenolphthalein indicator solution, and titrate with 0.5 *N* HCl. Reserve the titrated solution for the determination of the synthetic detergent or wetting agent (Section 61).

60. Calculation

60.1 Calculate the percentage of free alkali as sodium hydroxide (NaOH) as follows:

$$\text{Free alkali as NaOH, \%} = [(A \times 0.0200)/W] \times 100 \tag{18}$$

where:

A = millilitres of 0.5 *N* HCl required for titration of the sample, and
W = grams of sample used.

SYNTHETIC DETERGENT OR WETTING AGENT

61. Procedure

61.1 Transfer the titrated solution obtained in accordance with Section 59 to a tared beaker or evaporating dish, and evaporate to dryness on a steam bath. Dry to constant weight at 105°C.

62. Calculation

62.1 Calculate the percentage of synthetic detergent or wetting agent as follows (Note 6):

$$A = \frac{[E - (G + H)]}{W} \times 100 \tag{19}$$

where:

A = percentage synthetic detergent or wetting agent,
E = grams of titrated alcohol extract,
G = grams of NaCl,
H = grams of soap and rosin soap, and
W = grams of sample used.

NOTE 6—Since soap and rosin soap are also alcohol soluble, and since the known amount of NaCl was formed by the titration in the determination of free alkali (Section 59), also determined in the procedure for chlorides (Section 39), correction for soap, rosin soap, and chlorides may be made for each of these constituents. One millilitre of 0.5 *N* HCl (see Section 59) is equivalent to 0.0292 g of NaCl.

VOLATILE HYDROCARBONS

63. Summary of Test Method

63.1 This procedure requires a source of dry, oil-free steam which is passed through the sample treated with acid, sufficient to liberate the fatty acids from the cleaner. The steam is next passed through strong sodium hydroxide solution to scrub out any volatile fatty acids, while the volatile hydrocarbons are condensed with the steam in a suitable arrangement which allows the excess water to flow away, leaving the volatile hydrocarbons in the measuring buret. The procedure may be applied to samples containing substances immiscible with water and volatile with steam. (For solvents heavier than water

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[4] Fischer Scientific A962, or its equivalent, is suitable for this purpose.

the trap as shown in Fig. 1 for determining water by the distillation method (Sections 44-47) should be used.)

64. Apparatus

64.1 The apparatus and its arrangement are shown in Fig. 2. The following are the important items:

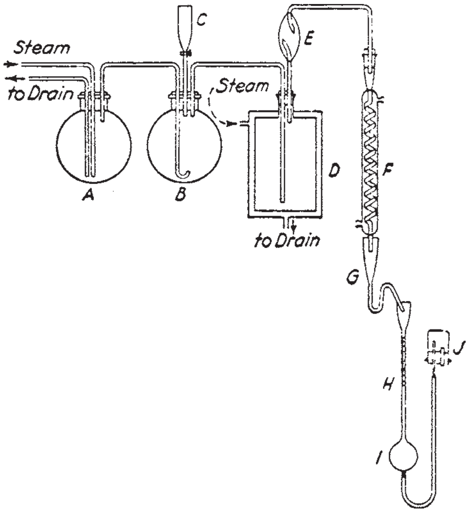
64.1.1 *Steam Trap, A*—A 1-L round-bottom, ring-neck flask equipped with a siphon tube to the drain from the bottom of the flask and provided with a means of regulating the steam flow into the flask.

64.1.2 *Evolution or Sample Flask, B*—A 1-L round-bottom, ring-neck flask. In case large samples are desirable the size of this flask may be increased.

64.1.3 *Caustic Scrubber Flask, D*—A steam-jacketed metal flask is preferred, but a 1-L Florence flask provided with a steam coil of 0.32-cm (0.125-in.) copper tubing around the upper half may be used. If the glass flask is used it should be provided with a safety bucket below it and should be renewed frequently since the strong caustic dissolves the glass rather rapidly. This flask should be connected to the condenser by a Kjeldahl connecting tube, *E*, or similar safety device. The inlet for the steam into the evolution and scrubber flasks should extend nearly to the bottom of the flasks and should be bent at right angles and parallel to the sides of the flask.

