Designation: E 200 – 97 (Reapproved 2001)ε1

Standard Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[1]

This standard is issued under the fixed designation E 200; 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.

ε[1] NOTE—Editorial corrections were made in October 2001.

1. Scope

1.1 This practice covers procedures for the preparation, standardization, and storage of the standard volumetric solutions and reagent testing solutions commonly used in chemical analysis.

1.2 The information in this practice is arranged as follows:

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.* Specific warning statements are given throughout this practice.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[3]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[4]

E 203 Test Method for Water Using Karl Fischer Reagent[4]

E 694 Specification for Volumetric Ware[5]

2.2 *Other Document:*

Reagent Chemicals, American Chemical Society Specifications (ACS)[6]

3. Terminology

3.1 *Definition:*

3.1.1 *standard volumetric solution*—a solution of accurately determined concentration used in the quantitative analysis of chemicals and other products. The concentration of such solutions is usually expressed in terms of normality or molarity.

4. Significance and Use

4.1 The accuracy of many analytical measurements is dependent upon the manner in which the standard solutions are prepared and stored, and the accuracy with which they are standardized. Combining the methods recommended for the preparation and handling of such solutions into one practice eliminates the necessity for covering such details in all of the methods wherein the solutions are used.

5. Apparatus

5.1 *Volumetric Glassware*—The use of ordinary volumetric glassware will meet the accuracy requirements of many test methods.

[1] This practice is under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicalsand is the direct responsibility of Subcommittee E15.01 on General Standards.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 03.05.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Available from American Chemical Society, 1155 16th St., N.W., Washington, DC 20036.

NOTE 1—For dependable accuracy, volumetric glassware meeting the requirements for Class A items given in Specification E 694 should be used. While for normal work apparatus meeting these specifications can be used without calibration corrections, it is preferable that such calibration corrections be used in standardizing volumetric solutions. Such corrections may be of significance when the volumetric ware is frequently used with alkali solutions, for the corrosive effect of the alkali upon the glass may result in changes in the apparent volume. It is recommended, therefore, that volumetric glassware, particularly burets and transfer pipets, be recalibrated at 3-month intervals if it is frequently used to measure alkali solution volumes.

5.2 *Buret*—A 50-mL buret, or alternatively, a 100-mL buret with a 50-mL bulb at the top and a 50-mL stem below, may be used. For use with alkali solutions, burets equipped with TFE-fluorocarbon stopcock plugs are preferable.

6. Temperature Effects

6.1 Volumetric solutions are often used at temperatures differing from those at which the standardization was carried out. Significant errors may be introduced when the solutions are used at these other temperatures. Values for the change of normality with temperature (ΔN/°C) have been established for the volumetric solutions described herein, and are listed in Table 1. When warranted by the desired accuracy of the work, normalities of standard solutions may be corrected to the temperature at which they are used as follows:

$$N_{t2} = N_{t1} + (t_1 - t_2)(F) \quad (1)$$

where:

N_{t1} = normality of solution when standardized,
N_{t2} = normality of solution when used,
t_1 = temperature of solution during standardization,° C
t_2 = temperature of solution during use, °C, and
F = factor to correct for thermal expansion of the solution (ΔN/°C values from Table 1).

6.2 From the above equation it will be seen that the correction is to be added to the normality of the solution when standardized if the temperature of use is lower than the temperature of standardization while the correction is to be subtracted if the temperature of use is higher than the temperature of standardization.

7. Measurements

7.1 *Weighings*—When it is directed that a chemical should be "accurately weighed," the weighing is to be performed in a manner so as to limit the error to 0.1 % or less. Where a specific weight of substance is designated in a procedure, it is intended, unless otherwise specified in the individual procedure, that a quantity within ±5 % of the designated weight be used, and that this quantity be "accurately weighed" as just defined.

TABLE 1 Temperature Correction Factors (F)

Approximate Normality	Solute	ΔN/°C for 20 to 30°C
1.0	NaOH, HCl, H_2SO_4	0.00035
0.5	NaOH, HCl, H_2SO_4	0.00014
0.1	all aqueous	0.00002
0.05	all aqueous	0.00001
0.01	all aqueous	0.00000
0.5 (in methanol)	NaOH	0.00045
0.1 (in 1 *N* H_2SO_4)	$Ce(SO_4)_2$	0.000035
0.1 (in glacial acetic acid)	$HClO_4$	0.00011

NOTE 2—In weighing primary standards to be used in standardizing volumetric solutions many laboratories customarily weigh to the nearest 0.1 mg even though such increased accuracy of weighing does not improve the accuracy or precision of the standardization.

7.2 *Buret Readings*—When buret readings are specified, or when the procedure infers that a specific volume be measured from a buret, the reading is to be estimated to one fifth of the smallest volume subdivision marked on the buret. In reading a 50-mL buret having subdivisions of 0.10 mL, therefore, the reading should be estimated to the nearest 0.02 mL.

7.3 *Expression of Results*—It is customary to express the normality and molarity of standard solutions to 1 part in 1000.

8. Reagents

NOTE 3—Additional information on reagents is given in Practices E 50.

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[7] 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II or Type III reagent water conforming to Specification D 1193. Where specified, carbon dioxide-free water is to be prepared by heating distilled water to boiling in a conical flask, and boiling for 20 min. The boiling water is cooled in the flask which is stoppered with a 1-hole rubber stopper fitted to a soda lime-ascarite drying tube. For larger (10 to 20-L) volumes of carbon dioxide-free water, the absorbed carbon dioxide may be removed by inserting a fritted-glass gas-dispersion tube to the bottom of the container and bubbling nitrogen through the water for 1 or 2 h.

8.3 *Primary Standards*—The National Institute of Standards and Technology offers for sale certified standard samples of arsenic trioxide, benzoic acid, potassium hydrogen phthalate, potassium dichromate, sodium oxalate, and tris(hydroxymethyl)aminomethane. Where specified, these samples, or samples of commercially available primary standards, are to be used in standardizing the volumetric solutions.

9. Concentration of Solutions

9.1 *Standard Solutions*—Directions are given for the preparation of the most commonly used concentrations of the standard volumetric solutions. Stronger or weaker solutions are prepared and standardized in the same general manner as described, using proportionate amounts of the reagents. Similarly, if quantities larger than 1 L are to be prepared, proportionate amounts of the reagents should be used.

9.2 *Diluted Acids and Ammonium Hydroxide*—Concentrations of diluted acids and ammonium hydroxide, except when standardized, shall be specified as a ratio stating

[7] 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."

the number of volumes of the concentrated reagent to be diluted with a given number of volumes of water, as in the following example: HCl (5 + 95) means 5 volumes of concentrated HCl (sp gr 1.19) diluted with 95 volumes of water.

10. Mixing of Solutions

10.1 When quantities of solution larger than 1 to 2 L are prepared, special problems are encountered in being sure that they are well mixed before being standardized. While blade stirrers with glass or metal shafts are suitable for many solutions, they are not suitable in every case. In those cases where contact of a glass or metal stirrer with the solution would be undesirable it may be possible to use a sealed polyolefin-coated stirrer. In those cases where only contact of the solution with metal must be avoided, the solution can be mixed by inserting a fritted-glass gas-dispersion tube to the bottom of the container and bubbling nitrogen through the solution for 1 or 2 h.

11. Storage of Solutions

11.1 Glass containers are suitable for the storage of most of the standard solutions, although the use of polyolefin containers is recommended for alkali solutions.

11.2 When large quantities of solutions are prepared and standardized, it is necessary to provide protection against changes in normality due to absorption of gases or water vapor from the laboratory air. As volumes of solution are withdrawn from the container, the replacement air should be passed through a drying tube filled with equal parts of 8 to 20-mesh soda lime, oxalic acid, and 4 to 8-mesh anhydrous calcium chloride, each product being separated from the other by a glass wool plug or use equivalent commercially available absorption tubes.

12. Preparation and Standardization of Solutions

12.1 Methods of standardization are given for each volumetric solution even though the methods of preparation for some of these solutions specify that they be prepared on a determinate basis. Since it is not possible to prepare large volumes of solutions on a determinate basis, a method of standardization is provided for those solutions that are prepared in such large volumes that accurate measurements of the solution volumes cannot be made.

