water until a colorless solution is obtained. Dilute with water to 100 mL.

***p*-Toluenesulfonic Acid TS**—Dissolve 2 g of *p*-toluenesulfonic acid in 10 mL of a mixture of 7 parts of acetone and 3 parts of water.

Triketohydrindene Hydrate TS *(Ninhydrin TS)*—Dissolve 200 mg of triketohydrindene hydrate in water to make 10 mL. Prepare this solution fresh.

Trinitrophenol TS *(Picric Acid TS)*—Dissolve the equivalent of 1 g of anhydrous trinitrophenol in 100 mL of hot water. Cool the solution, and filter if necessary.

Triphenyltetrazolium Chloride TS—Dissolve 500 mg of triphenyltetrazolium chloride in dehydrated alcohol to make 100 mL.

Xylenol Orange TS—Dissolve 100 mg of xylenol orange in 100 mL of alcohol.

Zinc Uranyl Acetate TS—Dissolve 50 g of uranyl acetate in a mixture of 15 mL of glacial acetic acid and water to make 500 mL. Then dissolve 150 g of zinc acetate in a mixture of 15 mL of glacial acetic acid and water to make 500 mL. Mix the two solutions, allow to stand overnight, and pass through a dry filter, if necessary.

VOLUMETRIC SOLUTIONS

Normal Solutions—Normal solutions are solutions that contain 1 gram equivalent weight of the active substance in each 1000 mL of solution; that is, an amount equivalent to 1.0079 g of hydrogen or 7.9997 g of oxygen. Normal solutions and solutions bearing a specific relationship to normal solutions, and used in volumetric determinations, are designated as follows: normal, 1 N; double-normal, 2 N; half-normal, 0.5 N; tenth-normal, 0.1 N; fiftieth-normal, 0.02 N; hundredth-normal, 0.01 N; thousandth-normal, 0.001 N.

Molar Solutions—Molar solutions are solutions that contain, in 1000 mL, 1 gram-molecule of the reagent. Thus, each liter of a molar solution of sulfuric acid contains 98.07 g of H_2SO_4 and each liter of a molar solution of potassium ferricyanide contains 329.25 g of $K_3Fe(CN)_6$. Solutions containing, in 1000 mL, one-tenth of a gram-molecule of the reagent are designated “tenth-molar,” 0.1 M; and other molarities are similarly indicated.

Empirical Solutions—It is frequently difficult to prepare standard solutions of a desired theoretical normality, and this is not essential. A solution of approximately the desired normality is prepared and standardized by titration against a primary standard solution. The normality factor so obtained is used in all calculations where such empirical solutions are employed. If desired, an empirically prepared solution may be adjusted downward to a given normality provided it is strong enough to permit dilution.

All volumetric solutions, whether made by direct solution or by dilution of a stronger solution, must be thoroughly mixed by shaking before standardization. As the strength of a standard solution may change upon standing, the factor should be redetermined frequently.

When solutions of a reagent are used in several normalities, the details of the preparation and standardization are usually given for the normality most frequently required. Stronger or weaker solutions are prepared and standardized in the same general manner as described, using proportionate amounts of the reagent. It is possible in many instances to prepare lower normalities accurately by making an exact dilution of a stronger solution. Volumetric solutions prepared by dilution should be restandardized either as directed for the stronger solution or by comparison with another volumetric solution having a known ratio to the stronger solution.

Dilute solutions that are not stable, as, for instance, potassium permanganate 0.01 N and more dilute sodium thiosulfate, are preferably prepared by exactly diluting the higher normality with thoroughly boiled and cooled water on the same day they are required for use.

Blank Determinations—Where it is directed that “any necessary correction” be made by a blank determination, the determination is to be conducted with the use of the same quantities of the same reagents treated in the same manner as the solution or mixture containing the portion of the substance under assay or test, but with the substance itself omitted. Appropriate blank corrections are to be made for all Pharmacopeial titrimetric assays (see *Titrimetry* ⟨541⟩).

All Pharmacopeial assays that are volumetric in nature indicate the weight of the substance being assayed to which each mL of the primary volumetric solution is equivalent. In general, these equivalents may be derived by simple calculation from the data given under *Molecular Formulas and Weights,* in the *Reference Tables.*

Preparation and Methods of Standardization of Volumetric Solutions

The following directions give only one method for standardization, but other methods of standardization, capable of yielding at least the same degree of accuracy, may be used. The values obtained in the standardization of volumetric solutions are valid for all Pharmacopeial uses of these solutions, regardless of the instrumental or chemical indicators employed in the individual monographs. Where the apparent normality or molarity of a titrant depends upon the special conditions of its use, the individual monograph sets forth the directions for standardizing the reagent in the specified context. For those salts that usually are available as certified primary standards, or that are available as highly purified salts of primary standard quality, it is permissible to prepare solutions by accurately weighing a suitable quantity of the salt and dissolving it to produce a specific volume of solution of known concentration. Acetic, hydrochloric, and sulfuric acids may be standardized against a sodium hydroxide solution that recently has been standardized against a certified primary standard.

All volumetric solutions, if practicable, are to be prepared, standardized, and used at the standard temperature of 25°. If a titration is carried out with the volumetric solution at a markedly different temperature, standardize the volumetric solution used as the titrant at that different temperature, or make a suitable temperature correction.

Acetic Acid, Double-Normal (2 N)

$C_2H_4O_2$, **60.05**
120.10 g in 1000 mL

Add 116 mL of glacial acetic acid to sufficient water to make 1000 mL after cooling to room temperature.

Ammonium Thiocyanate, Tenth-Normal (0.1 N)

NH_4SCN, **76.12**
7.612 g in 1000 mL

Dissolve about 8 g of ammonium thiocyanate in 1000 mL of water, and standardize the solution as follows.

Accurately measure about 30 mL of 0.1 N silver nitrate VS into a glass-stoppered flask. Dilute with 50 mL of water, then add 2 mL of nitric acid and 2 mL of ferric ammonium sulfate TS, and titrate with the ammonium thiocyanate solution to the first appearance of a red-brown color.

$$N = \frac{\text{mL AgNO}_3 \text{ x N AgNO}_3}{\text{mL NH}_4\text{SCN Solution}}$$

If desirable, 0.1 N ammonium thiocyanate may be replaced by 0.1 N potassium thiocyanate where the former is directed in various tests and assays.

Bismuth Nitrate, 0.01 M

$Bi(NO_3)_3 \cdot 5H_2O$, **485.07**
1000 mL of this solution contains 4.851 g of bismuth nitrate pentahydrate

Dissolve 4.86 g of bismuth nitrate pentahydrate in 60 mL of dilute nitric acid, add 0.01 N nitric acid to make 1000 mL, and standardize the solution as follows.

Accurately measure 25 mL of the prepared bismuth nitrate solution, add 50 mL of water and 1 drop of xylenol orange TS, and titrate the solution with 0.01 M edetate disodium VS until the red color changes to yellow. Calculate the molarity factor.

