Designation: E200 – 23

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

This standard is issued under the fixed designation E200; 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 U.S. Department of Defense.

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 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given throughout this practice. Consult current OSHA regulations, suppliers' Safety Data Sheets, and local regulations for all materials used in this specification.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E203 Test Method for Water Using Volumetric Karl Fischer Titration

E694 Specification for Laboratory Glass Volumetric Apparatus

2.2 *Other Document:*[4]

Reagent Chemicals American Chemical Society Specifications (ACS)

3. Terminology

3.1 *Definitions:*

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[1] This practice is under the jurisdiction of ASTM Committee D16 on Aromatic, Industrial, Specialty and Related Chemicals and is the direct responsibility of Subcommittee D16.04 on Instrumental Analysis.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from American Chemical Society (ACS), 1155 Sixteenth St., NW, Washington, DC 20036, http://www.acs.org.

***A Summary of Changes section appears at the end of this standard**

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 meq/mL (*N*) normality or mol/L (*M*) 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.

NOTE 1—For dependable accuracy, volumetric glassware meeting the requirements for Class A items given in Specification E694 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} = meq/mL (N) of solution when standardized,
N_{t2} = meq/mL (N) 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 meq/mL (*N*) 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.

TABLE 1 Temperature Correction Factors (F)

Approximate meq/mL (*N*)	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

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.

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 recommended to express the concentration of the solutions as the normality in the equivalent SI units as meq/mL (*N*) and molarity as mol/L (*M*).

8. Reagents

NOTE 3—Additional information on reagents is given in Practices E50.

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.[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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type I or Type II reagent water conforming to Specification D1193. Where specified, carbon dioxide-free water is to be prepared by heating Type I

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[5] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

or Type II 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 L 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*—Use either independently prepared or commercially available certified reference materials.

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 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 L 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 standardization concentration 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[6]

13.1 *Precision*—Precision for standardizing the volumetric solutions in this practice was determined in accordance with Practice E180 – 90 and the forms of the statements conform with that suggested in Practice E180 – 90.

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.

STANDARD VOLUMETRIC SOLUTIONS
SODIUM HYDROXIDE SOLUTION, 0.02 TO 1.0 meq/mL *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 meq/mL (*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.

TABLE 2 Sodium Hydroxide Dilution Requirements

Desired meq/mL (*N*)	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

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E15-1039. Contact ASTM Customer Service at service@astm.org.

15. Standardization

15.1 Crush 10 g to 20 g of primary standard potassium hydrogen phthalate[7] ($KHC_8H_4O_4$) 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 meq/mL (*N*) solution, weigh accurately 0.95 g ± 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.

TABLE 3 Weights of Dried Potassium Hydrogen Phthalate

meq/mL (*N*) 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.

16. pH 8.6 End Point Color Standard

16.1 Mix 25 mL of a solution 0.2 mol/L (*M*) in boric acid (H_3BO_3) and 0.2 mol/L (*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 meq/mL (*N*) of the NaOH solution, as follows:

$$A = \frac{B}{0.20423 \times C} \tag{2}$$

where:

A = meq/mL (*N*) 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 meq/mL (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 meq/mL (*N*) normality units at 36 df. The 95 % limit for the difference between two such determinations is 0.0020 meq/mL (*N*) normality units.

19.1.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—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 meq/mL (*N*) normality units at 18 df. The 95 % limit for the difference between two such averages is 0.0020 meq/mL (*N*) 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 meq/mL (*N*) normality units at 17 df. The 95 % limit for the difference between two such averages is 0.0028 meq/mL (*N*) normality units.

19.1.2 *Sodium Hydroxide* (0.1 meql/mL (*N*)):

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

19.1.2.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—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 meq/mL (*N*) normality units at 14 df. The 95 % limit for the difference between two such averages is 0.0003 meq/mL (*N*) 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 meq/mL (*N*) at 13 df. The 95 % limit for the difference between two such averages is 0.0005 meq/mL (*N*) 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 E180 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 E180 was used in developing these statements.

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[7] 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.

HYDROCHLORIC ACID,
0.02 to 1.0 meq/mL (*N*)

20. Preparation

20.1 To prepare a 0.1 meq/mL (*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.

TABLE 4 Hydrochloric Acid Dilution Requirements

Desired meq/mL (*N*)	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

21. Standardization with Sodium Carbonate [8]

21.1 Transfer 2 g to 4 g of primary standard anhydrous sodium carbonate (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 meq/mL (*N*) solution, weigh accurately 0.22 g ± 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.

22. Calculation

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

$$A = \frac{B}{0.053 \times C} \tag{3}$$

where:

A = meq/mL (*N*) normality of the HCl solution,
B = grams of Na_2CO_3 used, and
C = millilitres of HCl solution consumed.