13. Precision and Bias

13.1 *Precision*—Precision for standardizing the volumetric solutions in this practice was determined in accordance with Practice E 180 – 90 and the forms of the statements conform with that suggested in Practice E 180 – 90.[8]

13.2 *Bias*—No information concerning the bias of these standardization methods is available because certified reference solutions suitable for this practice are not available.

[8] Data supporting the precision statements are available from ASTM Headquarters. Request RR: E-15-1039.

STANDARD VOLUMETRIC SOLUTIONS
SODIUM HYDROXIDE SOLUTION, 0.02 TO 1.0 *N*

14. Preparation of 50 % NaOH Solution and of Standard Solutions

14.1 Dissolve 162 g of sodium hydroxide (NaOH) in 150 mL of carbon dioxide-free water. Cool the solution to 25°C and filter through a hardened filter paper or other suitable medium. Alternatively, commercial 50 % NaOH solution may be used.

14.2 To prepare a 0.1 *N* solution, dilute 5.45 mL of the clear solution to 1 L with carbon dioxide-free water, mix well, and store in a tight polyolefin container.

14.3 For other normalities of NaOH solution, use the requirements given in Table 2.

15. Standardization

15.1 Crush 10 to 20 g of primary standard potassium hydrogen phthalate[9] ($KHC_8H_4O_4$) to 100-mesh fineness, and dry in a glass container at 120°C for 2 h. Stopper the container and cool in a desiccator.

15.2 To standardize a 0.1 *N* solution, weigh accurately 0.95 ± 0.05 g of the dried $KHC_8H_4O_4$, and transfer to a 500-mL conical flask. Add 100 mL of carbon dioxide-free water, stir gently to dissolve the sample, add 3 drops of a 1.0 % solution of phenolphthalein in alcohol, and titrate with NaOH solution to a color that matches that of an end point color standard.

15.3 The weights of dried $KHC_8H_4O_4$ suitable for other normalities of NaOH solution are given in Table 3.

16. pH 8.6 End Point Color Standard

16.1 Mix 25 mL of a solution 0.2 *M* in boric acid (H_3BO_3) and 0.2 *M* in potassium chloride (KCl), (1.24 g H_3BO_3 and 1.49 KCl in 100 mL water) with 12 mL of 0.1 *N* NaOH solution, add 3 drops of a 1.0 % solution of phenolphthalein in alcohol, and dilute to 100 mL with carbon dioxide-free water.

17. Calculation

17.1 Calculate the normality of the NaOH solution, as follows:

$$A = \frac{B}{0.20423 \times C} \quad (2)$$

[9] A primary standard grade of this chemical (and many others) is available from the Office of Standard Reference Materials, National Institute of Standards and Technology, Gaithersburg, MD 20899.

TABLE 2 Sodium Hydroxide Dilution Requirements

Desired Normality	Grams of NaOH Required/1 L of Solution	Volume of 50 % NaOH Solution (25°C) Required/1 L of Solution, mL
0.02	0.8	1.1
0.04	1.6	2.2
0.05	2.0	2.7
0.1	4.0	5.4
0.2	8.0	10.9
0.25	10.0	13.6
0.5	20.0	27.2
1.0	40.0	54.5

TABLE 3 Weights of Dried Potassium Hydrogen Phthalate

Normality of Solution	Weight of Dried $KHC_8H_4O_4$ to Be Used, g[A]
0.02	0.19 ± 0.005
0.04	0.38 ± 0.005
0.05	0.47 ± 0.005
0.1	0.95 ± 0.05
0.2	1.90 ± 0.05
0.25	2.35 ± 0.05
0.5	4.75 ± 0.05
1.0	9.00 ± 0.05

[A]The listed weights are for use when a 50-mL buret is to be used. If a 100-mL buret is to be used, the weights should be doubled.

where:
A = normality of the NaOH solution,
B = grams of $KHC_8H_4O_4$ used, and
C = millilitres of NaOH solution consumed.

18. Stability

18.1 The use of polyolefin containers eliminates some of the difficulties attendant upon the use of glass containers, and their use is recommended. Should glass containers be used, the solution must be standardized frequently if there is evidence of action on the glass container, or if insoluble matter appears in the solution.

19. Precision and Bias

19.1 The following criteria should be used for judging the acceptability of results:

19.1.1 *Sodium Hydroxide (1.0 N) (See Note 5)*:

19.1.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0007 normality units at 36 df. The 95 % limit for the difference between two such determinations is 0.0018 normality units.

19.1.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.0007 normality units at 18 df. The 95 % limit for the difference between two such averages is 0.0019 normality units.

19.1.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.001 normality units at 17 df. The 95 % limit for the difference between two such averages is 0.0029 normality units.

19.1.2 *Sodium Hydroxide* (0.1 *N*):

19.1.2.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00009 normality units at 28 df. The 95 % limit for the difference between two such determinations is 0.0003 normality units.

19.1.2.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00011 normality units at 14 df. The 95 % limit for the difference between two such averages is 0.0003 normality units.

19.1.2.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.00020 at 13 df. The 95 % limit for the difference between two such averages is 0.0005 normality units.

NOTE 4—Precision data have not been obtained for concentrations other than those listed.

NOTE 5—These precision estimates are based on an interlaboratory study conducted in 1962. One sample was analyzed. One analyst in each of 18 laboratories performed duplicate determinations and repeated them on a second day, for a total of 72 determinations. Practice E 180 was used in developing these statements.

NOTE 6—These precision estimates are based on an interlaboratory study conducted in 1962. One sample was analyzed. One analyst in each of 16 laboratories performed duplicate determinations and repeated them on a second day, for a total of 64 determinations. Practice E 180 was used in developing these statements.

HYDROCHLORIC ACID, 0.02 to 1.0 *N*

20. Preparation

20.1 To prepare a 0.1 *N* solution, measure 8.3 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) into a graduated cylinder and transfer it to a 1-L volumetric flask. Dilute to the mark with water, mix well, and store in a tightly closed glass container.

20.2 For other normalities of HCl solution, use the requirements given in Table 4.

21. Standardization with Sodium Carbonate[10]

21.1 Transfer 2 to 4 g of primary standard anhydrous sodium carbonate[11] (Na_2CO_3) to a platinum dish or crucible, and dry at 250°C for 4 h. Cool in a desiccator.

21.2 To standardize a 0.1 *N* solution, weigh accurately 0.22 ± 0.01 g of the dried Na_2CO_3, and transfer to a 500-mL conical flask. Add 50 mL of water, swirl to dissolve the carbonate, and add 2 drops of a 0.1 % solution of methyl red in alcohol. Titrate with the HCl solution to the first appearance of a red color, and boil the solution carefully, to avoid loss, until the color is discharged. Cool to room temperature, and continue the titration, alternating the addition of HCl solution and the boiling and cooling to the first appearance of a faint red color that is not discharged on further heating.

21.3 The weights of dried Na_2CO_3 suitable for other normalities of HCl solution are given in Table 5.

[10] A buret having a bent delivery tube is helpful in carrying out this standardization procedure.

[11] A primary standard grade of anhydrous sodium carbonate (Na_2CO_3) is available from Science Products Division Mallinckrodt Specialty Chemicals Co., P.O. Box M, Paris, KY 40361.

TABLE 4 Hydrochloric Acid Dilution Requirements

Desired Normality	Volume of HCl to Be Diluted to 1 L, mL
0.02	1.66
0.04	3.32
0.1	8.3
0.2	16.6
0.5	41.5
1.0	83.0

TABLE 5 Weights of Dried Sodium Carbonate

Normality of Solution	Weight of Dried Na_2CO_3 to Be Used, g
0.02	0.088 ± 0.001[A]
0.04	0.176 ± 0.001[A]
0.1	0.22 ± 0.01[B]
0.2	0.44 ± 0.01[B]
0.5	1.10 ± 0.01[B]
1.0	2.20 ± 0.01[B]

[A] A 100-mL buret should be used for this standardization.
[B] The listed weights are for use when a 50-mL buret is used. If a 100-mL buret is to be used, the weights should be doubled.

22. Calculation

22.1 Calculate the normality of the HCl solution, as follows:

$$A = \frac{B}{0.053 \times C} \quad (3)$$

where:
A = normality of the HCl solution,
B = grams of Na_2CO_3 used, and
C = millilitres of HCl solution consumed.