Bromine, Tenth-Normal (0.1 N)

Br, **79.90**
7.990 g in 1000 mL

Dissolve 3 g of potassium bromate and 15 g of potassium bromide in water to make 1000 mL, and standardize the solution as follows.

Accurately measure about 25 mL of the solution into a 500-mL iodine flask, and dilute with 120 mL of water. Add 5 mL of hydrochloric acid, insert the stopper in the flask, and shake it gently. Then add 5 mL of potassium iodide TS, again insert the stopper, shake the mixture, allow it to stand for 5 minutes, and titrate the liberated iodine with 0.1 N sodium thiosulfate VS, adding 3 mL of starch TS as the endpoint is approached.

Preserve in dark amber-colored, glass-stoppered bottles.

$$N = \frac{\text{mL } Na_2S_2O_3 \times N Na_2S_2O_3}{\text{mL } Br_2 \text{ Solution}}$$

Ceric Ammonium Nitrate, Twentieth-Normal (0.05 N)

$Ce(NO_3)_4 \cdot 2NH_4NO_3$, **548.22**
2.741 g in 100 mL

Dissolve 2.75 g of ceric ammonium nitrate in 1 N nitric acid to obtain 100 mL of solution, and filter. Standardize the solution as follows.

Accurately measure 10 mL of freshly standardized 0.1 N ferrous ammonium sulfate VS into a flask, and dilute with water to about 100 mL. Add 1 drop of nitrophenanthroline TS, and titrate with the ceric ammonium nitrate solution to a colorless endpoint.

$$N = \frac{\text{mL } Fe(NH_4)_2(SO_4)_2 \text{ x N } Fe(NH_4)_2(SO_4)_2}{\text{mL } Ce(NO_3)_4 \cdot 2NH_4NO_3}$$

Ceric Sulfate, Tenth-Normal (0.1 N)

$Ce(SO_4)_2$, **332.24**
33.22 g in 1000 mL

Use commercially available volumetric standard solution. Standardize the solution as follows.

Accurately weigh about 0.2 g of sodium oxalate, primary standard, dried according to the instructions on its label, and dissolve in 75 mL of water. Add, with stirring, 2 mL of sulfuric acid that has previously been mixed with 5 mL of water, mix well, add 10 mL of hydrochloric acid, and heat to between 70° and 75°. Titrate with 0.1 N ceric sulfate to a permanent slight yellow color. Each 6.700 mg of sodium oxalate is equivalent to 1 mL of 0.1 N ceric sulfate.

$$N = \frac{\text{mg } Na_2C_2O_4}{67.00 \text{ x mL } Ce(SO_4)_2 \text{ solution}}$$

Cupric Nitrate, Tenth-Normal (0.1 N)

$Cu(NO_3)_2 \cdot 2.5H_2O$, **232.59**
23.26 g in 1000 mL
$Cu(NO_3)_2 \cdot 3H_2O$, **241.60**
24.16 g in 1000 mL

Dissolve 23.3 g of cupric nitrate 2.5 hydrate, or 24.2 g of the trihydrate, in water to make 1000 mL. Standardize the solution as follows.

Transfer 20.0 mL of the solution to a 250-mL beaker. Add 2 mL of 5 M sodium nitrate, 20 mL of ammonium acetate TS, and sufficient water to make 100 mL. Titrate with 0.05 M edetate disodium VS. Determine the endpoint potentiometrically using a cupric ion-double junction reference electrode system. Perform a blank determination, and make any necessary correction.

$$N = \frac{\text{mL edetate disodium (corrected for the blank) x M edetate disodium}}{20.0}$$

Standard Dichlorophenol–Indophenol Solution

To 50 mg of 2,6-dichlorophenol–indophenol sodium that has been stored in a desiccator over soda lime add 50 mL of water containing 42 mg of sodium bicarbonate, shake vigorously, and when the dye is dissolved, add water to make 200 mL. Filter into an amber, glass-stoppered bottle. Use within 3 days and standardize immediately before use. Standardize the solution as follows.

Accurately weigh 50 mg of USP Ascorbic Acid RS, and transfer to a glass-stoppered, 50-mL volumetric flask with the aid of a sufficient volume of metaphosphoric–acetic acids TS to make 50 mL. Immediately transfer 2 mL of the ascorbic acid solution to a 50-mL conical flask containing 5 mL of the metaphosphoric–acetic acids TS, and titrate rapidly with the dichlorophenol–indophenol solution until a distinct rose-pink color persists for at least 5 seconds. Perform a blank titration by titrating 7 mL of the metaphosphoric–acetic acids TS plus a volume of water equal to the volume of the dichlorophenol solution used in titrating the ascorbic acid solution. Express the concentration of the standard solution in terms of its equivalent in mg of ascorbic acid.

Edetate Disodium, Twentieth-Molar (0.05 M)

$C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$, **372.24**
18.61 g in 1000 mL

Dissolve 18.6 g of edetate disodium in water to make 1000 mL, and standardize the solution as follows.

Accurately weigh about 200 mg of chelometric standard calcium carbonate, previously dried at 110° for 2 hours and cooled in a desiccator, transfer to a 400-mL beaker, add 10 mL of water, and swirl to form a slurry. Cover the beaker with a watch glass, and introduce 2 mL of diluted hydrochloric acid from a pipet inserted between the lip of the beaker and the edge of the watch glass. Swirl the 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 with water, and dilute with water to about 100 mL. While stirring the solution, preferably with a magnetic stirrer, add about 30 mL of the edetate disodium solution from a 50-mL buret. Add 15 mL of sodium hydroxide TS and 300 mg of hydroxy naphthol blue, and continue the titration with the edetate disodium solution to a blue endpoint.

$$M = \frac{(\text{g } CaCO_3)(1000)}{100.09 \text{ x mL EDTA}}$$

Ferric Ammonium Sulfate, Tenth-Normal (0.1 N)

$FeNH_4(SO_4)_2 \cdot 12H_2O$, **482.19**
48.22 g in 1000 mL

Dissolve 50 g of ferric ammonium sulfate in a mixture of 300 mL of water and 6 mL of sulfuric acid, dilute with water to 1000 mL, and mix. Standardize the solution as follows.

Accurately measure about 40 mL of the solution into a glass-stoppered flask, add 5 mL of hydrochloric acid, mix, and add a solution of 3 g of potassium iodide in 10 mL of water. Insert the stopper, allow to stand for 10 minutes, then titrate the liberated iodine with 0.1 N sodium thiosulfate VS, adding 3 mL of starch TS as the endpoint is approached. Correct for a blank run on the same quantities of the same reagents.

Store in tight containers, protected from light.

$$N = \frac{mL\ Na_2S_2O_3 \times N\ Na_2S_2O_3}{mL\ FeNH_4(SO_4)_2}$$

Ferrous Ammonium Sulfate, Tenth-Normal (0.1 N)

$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$, **392.14**
39.21 g in 1000 mL

Dissolve 40 g of ferrous ammonium sulfate in a previously cooled mixture of 40 mL of sulfuric acid and 200 mL of water, dilute with water to 1000 mL, and mix. On the day of use, standardize the solution as follows.

