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Standard Test Methods of Chemical Analysis of Industrial Metal Cleaning Compositions¹

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 alkalis, 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 ₂ O	5-7
Total Fatty Acids (and Rosin)	8 and 9
Na ₂ O Combined with Fatty Acids (and Rosin)	10 and 11
Anhydrous Soap	12
—Rosin (McNicoll Test Method):	
Qualitative	13 and 14
Quantitative	15-18
Total Silica Calculated as SiO ₂	19-22
Phosphates:	
Qualitative	23 and 24
Quantitative	25-17
Combined Sodium and Potassium Oxides	33-36
Chlorides	37-39
Sulfates	40-42
Water, Distillation Test Method	43-46
Carbon Dioxide by Evolution-Absorption Test Method	47-51
Loss at 105°C	52 and 53
Total Matter Insoluble in Alcohol	54-56

Free Alkali	57-59
Synthetic Detergent or Wetting Agent	60 and 61
Volatile Hydrocarbons	62-66
Loss on Ignition	67 and 68
Report	69 and 70

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.

2. Referenced Documents

2.1 ASTM Standards:

- D 500 Test Methods of Chemical Analysis of Sulfonated and Sulfated Oils²
- D 841 Specification for Nitration Grade Toluene³
- D 843 Specification for Nitration Grade Xylene³
- D 1193 Specification for Reagent Water⁴

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.⁵ 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.

² *Annual Book of ASTM Standards*, Vol 15.04.

³ *Annual Book of ASTM Standards*, Vol 06.04.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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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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 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 43-46), loss at 105°C (52 and 53), and volatile hydrocarbons (Sections 62-66).

TOTAL ALKALINITY AS Na₂O

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. Procedure

6.1 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 (CO₂). 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.

7. Calculation

7.1 Calculate the total alkalinity as percentage of sodium oxide (Na₂O) as follows:

$$\text{Total alkalinity as Na}_2\text{O, \%} = [(A - B) \times 0.0155 \times 100]/W \quad (1)$$

where:

- A = millilitres of 0.5 N HCl (1 mL of 0.5 N HCl = 0.0155 g of Na₂O),
- B = millilitres of 0.5 N NaOH solution, and
- W = grams of sample used.

TOTAL FATTY ACIDS (AND ROSIN ACIDS)

8. Procedure

8.1 Pour into a separatory funnel the neutral ethyl ether-water mixture obtained from the titration in Section 6, 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.

9. Calculation

9.1 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.

Na₂O COMBINED WITH FATTY ACIDS (AND ROSIN ACIDS)

10. Procedure

10.1 Dissolve the fatty acid (and rosin acid) residue obtained under Section 8 in warm neutral alcohol, add six drops of phenolphthalein indicator solution, and titrate with 0.5 N NaOH solution.

11. Calculation

11.1 Calculate the percentage of Na₂O combined with the fatty acids (and rosin acids) as follows:

$$\begin{aligned} \text{Na}_2\text{O combined with fatty acids (and rosin acids), \%} \\ = (B \times 0.0155)/W \times 100 \end{aligned} \quad (3)$$

where:

- B = millilitres of 0.5 N NaOH solution (1 mL of 0.5 N NaOH = 0.0155 g of Na₂O), and
- W = grams of sample used.

ANHYDROUS SOAP

12. Calculation

12.1 Calculate the percentage of anhydrous soap as follows:

$$\begin{aligned} H &= C \times 0.29 \\ F &= R - H \\ P &= F + C \end{aligned} \quad (4)$$

where:

- C = percentage of Na₂O combined with fatty acids (and rosin acids) (Section 10),
- F = percentage of fatty acid anhydride,
- H = percentage of water equivalent,
- R = percentage of fatty acids (and rosin acids) (Section 9), and

P = percentage of anhydrous soap.

ROSIN (McNICOLL TEST METHOD)

Qualitative

13. Reagents

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

13.2 *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.

14. Procedure

14.1 The presence of rosin may be determined qualitatively by testing a portion of the residue obtained under Section 8 by the Liebermann-Storch reaction as follows:

14.1.1 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 15-18.

Quantitative

15. Apparatus

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

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

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

16. Reagents

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

16.2 *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.

16.3 *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).

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

17. Procedure

17.1 *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.

17.2 *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.

18. Calculation

18.1 Calculate the results as follows:

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

$$R_1 = R - 1.0$$

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

$$R_S = R_2 \times 1.064$$

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 12).

18.2 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

19. Reagents

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

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

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

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

20. Preparation of Sample

20.1 *When Soap is Present*—If soap is present in the cleaner, draw off the water solution from the total alkalinity titration (Section 6) 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 21. The weight of the sample will be 1.000 g.

20.2 *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 21. The weight of the sample will be 2.000 g.

21. Procedure

21.1 Triturate the dehydrated residue, obtained as described in 20.1 or 20.2, 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.

21.2 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 (30.2) and sulfates (Section 41).

21.3 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.

21.4 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.

22. Calculation

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

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

where:

- A = grams of ignited residue before treatment with HF (21.3),
- B = grams of ignited residue after treatment with HF (21.4), and
- C = grams of sample used.

PHOSPHATES Qualitative

23. Reagents

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

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

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

24. Procedure

24.1 The presence of an orthophosphate (Na_3PO_4) or pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7$) 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 31.

Quantitative

25. Application

25.1 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 30).

26. Summary of Test Method

26.1 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.

27. Interferences

27.1 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.

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

28. Apparatus

28.1 *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.

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

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

28.4 *Motor Stirrer*, air or electric.

29. Reagents

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

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

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

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

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

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

29.2.5 *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.

29.3 *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.

29.4 *Sodium Hydroxide Solution* (1 + 1)—See 23.3. A more-dilute solution may be used. NaOH solutions must be protected from carbon dioxide (CO₂) contamination.

30. Preparation of Sample Solution

30.1 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:

where:

N = normality of the NaOH solution to be used in the titration, and

P = percentage of P₂O₅ expected in the sample.

30.2 Soap products may be analyzed by using the filtrate from the silicon dioxide (SiO₂) determination (see 21.2). Use care not to exceed the sample weight prescribed in 30.1. Alternatively the sample may be prepared as described in 30.3.

30.3 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 30.1 (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.

31. Procedure

31.1 Each solution in a 400-mL beaker, prepared as described in 30.1, 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).

31.2 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.

32. Calculation

32.1 Calculate the percentage of total P₂O₅ as follows:

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

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

33. Reagents

33.1 *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.)

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

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

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

33.5 *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.

34. Preparation of Sample

34.1 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.

35. Procedure

35.1 Acidify an accurately measured 10-mL aliquot of the solution obtained in Section 33 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.

35.2 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, 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.

36. Calculation

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

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

where:

W = grams of K_2PtCl_6 used.

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

$$Na_2O, \% = C - (D \times 0.6582) \quad (10)$$

where:

C = percentage total alkalinity as Na_2O (Section 7), and

D = percentage combined K_2O (36.1) converted to equivalent Na_2O .

CHLORIDES

37. Reagents

37.1 *Magnesium Nitrate Solution (200 g/L)*—Dissolve 200 g of chloride-free magnesium nitrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) in water and dilute to 1 L.

37.2 *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.

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

38. Procedure

38.1 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 $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ 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.

39. Calculation

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

$$NaCl, \% = [(S \times 0.00585)/W] \times 100 \quad (11)$$

where:

S = millilitres of 0.1 N AgNO_3 solution required for titration of the sample, and

W = grams of sample used.

SULFATES

40. Reagents

40.1 *Barium Chloride Solution (100 g/L)*—Dissolve 100 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 L.

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