23. Stability

23.1 Restandardize monthly.

24. Precision and Bias (See Note 4)

24.1 The following criteria should be used for judging the acceptability of results:

24.1.1 *Hydrochloric Acid (1.0 N) (See Note 5)*:

24.1.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0004 normality units at 36 df. The 95 % limit for the difference between two such determinations is 0.0011 normality units.

24.1.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.0006 normality units at 18 df. The 95 % limit for the difference between two such averages is 0.0017 normality units.

24.1.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be 0.0015 normality units at 17 df. The 95 % limit for the difference between two such averages is 0.0042 normality units.

24.1.2 *Hydrochloric Acid (0.1 N) (See Note 6)*:

24.1.2.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00010 normality units at 28 df. The 95 % limit for the difference between two such determinations is 0.0003 normality units.

24.1.2.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00007 normality units at 14 df. The 95 % limit for the difference between two such averages is 0.0002 normality units.

24.1.2.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be 0.00017 normality units at 13 df. The 95 % limit for the difference between two such averages is 0.0005 normality units.

25. Standardization with Tris(hydroxymethyl)-Aminomethane:

25.1 Transfer 8 to 10 g of primary standard tris(hydroxymethyl)aminomethane[9] $[(HOCH_2)_3CNH_2]$ to a suitable dish or crucible, and dry in a vacuum at 70°C for 24 h. Cool in a desiccator.

25.2 To standardize a 0.1 *N* solution, weigh accurately 0.40 ± 0.02 g of the dried tris(hydroxymethyl)aminomethane, and transfer to a 250-mL beaker. Dissolve in 50 mL of ammonia- and carbon dioxide-free water, and titrate with the HCl solution to a pH of 4.70 using a suitable pH meter.

25.3 The weights of dried tris(hydroxymethyl)aminomethane suitable for other normalities of HCl solution are given in Table 6.

26. Calculation

26.1 Calculate the normality of the HCl solution, as follows:

$$A = \frac{B}{0.1211 \times C} \quad (4)$$

where:
A = normality of the HCl solution,
B = grams of tris(hydroxymethyl)aminomethane used, and
C = millilitres of HCl solution consumed.

27. Stability

27.1 Restandardize monthly.

28. Precision and Bias (See Notes 7 and 8)

28.1 The following criteria should be used for judging the acceptability of results:

28.1.1 *Hydrochloric Acid (1.0 N)*:

28.1.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0006 normality units at 16 df. The 95 % limit for the difference between two such determinations is 0.0016 normality units.

28.1.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been

TABLE 6 Weights of Dried Tris(Hydroxymethyl)Aminomethane

Normality of Solution	Weight of Dried $(HOCH_2)_3CNH_2$ to be Used, g
0.02	0.16 ± 0.008[A]
0.04	0.32 ± 0.016[A]
0.1	0.40 ± 0.02[B]
0.2	0.80 ± 0.04[B]
0.5	2.0 ± 0.1[B]
1.0	4.0 ± 0.2[B]

[A] A 100-mL buret should be used for this standardization.
[B] The listed weights are for use when a 50-mL buret is used. If a 100-mL buret is to be used, the weights should be doubled.

estimated to be 0.0007 normality units at 8 df. The 95 % limit for the difference between two such averages is 0.0019 normality units.

28.1.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be 0.0015 normality units at 7 df. The 95 % limit for the difference between two such averages is 0.0043 normality units.

28.1.2 *Hydrochloric Acid (0.1 N)*:

28.1.2.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00010 normality units at 16 df. The 95 % limit for the difference between two such determinations is 0.0003 normality units.

28.1.2.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00012 normality units at 8 df. The 95 % limit for the difference between two such averages is 0.0003 normality units.

28.1.2.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be 0.00024 normality units at 7 df. The 95 % limit for the difference between two such averages is 0.0007 normality units.

NOTE 7—These precision estimates are based on an interlaboratory study conducted in 1973. One sample of each concentration was analyzed. One analyst in each of 9 laboratories performed duplicate determinations and repeated them on a second day, for a total of 36 determinations for each concentration herein. Practice E 180 was used in developing these statements.

NOTE 8—Precision data have not been obtained for concentrations other than those listed in Section 28.

SULFURIC ACID, 0.02 TO 1.0 *N*

29. Preparation

29.1 To prepare a 0.1 *N* solution, measure 3.0 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) into a graduated cylinder and slowly add it to 400 mL of water in a 600-mL beaker. Rinse the cylinder into the beaker with water. Mix the acid-water mixture, allow it to cool, and transfer to a 1-L volumetric flask. Dilute to the mark with water, mix well, and store in a tightly closed glass container.

29.2 For other normalities of the H_2SO_4 solution, use the requirements given in Table 7.

30. Standardization [10]

30.1 Transfer 2 to 4 g of primary standard anhydrous sodium carbonate[11] (Na_2CO_3) to a platinum dish or crucible, and dry at 250°C for 4 h. Cool in a desiccator.

30.2 For standardization of a 0.1 *N* solution, weigh accurately 0.22 ± 0.01 g of the dried Na_2CO_3 and transfer to a 500-mL conical flask. Add 50 mL of water, swirl to dissolve the Na_2CO_3, and add 2 drops of a 0.1 % solution of methyl red in alcohol. Titrate with the H_2SO_4 solution to the first appearance of a red color, and boil the solution carefully, to avoid loss, until the color is discharged. Cool to room temperature and continue the titration alternating the addition of H_2SO_4 solution and the boiling and cooling, to the first appearance of a faint red color that is not discharged on further heating.

30.3 The weights of dried Na_2CO_3 suitable for other normalities of H_2SO_4 solution are given in Table 5.

TABLE 7 Sulfuric Acid Dilution Requirements

Desired Normality	Volume of H_2SO_4 to Be Diluted to 1 L, mL
0.02	0.60
0.1	3.0
0.2	6.0
0.5	15.0
1.0	30.0

31. Calculation

31.1 Calculate the normality of the H_2SO_4 solution, as follows:

$$A = \frac{B}{0.053 \times C} \quad (5)$$

where:

A = normality of the H_2SO_4 solution,
B = grams of Na_2CO_3 used, and
C = millilitres of H_2SO_4 solution consumed.

32. Stability

32.1 Restandardize monthly.

NOTE 9—A solution of 0.1 *N* sulfuric acid may be standardized using dried tris(hydroxymethyl)aminomethane by the same procedure used to standardize 0.1 *N* hydrochloric acid in Section 25.

33. Precision and Bias

33.1 The following criteria should be used for judging the acceptability of results:

33.1.1 *Sulfuric Acid (1.0 N)*:

33.1.1.1 *Repeatability (Single Analyst)*—See 24.1.1.1.

33.1.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—See 24.1.1.2.

33.1.1.3 *Reproducibility (Multilaboratory)*—See 24.1.1.3.

33.1.2 *Sulfuric Acid (0.1 N)*:

33.1.2.1 *Repeatability (Singly Analyst)*—See 24.1.2.1.

33.1.2.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—See 24.1.2.2.

33.1.2.3 *Reproducibility (Multilaboratory)*—See 24.1.2.3.

HYDROCHLORIC ACID, SPECIAL 1 *N*

NOTE 10—This solution is not for general use but is designed to satisfy the special requirements of ASTM Committee E-15, Subcommittee E15.52 on Alkalies.

34. Preparation

34.1 Measure 83.0 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) into a graduated cylinder and transfer it to a 1-L volumetric flask. Dilute to the mark with water, mix well, and store in a tightly closed glass container.

35. Standardization

35.1 Transfer 5 g of primary standard anhydrous sodium

carbonate[11] (Na_2CO_3) to a platinum dish or crucible, and dry at 250°C for 4 h (see Table 5). Cool in a desiccator. Weigh accurately 2.2 ± 0.1 g of the dried Na_2CO_3, and transfer to a 500-mL conical flask. Add 75 mL of water, swirl to dissolve the Na_2CO_3, and add 3 drops of a 0.1 % solution of methyl orange indicator. Titrate with HCl solution to a pink color.

35.2 Methyl orange indicator solution modified with xylene cyanole FF, suitable for use as an alternative indicator in this procedure, is described in 96.17. Titrate with HCl solution to a magenta color.

36. Calculation

36.1 Calculate the normality of the HCl solution, as follows:

$$A = \frac{B}{0.053 \times C} \quad (6)$$

where:
A = normality of the HCl solution,
B = grams of Na_2CO_3 used, and
C = millilitres of HCl solution consumed.