Accurately measure 25 to 30 mL of the solution into a flask, add 2 drops of orthophenanthroline TS, and titrate with 0.1 N ceric sulfate VS until the red color is changed to pale blue.

$$N = \frac{mL\ Ce^{IV} \times N\ Ce^{IV}}{mL\ Fe^{II}\ Solution}$$

Hydrochloric Acid, Alcoholic, Tenth-Molar (0.1 M)

HCl, **36.46**

Dilute 9.0 mL of hydrochloric acid to 1000 mL with aldehyde–free alcohol.

Change to read:

Hydrochloric Acid, Half-Normal (0.5 N)

HCl, **36.46**
18.23 g in 1000 mL

To a 1000-mL volumetric flask containing 40 mL of water slowly add 43 mL of hydrochloric acid. Cool, and add water to volume. Standardize the solution as follows.

Accurately weigh about 2.5 g of tromethamine, dried according to the label instructions. ▲Dissolve in 50 mL of water, and add 2 drops of bromocresol green TS. Titrate with 0.5 N hydrochloric acid to a pale yellow endpoint. Each 60.57 mg of tromethamine is equivalent to 1 mL of 0.5 N hydrochloric acid.▲*USP35*

$$N = \frac{mg\ tromethamine}{121.14 \times mL\ HCl}$$

Hydrochloric Acid, Half-Normal (0.5 N) in Methanol

HCl, **36.46**
18.23 g in 1000 mL

To a 1000-mL volumetric flask containing 40 mL of water slowly add 43 mL of hydrochloric acid. Cool, and add methanol to volume. Standardize the solution as follows.

Accurately weigh about 2.5 g of tromethamine, dried according to the label instructions. Proceed as directed under *Hydrochloric Acid, Normal (1 N),* beginning with “Dissolve in 50 mL of water.”

$$N = \frac{mg\ tromethamine}{121.14 \times mL\ HCl}$$

Hydrochloric Acid, Normal (1 N)

HCl, **36.46**
36.46 g in 1000 mL

Dilute 85 mL of hydrochloric acid with water to 1000 mL. Standardize the solution as follows.

Accurately weigh about 5.0 g of tromethamine, dried according to the label instructions. Dissolve in 50 mL of water, and add 2 drops of bromocresol green TS. Titrate with 1 N hydrochloric acid to a pale yellow endpoint. Each 121.14 mg of tromethamine is equivalent to 1 mL of 1 N hydrochloric acid.

$$N = \frac{mg\ tromethamine}{121.14 \times mL\ HCl}$$

Iodine, Tenth-Normal (0.1 N)

I, **126.90**
12.69 g in 1000 mL

Dissolve about 14 g of iodine in a solution of 36 g of potassium iodide in 100 mL of water, add 3 drops of hydrochloric acid, dilute with water to 1000 mL, and standardize the solution as follows.

Transfer 25.0 mL of the iodine solution to a 250-mL flask, dilute with water to 100 mL, add 1 mL of 1 N hydrochloric acid, swirl gently to mix, and titrate with 0.1 N sodium thiosulfate VS until the solution has a pale yellow color. Add 2 mL of starch TS and continue titrating until the solution is colorless.

Preserve in amber-colored, glass-stoppered bottles.

$$N = \frac{mL\ Na_2S_2O_3 \times N\ Na_2S_2O_3}{25}$$

Iodine, Twentieth-Normal (0.05 N)

I, **126.90**
6.33 g in 1000 mL

Dissolve about 6.5 g of iodine in a solution of 18 g of potassium iodide in 100 mL of water, add 3 drops of hydrochloric acid, dilute with water to 1000 mL, and standardize the solution as follows.

Transfer 50.0 mL of the iodine solution to a 250-mL flask, dilute with water to 100 mL, add 1 mL of 1 N hydrochloric acid, swirl gently to mix, and titrate with 0.1 N sodium thiosulfate VS until the solution has a pale yellow color. Add 2 mL of starch TS, and continue titrating until the solution is colorless.

$$N = \frac{mL\ Na_2S_2O_3 \times N\ Na_2S_2O_3}{50}$$

Iodine, Hundredth-Normal (0.01 N)

I, **126.90**
1.269 g in 1000 mL

Dissolve about 1.4 g of iodine in a solution of 3.6 g of potassium iodide in 100 mL of water, add 3 drops of hydrochloric acid, dilute with water to 1000 mL, and standardize the solution as follows.

Transfer 100.0 mL of iodine solution to a 250-mL flask, add 1 mL of 1 N hydrochloric acid, swirl gently to mix, and titrate with 0.1 N sodium thiosulfate VS until the solution has a pale yellow color. Add 2 mL of starch TS, and continue titrating until the solution is colorless.

Preserve in amber-colored, glass-stoppered bottles.

$$N = \frac{mL\ Na_2S_2O_3 \times N\ Na_2S_2O_3}{100}$$

Lead Nitrate, Hundredth-Molar (0.01 M)

$Pb(NO_3)_2$, **331.21**
3.312 g in 1000 mL

Xylenol Orange Triturate—Triturate 1 part of xylenol orange with 99 parts of potassium nitrate.

0.1 M Lead Nitrate—Dissolve 33 g of lead nitrate in 1000 mL of water. Standardize the solution as follows. To 20.0 mL of the lead nitrate solution add 300 mL of water. Add about 50 mg of *Xylenol Orange Triturate,* and add methenamine until the solution becomes violet-pink. Titrate with 0.1 M edetate disodium VS to the yellow endpoint. Calculate the molarity.

Dilute 50.0 mL of *0.1 M Lead Nitrate* to 500.0 mL with water.

Lead Perchlorate, Tenth-Molar (0.1 M)

$Pb(ClO_4)_2 \cdot 3H_2O$, **460.15**
46.01 g in 1000 mL

Dissolve 46 g of lead perchlorate in water, and dilute with water to 1000.0 mL. Accurately weigh about 150 mg of sodium sulfate, previously dried at 105° for 4 hours, and dissolve in 50 mL of water. Add 50 mL of a mixture of water and formaldehyde (1:1), and stir for about 1 minute. Determine the endpoint potentiometrically using a lead ion selective electrode. Perform a blank determination, and make any necessary corrections. Each 14.204 mg of sodium sulfate is equivalent to 1 mL of 0.1 M lead perchlorate.

$$M = \frac{\text{mg sodium sulfate}}{142.04 \times \text{mL lead perchlorate}}$$

Lead Perchlorate, Hundredth-Molar (0.01 M)

$Pb(ClO_4)_2$, **406.10**

Accurately pipet 100 mL of commercially available 0.1 M lead perchlorate solution into a 1000-mL volumetric flask, add a sufficient quantity of water to make 1000 mL, and standardize the solution as follows.