64.1.4 *Condenser, F*—A 30.5-cm (12-in.) or longer spiral condenser of sufficient bore so the condensate will not readily close it.

64.1.5 *Measuring Buret, H*—A 10-mL buret calibrated to 0.1 mL and carrying a bulb, *I*, of approximately 100-mL capacity, at the lower end. If desired, an ordinary 10-mL Mohr-type buret may be used having attached to it by rubber tubing a bulb of proper capacity which has been blown in the laboratory. An ordinary buret funnel may be placed in the top of the buret in place of the special flared-out top shown in Fig. 2. The stoppers used should be of a good grade of rubber and should have been thoroughly cleaned free of any surface sulfur and should be given a steam distillation in position for several hours before use on a sample. Insulating the flasks and tubing to reduce condensation aids distillation and its control.



A—Steam Trap
B—Sample Flask
C—Dropping Funnel
D—Caustic Scrubber-Iron
E—Kjeldahl Trap
F—Condenser
G—Siphon
H—Buret
I—Expansion Bulb
J—Automatic Overflow

FIG. 2 Volatile Hydrocarbon Apparatus

65. Reagents

65.1 *Sodium Hydroxide Solution (650 g/L)*—Dissolve 650 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

65.2 *Sodium Hydroxide (NaOH)*, sticks or pellets.

65.3 *Sulfuric Acid (1 + 3)*—Mix 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) carefully with stirring into 3 volumes of water.

66. Procedure

66.1 Place 150 mL of NaOH solution and several sticks of solid NaOH to provide against dilution in the scrubber flask. Rinse out the condenser and buret with acetone. Attach a rubber tubing to the lower end of the buret, fill the buret and tubing with water, and raise the outer end of the tubing so that the water level in the buret is near the top of the scale when the water is flowing to the drain from the automatic overflow, *J*. Be sure that the connections are tight and that the tubing contains no air bubbles. Place the condenser in position so that the lower end extends directly into the upper end of the buret just above the water level or connect to an adapter siphon, *G*, which discharges into the buret. The cooling water should be 15.5°C or colder. Ice water may be desirable for low-boiling hydrocarbons.

66.2 Weigh a sufficient portion of the sample to give the equivalent of 50 g of the dry material, and transfer to the evolution flask (Note 7). Add about 10 g of gum arabic (commercial) and 100 mL of water. Place the flask in position with 100 mL of H_2SO_4 in a dropping funnel, *C*, carried in the stopper. Connect with the steam and wash the flasks and the condenser, making sure that the stoppers are tightly fitting and held in place by wiring. Unnecessary rubber connections in the lines between the evolution flask and condenser should be avoided by placing the glass portions as tightly together as possible.

NOTE 7—For some samples the volatile hydrocarbon content may be so low that a larger sample than 50 or 100 g is desirable. The size of the evolution flask may need to be increased if larger samples are used. The amount of water in the evolution flask and acid used should also be correspondingly increased.

66.3 Add the acid to the sample slowly to avoid excessive frothing. While adding the acid, turn on the steam cautiously, so adjusting the pressure by a bleeder valve that just enough steam flows to prevent any liquid from backing into the steam trap flask.

66.4 When all the acid has been added, turn on enough steam to cause brisk distillation, taking care that no liquid is carried over from the evolution and wash flasks and that the condenser water does not become warm.

66.5 Continue the distillation until there is no increase in the volume of the upper layer for 45 min or no small droplets can be noted in the condensate.

66.6 When distillation is completed, shut off and drain the condenser water, and allow the steam to heat up the condenser to drive out the last traces of volatile hydrocarbon. Shut off the steam as soon as vapor begins to issue from the lower end of the condenser. Immediately open the stopcock of the dropping funnel to prevent NaOH solution from being drawn into the evolution flask.

66.7 Stopper the buret and allow its contents to come to room temperature or bring them to a definite temperature by immersing the buret for 1 to 2 h in a water bath held at 25°C.