37. Stability

37.1 Restandardize monthly.

38. Precision and Bias (See Note 11)

38.1 The following criteria should be used for judging the acceptability of results:

38.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00064 normality units at 34 df. The 95 % limit for the difference between two such determinations is 0.0018 normality units.

38.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00081 normality units at 18 df. The 95 % limit for the difference between two such averages is 0.0023 normality units.

38.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be 0.0022 normality units at 16 df. The 95 % limit for the difference between two such averages is 0.0062 normality units.

NOTE 11—These precision estimates are based on an interlaboratory study conducted in 1962. One sample was analyzed. One analyst in each of 18 laboratories performed duplicate determinations and repeated them on a second day, for a total of 72 determinations. Practice E 180 was used in developing these statements.

SULFURIC ACID, SPECIAL 1 *N* (See Note 10)

39. Preparation

39.1 Measure 30.0 mL of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) into a graduated cylinder, and slowly add it to one half the desired volume of water in a 600-mL beaker. Rinse the cylinder into the beaker with water. Mix the acid-water mixture, allow it to cool, and transfer to a 1-L volumetric flask. Dilute to the mark with water, mix well, and store in a tightly closed glass container.

40. Standardization

40.1 Transfer 5 g of primary standard anhydrous sodium carbonate[11] (Na_2CO_3) to a platinum dish or crucible, and dry at 250°C for 4 h (see Table 5). Cool in a desiccator. Weigh accurately 2.2 ± 0.1 g of the dried Na_2CO_3, and transfer to a 500-mL conical flask. Add 75 mL of water, swirl to dissolve the Na_2CO_3, and add 3 drops of a 0.1 % solution of methyl orange. Titrate with H_2SO_4 solution to a pink color.

40.2 Methyl orange indicator solution modified with xylene cyanole FF, suitable for use as an alternative indicator in this procedure, is described in 96.17. Titrate with H_2SO_4 solution to a magenta color.

41. Calculation

41.1 Calculate the normality of the H_2SO_4 solution, as follows:

$$A = \frac{B}{0.053 \times C} \quad (7)$$

where:
A = normality of the H_2SO_4 solution,
B = grams of Na_2CO_3 used, and
C = millilitres of H_2SO_4 solution consumed.

42. Stability

42.1 Restandardize monthly.

43. Precision and Bias

43.1 The following criteria should be used for judging the acceptability of results:

43.1.1 *Repeatability (Single Analyst)*—See 38.1.1.

43.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—See 38.1.2.

43.1.3 *Reproducibility (Multilaboratory)*—See 38.1.3.

SILVER NITRATE SOLUTION, 0.1 *N*

44. Preparation

44.1 Dry 17.5 g of silver nitrate ($AgNO_3$) at 105°C for 1 h. Cool in a desiccator. Transfer 16.99 g of the dried $AgNO_3$ to a 1-L volumetric flask. Add 500 mL of water, swirl to dissolve the $AgNO_3$, dilute to the mark with water, and mix. Store the solution in a tightly stoppered amber-glass bottle.

NOTE 12—If desired the solution may also be prepared on a determinate basis by weighing the dried silver nitrate accurately and diluting the solution carefully to volume.

45. Standardization

45.1 **Warning**—Nitrobenzene, used in this section, is extremely hazardous when absorbed through the skin or when its vapor is inhaled. Such exposure may cause cyanosis; prolonged exposure may cause anemia. Do not get in eyes, on skin, or on clothing. Avoid breathing vapor. Use only with adequate ventilation.

45.2 Dry 0.3 g of sodium chloride (NaCl) at 105°C for 2 h. Cool in a desiccator. Weigh accurately 0.28 ± 0.01 g of the

dried NaCl and transfer to a 250-mL glass-stoppered conical flask. Add 25 mL of water, swirl to dissolve the NaCl, and add 2 mL of nitric acid (HNO_3). Add from a volumetric pipet, 50 mL of the $AgNO_3$ solution, while mixing thoroughly, add 1 mL of ferric ammonium sulfate solution ($FeNH_4(SO_4)_2 \cdot 12H_2O$, 80 g/L) and 5 mL of nitrobenzene (**Warning**, see 45.1). Stopper the flask and shake vigorously to coagulate the precipitate. Rinse the stopper into the flask with a few millilitres of water and titrate the excess of $AgNO_3$ with ammonium thiocyanate solution (NH_4SCN) until the first permanent reddish-brown color appears and persists after vigorous shaking for 1 min. See 49.1 for preparation of ammonium thiocyanate. Solution does not need to be standardized for use here. (See Note 13.) Designate the volume of NH_4SCN solution required for the titration as Volume I.

45.3 Using the same volumetric pipet used in 45.2, transfer 50 mL of the $AgNO_3$ solution to a clean, dry, 250-mL, glass-stoppered conical flask. Add 25 mL of water, 2 mL of HNO_3, 1 mL of $FeNH_4(SO_4)_2 \cdot 12H_2O$ solution, stopper the flask, and shake vigorously. Rinse the stopper into the flask with a few millilitres of water and titrate the $AgNO_3$ solution with NH_4SCN solution until the first permanent reddish-brown color appears and persists after vigorous shaking for 1 min. Designate the volume of NH_4SCN solution consumed as Volume II.

45.4 Measure accurately, from either a buret or a volumetric pipet, 2.0 mL of the $AgNO_3$ solution, designate the exact volume as Volume III, and transfer to a 100-mL, glass-stoppered conical flask. Add 25 mL of water, 2 mL of HNO_3, 1 mL of $FeNH_4(SO_4)_2 \cdot 12H_2O$ solution, and 5 mL of nitrobenzene, (**Warning**, see 45.1) stopper the flask, and shake vigorously. Rinse the stopper into the flask with a few millilitres of water and titrate the $AgNO_3$ solution with NH_4SCN solution until the first permanent reddish-brown color appears and persists after vigorous shaking for 1 min. Designate the volume of NH_4SCN solution consumed as Volume IV.

NOTE 13—The ammonium thiocyanate titrant used in the three titrations must be from the same, well-mixed solution. The nitrobenzene used in each titration must also be from the same, well-mixed container.

46. Calculation

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

$$A = \frac{B}{0.05844 \times (C - D)} \quad (8)$$

where:

A = normality of the $AgNO_3$ solution,
B = grams of NaCl used,
C = volume of $AgNO_3$ solution consumed by the total chloride = 50 − [Volume I × (50/Volume II)], and
D = volume of $AgNO_3$ solution consumed by any chloride ion in the nitrobenzene = Volume III − [Volume IV × (50/Volume II)].

47. Stability

47.1 Restandardize monthly.

48. Precision and Bias (See Note 14)

48.1 The following criteria should be used for judging the acceptability of results:

48.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00010 normality units at 34 df. The 95 % limit for the difference between two such determinations is 0.0003 normality units.

48.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00017 normality units at 17 df. The 95 % limit for the difference between two such averages is 0.0005 normality units.

48.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be 0.00035 normality units at 16 df. The 95 % limit for the difference between two such averages is 0.0010 normality units.

NOTE 14—These precision estimates are based on an interlaboratory study conducted in 1963. One sample was analyzed. One analyst in each of 19 laboratories performed duplicate determinations and repeated them on a second day, for a total of 76 determinations. Practice E 180 was used in developing these statements.

AMMONIUM THIOCYANATE SOLUTION, 0.1 *N*

49. Preparation

49.1 Transfer 7.8 g of ammonium thiocyanate (NH_4SCN) to a flask, add 100 mL of water, and swirl to dissolve the NH_4SCN. When solution is complete, filter through a hardened filter paper, or other suitable medium. Dilute the clear filtrate to 1 L with water and mix. Store the solution in a tightly stoppered glass bottle.

50. Standardization

50.1 Measure accurately about 40 mL of freshly standardized 0.1 *N* silver nitrate ($AgNO_3$) solution and transfer to a 250-mL conical flask. Add 50 mL of water, swirl to mix the solution, and add 2 mL of nitric acid (HNO_3) and 1 mL ferric ammonium sulfate solution ($FeNH_4(SO_4)_2 \cdot 12H_2O$, 80 g/L). Titrate the $AgNO_3$ solution with the NH_4SCN solution until the first permanent reddish-brown color appears and persists after vigorous shaking for 1 min.