Accurately pipet 50 mL of 0.01 M lead perchlorate solution, as prepared above, into a 250-mL conical flask. Add 3 mL of aqueous hexamethylenetetramine solution (2.0 g per 100 mL) and 4 drops of 0.5% xylenol orange indicator prepared by adding 500 mg of xylenol orange to 10 mL of alcohol and diluting with water to 100 mL. (Omit the alcohol if the sodium salt of the indicator is used). Titrate with 0.05 M edetate disodium VS to a yellow endpoint.

$$M = \frac{\text{mL edetate disodium} \times \text{M edetate disodium}}{50.0}$$

Lithium Methoxide, Fiftieth-Normal (0.02 N) in Methanol

CH_3LiO, **37.97**
759.6 mg in 1000 mL

Dissolve 0.12 g of freshly cut lithium metal in 150 mL of methanol, cooling the flask during addition of the metal. When the reaction is complete, add 850 mL of methanol, and mix. Store the solution preferably in the reservoir of an automatic delivery buret suitably protected from carbon dioxide and moisture. Standardize the solution by titration against benzoic acid as described under *Sodium Methoxide, Tenth-Normal (0.1 N) (in Toluene),* but use only 100 mg of benzoic acid. Each 2.442 mg of benzoic acid is equivalent to 1 mL of 0.02 N lithium methoxide.

[NOTE—Restandardize the solution frequently.]

$$N = \frac{\text{mg benzoic acid}}{121.1 \times \text{mL lithium methoxide (corrected for the blank)}}$$

Lithium Methoxide, Tenth-Normal (0.1 N) in Chlorobenzene

CH_3OLi, **37.97**
3.798 g in 1000 mL

Dissolve 700 mg of freshly cut lithium metal in 150 mL of methanol, cooling the flask during addition of the metal. When the reaction is complete, add 850 mL of chlorobenzene. If cloudiness or precipitation occurs, add sufficient methanol to clarify the solution. Store preferably in the reservoir of an automatic delivery buret suitably protected from carbon dioxide and moisture. Standardize the solution by titration against benzoic acid as described under *Sodium Methoxide, Tenth-Normal (0.1 N) (in Toluene).*

[NOTE—Restandardize the solution frequently.]

$$N = \frac{\text{mg benzoic acid}}{121.1 \times \text{mL lithium methoxide (corrected for the blank)}}$$

Lithium Methoxide, Tenth-Normal (0.1 N) in Methanol

CH_3OLi, **37.97**
3.798 g in 1000 mL

Dissolve 700 mg of freshly cut lithium metal in 150 mL of methanol, cooling the flask during addition of the metal. When the reaction is complete, add 850 mL of methanol. If cloudiness or precipitation occurs, add sufficient methanol to clarify the solution. Store preferably in the reservoir of an automatic delivery buret suitably protected from carbon dioxide and moisture. Standardize the solution by titration against benzoic acid as described under *Sodium Methoxide, Tenth-Normal* (*0.1 N*) (*in Toluene*).

[NOTE—Restandardize the solution frequently.]

$$N = \frac{\text{mg benzoic acid}}{121.1 \times \text{mL lithium methoxide (corrected for the blank)}}$$

Lithium Methoxide, Tenth-Normal (0.1 N) in Toluene

CH_3OLi, **37.97**
3.798 g in 1000 mL

Dissolve 700 mg of freshly cut lithium metal in 150 mL of methanol, cooling the flask during addition of the metal. When reaction is complete, add 850 mL of toluene. If cloudiness or precipitation occurs, add sufficient methanol to clarify the solution. Store preferably in the reservoir of an automatic delivery buret suitably protected from carbon dioxide and moisture. Standardize the solution by titration against benzoic acid as described under *Sodium Methoxide, Tenth-Normal (0.1 N) (in Toluene).*

[NOTE—Restandardize the solution frequently.]

$$N = \frac{\text{mg benzoic acid}}{121.1 \times \text{mL lithium methoxide (corrected for the blank)}}$$

Magnesium Chloride, 0.01 M

$MgCl_2 \cdot 6H_2O$, **203.30**
2.0330 g in 1000 mL

Dissolve about 2.04 g of magnesium chloride in 1000 mL of freshly boiled and cooled water, and standardize the solution as follows.

Accurately measure 25 mL of the prepared magnesium chloride solution. Add 50 mL of water, 3 mL of ammonia–ammonium chloride buffer TS and 0.04 g of eriochrome black T–sodium chloride reagent. Titrate with 0.05 M edetate disodium VS until the red-purple color of the solution changes to blue-purple.

$$M = \frac{(\text{mL edetate disodium VS}) \times (\text{M edetate disodium})}{\text{mL magnesium chloride}}$$

Mercuric Nitrate, Tenth-Molar (0.1 M)
$Hg(NO_3)_2$, **324.60**
32.46 g in 1000 mL

Dissolve about 35 g of mercuric nitrate in a mixture of 5 mL of nitric acid and 500 mL of water, and dilute with water to 1000 mL. Standardize the solution as follows.

Transfer an accurately measured volume of about 20 mL of the solution to a conical flask, and add 2 mL of nitric acid and 2 mL of ferric ammonium sulfate TS. Cool to below 20°, and titrate with 0.1 N ammonium thiocyanate VS to the first appearance of a permanent brownish color.

$$M = \frac{\text{mL } NH_4SCN \times \text{N } NH_4SCN}{\text{mL } Hg(NO_3)_2 \times 2}$$

Oxalic Acid, Tenth-Normal (0.1 N)
$H_2C_2O_4 \cdot 2H_2O$, **126.07**
6.303 g in 1000 mL

Dissolve 6.45 g of oxalic acid in water to make 1000 mL. Standardize by titration against freshly standardized 0.1 N potassium permanganate VS as directed under *Potassium Permanganate, Tenth-Normal (0.1 N).*

Preserve in glass-stoppered bottles, protected from light.

$$N = \frac{\text{mL } KMnO_4 \times \text{N } KMnO_4}{\text{mL } H_2C_2O_4}$$

Perchloric Acid, Tenth-Normal (0.1 N) in Dioxane

Mix 8.5 mL of perchloric acid with sufficient dioxane to make 1000 mL. Standardize the solution as follows.

Accurately weigh about 700 mg of potassium biphthalate, previously crushed lightly and dried at 120° for 2 hours, and dissolve in 50 mL of glacial acetic acid in a 250-mL flask. Add 2 drops of crystal violet TS, and titrate with the perchloric acid solution until the violet color changes to bluish green. Carry out a blank determination. Each 20.423 mg of potassium biphthalate is equivalent to 1 mL of 0.1 N perchloric acid.

$$N = \frac{\text{g } KHC_8H_4O_4}{0.20423 \times \text{mL } HClO_4 \text{ solution (corrected for the blank)}}$$

Perchloric Acid, Tenth-Normal (0.1 N) in Glacial Acetic Acid
$HClO_4$, **100.46**
10.05 g in 1000 mL

[NOTE—Where called for in the tests and assays, this volumetric solution is specified as "0.1 N perchloric acid." Thus, where 0.1 N or other strength of this volumetric solution is specified, the solution in glacial acetic acid is to be used, unless the words "in dioxane" are stated. [See also *Perchloric Acid, Tenth-Normal (0.1 N) in Dioxane*.]]

