



Standard Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis¹

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This standard has been approved for use by agencies of the 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:

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Sodium hydroxide solution, 0.02 to 1.0 meq/mL (M)	14 to 19
Hydrochloric acid, 0.02 to 1.0 meq/mL (M)	20 to 28
Sulfuric acid, 0.02 to 1.0 meq/mL (M)	29 to 33
Hydrochloric acid, special 1 meq/mL (M)	34 to 38
Sulfuric acid, special 1 meq/mL (M)	39 to 43
Silver nitrate solution, 0.1 meq/mL (M)	44 to 48
Ammonium thiocyanate solution, 0.1 meq/mL (M)	49 to 53
Iodine solution, 0.1 meq/mL (M)	54 to 58
Sodium thiosulfate solution, 0.1 meq/mL (M)	59 to 63
Potassium permanganate solution, 0.1 meq/mL (M)	64 to 68
Potassium dichromate solution, 0.1 meq/mL (M)	69 to 73
Methanolic sodium hydroxide solution, 0.5 meq/mL (M)	74 to 79
Ceric sulfate solution, 0.1 meq/mL (M)	80 to 84
Acetous perchloric acid, 0.1 meq/mL (M)	85 to 89
Disodium ethylenediaminetetraacetate solution, 0.05 mol/L (M)	90 to 94
Standard ion solutions	95
Nonstandardized reagent solutions and indicator solutions	96

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 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:²

D 1193 Specification for Reagent Water

E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals

E 203 Test Method for Water Using Volumetric Karl Fischer Titration

E 694 Specification for Laboratory Glass Volumetric Apparatus

2.2 Other Document:

Reagent Chemicals, American Chemical Society Specifications (ACS)³

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 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

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

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

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

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 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 ($\Delta N/^\circ\text{C}$) 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:

$$N_{i2} = N_{i1} + (t_1 - t_2)(F) \quad (1)$$

where:

- N_{i1} = meq/mL (N) of solution when standardized,
- N_{i2} = meq/mL (N) of solution when used,
- t_1 = temperature of solution during standardization, $^\circ\text{C}$
- t_2 = temperature of solution during use, $^\circ\text{C}$, and
- F = factor to correct for thermal expansion of the solution ($\Delta N/^\circ\text{C}$ 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	$\Delta N/^\circ\text{C}$ for 20 to 30 $^\circ\text{C}$
1.0	NaOH, HCl, H ₂ SO ₄	0.00035
0.5	NaOH, HCl, H ₂ SO 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 ₂ SO 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 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.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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.

⁴ For suggestions on the testing of reagents not listed by the American Chemical Society, see “Analytical Standards for Laboratory Chemicals,” BDH Ltd., Poole, Dorset, U.K., and the “United States Pharmacopeia.”

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 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

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 – 90.⁵

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.

15. Standardization

15.1 Crush 10 to 20 g of primary standard potassium hydrogen phthalate⁶ (KHC₈H₄O₄) 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 ± 0.05 g of the dried KHC₈H₄O₄, 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 KHC₈H₄O₄ suitable for other normalities of NaOH solution are given in Table 3.

TABLE 2 Sodium Hydroxide Dilution Requirements

Desired meq/mL (<i>M</i>)	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

16. pH 8.6 End Point Color Standard

16.1 Mix 25 mL of a solution 0.2 mol/L (*M*) in boric acid (H₃BO₃) and 0.2 mol/L (*M*) in potassium chloride (KCl), (1.24

⁵ Data supporting the precision statements are available from ASTM Headquarters. Request RR: E-15-1039.

⁶ 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.