51. Calculation

51.1 Calculate the normality of the NH_4SCN solution, as follows:

$$A = \frac{B \times C}{D} \quad (9)$$

where:

A = normality of the NH_4SCN solution,
B = millilitres of $AgNO_3$ used,
C = normality of the $AgNO_3$ solution, and
D = millilitres of NH_4SCN solution required for titration of the solution.

52. Stability

52.1 Restandardize monthly.

53. Precision and Bias (See Note 14)

53.1 The following criteria should be used for judging the acceptability of results:

53.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00010 normality units at 38 df. The 95 % limit for the difference between two such determinations is 0.00028 normality units.

53.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00035 normality units at 19 df. The 95 % limit for the difference between two such averages is 0.00099 normality units.

53.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be 0.00046 normality units at 18 df. The 95 % limit for the difference between two such averages is 0.00130 normality units.

IODINE SOLUTION, 0.1 *N*

54. Preparation

54.1 Transfer 12.7 g of iodine and 60 g of potassium iodide (KI) to an 800-mL beaker, add 30 mL of water, and stir until solution is complete. Dilute with water to 500 mL, and filter through a sintered-glass filter. Wash the filter with about 15 mL of water, transfer the combined filtrate and washing to a 1-L volumetric flask, dilute to the mark with water, and mix. Store the solution in a glass-stoppered, amber-glass bottle in a cool place.

55. Standardization

55.1 **Warning**—Arsenic trioxide is extremely toxic, avoid ingestion.

55.2 Transfer 1 g of primary standard arsenic trioxide[9] (As_2O_3) (**Warning**, see 55.1) to a platinum dish, and dry at 105°C for 1 h. Cool in a desiccator. Weigh accurately 0.20 ± 0.01 g of the dried As_2O_3 and transfer to a 500-mL conical flask. Add 10 mL of sodium hydroxide solution (NaOH, 40 g/L), and swirl to dissolve. When solution is complete, add 100 mL of water and 10 mL of sulfuric acid (H_2SO_4, 1 + 35), and mix. Slowly add sodium bicarbonate ($NaHCO_3$) until effervescence ceases, add 2 g of $NaHCO_3$ in excess, and stir until dissolved. Add 2 mL of starch solution (10 g/L) and titrate with the iodine solution to the first permanent blue color.

56. Calculation

56.1 Calculate the normality of the iodine solution, as follows:

$$A = \frac{B}{0.04946 \times C} \quad (10)$$

where:

A = normality of the iodine solution,
B = grams of As_2O_3 used, and
C = millilitres of iodine solution required for titration of the solution.

57. Stability

57.1 Restandardize sealed bottles monthly.

57.2 Restandardize open bottles weekly.

58. Precision and Bias (See Note 15)

58.1 The following criteria should be used for judging the acceptability of results:

58.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00015 normality units at 32 df. The 95 % limit for the difference between two such averages is 0.0004 normality units.

58.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00016 normality units at 16 df. The 95 % limit for the difference between two such averages is 0.0004 normality units.

58.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be 0.00021 normality units at 15 df. The 95 % limit for the difference between two such averages is 0.0006 normality units.

NOTE 15—These precision estimates are based on an interlaboratory study conducted in 1962. One sample was analyzed. One analyst in each of 16 laboratories performed duplicate determinations and repeated them on a second day, for a total of 64 determinations. Practice E 180 was used in developing these statements.

SODIUM THIOSULFATE SOLUTION, 0.1 *N*

59. Preparation

59.1 Dissolve 25 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) in 500 mL of freshly boiled and cooled water, and add 0.11 g of sodium carbonate (Na_2CO_3). Dilute to 1 L with freshly boiled and cooled water, and let stand for 24 h. Store the solution in a tightly closed glass bottle.

60. Standardization

60.1 Pulverize 2 g of primary standard potassium dichromate[9] ($K_2Cr_2O_7$), transfer to a platinum dish, and dry at 120°C for 4 h. Cool in a desiccator. Weigh accurately 0.21 ± 0.01 g of the dried $K_2Cr_2O_7$, and transfer to a 500-mL glass-stoppered conical flask. Add 100 mL of water, swirl to dissolve, remove the stopper, and quickly add 3 g of potassium iodide (KI), 2 g of sodium bicarbonate ($NaHCO_3$), and 5 mL of hydrochloric acid (HCl). Stopper the flask quickly, swirl to ensure mixing, and let stand in the dark for 10 min. Rinse the stopper and inner walls of the flask with water and titrate with the $Na_2S_2O_3$ solution until the solution is yellowish green. Add 2 mL of starch solution (10 g/L), and continue the titration to the disappearance of the blue color.

61. Calculation

61.1 Calculate the normality of the $Na_2S_2O_3$ solution, as follows:

$$A = \frac{B}{0.04904 \times C} \quad (11)$$

where:
A = normality of the $Na_2S_2O_3$ solution,
B = grams of $K_2Cr_2O_7$ used, and
C = millilitres of $Na_2S_2O_3$ solution required for titration of the solution.

62. Stability

62.1 Restandardize weekly.

63. Precision and Bias (See Note 16)

63.1 The following criteria should be used for judging the acceptability of results:

63.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00009 normality units at 32 df. The 95 % limit for the difference between two such determinations is 0.0003 normality units.

63.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00014 normality units at 16 df. The 95 % limit for the difference between two such averages is 0.0004 normality units.

63.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be 0.00024 normality units at 15 df. The 95 % limit for the difference between two such averages is 0.0007 normality units.

NOTE 16—These precision estimates are based on an interlaboratory study conducted in 1962. One sample was analyzed. One analyst in each of 16 laboratories performed duplicate determinations and repeated them on a second day, for a total of 64 determinations. Practice E 180 was used in developing these statements.

POTASSIUM PERMANGANATE SOLUTION, 0.1 *N*

64. Preparation

64.1 Dissolve 3.2 g of potassium permanganate ($KMnO_4$) in 100 mL of water and dilute the solution with water to 1 L. Allow the solution to stand in the dark for two weeks and then filter through a fine-porosity sintered-glass crucible. *Do not wash the filter*. Store the solution in glass-stoppered, amber-colored glass bottles.

NOTE 17—Do not permit the filtered solution to come into contact with paper, rubber, or other organic material.

65. Standardization [10]

65.1 Transfer 2 g of primary standard sodium oxalate[9] ($Na_2C_2O_4$) to a platinum dish and dry at 105°C for 1 h. Cool in a desiccator. Weigh accurately 0.30± 0.01 g of the dried $Na_2C_2O_4$ and transfer to a 500-mL glass container. Add 250 mL of sulfuric acid (H_2SO_4, 1 + 19) that was previously boiled for 10 to 15 min and then cooled to 27 ± 3°C, and stir until the sample is dissolved. Add 39 mL of the $KMnO_4$ solution at a rate of 30± 5 mL/min, while stirring slowly, and let stand for about 45 s until the pink color disappears. Heat the solution to 60°C, and complete the titration by adding $KMnO_4$ solution until a faint pink color persists for 30 s. Add the final 0.5 to 1.0 mL dropwise, and give the solution time to decolorize before adding the next drop.

65.2 Carry out a blank determination on a second 250-mL portion of the H_2SO_4(1 + 19), and make sure that the pink color at the end point matches that of the standardization solution. Correct the sample titration volume as shown to be necessary.

NOTE 18—If the pink color of the solution persists more than 45 s after the addition of the first 39 mL of $KMnO_4$ solution is complete, discard the solution and start over with a fresh solution of the $Na_2C_2O_4$, but add less of the $KMnO_4$ solution.

NOTE 19—The blank correction usually amounts to 0.03 to 0.05 mL.

66. Calculation

66.1 Calculate the normality of the $KMnO_4$ solution, as follows:

$$A = \frac{B}{0.06701\ (C - D)} \quad (12)$$

where:
A = normality of the $KMnO_4$ solution,
B = grams of $Na_2C_2O_4$ used,
C = millilitres of $KMnO_4$ solution required for titration of the solution, and
D = millilitres of $KMnO_4$ solution required for blank titration.

67. Stability

67.1 Restandardize weekly.

68. Precision and Bias (See Note 20)

68.1 The following criteria should be used for judging the acceptability of results:

68.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00009 normality units at 32 df. The 95 % limit for the difference between two such determinations is 0.0002 normality units.