Mix 8.5 mL of perchloric acid with 500 mL of glacial acetic acid and 21 mL of acetic anhydride, cool, and add glacial acetic acid to make 1000 mL. Alternatively, the solution may be prepared as follows. Mix 11 mL of 60 percent perchloric acid with 500 mL of glacial acetic acid and 30 mL of acetic anhydride, cool, and add glacial acetic acid to make 1000 mL.

Allow the prepared solution to stand for 1 day for the excess acetic anhydride to be combined, and determine the water content by *Method I* (see *Water Determination* ⟨921⟩), except to use a test specimen of about 5 g of the 0.1 N perchloric acid that is expected to contain approximately 1 mg of water and the *Reagent* (see *Reagent* under *Method Ia* in *Water Determination* ⟨921⟩) diluted such that 1 mL is equivalent to about 1 to 2 mg of water. If the water content exceeds 0.5%, add more acetic anhydride. If the solution contains no titratable water, add sufficient water to obtain a content of between 0.02% and 0.5% of water. Allow the solution to stand for 1 day, and again titrate the water content. The solution so obtained contains between 0.02% and 0.5% of water, indicating freedom from acetic anhydride.

Standardize the solution as follows.

Accurately weigh about 700 mg of potassium biphthalate, previously crushed lightly and dried at 120° for 2 hours, and dissolve it in 50 mL of glacial acetic acid in a 250-mL flask. Add 2 drops of crystal violet TS, and titrate with the perchloric acid solution until the violet color changes to blue-green. Deduct the volume of the perchloric acid consumed by 50 mL of the glacial acetic acid. Each 20.423 mg of potassium biphthalate is equivalent to 1 mL of 0.1 N perchloric acid.

$$N = \frac{\text{g } KHC_8H_4O_4}{0.20423 \times \text{mL } HClO_4 \text{ solution (corrected for the blank)}}$$

Potassium Arsenite, Tenth-Normal (0.1 N)
$KAsO_2$, **146.02**
7.301 g in 1000 mL

Dissolve 4.9455 g of arsenic trioxide primary standard, previously dried at 105° for 1 hour, in 75 mL of 1 N potassium hydroxide. Add 40 g of potassium bicarbonate, dissolved in about 200 mL of water, and dilute with water to 1000.0 mL.

Potassium Bromate, Tenth-Normal (0.1 N)
$KBrO_3$, **167.00**
2.784 g in 1000 mL

Dissolve 2.784 g of potassium bromate in water to make 1000 mL, and standardize the solution as follows.

Transfer an accurately measured volume of about 40 mL of the solution to a glass-stoppered flask, add 3 g of potassium iodide, and follow with 3 mL of hydrochloric acid. Allow to stand for 5 minutes, then titrate the liberated iodine with 0.1 N sodium thiosulfate VS, adding 3 mL of starch TS as the endpoint is approached. Correct for a blank run on the same quantities of the same reagents, and calculate the normality.

$$N = \frac{\text{mL } Na_2S_2O_3 \times \text{N } Na_2S_2O_3}{\text{mL } KBrO_3 \text{ Solution}}$$

Potassium Bromide–Bromate, Tenth-Normal (0.1 N)

Dissolve 2.78 g of potassium bromate ($KBrO_3$) and 12.0 g of potassium bromide (KBr) in water, and dilute with water to 1000 mL. Standardize by the procedure set forth for *Potassium Bromate, Tenth-Normal (0.1 N).*

$$N = \frac{\text{mL } Na_2S_2O_3 \times \text{N } Na_2S_2O_3}{\text{mL } KBrO_3/KBr}$$

Potassium Dichromate, Tenth-Normal (0.1 N)
$K_2Cr_2O_7$, **294.18**
4.903 g in 1000 mL

Dissolve about 5 g of potassium dichromate in 1000 mL of water. Standardize the solution as follows.

Transfer 25.0 mL of this solution to a glass-stoppered, 500-mL flask, add 2 g of potassium iodide (free from iodate), dilute with 200 mL of water, add 5 mL of hydrochloric acid, allow to stand for 10 minutes in a dark place, and titrate the liberated iodine with 0.1 N sodium thiosulfate VS, adding 3 mL of starch TS as the endpoint is approached. Carry out a blank determination.

$$N = \frac{\text{mL } Na_2S_2O_3 \times N\ Na_2S_2O_3}{25.0}$$

Potassium Ferricyanide, Twentieth-Molar (0.05 M)

$K_3Fe(CN)_6$, **329.24**
16.46 g in 1000 mL

Dissolve about 17 g of potassium ferricyanide in water to make 1000 mL. Standardize the solution as follows.

Transfer 50.0 mL of this solution to a glass-stoppered, 500-mL flask, dilute with 50 mL of water, add 10 mL of potassium iodide TS and 10 mL of dilute hydrochloric acid, and allow to stand for 1 minute. Then add 15 mL of zinc sulfate solution (1 in 10), and titrate the liberated iodine with 0.1 N sodium thiosulfate VS, adding 3 mL of starch TS as the endpoint is approached.

Protect from light, and restandardize before use.

$$M = \frac{\text{mL } Na_2S_2O_3 \times N\ Na_2S_2O_3}{50.0}$$

Potassium Hydroxide, Alcoholic, Half-Normal (0.5 N)

28.06 g in 1000 mL

Dissolve about 34 g of potassium hydroxide in 20 mL of water, and add aldehyde-free alcohol to make 1000 mL. Allow the solution to stand in a tightly stoppered bottle for 24 hours. Then quickly decant the clear supernatant into a suitable, tight container, and standardize the solution as follows.

Accurately measure about 25 mL of 0.5 N hydrochloric acid VS. Dilute with 50 mL of water, add 2 drops of phenolphthalein TS, and titrate with the alcoholic potassium hydroxide solution until a permanent, pale pink color is produced.

[NOTE—Store in tightly stoppered bottles, protected from light.]

$$N = \frac{\text{mL HCl} \times N\ \text{HCl}}{\text{mL KOH}}$$

Potassium Hydroxide, Alcoholic, Tenth-Molar (0.1 M)

KOH, **56.11**

Dilute 20 mL of 0.5 M alcoholic potassium hydroxide to 100.0 mL with aldehyde-free alcohol.

Potassium Hydroxide, Methanolic, Tenth-Normal (0.1 N)

5.612 g in 1000 mL

Dissolve about 6.8 g of potassium hydroxide in 4 mL of water, and add methanol to make 1000 mL. Allow the solution to stand in a tightly stoppered bottle for 24 hours. Then quickly decant the clear supernatant into a suitable, tight container, and standardize the solution as follows.

Accurately measure about 25 mL of 0.1 N hydrochloric acid VS. Dilute with 50 mL of water, add 2 drops of phenolphthalein TS, and titrate with the methanolic potassium hydroxide solution until a permanent, pale pink color is produced.