TABLE 5 Weights of Dried Sodium Carbonate

meq/mL (*N*) 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.

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 meq/mL (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 meq/mL (*N*) normality units at 36 df. The 95 % limit for the difference between two such determinations is 0.0011 meq/mL (*N*) normality units.

24.1.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—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 meq/mL (*N*) normality units at 18 df. The 95 % limit for the difference between two such averages is 0.0017 meq/mL (*N*) 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 meq/mL (*N*) normality units at 17 df. The 95 % limit for the difference between two such averages is 0.0042 meq/mL (*N*) normality units.

24.1.2 *Hydrochloric Acid (0.1 meql/mL (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 meq/mL (*N*) normality units at 28 df. The 95 % limit for the difference between two such determinations is 0.0003 meq/mL (*N*) 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 meq/mL (*N*) normality units at 14 df. The 95 % limit for the difference between two such averages is 0.0002 meq/mL (*N*) 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

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

0.00017 meq/mL (*N*) normality units at 13 df. The 95 % limit for the difference between two such averages is 0.0005 meq/mL (*N*) normality units.

25. Standardization with Tris(hydroxymethyl)-Aminomethane

25.1 Transfer 8 g to 10 g of primary standard tris(hydroxymethyl)aminomethane[7] [$(HOCH_2)_3CNH_2$] 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 meq/mL (*N*) solution, weigh accurately 0.40 g ± 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 meq/mL (*N*) normality of the HCl solution, as follows:

$$A = \frac{B}{0.1211 \times C} \tag{4}$$

where:

A = meq/mL (*N*) 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 meq/mL (N)):*

28.1.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0006 meq/mL (*N*) normality units at 16 df. The 95 % limit for the difference between two such determinations is 0.0017 meq/mL (*N*) normality units.

28.1.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—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 meq/mL (*N*) normality units at 8 df. The 95 % limit for the difference between two such averages is 0.0020 meq/mL (*N*) normality units.

TABLE 6 Weights of Dried Tris(Hydroxymethyl)Aminomethane

meq/mL (*N*) 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.

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 meq/mL (*N*) normality units at 7 df. The 95 % limit for the difference between two such averages is 0.0042 meq/mL (*N*) normality units.

28.1.2 *Hydrochloric Acid (0.1 meq/mL (N):*

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

28.1.2.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—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 meq/mL (*N*) normality units at 8 df. The 95 % limit for the difference between two such averages is 0.0003 meq/mL (*N*) 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 meq/mL (*N*) normality units at 7 df. The 95 % limit for the difference between two such averages is 0.0007 meq/mL (*N*) normality units.

TABLE 7 Sulfuric Acid Dilution Requirements

Desired meq/mL (*N*)	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

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 E180 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 meq/mL (*N*)

29. Preparation

29.1 To prepare a 0.1 meq/mL (*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 meq/mL (*N*) normalities of the H_2SO_4 solution, use the requirements given in Table 7.

30. Standardization [8]

30.1 Transfer 2 g to 4 g of primary standard anhydrous sodium carbonate (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 meq/mL (*N*) solution, weigh accurately 0.22 g ± 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 meq/mL (*N*) normalities of H_2SO_4 solution are given in Table 5.

31. Calculation

31.1 Calculate the meq/mL (*N*) normality of the H_2SO_4 solution, as follows:

$$A = \frac{B}{0.053 \times C} \tag{5}$$

where:

A = meq/mL (*N*) 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 meq/mL (*N*) sulfuric acid may be standardized using dried tris(hydroxymethyl)aminomethane by the same procedure used to standardize 0.1 meq/mL (*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 meq/mL (N)):*

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

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

33.1.1.3 *Reproducibility (Multilaboratory)*—See 24.1.1.3.

33.1.2 *Sulfuric Acid (0.1 meq/mL (N)):*

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

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

33.1.2.3 *Reproducibility (Multilaboratory)*—See 24.1.2.3.

HYDROCHLORIC ACID, SPECIAL 1 meq/mL (*N*)

NOTE 10—This solution is not for general use but is designed to satisfy the special requirements of ASTM Committee E15, 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 (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 g ± 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 meq/mL (*N*) normality of the HCl solution, as follows:

$$A = \frac{B}{0.053 \times C} \tag{6}$$

where:

A = meq/mL (*N*) 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 meq/mL (*N*) normality units at 34 df. The 95 % limit for the difference between two such determinations is 0.0018 meq/mL (*N*) normality units.

38.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability)*—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 meq/mL (*N*) normality units at 18 df. The 95 % limit for the difference between two such averages is 0.0023 meq/mL (*N*) 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 meq/mL (*N*) normality units at 16 df. The 95 % limit for the difference between two such averages is 0.0062 meq/mL (*N*) 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 E180 was used in developing these statements.