68.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00009 normality units at 16 df. The 95 % limit for the difference between two such averages is 0.0002 normality units.

68.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be 0.00013 normality units at 15 df. The 95 % limit for the difference between two such averages is 0.0004 normality units.

NOTE 20—These precision estimates are based on an interlaboratory study conducted in which one sample was analyzed. One analyst in each of 18 laboratories performed duplicate determinations and repeated them on a second day, for a total of 72 determinations. Practice E 180 was used in developing these statements.

POTASSIUM DICHROMATE SOLUTION, 0.1 *N*

69. Preparation

69.1 Transfer 6 g of potassium dichromate ($K_2Cr_2O_7$) to a platinum dish and dry at 120°C for 4 h. Cool in a desiccator. Place 4.9 g of the dried $K_2Cr_2O_7$ in a 1-L volumetric flask, and add 100 mL of water. Swirl to dissolve and when solution is complete, dilute to the mark with water and mix. Store the solution in a glass-stoppered bottle.

NOTE 21—If desired, the solution also may be prepared on a determinate basis by accurately weighing dried primary standard potassium dichromate,[9] and diluting the solution carefully to volume.

70. Standardization

70.1 Place 40 mL of water in a 250-mL glass-stoppered conical flask, and add 40 mL, accurately measured, of the $K_2Cr_2O_7$ solution. Stopper the flask, swirl to mix, remove the stopper, and add 3 g of potassium iodide (KI), 2 g of sodium bicarbonate ($NaHCO_3$), and 5 mL of hydrochloric acid (HCl). Stopper the flask quickly, swirl to ensure mixing, and let stand in the dark for 10 min. Rinse the stopper and inner walls of the flask with water and titrate with freshly standardized sodium thiosulfate solution ($Na_2S_2O_3$) until the solution is yellowish green. Add 2 mL of starch solution (10 g/L), and continue the titration to the disappearance of the blue color.

71. Calculation

71.1 Calculate the normality of the $K_2Cr_2O_7$ solution, as follows:

$$A = \frac{B \times C}{D} \tag{13}$$

where:

A = normality of the $K_2Cr_2O_7$ solution,
B = millilitres of $Na_2S_2O_3$ solution required for titration of the solution,
C = normality of the $Na_2S_2O_3$ solution, and
D = millilitres of $K_2Cr_2O_7$ solution used.

72. Stability

72.1 Restandardize monthly.

73. Precision and Bias (See Note 22)

73.1 The following criteria should be used for judging the acceptability of results:

73.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00007 normality units at 28 df. The 95 % limit for the difference between two such determinations is 0.00019 normality units.

73.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00007 normality units at 14 df. The 95 % limit for the difference between two such averages is 0.00019 normality units.

73.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be 0.00009 normality units at 13 df. The 95 % limit for the difference between two such averages is 0.00026 normality units.

NOTE 22—These precision estimates are based on an interlaboratory study conducted in 1963. One sample was analyzed. One analyst in each of 18 laboratories performed duplicate determinations and repeated them on a second day, for a total of 72 determinations. Practice E 180 was used in developing these statements.

METHANOLIC SODIUM HYDROXIDE SOLUTION, 0.5 *N*

NOTE 23—Because of the magnitude of the temperature correction factor (ΔN/°C) for this normality, volume corrections should be utilized if this solution is used at temperatures differing from that of standardization (see 6.1 and Table 1).

74. Preparation

74.1 Dilute 28 mL of clear 50 % NaOH solution (see 14.1) with 71 mL of water, add 90 mL of absolute methanol, and mix thoroughly in a hard glass container having a vented closure. Store the solution in a light-resistant hard glass bottle fitted with a delivery tube and a guard tube containing a carbon dioxide absorbent.

NOTE 24—Mixing of the solution may be accompanied by pressure build-up and should be done with a vented system.

75. Standardization

75.1 See Section 15, but use 4.75 ± 0.05 g of the dried $KHC_8H_4O_4$.

76. pH 8.6 End Point Color Standard

76.1 See Section 16.

77. Calculation

77.1 See Section 17.

78. Stability

78.1 The solution must be standardized frequently if there is evidence of action on the glass container, or if insoluble matter appears in the solution.

79. Precision and Bias (See Note 25)

79.1 The following criteria should be used for judging the acceptability of results:

79.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0004 normality units at 30 df. The 95 % limit for the difference between two such determinations is 0.0010 normality units.

79.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00061 normality units at 15 df. The 95 % limit for the difference between two such averages is 0.0017 normality units.

79.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be

0.00132 normality units at 8 df. The 95 % limit for the difference between two such averages is 0.0037 normality units.

NOTE 25—These precision estimates are based on an interlaboratory study in which one sample was analyzed. Two analysts in each of 9 laboratories performed duplicate determinations and repeated them on a second day, for a total of 72 determinations. Practice E 180 was used in developing these statements.

CERIC SULFATE SOLUTION, 0.1 *N* (in 1 *N* H_2SO_4)

80. Preparation

80.1 **Warning**—The addition of water to the sulfuric acid slurry in this procedure should be carried out slowly and cautiously, as the resulting solution becomes very hot and may spatter. Wear a face-shield during the operation.

80.2 To 60 g of ceric ammonium nitrate [$(NH_4)_2 \cdot Ce(NO_3)_6$] add 30 mL of concentrated sulfuric acid (H_2SO_4 sp gr 1.84), and stir until a smooth slurry is formed. Add, cautiously and with constant stirring, 100 mL of water (**Warning**, see 80.1) and stir for 2 min when addition is complete. Add 600 mL of additional water in three portions, adding the water slowly, and stirring for 2 min after each 200 mL is added. Dilute with water to 900 mL, cool, filter through a glass microfiber filter, and let the filtrate stand undisturbed in a tightly-closed glass container for 2 or 3 days. If the solution is clear after standing, dilute it with water to 1 L. Store the solution in tightly-closed glass containers. If any insoluble matter precipitates during the standing, filter as above and dilute the filtrate with water to 1 L.

81. Standardization

81.1 **Warning**—The preparation of the osmium tetroxide solution used in this procedure should be carried out in a well-ventilated hood because of the poisonous and irritating vapors given off by this compound.

81.2 Transfer about 1 g of primary standard arsenic trioxide[9] (As_2O_3) (**Warning**, see 55.1) to a platinum dish, and dry at 105°C for 1 h. Cool in a desiccator. Weigh accurately 0.20 ± 0.01 g of the dried As_2O_3 and transfer to a 500-mL conical flask. Rinse the walls of the flask with 25 mL of water containing 2 g of sodium hydroxide (NaOH), and swirl to dissolve. When solution is complete, dilute with 100 mL of water, add 10 mL of sulfuric acid (H_2SO_4, 1 + 1), 3 drops of a solution of 0.01 *M* osmium tetroxide (OsO_4) (**Warning**, see 81.1) in 0.1 *N* H_2SO_4 (see 96.20), and 2 drops of 0.025 *M*, 1,10-phenanthroline ferrous sulfate indicator solution (see 96.21). Titrate slowly with the ceric sulfate solution to the sharp color change from pink to very pale blue.

82. Calculation

82.1 Calculate the normality of the ceric sulfate solution, as follows:

$$A = \frac{B}{0.04946 \times C} \tag{14}$$

where:

A = normality of the ceric sulfate solution,
B = grams of As_2O_3 used, and
C = millilitres of ceric sulfate solution required for titration of the solution.

83. Stability

83.1 Restandardize monthly.

84. Precision and Bias (See Note 26)

84.1 The following criteria should be used for judging the acceptability of results:

84.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00013 normality units at 36 df. The 95 % limit for the difference between two such determinations is 0.0004 normality units.

84.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00012 normality units at 18 df. The 95 % limit for the difference between two such averages is 0.0004 normality units.

84.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be 0.00021 normality units at 17 df. The 95 % limit for the difference between two such averages is 0.0006 normality units.

NOTE 26—These precision estimates are based on an interlaboratory study conducted in 1965. One sample was analyzed. One analyst in each of 18 laboratories performed duplicate determinations and repeated them on a second day, for a total of 72 determinations. Practice E 180 was used in developing these statements.

ACETOUS PERCHLORIC ACID, 0.1 *N*

85. Preparation

85.1 **Warning**—Use chemical safety goggles and long-sleeved rubber gloves in the preparation of this solution. Perchloric acid in contact with certain organic materials can form explosive mixtures. All glassware that has been in contact with perchloric acid and its solutions should be rinsed with water before being set aside.