[NOTE—Store in tightly stoppered bottles, protected from light.]

$$N = \frac{\text{mL HCl} \times N\ \text{HCl}}{\text{mL KOH}}$$

Potassium Hydroxide, Normal (1 N)

KOH, **56.11**
56.11 g in 1000 mL

Dissolve 68 g of potassium hydroxide in about 950 mL of water. Add a freshly prepared saturated solution of barium hydroxide until no more precipitate forms. Shake the mixture thoroughly, and allow it to stand overnight in a stoppered bottle. Decant the clear liquid, or filter the solution in a tight, polyolefin bottle, and standardize by the procedure set forth for *Sodium Hydroxide, Normal (1 N).*

$$N = \frac{\text{g } KHC_8H_4O_4}{0.20423 \times \text{mL KOH}}$$

Potassium Iodate, Twentieth-Molar (0.05 M)

KIO_3, **214.00**
10.70 g in 1000 mL

Dissolve 10.700 g of potassium iodate, previously dried at 110° to constant weight, in water to make 1000.0 mL. Standardize the solution as follows: to 15.0 mL of solution in a 250-mL iodine flask, add 3 g of potassium iodide and 3 mL of hydrochloric acid previously diluted with 10 mL of water. Stopper immediately, and allow to stand in the dark for 5 minutes. Then add 50 mL of cold water, and titrate the liberated iodine with freshly standardized 0.1 N sodium thiosulfate. Add 3 mL of starch indicator solution near the end of the titration, and continue to the absence of the blue-starch-iodine complex.

$$M = \frac{\text{mL} \times N\ Na_2S_2O_3}{\text{mL } KIO_3 \times 6}$$

Potassium Permanganate, Tenth-Normal (0.1 N)

$KMnO_4$, **158.03**
3.161 g in 1000 mL

Dissolve about 3.3 g of potassium permanganate in 1000 mL of water in a flask, and boil the solution for about 15 minutes. Insert the stopper in the flask, allow it to stand for at least 2 days, and filter through a fine-porosity, sintered-glass crucible. If necessary, the bottom of the sintered-glass crucible may be lined with a pledget of glass wool. Standardize the solution as follows.

Accurately weigh about 200 mg of sodium oxalate, dried according to the instructions on its label, and dissolve it in 250 mL of water. Add 7 mL of sulfuric acid, heat to about 70°, and then slowly add the permanganate solution from a buret, with constant stirring, until a pale pink color, which persists for 15 seconds, is produced. The temperature at the conclusion of the titration should be not less than 60°. Calculate the normality. Each 6.700 mg of sodium oxalate is equivalent to 1 mL of 0.1 N potassium permanganate.

Since potassium permanganate is reduced on contact with organic substances such as rubber, the solution must be handled in apparatus entirely of glass or other suitably inert material. It should be frequently restandardized. Store in glass-stoppered, amber-colored bottles.

$$N = \frac{\text{g } Na_2C_2O_4}{\text{mL } KMnO_4 \text{ solution} \times 0.06700}$$

Potassium Thiocyanate, Tenth-Normal (0.1 N)

KSCN, **97.18**
9.72 g in 1000 mL

Weigh exactly 9.72 g of potassium thiocyanate, previously dried for 2 hours at 110°, transfer to a 1-L volumetric flask, dilute with water to volume, and mix well. Standardize as follows: transfer 40.0 mL of freshly standardized 0.1 N silver nitrate VS to a 250-mL Erlenmeyer flask; and add 100 mL of water, 1 mL of nitric acid, and 2 mL of ferric ammonium sulfate TS. Titrate with the potassium thiocyanate solution, with agitation, to a permanent light pinkish-brown color of the supernatant.

$$N = \frac{\text{mL x N AgNO}_3}{\text{mL KSCN}}$$

Silver Nitrate, Tenth-Normal (0.1 N)

$AgNO_3$, **169.87**
16.99 g in 1000 mL

Dissolve about 17.5 g of silver nitrate in 1000 mL of water, and standardize the solution as follows.

Transfer about 100 mg, accurately weighed, of reagent-grade sodium chloride, previously dried at 110° for 2 hours, to a 150-mL beaker, dissolve in 5 mL of water, and add 5 mL of acetic acid, 50 mL of methanol, and about 0.5 mL of eosin Y TS. Stir, preferably with a magnetic stirrer, and titrate with the silver nitrate solution.

$$N = \frac{\text{mg NaCl}}{\text{mL AgNO}_3 \text{ x } 58.44}$$

Sodium Arsenite, Twentieth-Molar (0.05 M)

$NaAsO_2$ **129.91**
6.496 g in 1000 mL

Transfer 4.9455 g of arsenic trioxide, which has been pulverized and dried at 100° to constant weight, to a 1000-mL volumetric flask, dissolve it in 40 mL of 1 N sodium hydroxide, and add 1 N sulfuric acid or 1 N hydrochloric acid until the solution is neutral or only slightly acid to litmus. Add 30 g of sodium bicarbonate, dilute with water to volume, and mix.

Sodium Hydroxide, Alcoholic, Tenth-Normal (0.1 N)

NaOH, **40.00**

To 250 mL of alcohol add 2 mL of a 50% (w/v) solution of sodium hydroxide.

Dissolve about 200 mg of benzoic acid, accurately weighed, in 10 mL of alcohol and 2 mL of water. Add 2 drops of phenolphthalein TS, and titrate with the alcoholic sodium hydroxide solution until a permanent pale pink color is produced.

$$N = \frac{\text{mg benzoic acid}}{122.1 \text{ x mL sodium hydroxide}}$$

Change to read:

Sodium Hydroxide, Normal (1 N)

NaOH, **40.00**
40.00 g in 1000 mL

Dissolve 162 g of sodium hydroxide in 150 mL of carbon dioxide-free water, cool the solution to room temperature, and filter through hardened filter paper. Transfer 54.5 mL of the clear filtrate to a tight, polyolefin container, and dilute with carbon dioxide-free water to 1000 mL.

Accurately weigh about 5 g of potassium biphthalate, previously crushed lightly and dried at 120° for 2 hours, and dissolve in 75 mL of carbon dioxide-free water. Add 2 drops of phenolphthalein TS, and titrate with the sodium hydroxide solution to the production of a permanent pink color. Each ▲204.22 mg▲*USP35* of potassium biphthalate is equivalent to 1 mL of 1 N sodium hydroxide.

▲

$$N = \frac{\text{g KHC}_8\text{H}_4\text{O}_4}{0.20422 \times \text{mL NaOH solution}}$$

▲*USP35*

[NOTES—(1) Solutions of alkali hydroxides absorb carbon dioxide when exposed to air. They should be preserved in bottles having well-fitted, suitable stoppers, provided with a tube filled with a mixture of sodium hydroxide and lime (soda-lime tubes) so that air entering the container must pass through this tube, which will absorb the carbon dioxide. (2) Prepare solutions of lower concentration (e.g., 0.1 N, 0.01 N) by quantitatively diluting accurately measured volumes of the 1 N solution with sufficient carbon dioxide-free water to yield the desired concentration.]

Restandardize the solution frequently.