66.8 Read the volume of the upper layer to the nearest 0.01 mL. The volume multiplied by the specific gravity equals the weight of the volatile hydrocarbons. The specific gravity should be determined at the temperature at which the volume is read. A small Sprengel tube made of 3-mm glass tubing is convenient for this purpose.

67. Calculation

67.1 Calculate the percentage of volatile hydrocarbons as follows:

$$V = [(M \times S)/W] \times 100 \tag{20}$$

where:
V = percentage of volatile hydrocarbons,
M = millilitres of volatile hydrocarbons,
S = specific gravity of volatile hydrocarbons, and
W = grams of sample used.

LOSS ON IGNITION

68. Procedure

68.1 Weigh accurately about 2 g of sand in a clean platinum crucible with a tight-fitting lid, and ignite to constant weight. Weigh accurately about 2 g of the cleaner in the crucible, and heat with a low flame. Increase the heat gradually as the water is driven off, care being taken to prevent spattering. Ignite to constant weight. Cool in a desiccator, and weigh.

69. Calculation

69.1 Calculate the loss on ignition as follows:

$$\text{Loss on ignition, \%} = (L/W) \times 100 \tag{21}$$

where:
L = grams loss in weight, and
W = grams of sample used.

REPORT

70. Composition of Cleaner

70.1 *Paste or Liquid Cleaners*—The sum of the water, soap (and rosin soap), volatile hydrocarbons, synthetic detergent or wetting agent, and total matter insoluble in alcohol shall be reported and should represent 100 % but seldom totals this amount. The constituents of the total matter insoluble in alcohol may be reported as described in 70.2 for the constituents of solid cleaners.

70.2 *Solid Cleaners*—The following constituents shall be reported (Note 8):

70.2.1 Percentage of total sodium oxide (Na_2O),
70.2.2 Percentage of potassium oxide (K_2O),
70.2.3 Percentage of free sodium hydroxide (NaOH),
70.2.4 Percentage of water (distillation test method),
70.2.5 Percentage of loss at 105°C,
70.2.6 Percentage of loss on ignition,
70.2.7 Percentage of matter insoluble in alcohol,
70.2.8 Percentage of fatty acids (and rosin acids),
70.2.9 Percentage of rosin,
70.2.10 Percentage of synthetic detergent or wetting agent,
70.2.11 Percentage of silica (SiO_2),
70.2.12 Percentage of phosphorus pentoxide (P_2O_5),
70.2.13 Percentage of carbon dioxide (CO_2),
70.2.14 Percentage of sodium chloride (NaCl),
70.2.15 Percentage of sodium sulfate (Na_2SO_4), and
70.2.16 Percentage of volatile hydrocarbons.

NOTE 8—Of the several determinations water, free NaOH, loss at 105°C, loss on ignition, matter insoluble in alcohol, rosin, synthetic detergent or wetting agent, NaCl, Na_2SO_4, and volatile hydrocarbons are complete determinations in themselves. Water may be free or combined as water of crystallization. Loss at 105°C will show the total free or uncombined volatile matter, while loss on ignition will indicate the amount of combined water and organic or inorganic matter present. The determination of material insoluble in alcohol will account for most of the alkalies except sodium hydroxide, and is a means for checking the matter other than soap or synthetic agent which may be present in a composition. Since rosin is often added as such to metal cleaning compositions, it is not always satisfactory to calculate it as rosin soap.

71. Conversion Factors

71.1 Conversion of the various constituents determined to their equivalents as chemical compounds for the purpose of reporting may be made as follows:

71.1.1 *Soap*—See Section 13.

71.1.2 *Sodium Silicate*—To calculate sodium oxide (Na_2O) combined with silica (SiO_2):

Use ratio of 1 Na_2O to 3.25 SiO_2 for liquids and pastes. This silicate is most commonly used:

$$Na_2O, \% = SiO_2, \% \times 0.3077 \tag{22}$$

SiO_2 to Na_2SiO_3, if identified:

$$Na_2O, \% = 0.5082 \times Na_2SiO_3, \% \tag{23}$$

SiO_2 to $(Na_2O)_{1.5}(SiO_2)$, if identified:

$$Na_2O, \% = 0.6075 \times \text{sesquisilicate, \%} \tag{24}$$

SiO_2 to Na_4SiO_4, if identified:

$$Na_2O, \% = 0.6737 \times Na_4SiO_4, \% \tag{25}$$

71.1.3 *Sodium Phosphate*—To calculate sodium oxide (Na_2O) combined with phosphorus pentoxide (P_2O_5):