85.2 To approximately 500 mL of glacial acetic acid (CH_3COOH, sp gr 1.05) in a 1-L volumetric flask, add 8.5 mL of 70 % perchloric acid ($HClO_4$, sp gr 1.67) and mix the solution by swirling. Dilute to volume with glacial acetic acid and mix again.

85.3 Add 25.0 mL of the acetous perchloric acid solution to a flask containing 25.0 mL of pyridine and determine the percent water (w/w) by titration with Karl Fischer reagent (Test Method E 203). Make any necessary blank correction after titrating a separate 25.0-mL portion of pyridine with the Karl Fischer reagent.

85.4 Calculate the amount of acetic anhydride ($C_4H_6O_3$) required to react with all except 0.035 % (w/w) of the water in the acetous perchloric acid solution. The following formula, based on acetic anhydride having a specific gravity of 1.08 and an assay of 100 % may be used:

$$A = 52.5 \times B \tag{15}$$

where:
A = millilitres of acetic anhydride to be added to 1000 mL of the acetous perchloric acid, and
B = percent (w/w) of water in the acetous perchloric acid.

85.5 Add with constant stirring, the calculated amount of acetic anhydride ($C_4H_6O_3$) in successive small portions to the acetous perchloric acid. Cool, mix the solution thoroughly and determine the water content with Karl Fischer reagent as described in 85.3. If the water content exceeds 0.05 %[12] add more acetic anhydride, but if the solution contains less than 0.02 % water, add sufficient water to make the content between 0.02 and 0.05 % of water. Mix the solution thoroughly, and again determine the water content by titration. When the water content of the solution is between 0.02 and 0.05 %, standardize the solution by the following procedure and protect it from atmospheric moisture by a guard tube containing silica gel.

86. Standardization

86.1 Weigh accurately about 0.7 g of potassium hydrogen phthalate[9] ($KHC_8H_4O_4$), previously dried at 105°C for 3 h, and dissolve it in 50 mL of glacial CH_3COOH in a 250-mL flask. Add 2 drops of crystal violet indicator solution (10 g/L in glacial acetic acid), titrate with the $HClO_4$ solution until the violet color changes to emerald-green. Determine the volume of $HClO_4$ solution consumed by a blank using 50 mL of the glacial CH_3COOH.

87. Calculation

87.1 Calculate the normality of the $HClO_4$ solution, as follows:

$$A = \frac{B}{0.2042 \times (C - D)} \quad (16)$$

where:
A = normality of the $HClO_4$ solution,
B = grams of $KHC_8H_4O_4$ used,
C = millilitres of $HClO_4$ solution consumed by the $KHC_8H_4O_4$ solution, and
D = millilitres of $HClO_4$ solution consumed by 50 mL of glacial CH_3COOH.

88. Stability

88.1 Restandardize monthly.

89. Precision and Bias (See Note 27)

89.1 The following criteria should be used for judging the acceptability of results:

89.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00008 normality units at 34 df. The 95 % limit for the difference between two such determinations is 0.0002 normality units.

89.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00008 normality units at 17 df. The 95 % limit for the difference between two such averages is 0.0002 normality units.

89.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be 0.00016 normality units at 16 df. The 95 % limit for the difference between two such averages is 0.0005 normality units.

NOTE 27—These precision estimates are based on an interlaboratory study conducted in 1966. One sample was analyzed. One analyst in each of 20 laboratories performed duplicate determinations and repeated them on a second day, for a total of 80 determinations. Practice E 180 was used in developing these statements.

DISODIUM ETHYLENEDIAMINE TETRAACETATE SOLUTION, 0.05 *M*

90. Preparation

90.1 Dissolve 18.6 g of disodium ethylenediaminetetraacetate dihydrate ($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$)[13] in sufficient water to make 1000 mL, and store in a polyethylene container.

91. Standardization

91.1 Weigh accurately about 200 mg of calcium carbonate[14] transfer to a 400-mL beaker, add 10 mL of water, and swirl to form a slurry. Cover the beaker with a water glass and introduce 2 mL of hydrochloric acid (HCl, 3 + 10) from a pipet inserted between the lip of the beaker and edge of the watch glass. Swirl contents of the beaker to dissolve the calcium carbonate. Wash down the sides of the beaker, the outer surface of the pipet, and the watch glass, and dilute to about 100 mL. While stirring the solution, preferably with a magnetic stirrer, add about 30 mL of the disodium ethylenediaminetetraacetate solution from a 50-mL buret. Add 15 mL of sodium hydroxide solution (NaOH, 40 g/L), 300 mg of hydroxy naphthol blue indicator, and continue the titration with the disodium ethylenediaminetetraacetate solution to a blue endpoint.

92. Calculation

92.1 Calculate the molarity of the disodium ethylenediaminetetraacetate solution as follows:

$$M = \frac{W}{100.09\ V} \quad (17)$$

where:
M = molarity of the disodium ethylenediaminetetraacetate solution,
W = milligrams of $CaCO_3$ in the sample of calcium carbonate taken, and
V = millilitres of disodium ethylenediaminetetraacetate solution consumed.

[12] When the reagent is used for the titration of strong bases, higher contents are permissible.

[13] Correct American Chemical Society name: (Ethylenedinitrilo) Tetraacetic Acid Disodium Salt Dihydrate.

[14] Use chelometric standard grade of calcium carbonate ($CaCO_3$).

93. Stability

93.1 Restandardize monthly.

94. Precision and Bias (See Note 28)

94.1 The following criteria should be used for judging the acceptability of results:

94.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.00004 molarity units at 40 df. The 95 % limit for the difference between two such determinations is 0.0001 molarity units.

94.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability), formerly called Repeatability*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days, has been estimated to be 0.00006 molarity units at 20 df. The 95 % limit for the difference between two such averages is 0.0002 molarity units.

94.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories has been estimated to be 0.00006 molarity units at 19 df. The 95 % limit for the difference between two such averages is 0.0002 molarity units.

NOTE 28—These precision estimates are based on an interlaboratory study conducted in 1966. One sample was analyzed. One analyst in each of 20 laboratories performed duplicate determinations and repeated them on a second day, for a total of 80 determinations. Practice E 180 was used in developing these statements.

REAGENT TESTING SOLUTIONS

95. Standard Ion Solutions

95.1 *Arsenic, Standard Solution* (1 mL = 0.001 mg As)—Dissolve 0.1320 g of arsenic trioxide (As_2O_3) (**Warning**, see 55.1) in 10 mL of sodium hydroxide solution (NaOH, 40 g/L), neutralize with sulfuric acid (H_2SO_4, 1 + 15), add 10 mL of the acid in excess, and dilute with water to 1 L. To 10 mL of this solution (1 mL = 0.1 mg As) add 10 mL of H_2SO_4 (1 + 15), and dilute with water to 1 L.

95.2 *Chloride, Standard Solution* (1 mL = 0.005 mg Cl^-)—Dissolve 0.1650 g of sodium chloride (NaCl) in water, and dilute to 1 L. Dilute 5 mL of this solution to 100 mL.

95.3 *Iron, Standard Solution* (1 mL = 0.01 mg Fe)—Dissolve 0.1000 g of iron in 10 mL of hydrochloric acid (HCl, 1 + 1) and 1 mL of bromine water. Boil until the excess bromine is removed. Add 200 mL of HCl, cool, and dilute to 1 L in a volumetric flask. Dilute 100 mL of this solution to 1 L.

95.3.1 *Alternative Method*—As an alternative, the standard iron solution may be prepared by weighing exactly 0.7022 g of ferrous ammonium sulfate hexahydrate ($FeSO_4·(NH_4)_2SO_4·6H_2O$, minimum purity, 99.5 %), dissolving in 500 mL of water containing 20 mL of sulfuric acid (H_2SO_4, sp gr 1.84) and diluting to 1 L with water. Dilute 100 mL of this solution to 1 L.

95.4 *Lead, Standard Solution* (1 mL = 0.01 mg Pb)—Dissolve 0.160 g of lead nitrate ($Pb(NO_3)_2$) in 100 mL of nitric acid (HNO_3, 1 + 99), and dilute to 1 L. Dilute 10 mL of this solution with HNO_3 (1 + 99) to 100 mL. Prepare the dilute solution immediately before use.