Sodium Methoxide, Half-Normal (0.5 N) in Methanol

CH_3ONa, **54.02**
27.01 g in 1000 mL

Weigh 11.5 g of freshly cut sodium metal, and cut into small cubes. Place about 0.5 mL of anhydrous methanol in a round-bottom, 250-mL flask equipped with a ground-glass joint, add 1 cube of the sodium metal, and, when the reaction has ceased, add the remaining sodium metal to the flask. Connect a water-jacketed condenser to the flask, and slowly add 250 mL of anhydrous methanol, in small portions, through the top of the condenser. Regulate the addition of the methanol so that the vapors are condensed and do not escape through the top of the condenser. After addition of the methanol is complete, connect a drying tube to the top of the condenser, and allow the solution to cool. Transfer the solution to a 1-L volumetric flask, dilute with anhydrous methanol to volume, and mix. Standardize the solution as follows.

Accurately measure about 20 mL of freshly standardized 1 N hydrochloric acid VS into a 250-mL conical flask, add 0.25 mL of phenolphthalein TS, and titrate with the sodium methoxide solution to the first appearance of a permanent pink color.

$$N = \frac{\text{mL HCl x N HCl}}{\text{mL sodium methoxide}}$$

Sodium Methoxide, Tenth-Normal (0.1 N) in Toluene

CH_3ONa, **54.02**
5.402 g in 1000 mL

Cool in ice-water 150 mL of methanol contained in a 1000-mL volumetric flask, and add, in small portions, about 2.5 g of freshly cut sodium metal. When the metal has dissolved, add toluene to make 1000 mL, and mix. Store preferably in the reservoir of an automatic delivery buret suitably protected from carbon dioxide and moisture. Standardize the solution as follows.

Accurately weigh about 400 mg of primary standard benzoic acid, and dissolve in 80 mL of dimethylformamide in a flask. Add 3 drops of a 1 in 100 solution of thymol blue in dimethylformamide, and titrate with the sodium methoxide to a blue endpoint. Correct for the volume of the sodium methoxide solution consumed by 80 mL of the dimethylformamide. Each 12.21 mg of benzoic acid is equivalent to 1 mL of 0.1 N sodium methoxide.

$$N = \frac{\text{mg benzoic acid}}{122.1 \text{ x mL sodium methoxide (corrected for the blank)}}$$

[NOTES—(1) To eliminate any turbidity that may form following dilution with toluene, add methanol (25 to 30 mL usually suffices) until the solution is clear. (2) Restandardize the solution frequently.]

Sodium Nitrite, Tenth-Molar (0.1 M)

$NaNO_2$, **69.00**
6.900 g in 1000 mL

Dissolve 7.5 g of sodium nitrite in water to make 1000 mL, and standardize the solution as follows.

Accurately weigh about 500 mg of USP Sulfanilamide RS, previously dried at 105° for 3 hours, and transfer to a suitable beaker. Add 20 mL of hydrochloric acid and 50 mL of water, stir until dissolved, and cool to 15°. Maintaining the temperature at about 15°, titrate slowly with the sodium nitrite solution, placing the buret tip below the surface of the solution to preclude air oxidation of the sodium nitrite, and stir the solution gently with a magnetic stirrer, but avoid pulling a vortex of air beneath the surface. Use the indicator specified in the individual monograph, or, if a potentiometric procedure is specified, determine the endpoint electrometrically, using platinum–calomel or platinum–platinum electrodes. When the titration is within 1 mL of the endpoint, add the titrant in 0.1-mL portions, and allow 1 minute between additions. Each 17.22 mg of sulfanilamide is equivalent to 1 mL of 0.1000 M sodium nitrite.

$$M = \frac{\text{mg of sulfanilamide}}{172.22 \times \text{mL } NaNO_2}$$

Sodium Tetraphenylboron, Fiftieth-Molar (0.02 M)

$NaB(C_6H_5)_4$, **342.22**
6.845 g in 1000 mL

Dissolve an amount of sodium tetraphenylboron, equivalent to 6.845 g of $NaB(C_6H_5)_4$, in water to make 1000 mL, and standardize the solution as follows.

Pipet two 75-mL portions of the solution into separate beakers, and to each add 1 mL of acetic acid and 25 mL of water. To each beaker add, slowly and with constant stirring, 25 mL of potassium biphthalate solution (1 in 20), and allow to stand for 2 hours. Filter one of the mixtures through a filtering crucible, and wash the precipitate with cold water. Transfer the precipitate to a container, add 50 mL of water, shake intermittently for 30 minutes, filter, and use the filtrate as the saturated potassium tetraphenylborate solution in the following standardization procedure. Filter the second mixture through a tared filtering crucible, and wash the precipitate with three 5-mL portions of saturated potassium tetraphenylborate solution. Dry the precipitate at 105° for 1 hour. Each g of potassium tetraphenylborate (KTPB) is equivalent to 955.1 mg of sodium tetraphenylboron.

$$M = \frac{\text{g KTPB} \times 0.9551}{342.22 \times 0.075}$$

[NOTE—Prepare this solution just before use.]

Sodium Thiosulfate, Tenth-Normal (0.1 N)

$Na_2S_2O_3 \cdot 5H_2O$, **248.19**
24.82 g in 1000 mL

Dissolve about 26 g of sodium thiosulfate and 200 mg of sodium carbonate in 1000 mL of recently boiled and cooled water. Standardize the solution as follows.

Accurately weigh about 210 mg of primary standard potassium dichromate, previously pulverized and dried according to the instructions on its label, if necessary, and dissolve in 100 mL of water in a glass-stoppered, 500-mL flask. Swirl to dissolve the solid, remove the stopper, and quickly add 3 g of potassium iodide, 2 g of sodium bicarbonate, and 5 mL of hydrochloric acid. Insert the stopper gently in the flask, swirl to mix, and allow to stand in the dark for exactly 10 minutes. Rinse the stopper and the inner walls of the flask with water, and titrate the liberated iodine with the sodium thiosulfate solution until the solution is yellowish green in color. Add 3 mL of starch TS, and continue the titration until the blue color is discharged. Perform a blank determination.

Restandardize the solution as frequently as supported by laboratory stability data. In the absence of such data, restandardize the solution weekly.

$$N = \frac{\text{mg } K_2Cr_2O_7}{49.04 \times \text{mL } Na_2S_2O_3}$$

Sulfuric Acid, Half-Normal (0.5 N) in Alcohol

H_2SO_4, **98.08**
24.52 g in 1000 mL

Add slowly, with stirring, 13.9 mL of sulfuric acid to a sufficient quantity of dehydrated alcohol to make 1000 mL. Cool, and standardize against tromethamine as described under *Hydrochloric Acid, Half-Normal (0.5 N) in Methanol.*

$$N = \frac{\text{mg tromethamine}}{121.14 \times \text{mL } H_2SO_4}$$

Sulfuric Acid, Normal (1 N)

H_2SO_4, **98.08**
49.04 g in 1000 mL

Add slowly, with stirring, 30 mL of sulfuric acid to about 1020 mL of water, allow to cool to 25°, and determine the normality by titration against tromethamine as described under *Hydrochloric Acid, Normal (1 N).*

Tetrabutylammonium Hydroxide, Tenth-Normal (0.1 N)