Calculate P_2O_5 to Na_3PO_4, if identified:

$$\text{Titratable } Na_2O, \% = 0.3778 \times Na_3PO_4, \% \tag{26}$$

Calculate P_2O_5 to $Na_4P_2O_7$, if identified:

$$\text{Titratable } Na_2O, \% = 0.2331 \times Na_4P_2O_7, \% \tag{27}$$

Calculate P_2O_5 to Na_3PO_4 and $Na_4P_2O_7$, if identified

NOTE 9—If the total P_2O_5 is known, and the sum of the two phosphates determined (this can often be obtained by subtracting the total of all the other known compounds from 100, but may be far from accurate because of uncertainties surrounding such items as carbonates, silicates, etc.) this

is a simple algebraic calculation.
Calculate percentage of Na_2O as in Eq 5 and Eq 6

71.1.4 *Potassium*—If potassium was found it is very likely to be present as potassium soap. Calculate the potassium oxide (K_2O), if present, to soap in about the correct amount to combine with fatty acids:

$$\text{Molecular wt of potassium soap} = (F + 56.1 - 18 \qquad (28)$$

where:
F = molecular weight of fatty acids (and rosin acids) (Section 10).

$$\text{Wt of potassium soap} = (2 \times S \times K_2O, \%)/94.2 \qquad (29)$$

where:
S = molecular weight of potassium soap.

$$\text{Potassium soap}, \% = (S/W) \times 100 \qquad (30)$$

where:
S = grams of potassium soap, and
W = grams of sample (1.000 g).

71.1.5 *Residual K_2O*—Calculate residual potassium oxide (K_2O), if any remains, to potassium carbonate (K_2CO_3):

$$K_2CO_3, \% = K_2O, \% \times 1.467 \qquad (31)$$

71.1.6 *Sodium and Potassium Oxides*—Calculate carbon dioxide (CO_2) to sodium carbonate (Na_2Co_3) if no potassium carbonate (K_2CO_3) is present:

$$Na_2CO_3, \% = CO_2, \% \times 2.409 \qquad (32)$$

If K_2CO_3 is present, calculate the percentage of CO_2 combined with K_2O as follows:

$$CO_2, \% = K_2CO_3, \% \times 0.3184 \qquad (33)$$

Then subtract this percentage of CO_2 from the total CO_2 and calculate the balance to Na_2CO_3 as in Eq 31.

71.1.7 *Total Sodium Oxide (Na_2O)*—Total the calculated percentage of Na_2O combined with all the alkali titratable acid radicals:

		Na_2O %
Soap (Section 13)		____
Sodium silicate, (71.1.2)	1 + 3.25 ratio (Eq 22) or	____
	1 + 1 ratio, if identified (Eq 23), or	____
	1 + 5 + 1 ratio, if identified (Eq 24), or	____
	2 + 1 ratio, if identified (Eq 25)	____
Sodium phosphate[A] (71.1.3)	Na_3PO_4(Eq 26)	____
	$Na_4P_2O_7$(Eq 27)	____
K_2CO_3, (71.1.4)	K_2CO_3, % × 0.4485 (Equivalent to Na_2O) (Eq 31)	____
		____
Na_2CO_3, (71.1.5)	Na_2CO_3, % × 0.5848 (Eq 32)	____
		____
Total		____

[A] One Na for each P is not titratable.

71.1.8 If this total is in excess of the total alkalinity as percentage of Na_2O determined in accordance with Sections 7 and 8, the wrong silicate or phosphate has been used in the calculations.

71.1.9 If the total calculated percentage of Na_2O is less than the total Na_2O as determined under Sections 7 and 8 and sodium hydroxide has been proven to be present, calculate the residual alkalinity to NaOH as follows:

$$NaOH, \% = Na_2O, \% \times 1.2903 \qquad (34)$$