95.5 *Mercury, Standard Solution* (1 mL = 0.05 mg Hg)—Dissolve 1.35 g of mercuric chloride ($HgCl_2$) in water, add 8 mL of HCl, and dilute to 1 L. To 50 mL of this solution (1 mL = 1 mg Hg) add 8 mL of HCl, and dilute to 1 L. **Warning**—Mercuric chloride is very toxic if swallowed.

95.6 *Sulfate, Standard Solution* (1 mL = 0.01 mg SO_4^{--})—Dissolve 0.148 g of anhydrous sodium sulfate (Na_2SO_4) in water, and dilute to 100 mL. Dilute 10 mL of this solution to 1 L.

96. Nonstandardized Reagent Solutions and Indicator Solutions

96.1 *Acetic Acid Solution* (1 + 19)—Dilute 50 mL of glacial acetic acid with 950 mL of water, and mix.

96.2 *Ammonium Acetate Solution* (100 g/L)—Dissolve 100 g of ammonium acetate (CH_3COONH_4) in about 750 mL of water, filter, and dilute to 1 L.

96.3 *Ammonium Acetate—Acetic Acid Solution*—Dissolve 100 g of ammonium acetate (CH_3COONH_4) in about 600 mL of water, filter, add 200 mL of glacial acetic acid to the filtrate, and dilute to 1 L with water.

96.4 *Ammonium Hydroxide Solution* (1 + 1)—Dilute 500 mL of ammonium hydroxide (NH_4OH) with 500 mL of water, and mix.

96.5 *Ammonium Molybdate—Sulfuric Acid Solution* (50 g $(NH_4)_6\ Mo_7O_{24}·4H_2O$/L)—Transfer 50 g of ammonium molybdate tetrahydrate to a 1 L flask, add 800 mL of 1 *N* H_2SO_4 (see Section 39), shake to dissolve the salt, and dilute with 1 *N* H_2SO_4 to 1 L.

96.6 *Ammonium Thiocyanate Solution* (300 g/L)—Dissolve 300 g of ammonium thiocyanate (NH_4SCN) in about 750 mL of water, filter, and dilute to 1 L.

96.7 *Barium Chloride Solution* (120 g $BaCl_2·2H_2O$/L)—Dissolve 120 g of barium chloride dihydrate in about 750 mL of water, filter, and dilute to 1 L.

96.8 *Bromine Water (Saturated)*—To 1 L of water in a glass-stoppered bottle add bromine and shake until no more bromine is dissolved by the solution. Start with 10 mL of bromine. Keep a few drops of bromine on the bottom of the bottle, and use only the clear water solution.

96.9 *Crystal Violet Indicator Solution* (10 g/L)—Dissolve 1 g of crystal violet (hexamethyl-*p*-rosaniline chloride) in 100 mL of glacial acetic acid (CH_3COOH, sp gr 1.05) and filter if necessary.

96.10 *Ferric Ammonium Sulfate Indicator Solution* (80 g $FeNH_4(SO_4)_2·12H_2O$/L)—Dissolve 80 g of clear crystals of ferric ammonium sulfate dodecahydrate in about 750 mL of water, filter, add a few drops of sulfuric acid (H_2SO_4), if necessary, to clear the solution, and dilute to 1 L.

96.11 *Hydrogen Sulfide Solution (Saturated)*—Saturate water with hydrogen sulfide gas by bubbling the gas through the water. The solution must be freshly prepared.

96.12 *Hydroxylamine Hydrochloride Solution* (100 g/L)—Dissolve 100 g of hydroxylamine hydrochloride ($NH_2OH·HCl$) in about 600 mL of water, filter, and dilute to 1 L.

96.13 *Hydroxylamine Hydrochloride Solution* (300 g/L)—Dissolve 300 g hydroxylamine hydrochloride (NH_2–$OH·HCl$) in about 600 mL of water, filter, and dilute to 1 L.

96.14 *Mercuric Acetate Solution* (25 g/L)—Dissolve 25 g of mercuric acetate ($Hg(CH_3COO)_2$) in about 500 mL of water,

filter, and dilute to 1 L. (**Warning**—Mercuric acetate is very toxic if swallowed.)

96.15 *Mercuric Chloride Solution* (50 g/L)—Dissolve 50 g of mercuric chloride ($HgCl_2$) in about 750 mL of water, filter, and dilute to 1 L. (**Warning**—Mercuric chloride is very toxic if swallowed.)

96.16 *Methyl Orange Indicator Solution* (1 g/L)—Dissolve 0.1 g of methyl orange in 100 mL of water and filter if necessary.

96.17 *Methyl Orange Indicator Solution, Modified* (1 g/L)—Dissolve 0.1 g of methyl orange and 0.14 g of xylene cyanole FF dye in 100 mL of water and filter if necessary.

96.18 *Methyl Red Indicator Solution* (1 g/L)—Dissolve 1 g of methyl red in 1 L of ethanol (95 %).

NOTE 29—In most cases certain denatured alcohols such as specially denatured Formula Nos. 3A, 30, or 2B may be substituted for ethanol.

96.19 *Methyl Red Indicator Solution* (5 g/L)—Dissolve 5 g of methyl red in 1 L of ethanol (95 %) (Note 29).

96.20 *Osmium Tetroxide Solution* (0.01 *M*) (in 0.1 *N* H_2SO_4)—Dissolve 0.25 g of osmium tetroxide (OsO_4) in 100 mL of 0.1 *N* H_2SO_4 (see 29.1). (**Warning**—The preparation of the osmium tetroxide solution should be carried out in a well-ventilated hood because of the poisonous and irritating vapors given off by this compound.)

96.21 *1,10-Phenanthroline (o-Phenanthroline) Ferrous Sulfate Indicator Solution (0.025 M)*—Dissolve 1.485 g of 1,10-phenanthroline monohydrate in 100 mL of 0.025 *M* ferrous sulfate solution. The 0.025 *M* ferrous sulfate solution is prepared by dissolving 0.695 g of $FeSO_4 \cdot 7H_2O$ in 100 mL of water.

96.22 *1,10-Phenanthroline (o-Phenanthroline) Indicator Solution (3 g/L)*—Dissolve 3 g of 1,10-phenanthroline monohydrate in 500 mL of water, add 1 mL of hydrochloric acid (HCl), mix, filter, and dilute to 1 L.

96.23 *Phenolphthalein Indicator Solution* (10 g/L)—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %) (see Note 29), methanol, or isopropanol.

96.24 *Phenolphthalein Indicator Solution in Pyridine* (10 g/L)—Dissolve 1 g of phenolphthalein in pyridine and dilute with pyridine to 100 mL.

96.25 *Potassium Iodide Solution* (100 g/L)—Dissolve 100 g of potassium iodide (KI) in about 750 mL of water, filter, and dilute to 1 L.

96.26 *Potassium Iodide Solution* (300 g/L)—Dissolve 300 g of potassium iodide (KI) in about 750 mL of water, filter, and dilute to 1 L.

96.27 *Silver Nitrate Solution* (17 g/L)—Dissolve 17 g of silver nitrate ($AgNO_3$) in water, mix, dilute to 1 L, and store in a light-resistant glass container.

96.28 *Sodium Diethyldithiocarbamate Solution* (1 g/L)—Dissolve 1 g of sodium diethyldithiocarbamate in 750 mL of water, filter if necessary, and dilute to 1 L.

96.29 *Sodium Hydroxide Solution* (4 g/L)—Dissolve 4 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

96.30 *Sodium Hydroxide Solution* (40 g/L)—Dissolve 40 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

96.31 *Stannous Chloride Solution* (20 $SnCl_2 \cdot 2H_2O$/L)—Dissolve 20 g of stannous chloride dihydrate in 500 mL of hydrochloric acid (HCl), filter, if necessary, through a sintered-glass filter, and dilute with HCl to 1 L.

96.32 *Starch Indicator Solution* (10 g/L)—Mix 1 g of soluble starch with 5 mg of red mercuric iodide (HgI_2) and enough cold water to make a thin paste, and pour slowly, with constant stirring, into 100 mL of boiling water. Boil the mixture while stirring until a thin, translucent fluid is obtained. Cool before use. (**Warning**—Mercuric iodide is very toxic if swallowed.)

97. Keywords

97.1 indicator solutions; reagent solutions; standard solutions; titrants