$(C_4H_9)_4NOH$, **259.47**
25.95 g in 1000 mL

Dissolve 40 g of tetra-*n*-butylammonium iodide in 90 mL of anhydrous methanol in a glass-stoppered flask. Place in an ice bath, add 20 g of powdered silver oxide, insert the stopper in the flask, and agitate vigorously for 60 minutes. Centrifuge a few mL, and test the supernatant for iodide (see *Iodide* ⟨191⟩). If the test is positive, add an additional 2 g of silver oxide, and continue to allow to stand for 30 minutes with intermittent agitation. When all of the iodide has reacted, filter through a fine-porosity, sintered-glass funnel. Rinse the flask and the funnel with three 50-mL portions of anhydrous toluene, adding the rinsings to the filtrate. Dilute with a mixture of three volumes of anhydrous toluene and 1 volume of anhydrous methanol to 1000 mL, and flush the solution for 10 minutes with dry, carbon dioxide-free nitrogen. [NOTE—If necessary to obtain a clear solution, further small quantities of anhydrous methanol may be added.] Store in a reservoir protected from carbon dioxide and moisture, and discard after 60 days. Alternatively, the solution may be prepared by diluting a suitable volume of commercially available tetrabutylammonium hydroxide solution in methanol with a mixture of 4 volumes of anhydrous toluene and 1 volume of anhydrous methanol. [NOTE—If necessary to obtain a clear solution, further small quantities of methanol may be added.]

Standardize the solution on the day of use as follows.

Dissolve about 400 mg of primary standard benzoic acid, accurately weighed, in 80 mL of dimethylformamide, add 3 drops of a 1 in 100 solution of thymol blue in dimethylformamide, and titrate to a blue endpoint with the tetrabutylammonium hydroxide solution, delivering the titrant from a buret equipped with a carbon dioxide absorption trap. Perform a blank determination, and make any necessary correction. Each mL of 0.1 N tetrabutylammonium hydroxide is equivalent to 12.21 mg of benzoic acid.

$$N = \frac{\text{mg benzoic acid}}{122.1 \times \text{mL } (C_4H_9)_4NOH}$$

Tetrabutylammonium Hydroxide in Methanol/ Isopropyl Alcohol, 0.1 N

Prepare as described for *Tetrabutylammonium Hydroxide, Tenth-Normal (0.1 N)* using isopropyl alcohol instead of toluene, and standardize as described. Alternatively, the solution may be prepared by diluting a suitable volume of commercially available tetrabutylammonium hydroxide solution in methanol with 4 volumes of anhydrous isopropyl alcohol.

$$N = \frac{\text{mg benzoic acid}}{122.1 \times \text{mL } (C_4H_9)_4NOH}$$

Tetramethylammonium Bromide, Tenth-Molar (0.1 M)

$(CH_3)_4NBr$, **154.05**
15.41 g in 1000 mL

Dissolve 15.41 g of tetramethylammonium bromide in water to make 1000 mL, and standardize the solution as follows.

Transfer an accurately measured volume of about 40 mL of the solution to a beaker, add 10 mL of diluted nitric acid and 50.0 mL of 0.1 N silver nitrate VS, and mix. Add 2 mL of ferric ammonium sulfate TS, and titrate the excess silver nitrate with 0.1 N ammonium thiocyanate VS.

$$M = \frac{\text{mL } AgNO_3 \times \text{N } AgNO_3}{\text{mL } (CH_3)_4NBr}$$

Tetramethylammonium Chloride, Tenth-Molar (0.1 M)

$(CH_3)_4NCl$, **109.60**
10.96 g in 1000 mL

Dissolve 10.96 g of tetramethylammonium chloride in water to make 1000 mL, and standardize the solution as follows.

Transfer an accurately measured volume of about 40 mL of the solution to a flask, add 10 mL of diluted nitric acid and 50.0 mL of 0.1 N silver nitrate VS, and mix. Add 5 mL of nitrobenzene and 2 mL of ferric ammonium sulfate TS, shake, and titrate the excess silver nitrate with 0.1 N ammonium thiocyanate VS.

$$M = \frac{\text{mL } AgNO_3 \times \text{N } AgNO_3}{\text{mL } (CH_3)_4NCl}$$

Titanium Trichloride, Tenth-Normal (0.1 N)

$TiCl_3$, **154.23**
15.42 g in 1000 mL

Add 75 mL of titanium trichloride solution (1 in 5) to 75 mL of hydrochloric acid, dilute to 1000 mL, and mix. Standardize the solution as follows, using the special titration apparatus described.

Apparatus—Store the titanium trichloride solution in the reservoir of a closed-system titration apparatus in an atmosphere of hydrogen.

Use a wide-mouth, 500-mL conical flask as the titration vessel, and connect it by means of a tight-fitting rubber stopper to the titration buret, an inlet tube for carbon dioxide, and an exit tube. Arrange for mechanical stirring. All joints must be air-tight. Arrange to have both the hydrogen and the carbon dioxide pass through wash bottles containing titanium trichloride solution (approximately 1 in 50) to remove any oxygen.

If the solution to be titrated is to be heated before or during titration, connect the titration flask with an upright reflux condenser through the rubber stopper.

Standardization—Place an accurately measured volume of about 40 mL of 0.1 N ferric ammonium sulfate VS in the titration flask, and pass in a rapid stream of carbon dioxide until all the air has been removed. Add the titanium trichloride solution from the buret until near the calculated endpoint (about 35 mL), then add through the outlet tube 5 mL of ammonium thiocyanate TS, and continue the titration until the solution is colorless.

$$N = \frac{\text{mL } FeNH_4(SO_4)_2 \times \text{N } FeNH_4(SO_4)_2}{\text{mL } TiCl_3}$$

Zinc Sulfate, Twentieth-Molar (0.05 M)

$ZnSO_4 \cdot 7H_2O$, **287.56**
14.4 g in 1000 mL

Dissolve 14.4 g of zinc sulfate in water to make 1 L. Standardize the solution as follows.

Accurately measure about 10 mL of 0.05 M edetate disodium VS into a 125-mL conical flask, and add, in the order given, 10 mL of acetic acid–ammonium acetate buffer TS, 50 mL of alcohol, and 2 mL of dithizone TS. Titrate with the zinc sulfate solution to a clear, rose-pink color.

$$M = \frac{\text{mL edetate disodium} \times \text{M edetate disodium}}{\text{mL } ZnSO_4}$$

Chromatographic Columns

The following list of packings (L), phases (G), and supports (S) is intended to be a convenient reference for the chromatographer. [Note—Particle sizes given in this listing are those generally provided. Where other, usually finer, sizes are required, the individual monograph specifies the desired particle size. Within any category of packings or phases listed below, there may be a wide range of columns available. Where it is necessary to define more specifically the chromatographic conditions, the individual monograph so indicates.]

Packings

Change to read:

L1—Octadecyl silane chemically bonded to porous ▲or nonporous▲*USP35* silica or ceramic microparticles, 1.5 to 10 µm in diameter, or a monolithic silica rod.

L2—Octadecyl silane chemically bonded to silica gel of a controlled surface porosity that has been bonded to a solid spherical core, 30 to 50 µm in diameter.